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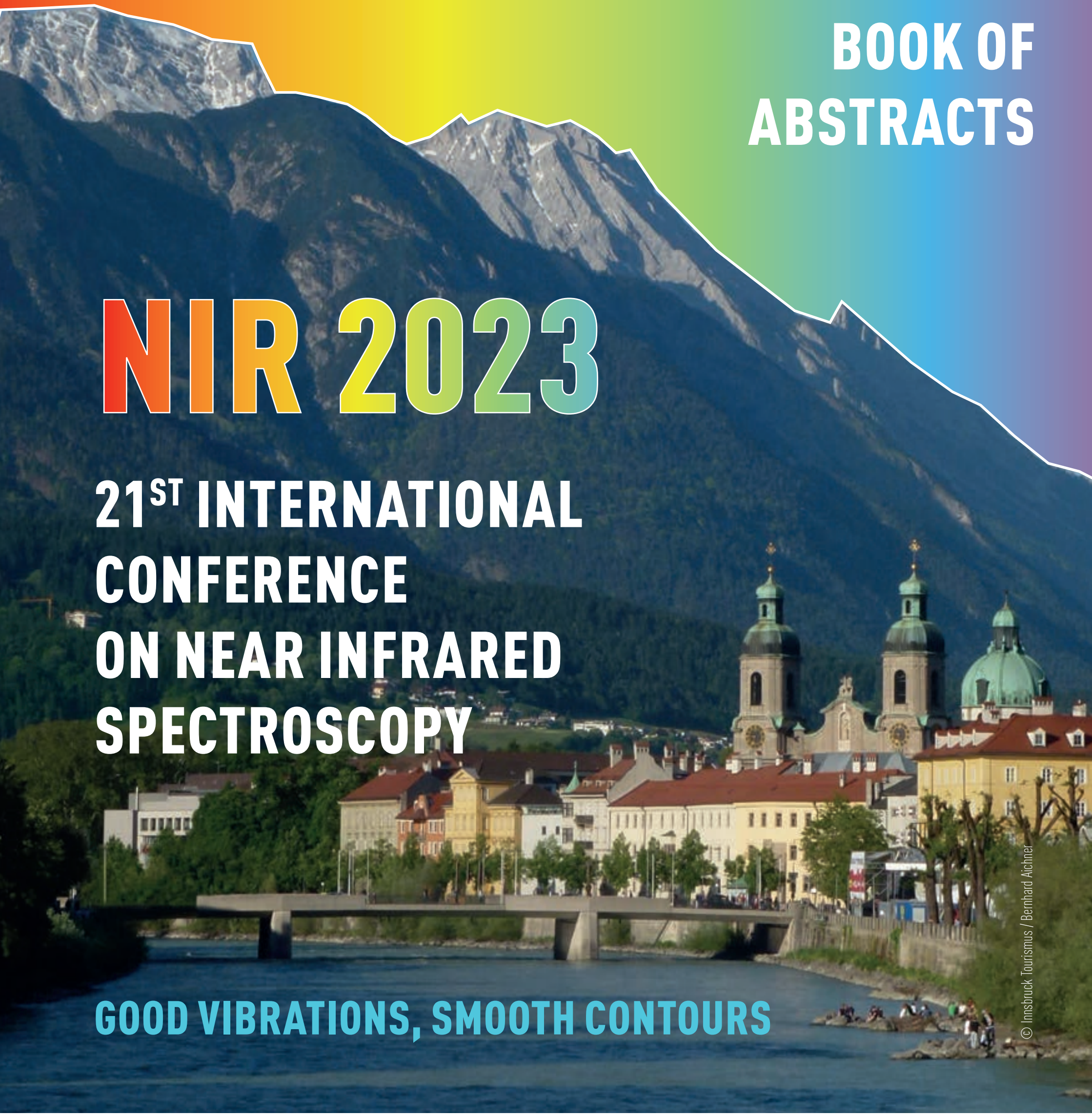
BOOK OF ABSTRACTS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NIR 2023

21ST INTERNATIONAL CONFERENCE ON NEAR INFRARED SPECTROSCOPY

GOOD VIBRATIONS, SMOOTH CONTOURS

August 20-24, 2023 INNSBRUCK - AUSTRIA

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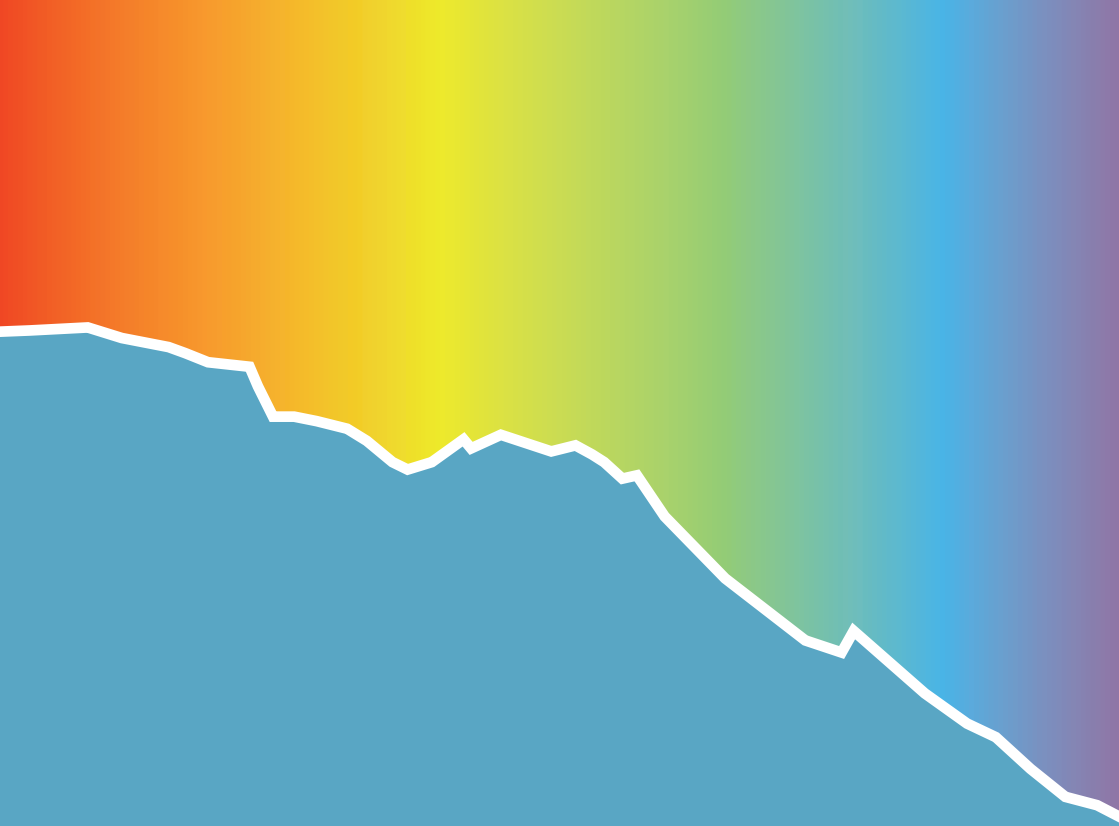

PLENARY SPEAKER

PL01 – PL08

PL01

NEAR INFRARED SPECTROSCOPY EXPLORES MYSTERIES OF ANCIENT AND MODERN MEDICINE

Gerhard Litscher[1]

[1]International Society for Medical Laser Applications, Medical University of Graz, Graz, Austria

Near Infrared Spectroscopy (NIRS) is a powerful technology used in many areas of medicine, including traditional and innovative medicine.

In ancient medicine, NIRS has been used to gain insights into diseases and medical treatments of the past. For example, in traditional medicine, NIRS can be used to determine effects of acupuncture and the quality of herbal medicines.

In modern medicine, NIRS is used to diagnose and monitor diverse conditions, including brain injuries, stroke, and cancer. Over the decades, the application of NIRS in neuroscientific and neurological research has gained importance, and the number of scientific studies has grown extensively.

Within the current opening lecture, a short review and highlights from 1995 to 2023 from an interdisciplinary research team at the Medical University of Graz, Austria, Europe, will be presented. Main topics are among others results from transcranial cerebral oximetry in the hyperbaric environment and the non-invasive assessment of oxygen metabolism using NIRS technology during high altitude trekking in the Nepal Himalayas. In addition, NIRS publications from intensive care medicine and needle and laser acupuncture are presented and discussed. Last but not least, the application of regional oxygen saturation in regenerative medicine is demonstrated.

NIRS could be a fruitful approach helping to explore deeper biological mysteries within the brain and the periphery. Overall, NIRS is a powerful tool that has the potential to unlock many mysteries of ancient and modern medicine. As technology continues to improve, it is likely that NIRS will become even more important in the diagnosis and treatment of a wide range of medical conditions.

PL02

GOOD VIBRATIONS IN THE GAS PHASE - FROM NIR TO MIR

Boris Mizaikoff[1]

[1]Ulm University & Hahn-Schickard, Ulm, Germany

Vibrational spectroscopies - and especially infrared spectroscopy - play an increasingly important role in modern biodiagnostics. This begs the question - quo vadis, where do we go from here?

Non- or minimally-invasive biomedical diagnostics are aiming at point-of-care (POC) and point-of-need (PON) are of particular interest. Exhaled breath analysis provides a diagnostic window into the physiological condition of an organism with the potential of monitoring heath conditions, diseases, and therapeutic progress. Near- and mid-infrared photonics and technologies range among the most flexible molecular sensing platforms nowadays available. With the emergence of laser technology across the infrared spectral band, even the on-chip hybridisation and/or integration of entire IR sensing devices is on the horizon ultimately leading to IR-lab-on-chip systems. The inherent molecular selectivity of NIR and MIR signatures provides particular access to small molecules (e.g., volatile organic compounds; VOCs) in the gas phase at unprecedented detail in a label-free and non-destructive fashion. Most importantly, the combination with orthogonal sensing concepts (e.g., eNoses) and advanced multivariate data evaluation algorithms enables analyses in real-world complex matrices of biomedial and clinical relevance. The discussion of latest IR photonic technologies we will be complemented by highlight applications in exhaled breath analysis underlining the utility of next-generation IR photonics.

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PL03

NEAR INFRARED SPECTROSCOPY: A “RESTLESS” ANALYTICAL TECHNIQUE FOR A MULTIPLICITY OF APPLICATIONS

Heinz Wilhelm Siesler[1]

[1]University Duisburg-Essen, Essen, Germany

This presentation will give a brief overview of the development of the NIR technique from its beginnings when spectrometers were just about to evolve from add-ons to UV-VIS or mid-IR spectrometers and appear as standalone instruments to its current miniaturization in pocket size. The initial organizational difficulties in introducing the NIR technique into industrial analytical laboratories are addressed in a humorous manner, and the new developments that have taken place over time (chemometric evaluation methods, fiber-optic coupled probes, imaging technique) are briefly discussed in light of some typical application examples.

The presentation will identify potential pitfalls, provide recommendations for data processing from the perspective of a conservative vibrational spectroscopist, and highlight the advantages and disadvantages compared to Raman and mid-IR spectroscopy.

Current miniaturization and opportunities for on-site and in-the-field measurements will be discussed with some selected examples. Special attention will thereby be paid to possible applications that will allow a clientele that is not necessarily scientifically trained to solve quality control and authentication problems with this technology in everyday life.

Finally, the danger is addressed not to fall into the exaggerated and sometimes unrealistic narrative of some direct-to-consumer companies, which has raised unrealistic expectations with full-bodied promises, but has harmed the very valuable technology of NIR spectroscopy, rather than promoting its further development.

PL04

NIRS: THE GREEN ANALYTICAL CHOICE FOR FUTURE SUSTAINABLE FOOD PRODUCTION

Søren Balling Engelsen[1]

[1]University of Copenhagen, Copenhagen, Denmark

In academia, NIR spectroscopy has not been considered serious due to strongly overlapping peaks with no baseline separations. On the industrial side, NIR spectroscopy has revolutionised quality control in practically all areas of primary food and feed production and has become the de facto standard for monitoring the quality of millions of samples of cereals, vegetables, milk, meat, powders and tablets with unprecedented precision and speed and with practically no environmental fingerprint. NIR spectroscopy can thus be considered as the ugly duckling amongst spectroscopies that has turned into a swan and revolutionized quality control.

The potential of introducing real-time process monitoring through fingerprinting of complex process streams is enormous, and the ultimate challenge for NIR spectroscopy is on-line monitoring in process analytical technology (PAT). In this setting, NIR spectroscopy represent a unique green analytical technology that will allow for controlling and optimizing processes toward increased sustainability - and there are practically no analytical alternatives.

This presentation will try to highlight the advantages and limitations of the use of NIRS in industrial process monitoring and control using examples primarily from the food and biotechnology production.

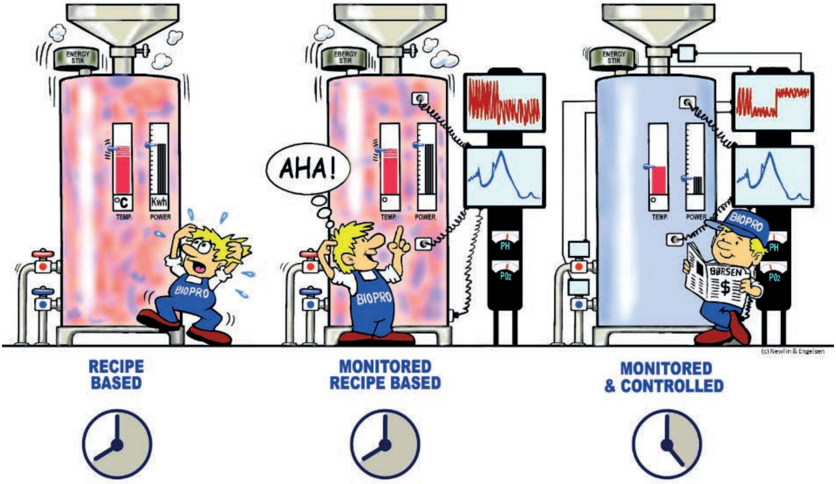

Illustration of the principle of monitoring batch processes by NIR spectroscopy.
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PL05

ANALYSIS OF CEREAL QUALITY USING NIR SPECTROSCOPY - A JOURNEY THAT BEGAN ON 1ST AUGUST 1975

Marena Manley[1]

[1]Stellenbosch University, Stellenbosch, South Africa

Following the discovery of the NIR region by Herschel, two significant events contributed to the discovery, development, and application of NIR technology. The late Karl Norris, an engineer who was unaware of the limitations of the NIR region, investigated its application for quantitative analysis leading to the development of the first commercial NIR instrument. During this period, Phil Williams who needed an alternative method to Kjeldahl to measure wheat protein content, purchased one of the first commercial NIR instruments and tested it on wheat flour. The first commercial use of NIR technology officially started on 1st August 1975. Karl showed, with the use of appropriate regression techniques, that relationships between absorption values at specific wavelengths and reference results of the constituent to be predicted could be established. Svante Wold created the term chemometrics in 1971 for this method of analysing spectral data. With the invention of the computer and its subsequent development, chemometrics developed into an independent field of study. One major development in the 1980s was the introduction of multivariate calibration methods, such as principal component analysis, partial least squares regression and multiplicative scatter correction. Compared to linear regression methods, these methods allowed for the analysis of multiple variables simultaneously, improving the accuracy and reliability of the calibration models. Also, during this time the first whole grain analyser became available. The subsequent development and use of chemometric software allowed for automated model development. Numerous spectroscopist and chemometricians continued to contribute significantly to the development of NIR spectroscopy. NIR hyperspectral imaging equipment became available for commercial use and investigated for cereal analysis in the early 2000s and since 2013 an increasing number of miniature devices have become available, some now dedicated to cereal analysis. In recent years, machine learning techniques, such as artificial neural networks and support vector machines have become increasingly popular allowing for the creation of highly complex models that can accurately predict the chemical composition of a sample, even in cases where the relationships between the variables are not well understood. Now, for almost 50 years, NIR spectroscopy has been an essential tool in the cereal and especially wheat and flour milling industries.

PL06

THE STATE-OF-THE-ART NIR IMAGING

Yukihiro Ozaki[1]

[1]*Kwansei Gakuin University, Nishinomiya, Japan*

For the last decade we have been involved extensively in the development of NIR imaging. Our research has two directions; one is the development of NIR data analysis methods for building NIR imaging. We proposed the following three data analysis methods;1) Two-dimensional (2D) band shift correlation spectroscopy for NIR imaging data of cellulose tablets. 2) NIR imaging analysis of cellulose tablets by a band position shift. 3) Self-modeling curve resolution (SMCR) analysis of NIR imaging data of pharmaceutical tablets. Another direction is to develop MIR imaging systems. We have been concerned with the development of three NIR imaging systems. The first one is a highly sensitive portable NIR imaging device; D-NIR (Yokogawa Co.). This system was used for the study of dissolution of water into a tablet and the detection of defective tablets. The second one is a high speed and wide area NIR imaging system (Compovision) (Sumitomo Electric Industries). This one is applicable also micro NIR imaging. We employed this system to explore the biomolecular distribution and molecular mechanism of embryonic development in fertilized fish eggs. Figure shows the results of NIR imaging obtained by this system. The last one is an imaging-type two-dimensional Fourier spectroscopy (ITFS) system proposed by Ishimaru. Using this system we explored nonstaining blood flow imaging and molecular distribution in developing fish egg embryos.

Acknowledgement; Dr. Hideyuki Shinzawa (AIST, Tsukuba), Dr. Kimie Awa (Dainihon-Sumitomo Pharma), Dr. Koudai Murayama (Yokogawa Co. Ltd.), Prof. Daitaro Ishikawa (Tohoku Univ.), Prof. Mika Ishigaki (Shimane Univ.), Prof. Ichiro Ishimaru (Kagawa Univ.)

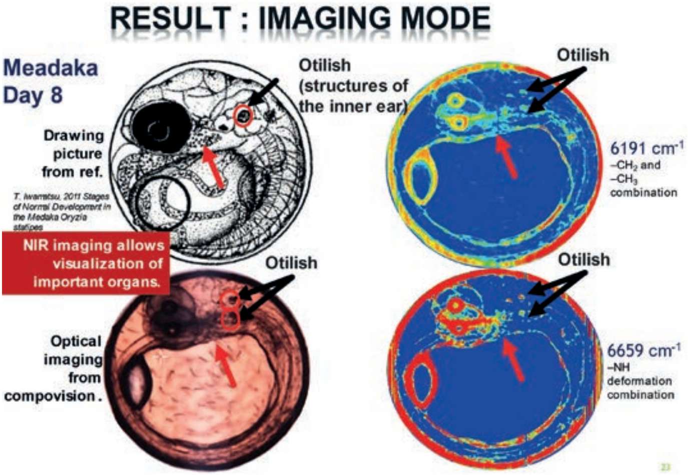

Figure 1. Drawing picture of a Medaka egg on day 8, its optical image, and two NIR images developed by peak intensities at 6191 and 6659 cm^{-1}.

PL07

SPECTROSCOPY THROUGH THE EYES OF A CHEMOMETRICIAN; HOW NIR CAN PROFIT FROM RECENT ADVANCES IN CHEMOMETRICS

Federico Marini[1]

[1]Dept. of Chemistry, University of Rome "La Sapienza", Rome, Italy

The present communication aims at presenting an overview of the potential of coupling some advanced chemometric tools with vibrational spectroscopy, in particular in the Near infrared range, to solve problems in different fields of application. Various recently developped techniques will be presented and illustrated by means of selective examples. In particular, attention will be focused on methods which span all the phases of the analytical pipeline, from experimental design/sampling to variable selection/putative marker identification and validation of the whole process.

Methods like ANOVA-simultaneous component analysis (ASCA) [1] or ANOVA-Target projection (ANOVA-TP [2]), which allow to partition the variability of multivariate data matrices collected according to an underlying experimental design, to evaluate the significance of the different model terms and to provide a straightforward interpretation of the observed effects, will be illustrated.

On the other hand, some recently proposed preprocessing approaches aimed at reducing the impact of unwanted variability on the data, at the same time preserving as much as possible of the relevant information will also be discussed [3], together with the possibility of exploiting the multi-block concept to avoid the need for selection of the optimal pre-processing strategy [4].

Some space will be devoted to recent advances in the field of variable selection, where extensions of the CovSel paradigm [5] have been recently proposed.

The advantages of exploiting the concepts of locally linear models for regression and classification in order to flexibly implement nonlinearities will also be discussed and lastly, a few words will be spent on some recent developments in the field of class modeling.

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PL08

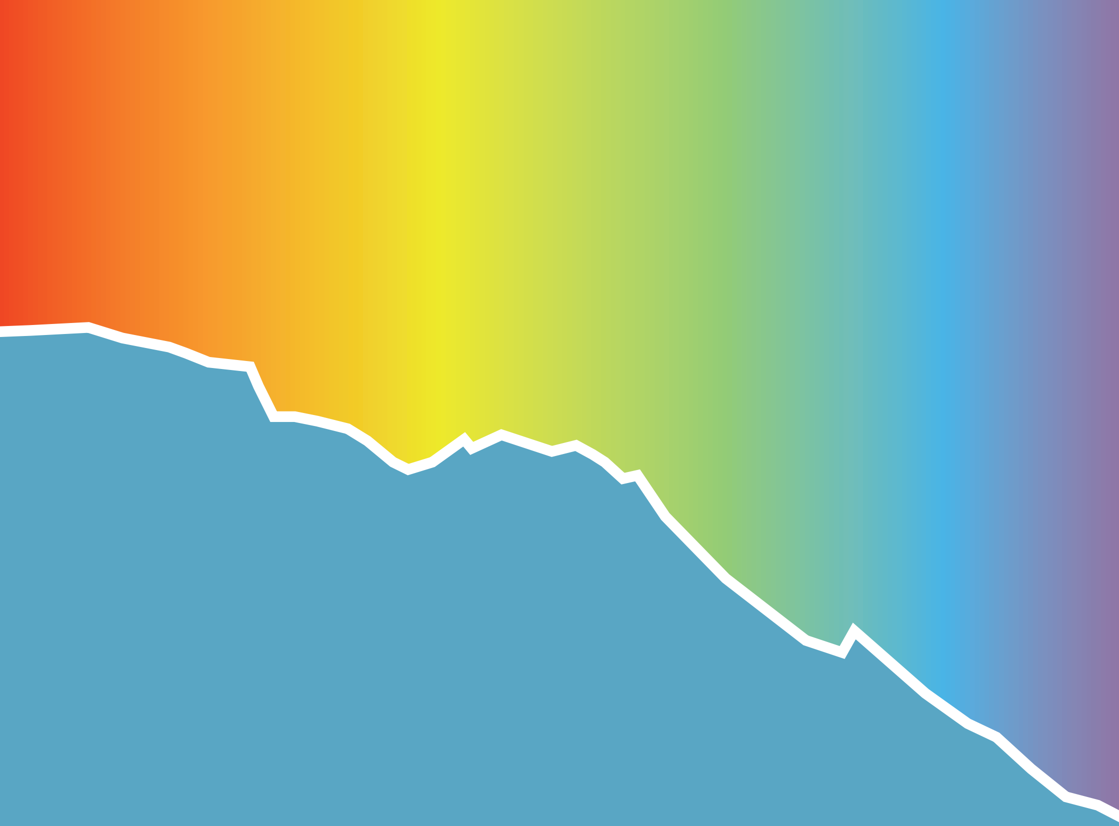
"NIRVOLUTION"

Michael Popp[1]

[1]Bionorica, Neumarkt, Germany

Medicinal plants and their extracted active pharmaceutical ingredients consists of more than a thousand chemical compounds. One of the few analytical methods which is able to measure the sample in a holistic way is NIR. To ensure a high quality, as well as safety and efficacy, it is necessary to define the quality of herbal products at the level of cultivation, dried herbal drug, extract and the final medicinal product.

The year 2000 marked the beginning of an exciting scientific journey and success story to reveal the full potential of NIR technology for Bionorica. Methods for measuring dried herbs, living plants, and final products have been developed at the Institute of Analytical Chemistry at the University of Innsbruck in collaboration with Prof. Bonn and Prof. Huck. Furthermore, the technique and know-how were transferred to Bionorica research GmbH, where a NIR team was formed for additional method diversification and refinement. Finally, NIR was established in the headquarter in Neumarkt/Germany with ready to use methods. Within this process high tech tailor made solutions for phytopharmaceutical industry could be introduced. As an example, in an outstanding multi-country field initiative cultivation data was collected with handheld devices over a time span of three years. With the obtained data models for measuring the quality of medicinal plants in real time directly on the field could be developed.



KEYNOTE SPEAKER

KN01 – KN14

KN01

NIRS - AQUAPHOTOMICS: NEW INTEGRATIVE SCIENCE AND TECHNOLOGY PLATFORM

Roumiana Tsenkova[1]

[1]University of Kobe, Kobe, Japan

Aquaphotomics is a new Integrative Science and Technology Platform presented in 2005 at International Conference of NIRS in New Zealand. The principal novelty of this field is placing the focus on water as a complimentary to other molecules in biological or aqueous systems. NIRS has been developed aiming at measuring the concentration of various components in systems. Water has been considered as very strong absorber of NIR light, therefore as an obstacle when measuring other molecules. In contrary, Aquaphotomics explores the fact that water is sensitive to any change the system experiences – external or internal. The interaction with NIR light transforms water in to an integrative sensor, a collective mirror. The molecular structure of water, revealed through its interaction with light as a multidimensional spectral pattern of all frequencies becomes a source of information about the state of the system. As the water spectral pattern in the NIR region can be measured and monitor non-invasively, it opens immense opportunity to be used as a multidimensional biomarker for various purposes of bio measurements, bio monitoring and bio diagnostics as well as to investigate any aqueous system. Together with the quantitative measurement of other molecules in the system routinely done by NIRS Aquaphotomics presents a complimentary approach in spectroscopy. As a result, direct explanation of the functionality of the system coupled with understanding about its relation to specific molecules and elements of the system becomes possible.

KN02

INTRODUCING NOVEL PHOTOTHERMAL SENSING STRATEGIES FOR THE ANALYSIS OF GASES AND LIQUIDS USING NIR AND MIR LASERS

Bernhard Lendl[1], Johannes Paul Waclawek[2], Harald Moser[2], Davide Pinto[1], Jesus Mendoza-Castro[1,3], Liam O'Faolain[3]

[1]TU Wien, Vienna, Austria, [2]CHASE GmbH, Vienna, Austria, [3]Munster Technological University, Cork, Ireland

NIR diode lasers as well as MIR quantum cascade lasers are highly interesting light sources for use in spectroscopy, providing high spectral power densities while allowing also for rapid wavelength adjustment through current modulation. NIR and MIR light photonic circuitry can be powered by these sources and provide additional degrees of freedom in the design of new, miniaturized sensing concepts for quantitative analysis of liquid and gaseous samples.

This presentation will focus on photothermal spectroscopy (PTS), a rapidly developing sensing scheme. In PTS modulated absorption of light in the sample induces changes of the sample´s refractive index, which result from absorption induced small temperature changes in the liquids or gases under investigation. The generated signal scales directly proportional with the power of the excitation source. These refractive index changes, preferably induced by NIR or MIR lasers, can be read out optically, e.g. using NIR lasers and resonant transducers for increased sensitivity. For gas sensing the concept of interferometric cavity assisted photothermal spectroscopy (ICAPS) will be presented, which uses an NIR Fabry-Perot cavity as transducer for sensitive detection of refractive index changes resulting from laser-based wavelength modulation spectroscopy carried out in the MIR or NIR spectral region. Results for sensing of sulphur dioxide (LOD: 3 ppb) will be shown using a cavity spacing (optical path) of just 1 mm. For liquid sensing new chip based concepts for sensitive refractive index sensing will be presented, which have been realized on a Silicon Nitride (SiN) platform. We have designed, fabricated and also successfully tested SiN photonic Fano resonators which were realized by combining photonic crystal structures coupled to micro ring resonators for the detection of small refractive index changes. First results for refractive index sensing in the NIR spectral region (1550 band) will be presented.

KN03

CHEMOMETRICS IS MORE THAN ALGORITHMS

Tom Fearn[1]

[1]*University College London, London, United Kingdom*

Everyone will have their own ideas about what chemometrics is. For me it is the use of tools from mathematics, statistics and computer science to help solve problems in chemistry. As these tools, especially those from machine learning, become ever more powerful, I believe we risk forgetting that they are tools for the chemist, or physicist or spectroscopist, and not complete solutions in themselves. At the risk of seeming a grumpy old man trying to turn back the tide of progress, I would like to suggest that the things about the underlying problem that we know and that the calibration algorithm, however intelligent it might appear, does not know, are also a crucial input to its solution. The following suggestions are offered in this spirit. None of them is new, but they bear repeating.

- The experimental design is crucial.
- Look at the spectra.
- Use the simplest algorithm that gives acceptable performance.
- Try to understand why the calibration works. If your algorithm doesn't help you to do this, try using one that does.
- Remember that NIR, with few exceptions, measures percents, not ppm.
- If the calibration ought not to work, it probably doesn't.

KN04

MAIN TRENDS AND OPPORTUNITIES IN THE USE OF NEW GENERATION OF NEAR-INFRARED SENSORS FOR THE CONTROL, AUTHENTICATION, AND MONITORING OF AGRI-FOOD PRODUCTS AND PROCESSES

Dolores Perez-Marin[1]

[1]*University of Cordoba, Cordoba, Spain*

Control, authentication, and monitoring products and processes are key issues for the agri-food industry, affecting its efficiency, trademark image, economic performance, and sustainability. Recent developments in spectral sensors technology, and advances in big data analytics, provide new opportunities to offer solutions to the industry in this challenging field, where it is also essential to control and trace the products along the entire food chain — "from farm to fork". Near infrared spectroscopy (NIRS) combined with data analytics is well suited for this, providing cost effective and added value solutions to a few current problems in the of food industry, as well as, at the same time, possibilities to gain a better understanding of their products and ingredients. In addition, advances in the development of a new generation of highly portable NIRS have opened many new opportunities for the on-site use of NIR sensors to effectively monitor, control, and trace food from source to consumption, including enhancement of the labelling of the final product. Importantly, some of the new applications were unthinkable just a few years ago, making food control systems now more robust, smart, massive, interoperable, and accessible. This lecture will present the main trends in the use of this new generation of NIR sensors to address specific food quality, safety and authenticity issues in agriculture and food products and processes.

KN05

ADVANCEMENT OF HYPERSPECTRAL IMAGING AND FUTURE TRENDS FOR FOOD SAFETY AND QUALITY APPLICATIONS

Moon Kim[1], Jianwei Qin[1], Insuck Baek[1], Kevin Chao[1], Diane Chan[1]

[1]United States Department of Agriculture, ARS, Beltsville, United States

Over the past two decades, USDA researchers in Beltsville, Maryland, in collaboration with industry and academic cooperators, have developed a variety of hyperspectral imaging technologies for food safety and quality evaluation of agricultural commodities. The sensing technology team at USDA has emphasized the research and development of spectral imaging and instrumentation platforms using spectral methods such as fluorescence, visible and near-infrared reflectance (VNIR), and Raman chemical imaging, in its work such as the pioneering online hyperspectral line-scan systems for automated food processing line applications such as wholesomeness inspection of broiler chickens, portable fluorescence imaging systems for in situ use in illuminated ambient conditions, and rapid Raman-based contamination detection for food powders. In considering potential applications of the spectral imaging technologies created, the team's in-house development of system- and application-specific software interfaces to control imaging instrument platforms and automate data acquisition and processing has been critical to developing effective, user-friendly technologies for real-world use. Most recently, new systems integrated with multimodal-based sensing and automated data analyses, such as embedded AI-based processing, have enabled the use of various spectral imaging technologies to further enhance applications in food safety and quality assessment. The advancement of the above spectral imaging technologies and future trends are presented.

KN06

ELASTIC AND INELASTIC LIGHT SCATTERING SPECTROSCOPY: THE HIDDEN CHAMPION IN PAT APPLICATIONS

Rudolf Kessler[1]

[1]Reutlingen University and Kessler ProData GmbH, Reutlingen, Germany

Optical spectroscopy is commonly used to get insight into the chemical composition of matter by analyzing the absorption spectra which are the result of inelastic interactions between photons and molecules. However, optical spectroscopy can also be used to characterize the nanoscopic texture of materials like particles or biological tissues by analyzing the scatter spectra which are the result of an elastic interaction between photons and matter.

The common procedure to get both information is to analyze the morphological structure of a system in the UV-Vis region due to the higher scatter cross sections at shorter wavelengths and additionally to measure the chemical signature of interest by NIR or MIR-spectroscopy due to the high absorption cross sections at longer wavelengths. The combined chemical and morphological information offer then the possibility to control processes on a molecular level and to assess inline the macroscopic fundamental functionalities. This is conclusive but can also be quite laborious and cost intensive for inline PAT-applications.

In NIRS the absorption cross sections decrease significantly towards shorter wavelengths. The result is a higher penetration depth of the photons into the material at shorter wavelengths (third overtone) and an increased probability of scatter events. Within the vibrational combination wavelength range, absorption is dominant and thus photon penetration is low and scatter events occur less often.

This effect is of advantage as one can measure with a single instrument the absorption and scatter spectra simultaneously. This saves time and costs in PAT measurements using only a single instrument. The only extra effort for the experimenter is a different chemometric spectral pretreatment for the absorption and scatter spectral region.

Examples of this combined elastic light scattering- and absorption-spectroscopy will be demonstrated for instance in the areas: medical and biotechnological fields e.g., for the mRNA vaccines, the characterization of pharmaceuticals (e.g. tablets), the marker free analysis of cancer tissues and other applications in the field of food industry (e.g., milk, fruit classification), wood and fiberboard industry, paper and pulp and also in the polymer industry.

Further reading:

Rudolf W. Kessler, Waltraud Kessler, Michael Maiwald, Inline and Online Process Analytical Technology with a Focus on Optical Spectroscopy, Encyclopedia of Analytical Chemistry, Online a9791, 2022 John Wiley & Sons, Ltd., DOI: 10.1002/9780470027318.a9791,
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KN07

SUBSAMPLING, SAMPLING, OVERSAMPLING? NIR OR HSI-NIR?

Daniele Tanzilli[1,2], **Jose Manuel Amigo**[3,4], Marina Cocchi[1],
Alessandro D'Alessandro[5]

[1]Department of Chemistry and Geology. University of Modena e Reggio Emilia. Via Campi 103., Modena, Italy, [2]LASIRE, Université de Lille, Cité Scientifique, 59650, Lille, France, [3]IKERBASQUE, Basque Society for the Promotion of Science, Plaza Euskadi, 5., Bilbao, Spain, [4]Department of Analytical Chemistry. University of the Basque Country. Barrio Sarriena S/N, Leioa, Spain, [5]Barilla G. and R. Fratelli, Via mantova 166, Parma, Italy

Near Infrared Hyperspectral Imaging (NIR-HSI) emerged from the Remote Sensing area in the late 70's and has been settled down in our analytical laboratories as a perfect tool for assessing the spatial distribution of components in a mixture. Its applicability in many situations, with the advance of new sensing methodologies and faster data acquisition and deployment techniques, makes NIR-HSI a plausible methodology to be implemented in-line for monitoring and controlling the quality of many different products.

The first inclusion of NIR-HSI in the industry was in the late 90's, when the pharmaceutical industry tested its ability to monitor and control in-line the distributional homogeneity of tablets in production lines. However, at the same time, food and other production industries started implementing NIR-HSI devices in their production line with different success ratios.

Behind this apparent success, several questions arise when one HSI-NIR device needs to be implemented in real-time production lines. This presentation will review the main points to consider (stability of the hardware in harsh environments, or deployment of data, among others) and focus the interest on one particular point that deserves equal attention: sampling.

One of the major advantages of HSI-NIR is the possibility of scanning the whole surface of a sample (therefore, being in a situation of oversampling) instead of measuring a single point of the sample (thus, subsampling). This point is especially crucial when the sample has a heterogeneous composition. This presentation will compare both methodologies (HSI-NIR from NIR) from different angles (e.g. adequacy of the hardware and software, price, stability of the models, etc.) to study whether HSI-NIR is absolutely needed or using single point NIR is enough. This discussion will be done by studying the production of Italian pesto sauce, a highly heterogeneous sample.

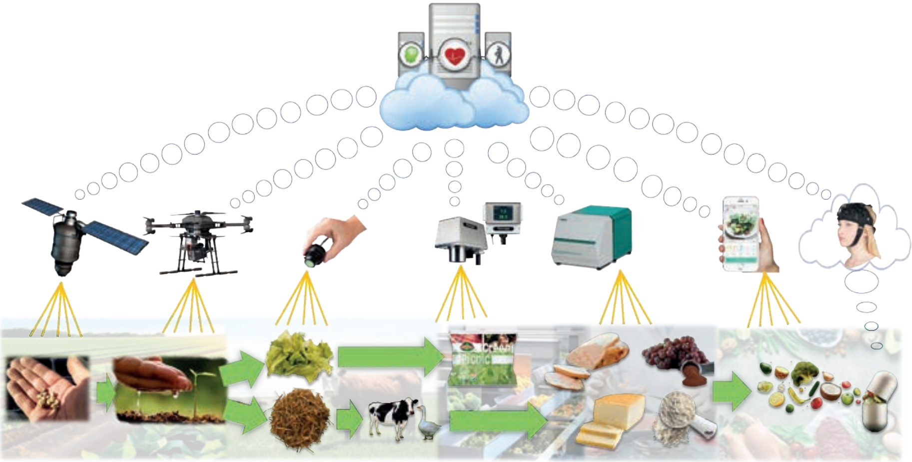
KN08

RECENT RESULTS OF NEAR INFRARED SPECTROSCOPY ON THE WAY "FROM FARM TO FORK" OR EVEN FURTHER

Zoltan Kovacs[1], George Bazar[2], Zoltan Gillay[1], Juan Pablo Aguinaga Bósquez[1], Matyas Lukacs[1], Mariem Majadi[1], Flora Vitalis[1]

[1]Department of Food Measurement and Process Control, Institute of Food Science and Technology, Hungarian University of Agriculture and Life Sciences, Budapest, Hungary, [2]ADEXGO Ltd., Balatonfüred, Hungary

Agri-food products undergo very complex journeys until they reach our kitchens. Initial challenges start on the farm and continue in processing, storage, transport, distribution and even consumption. Besides the origin of the raw materials, value chain processes influence the quality of our food and finally our health. Modern digitalization endeavors, however, provide great opportunities to ensure the quality and authenticity of the agri-food products. Development of digital fingerprinting approaches enable on-the-spot examinations providing comprehensive information about the product. This study aimed to provide insights about recent results of Near Infrared Spectroscopy (NIRS) supporting the monitoring of food supply "from farm to fork" – and beyond. The quality of forage fed to cattle determines milk yield and composition affecting quality and storability of dairy products or beef meat. This was confirmed in a study where the shelf-life of cheese produced from the milk of cattle subjected to different diets was studied under different temperatures using NIRS. Complimentary NIRS studies have confirmed that compound feeds of poultry or pig production highly influence the quality of animal products, e.g., egg, meat, lard. Subsequently, the blood content is critical in the case of premium quality foie gras, which depends on breeding and slaughtering conditions. Accurate prediction of liver blood content and its color change was possible with NIRS based regression models. Freshness is of paramount importance for foods, especially in the case of minimally processed leafy vegetables. The potential shelf-life extension was studied with argon gas over "conventional" modified atmosphere packaging during iceberg lettuce production. NIRS proved to be useful to confirm that leaves of different stages of development and type of packaging react differently. Nutraceuticals, or food products with added-nutritional value, have been rapidly growing recently with legislative framework and established quality control ever so lagging behind. Monitoring of adulterants in grape seed extract as well as the extract-fortification of fruit juices were possible with NIRS. Studies have proven that NIRS is one of the most comprehensive tools to evaluate the old saying, "you are what you eat". The most recent findings in the field of nutritional neuroscience even enables the technique to move beyond the confines of food quality assessment to directly correlate nutrition and health.



KN09

CONTROL OF POLYMERIZATION PROCESS AND STUDY ON POLYMER STRUCTURE USING NIR & IR

Longhai Guo[1], Hongfu Yuan[1]

[1]College of Material Science and Technology, Beijing University of Chemical Technology, Beijing, China

Polymer materials have been widely applied in many fields, and the development of polymeric synthetic materials has surpassed the traditional three basic materials of steel, cement and wood. How to on-line analyze the morphology of composite polymer particles, as well as the polymerization reaction process, are a great challenge to develop the high-performed polymer nanoparticle materials, not only academia aspect but also industrial applications. The molecular vibrational spectrum due to the groups with hydrogen atoms can be obtained by NIR spectroscopy, and using optical fiber and probe, the NIR spectrum of polymers can be in-situ recorded in reactors, especially for the polymer colloid particles in an aqueous dispersion system, which provide a significant method for the on-line control on polymerization process.

For emulsion polymerization system, the residual monomer concentration, latex particle diameter, and latex particle structure (core-shell and inverse core-shell) can be on-line detected by using NIR spectroscopy. Moreover, the weight fractions of core phase, interfacial layer, and shell layer (Wc, Wi, and Ws, respectively) in the multi-layered composite polymer particles prepared by semi-continuous seeded emulsion polymerization, where NIR calibration models on Wc, Wi, and Ws were successfully established, the determination coefficients of prediction (R^2)were all above 0.92, and the standard error of prediction (SEP) were 0.047, 0.035, and 0.012 respectively. The structural evolution of core-shell particles could be on-line detected and well-controlled.

For the synthesis of phenolic resins, the free formaldehyde content is the key parameter to control the polymerization. For the curing process of epoxy resin, gelation time and gelation point are key indicators for controlling their quality and cost. We proposed a method of near infrared (NIR) spectroscopy combined with multivariate curve resolution-alternating least squares (MCR-ALS) to on-line detect free formaldehyde content of phenolic resins and the curing reaction of primary and secondary amine.

Amphiphilic block copolymers are widely investigated due to their self-assembly performance and potential application as a kind of thermo-responsive hydrogel in biomedical science. However, the molecular mechanism of their structural change during the gelation process is still unclear, which is very significant for the structural design. The temperature-dependent IR and NIR spectra of PDMAA-b-PDAAM-b-PDMAA triblock copolymer were employed to investigate the LCST-type sol-gel transition with a self-assembled morphological transition. It is found that the dissociation of intermolecular hydrogen bonds within the PDAAM block is the driving force for the morphological transition.

KN10

POINT-OF-CARE APPLICATIONS OF NEAR-INFRARED SPECTROSCOPY IN CLINICAL MEDICINE

Bayden Wood[1], John Adegoke[1]

[1] *Monash University, Monash, Australia*

Malaria remains a major public health challenge, and the need for point-of-care diagnostic approaches that are sensitive to low parasitemia, affordable, and easy to use in field settings are urgently required. This study presents a novel approach for malaria diagnosis using a low-cost, miniaturized near-infrared (NIR) spectrophotometer, which was used for the first time to detect and quantify malaria infection in vitro from isolated dried red blood cells. The use of NIR offers several advantages, including wavelength accuracy and repeatability, speed, and resolution, as well as a greatly improved signal-to-noise ratio compared to existing spectroscopic options. Multivariate data analysis was used to discriminate control red blood cells from infected cells, and the technique established the limit of detection. Principal component analysis showed good separation between infected and uninfected RBCs, while partial least-squares regression analysis yielded a robust parasitemia prediction, with a root-mean-square error of prediction values of 0.446 and 0.001% for the higher and lower parasitemia models, respectively. The R^2 values of the higher and lower parasitemia models were 0.947 and 0.931, respectively. The estimated parasitemia detection limit was 0.00001%, and a quantification limit of 0.001% was achieved. Further clinical studies with larger patient numbers are required to ascertain the efficacy of the technique for point-of-care screening. This study represents an important step toward the development of a low-cost, easy-to-use, and sensitive diagnostic tool for malaria, which could significantly contribute to reducing malaria cases and related life losses globally by 2030, as per the World Health Organization's objective.

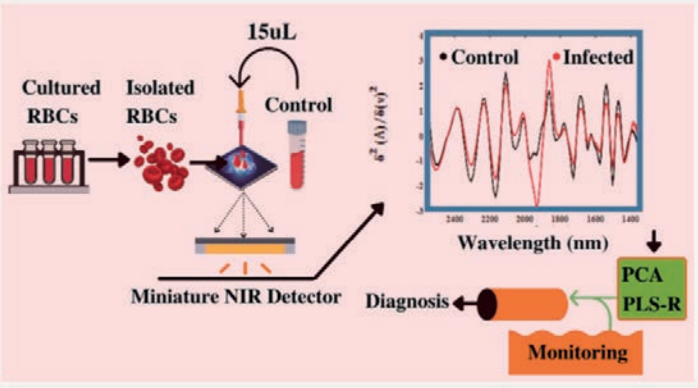


Figure 1. Concept figure for the development of a malaria diagnostic test using near-infrared spectroscopy.

KN11

MODEL BUILDING IN NIR SPECTRAL IMAGING: BASIC PRINCIPLES, DEVELOPMENTS AND SELECTED APPLICATIONS

Aoife Gowen[1]

[1] *University of Dublin, Ireland*

Spectral imaging expands NIR spectroscopy (NIRS) into the spatial domain through acquisition of spatially contiguous spectra over a sample surface. This technique enables investigation of the spatial distribution of components on or within a sample; however the scale and format of the resultant data is considerably challenging than traditional NIRS. This presentation provides an introduction to concepts of model building in NIR spectral imaging, including pretreatments to correct for instrumental artefacts in the data and approaches to build predictive classification/regression models. Peculiarities of spectral imaging data, as well as strategies to overcome the large data sets encountered, will be discussed. Recent developments in spectral image processing, data fusion and model interpretation will be presented and critically appraised. These novel approaches will be illustrated through several recent datasets from the UCD Spectral Imaging Research Group.

KN12

HYPERSPECTRAL IMAGING FOR PROCESS CONTROL IN COATING AND FINISHING TECHNOLOGY

Tom Scherzer[1], Olesya Daikos[1]

[1]Leibniz Institute of Surface Engineering (IOM), Leipzig, Germany

Most samples in NIR spectroscopy are rather thick or even 3-dimensional objects such as fruits, tablets, foodstuffs etc. In contrast, in coating, finishing, lamination, printing and other similar processes, flat web-type materials with low thickness are converted. This presentation is dealing with in-line monitoring of such processes by various NIR approaches for process control. In-process monitoring makes high demands on the sensitivity of instrumentation as well as on the chemometric approaches for quantification due to the rather high line speeds (up to some hundred m/min in printing) combined with low thicknesses of the materials (mostly in the µm range, sometimes less). Moreover, the width of the converted web may reach several meters in some cases. Simple budget-priced NIR spectrometers can monitor a small stripe only, which is not representative and consequently usually not sufficient for monitoring of large-area materials. Depending on the specific working width, we use either NIR cameras or NIR multiplex spectrometer systems based on numerous optical fibers, which are better suited due to their scalability and more cost efficient for extremely broad webs.

The application spectrum of hyperspectral imaging in surface analysis and for monitoring of other thin web-like materials is very wide-ranging.1 The lecture will provide selected examples from different specific application areas such as coating technology (UV curing of acrylates on various substrates, spray coating of urethanes, silicon-based release coatings), printing (sheet-fed offset, ink jet, printing of conductive polymers), lamination (textile composites for automotive engineering), adhesives and adhesion promoters (pressure-sensitive adhesives, tire cords), finishing of textiles (impregnation, Chromojet printing) or functionalization of technical membranes (hydrophilization). The parameters to be monitored depend on the specific system. Generally, they comprise chemical conversion (e.g. after UV curing), application weight (or thickness), degree of functionalization or residual moisture content after drying processes. Another very important objective is the control of the homogeneity of the converted materials (e.g. with respect to the thickness of coatings or the degree of functionalization) as well as the detection of any kind of irregularity such as impurities, surface defects, gradients, delaminations etc.

T. Scherzer, Applications of NIR techniques in polymer coatings and synthetic textiles, in: Y. Ozaki, C. Huck, S. Tsuchikawa, S.B. Engelsen (Eds.), Near-Infrared Spectroscopy: Theory, Spectral Analysis, Instrumentation, and Applications, Springer Nature, Singapore, 2021, chap. 21, pp. 475-516.

Figure 1. Spectral image of polyester fabric which was locally finished with a colorless flame retardant by Chromojet printing.

KN13

NIR SPECTROSCOPY CONTRIBUTED TO SDGS - FROM VIEWPOINT OF WOOD SCIENCE AND TECHNOLOGY

Satoru Tsuchikawa[1], Tetsuya Inagaki[1], Te Ma[1]

[1]Nagoya University, Nagoya, Japan

Wood science and technology help the Sustainable Development Goals (SDGs) in many ways. For example, they create new wood products and building methods that lower carbon emissions, which helps fight climate change. Near-Infrared (NIR) technology is useful for this.
Forests and wood products need to have consistent quality. To make sure they're high-quality, we need quick, non-destructive ways to check them. NIR spectroscopy is a great method for examining organic compounds, often used with advanced data analysis techniques. This talk shows how NIR spectroscopy can evaluate forest and wood products.
Thanks to improvements in spectral imaging and data analysis, NIR devices can do more than ever before. Handheld devices make NIR technology easy for everyone to use. While NIR has some limits for online grading, it's still useful for testing small sample batches. In the future, we expect more progress in this area, which will lead to more uses of NIR technology for monitoring quality in various fields.

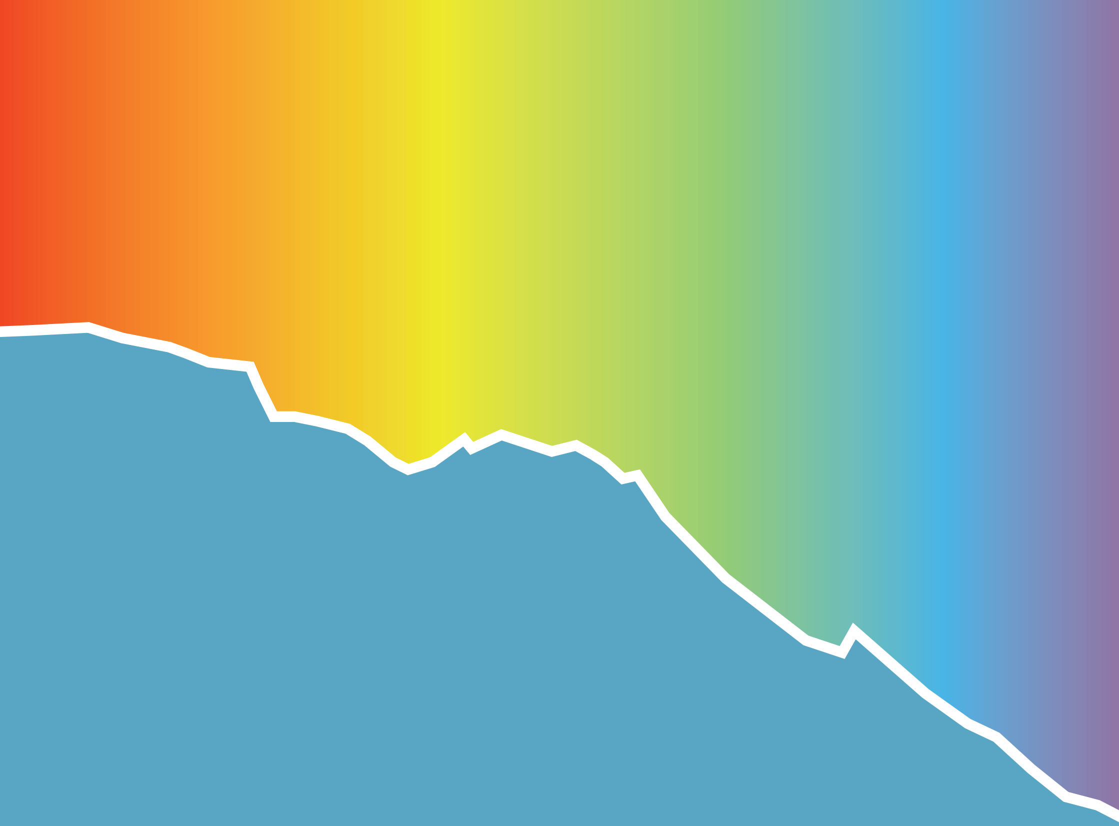
KN14

MACHINE LEARNING APPROACHES FOR NIR SPECTROSCOPY IN HERBAL MEDICINE QUALITY CONTROL

Stefan Schönbichler[1], Bernhard Huber[1], Simon Strasser[1], Marlies Stonig[1], Beatriz Barroso-Gstrein[1], Joy Gertzen[1], Dorothea Schneider[1]

[1]Bionorica research GmbH, Innsbruck, Austria

Quality control is an essential aspect of medicinal herb production, as it ensures that the herbs are safe and effective for consumption. Specifically the identity of the dried medicinal plant and the right content of the analytical marker compounds are of high importance. From a machine learning perspective identification is a classification task and content determination is a regression task. In this talk different machine learning algorithms for classification and regression based on examples from phytopharmaceutical industry are compared and discussed.



AWARDEE

AW01 – AW03

AW01

HYBRID SUBSPACE MODELLING- LINKING MULTIVARIATE REGRESSION AND EXPLAINABLE AI

Harald Martens[1]

[1]Norwegian University of Science and Technology NTNU, Trondheim, Norway

Can today's chemometric "interpretable machine learning with an eye for causality" handle tomorrow's torrents of informative but per se non-selective BIG DATA from high-speed NIR process analyzers and hyperspectral cameras?

NIR measurements need the selectivity enhancement that multichannel instruments can offer. Chemometric multivariate calibration can give that, based on fast, low -dimensional, semi-causal, transparent hybrid modelling using e.g. EMSC/OEMSC & PLSR, requiring limited amounts of calibration data. And so can AI/deep learning, with more flexibility but based on slow, high-dimensional, purely empirical black box artificial neural nets, i.e. machine learning requiring huge amounts of calibration data. When to do what?

High-speed multi-channel diffuse spectroscopy of real-world samples, be it from a process stream or a hyperspectral satellite camera, gives A LOT OF data. These raw intensity spectra are useless per se, because they reflect complex combinations of overlapping chemical and physical sample signals, mixed with instrumental and experimental variations in more or less non-linear ways.

Multivariate calibration models can be trained to turn these raw signals into useful linear quantifications, since the different types of NIR variation have different responses and can thus correct for each others' interfering effects and for non-linearities.

Karl Norris – the wonderful father of diffuse NIR spectroscopy – increased NIR selectivity via local spectral derivatives from his scanning spectrophotometer. Having an NIR instrument with only 19 fixed wavelength channels, spectral derivatives were impossible for me in 1981. Instead, I opted for linear subspace models (first unmixing and SMLR, then PLSR and OTFP).

Karl Norris also knew that according to the combined Beer-Lambert's law, unwanted scattering-induced path length variations gave a multiplicative absorbance effect which could not be eliminated by purely additive modelling. So he chose to divide the spectral derivative in one wavelength region by the derivative in another.

Since I could not do that, I developed the MSC instead. This model-based preprocessing was later extended into EMSC to accommodate known spectral variation sources, and then optimized into a simple hybrid subspace model (OEMSC) in order to combine prior insight and empirical data into predictive models and improved insight.

In my lecture I shall discuss when to use chemometrics and when to use AI for NIR data.

AW02

DEALING WITH LIGHT SCATTERING IN NIR SPECTROSCOPY: REMOVE OR EXPLOIT?

Wouter Saeys[1]

[1]KU Leuven Department of Biosystems, MeBioS, Leuven, Belgium

While the attenuation of NIR radiation in transparent media is well-described by the law of Lambert-Bouger and Beer, the relation between the concentration of an absorbing species and the attenuation in turbid media is considerably more complicated in turbid media. While empirical scatter correction methods have their merit in reducing the spectral variation due to light scattering, they rely on assumptions which may not be fulfilled in real-life systems. In this presentation, it will be shown that combination of multiple diffuse reflectance and/or transmittance measurements with light propagation models allows to separate the effects light absorption and scattering (Fig. 1). While the former can be used to obtain more reliable estimation of the chemical composition, the latter can provide information on the microstructural properties. Different measurement configurations and modelling approaches for estimating the scattering and absorption properties will be discussed and the characterization of microstructure traits from the scattering properties will be demonstrated for different applications from the agrifood domain.

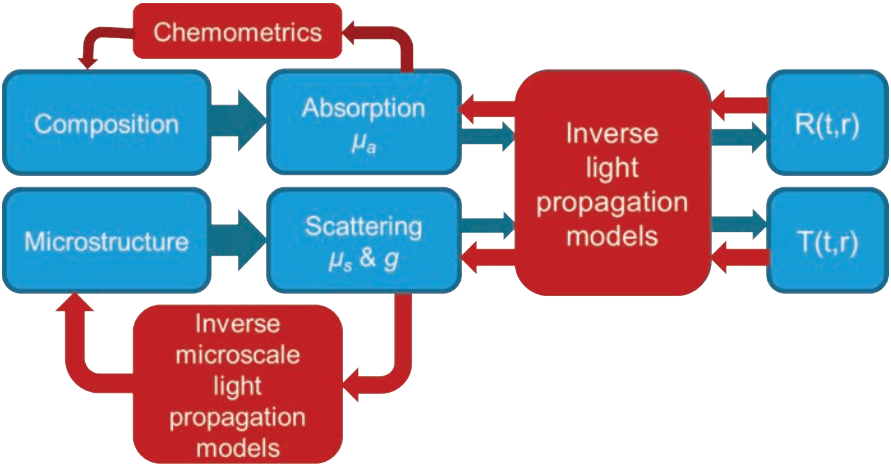


Figure 1. Schematic illustration of the model-based estimation composition and microstructure properties from diffuse reflectance and transmittance measurements.

AW03

A CAREER IN NXR SPECTROSCOPY: X = I,M

Roger Meder[1]

[1]Ridley Agriproducts / Meder Consulting / University of the Sunshine Coast, Narangba, Australia

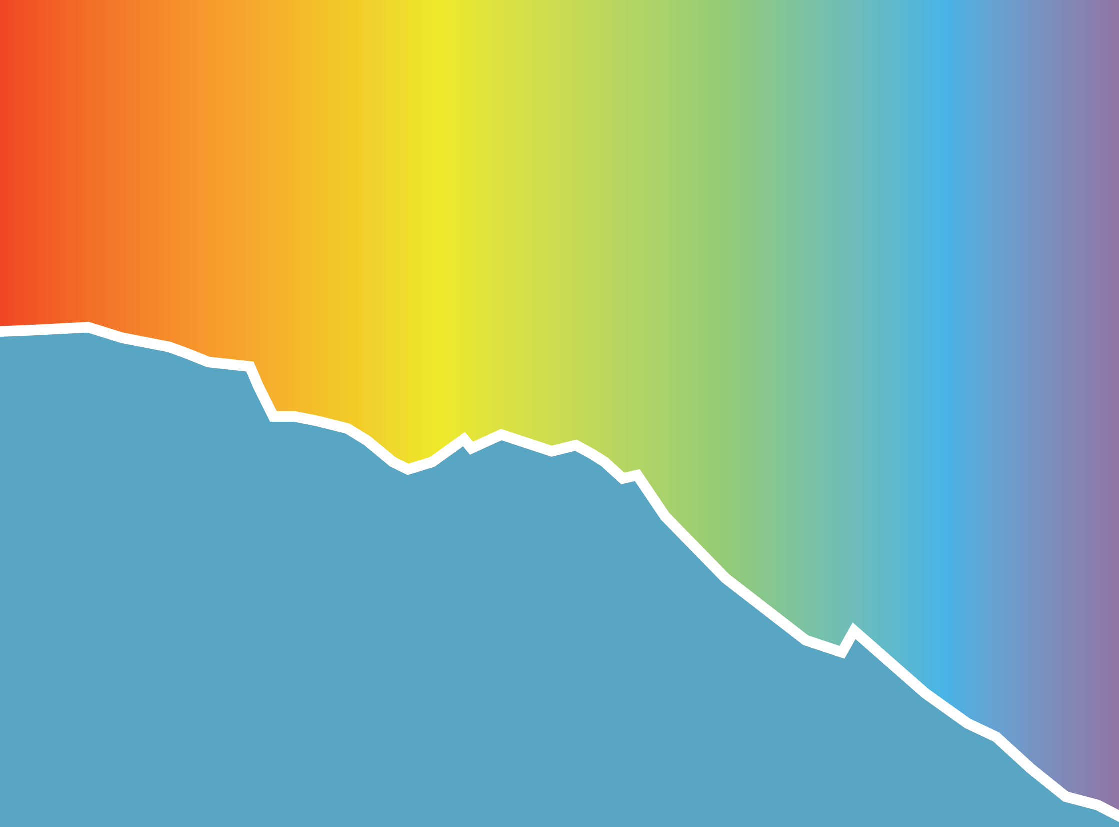
It is an honour to present the Tomas Hirschfeld award presentation for 2023. I would like to take moments and highlights from my NIR spectroscopy career, that has now spanned 30 years, to describe the thin threads that define and guide our scientific careers. This presentation will look at three key contributors to my career with examples of NIR (and NMR) spectroscopy

Find a Mentor
"If I have seen further it is by standing on the shoulders of giants."
Isaac Newton 1642-1726, English Mathematician/Physicist (attributed Bernard of Chartres 1159)
One thing I learned early in my career was to find good mentors – and not only other chemists. It is important to understand not only science, but other aspects of life. This helps place your work in greater context. Several people have helped me understand that. In this part of the presentation I will describe some of my early successful (and unsuccessful) developments in NIR spectroscopy. Don't be surprised if it doesn't always work.

Ask Deep Questions
"Think deeply about simple things."
Arnold Ross 1906-2002, American Mathematician
Children ask questions about everything. Their favourite word is "Why?". As adults we seem to lose the enquiring mind of children, yet as scientists, this is the trait we need to develop. I will present an area of calibration development that still intrigues me after 30 years, but by asking key questions it is possible to look at the problem afresh.

Solve Big Problems
"We can't solve problems by using the same kind of thinking we used when we created them."
Albert Einstein 1879-1955, German Physicist
There was a time when scientific discovery occurred within scientific disciplines. I started my career working with a group of chemists. However, the problems facing the world today require multidisciplinary teams and I have worked with physicists, geneticists, mechanical and electrical engineers, plant physiologists, biochemists, biologists and soil scientists. Today the exciting field of functional NIR brings together neuroscientists, human physiologists, physicists and chemists (or at least it should).

The future is promising once you look beyond your own field of expertise.



THEME 01

AGRICULTURE, DAIRY AND FOOD

ORAL PRESENTATION: 001.01 – 001.21
FLASH TALK: F01.01 – F01.07
POSTER PRESENTATION: P01.01 – P01.76

001.01

CONTRIBUTION OF NIRS TECHNIQUES TO ASSESS THE PRESENCE IN FEED OF AUTHORISED AND NON-AUTHORISED INSECT MEAL.

Vincent Baeten[1], Abiagaël Anselmo[1], Juan Antonio Fernández Pierna[1], Sébastien Gofflot[1], Audrey Pissard[1]

[1]*Walloon Agricultural Research Centre, Gembloux, Belgium*

Following the outbreak of the bovine spongiform encephalopathy (BSE), the European Commission took measures to protect the consumer. More recently, based on EFSA's recommendation, the European authorities agreed to introduce the use of insects in aquaculture (farmed fish) from July 2017. In August 2021, EC decided to revise the feed ban by authorising the use of insect-derived PAPs in feed for pigs and poultry and introducing an eighth insect species. The correct characterisation and identification of insect meals, the detection of foreign bodies and unauthorised feed materials as well as the detection the presence of insect particles in compound feed are required1.
In the framework of the EU Farmyng project and the European Union Reference Laboratory for Animal Protein in feedingstuffs (EURL-AP), different research studies have been carried out. 1) A sample bank of well-referenced and characterised insect meals has been built. This sample bank has been used as starting point to develop NIRS models to predict chemical composition of insect meals. The first results gave a RMSEP of 2.21, 2.97 and 1.22 % for protein, fat and chitin contents respectively. 2) In a second initiative, Near-Infrared Microscopy (NIRM), combined with chemometrics was investigated in order to develop a screening method to detect insect PAPs in feed without any chemical extraction step as is the case with current official methods. Different blends have been created from pig feed adulterated at levels of 1 %, 5 % and 10 % w/w with either H. illucens larvae meal or T. molitor larvae meal. Discriminant calibration models based on pure reference samples of insects and pig feed have been built using PLS-DA. Based on PLS-DA analysis, several spectra of insect particles in the blend were identified. An accuracy of 99 % and 98 % was achieved for the respective detection of T. molitor and H. illucens particles.

Keywords : insect meal, feed, NIRS, NIR microscopy, adulteration

[1] Anselmo A, Veys P, Fumière O, Lecrenier M-C, Cordonnier A, Michez D, Baeten V. 2023. Challenges related to the application of analytical methods to control insect meals in the context of European legislation. Submitted.

001.02

FOURIER TRANSFORM NEAR INFRARED SPECTROSCOPY OF OTOLITHS COUPLED WITH MACHINE LEARNING CAN PREDICT FISH AGE MORE EFFICIENTLY AND WITH COMPARABLE PRECISION COMPARED TO TRADITIONAL AGEING METHODS

Irina Benson[1], Thomas Helser[1], Beverly Barnett[2]

[1]Resource Ecology and Fisheries Management Division, Alaska Fisheries Science Center, National Marine Fisheries Service, NOAA, Seattle, United States, [2]Fisheries Assessment, Technology, and Engineering Support Division, Biology and Life History Branch, Southeast Fisheries Science Center, Panama City Facility, National Marine Fisheries Service, NOAA, Panama City, United States

Estimating the age of fishes is essential for understanding fisheries ecology and management. Traditionally, fishes have been aged using microscopic examination of otoliths, which are formed by daily deposits of alternating mineral-rich and protein-rich bands with annual periods of rapid and slower growth corresponding to opaque and translucent zones. The microscopic age estimation methods of fish otoliths are expensive, labor-intensive, and often subject to poor repeatability. We explore advanced technologies using Fourier transform near infrared (FT-NIR) spectroscopy coupled with machine learning to estimate fish age more rapidly and with greater efficiency than traditional approaches. New technology has recently been applied to the otoliths' diffuse reflectance measurements from walleye pollock (Gadus chalcogrammus), northern rockfish (Sebastes polyspinis), and red snapper (Lutjanus campechanus) (Table 1). Neural networks explored the underlying relationships between an otolith spectrum and fish age along with other biological and geospatial data that have an effect on fish growth. The proposed model was built on ingesting multimodal input of features. Convolutional neural network (CNN) was used for 1D spectral data because it is based on the shared-weight architecture of the convolutional kernels, which can easily lower data complexity and identify important spectral regions. Results from the hybrid CNN models between the FT-NIR predicted age and traditional age estimates for each of three species indicate that otolith spectra in the 4,000-7,000 cm^{-1} wavenumber region, which is associated with C-H and N-H functional groups, has the highest impact predicting fish age. Otolith weight, for which both fish age and somatic growth are determinants, has greater impact than fish length. The geographic coordinates or substock designation have an impact due to fish ontogenetic habitat shifts. Coefficients of determination (R^2)are 0.91 to 0.93 for train data and 0.89 to 0.92 for test data; root mean square errors (RMSE) are 0.83 to 3.37 for train data and 0.91 to 3.36 for test data (Table 1). Age estimation outcomes between the traditional and deep machine learning methods are largely indistinguishable based on Bland-Altman plots. Our results suggest that FT-NIR spectroscopy of otoliths coupled with deep machine learning can predict fish age ten times faster and with comparable precision.

Species	**Species Longevity**	**Collection Years**	**Features**	**n**	**R^2**		**RMSE**	
					Train	Test	Train	Test
walleye pollock	20+ years	2014-2018	FT-NIR spectra, fish length, latitude, gear depth, gear temperature	8,617	.93	.92	.83	.91
northern rockfish	80+ years	2015, 2017, 2019	FT-NIR spectra, otolith weight, fish length, latitude, gear depth	1,125	.92	.92	3.37	3.36
red snapper	50+ years	2017-2019	FT-NIR spectra, otolith weight, fish length, sub-stock	6,080	.91	.89	1.01	1.13

Table 1. Summary of available data and prediction results.

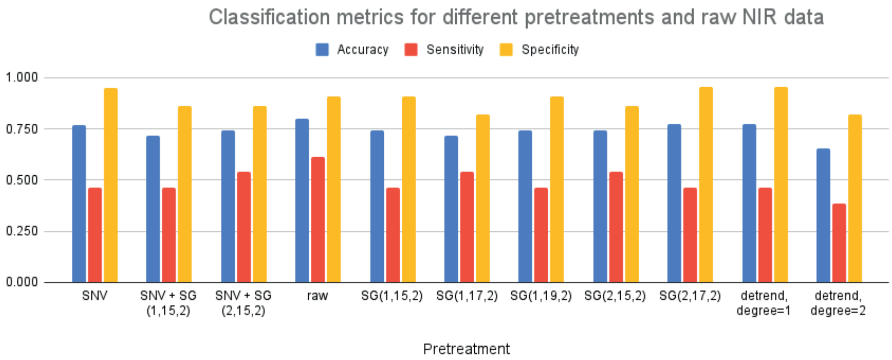
001.03

PREDICTING FUNGAL INFECTION SENSITIVITY OF SEPALS IN HARVESTED TOMATOES USING HYPERSPECTRAL IMAGING AND PARTIAL LEAST SQUARES DISCRIMINANT ANALYSIS

Mercedes Bertotto[1], Hendrik de Villiers[1], Aneesh Chauhan[1], Esther Hogeveen-van Echtelt[1], Manon Mensink[1], Željana Grbović[2], Dimitrije Stefanović[2], Marko Panić[2], Sanja Brdar[2]

[1]Wageningen University And Research, Wageningen, Netherlands, [2]BioSense Institute, University of Novi Sad, Novi Sad, Serbia

A new method was developed to classify sepal sensitivity to fungal infections of recently harvested tomatoes using spectral imaging and PLSDA. Previous work has been done by Brdar, S et al. (2021) and De Villiers, HAC et al. (2023), where the influence of variables were determined in the final model. However, in the present work an iterative process is used to select a sparse subset of important variables before their use by the final model. In this way, only a small subset of wavelengths needs to be measured in the unseen samples.



32 'Cappricia' tomatoes without any visible indications of fungal infection were imaged in two separate equally sized groups. Hyperspectral images were recorded on day one using a Specim FX17 NIR linescan camera. Subsequently, tomatoes were stored in controlled conditions encouraging fungal growth (20°C, in a closed box reaching 100% Relative Humidity, in a room at 60% RH, lights on during 7:00-19:00h, 15 $\mu mol \cdot s^{-1} \cdot m^{-2}$). Ground truth observations were made by experts on day three and four, comprised of severity scores from zero (no fungus) to three (severe infection). Ratings of the two days were averaged. Firstly, outliers were removed in each tomato, by PCA. The remaining pixels belonging to the same sepal were averaged giving rise to 167 rows of sepals.

Samples were distributed in two classes according to visual scoring. Class 1 (negative) included ratings of 0.5 or less. Class 2 (positive) included ratings of 1 or greater. The data set was then divided into calibration (70%) and validation (30%) sets, randomly, by tomato. Besides raw data, several preprocessing steps were performed (Figure 1). Models were built in the training set using 11 to 40 selected variables by CovSel. PLSDA latent variables were optimized as well, by cross-validation on each tomato. Figure 1 shows results of different models and the pretreatments used. In all of them the optimal number of variables was also optimized.

The important variables found in this work are (nm): 937, 944, 951, 971, 1089, 1152, 1306, 1356, 1391, 1440, 1540, 1675, 1704, 1711, 1718. The best results were obtained using raw data, the mentioned features, and 3 latent variables in PLSDA. The model presented high accuracy of validation, 0.80. Sensitivity and specificity were 0.62 and 0.91 respectively for class 1. Thus, the model presented potential as a fast alternative method to classify recently harvested tomatoes before the fungal infection is visually observed.

001.04

PREDICTIONS ACCURACY OF NUTRIENTS IS GREATLY REDUCED IN WET FEED AND FORAGES

Xueping Yang[1], Alejandra Arroyo-Cerezo[2], Luisa Magrin[1], **Paolo Berzaghi**[1]

[1]Department MAPS, University of Padua, Legnaro, Italy, [2]Department of Analytical Chemistry, University of Granada, Granada, Spain

Nutrients analysis in forages has been one of the first NIR successful applications. Traditionally forages are dried and then ground before any spectra collection, but wet forage analysis is gaining in popularity as it reduces time and workload, or it is necessary for on-site and on-line analysis.

Samples from chopped corn plant (CWP; n = 492) and high moisture corn (HMC; n = 405) were collected from farms in Italy over a period of 4 years. Samples collected during the last year of collection (CWP n = 46; HMC n = 41) were used as test set, and all the other samples as training set. Spectra were collected from undried unground and after drying and grinding (1mm screen) using two different NIR instruments (1100-2498 nm), one per type of sample. Reference chemical composition was expressed either as wet or also dry basis to evaluate their effect on NIR performance. Average moisture content was about 65% for CWP and about 35% for HMC with large variation for both products. Spectra of wet samples showed two large absorption peaks related to water, covering some of spectral features that were visible in dried samples. When expressed on the wet basis, nutrients had a lower concentration and lower standard deviation because of the dilution effect of water. Moisture content in wet samples was predicted well, with an error of 2% for CWP and 1.5% for HMC. When comparing performances on the dry matter basis, wet samples showed a marked decrease in accuracy of nutrients prediction compared to the same samples after drying and grinding. The increase in prediction errors was related to the moisture level of products, with a rise in errors, compared to the dry ground samples, of 60-70% for CWP and a limited increase of 10-15% for the drier HMC. In wet CWP both NDF and Starch had large prediction errors, which would be good only for qualitative evaluation. On calibration strategies for wet samples, there isn't a clear advantage using data expressed on wet or dry matter basis. The only difference is that on wet basis calibrations have greater RSQ as they are related to the dilution effect of water. In conclusion NIR analysis on very wet sample have a limited accuracy of prediction of nutrients and it may be used as qualitative evaluation, while for drier samples (water < 50%) NIR prediction of wet samples may represent an opportunity to simplify routine analysis.

001.05

MONITORING RAW MATERIAL VARIATION IN INDUSTRIAL ENZYMATIC PROTEIN HYDROLYSIS USING NEAR INFRARED SPECTROSCOPY

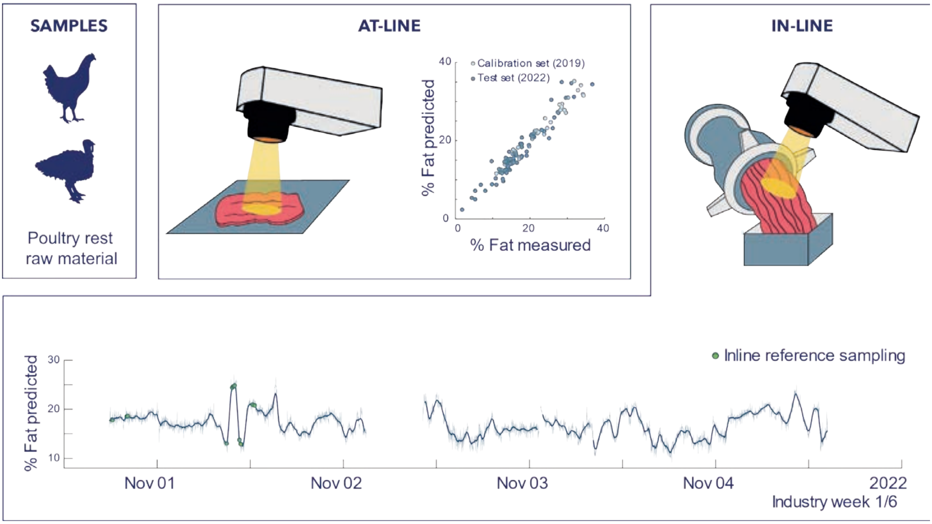
Katinka Dankel[1], Marco Cattaldo[1], Erik Tengstrand[1], Ingrid Måge[1],
Nils Kristian Afseth[1], Jens Petter Wold[1], Lars Erik Solberg[1]

[1]Nofima - The Norwegian Institute of Food, Fisheries and Aquaculture Research, Ås, Norway

A growing number of processing plants utilise enzymatic protein hydrolysis to upcycle rest raw materials from salmon and poultry production. Using catalytic amounts of proteases, valuable oils and protein products are extracted from chicken and turkey residues (carcasses, skin, and wings), or salmon trimmings (heads, backbones, skin, and belly flaps). For the industry, yield, degree of hydrolysis, bioactivity and taste of the water-soluble protein hydrolysates are typical examples of product parameters of interest. A slightly bitter flavour, commonly attributed to small peptides with hydrophobic side groups, is one of the challenges these industries seek to overcome prior to a large-scale launch in the human food market.

The rest raw materials used as starting materials in these reactions vary in gross composition due to the nature of the supply chains. Varying amount of fat, protein and water in the raw material causes variation in the end products, and near infrared spectroscopy (NIRS) is the obvious choice for in-line monitoring of these parameters.

An initial goal for the work presented here has been to model the correlation between raw material variation monitored with NIRS, process parameters, and product variation. A future ambition would be to use real-time NIRS monitoring of the raw material variation in a feed-forward manner, adjusting the process parameters accordingly to get a uniform and desired product.



When calibrating in-line partial least squares (PLS) prediction models from NIR spectra in these industrial settings, certain challenges have arisen. Collecting enough calibration data in-line to cover all future variation can be time-consuming. Also, when large volumes of heterogeneous material are ejected from a grinder and pass the NIRS sensor at a high speed, establishing a sound method for representative reference material sampling becomes a bit of a challenge. For these reasons, the current calibration work has started in an at-line fashion on designed samples prior to in-line implementation, calibration adjustments and finally independent test set validation.

The current presentation will be based on studies obtained in collaboration with two industry partners utilising enzymatic protein hydrolysis of salmon and poultry rest raw materials. We will give an overview of the calibration work performed, the challenges experienced and the implementation of in-line NIRS based prediction models in industrial settings.

001.06

USE OF HANDHELD NIR SPECTROSCOPY TO QUANTIFY AND CLASSIFY NUTRITIONAL PARAMETERS IN PASTA USING PLS, LW-PLS AND PLS-DA

Marina De Gea Neves[1], Heinz W. Siesler[1]

[1]University Duisburg-Essen, Essen, Germany

In times of increasing obesity, the assay of nutritional parameters such as carbohydrates, protein, fat, fiber, sugar, and energy in ready-to-eat-meals (e.g. pasta/sauce) by handheld near-infrared (NIR) spectrometers has recently become a realistic and potential tool for consumers to control and determine the quality of their meals based on the feeling of satiety, nutritional facts, and health considerations1. Partial Least-Squares (PLS) has been used to quantify the nutritional parameters, however, the question arises whether the spectra measured at different times, by different operators and with different pastas and ingredients can be combined in a master calibration.

To correct possible non-linearities and heterogeneities of samples and measurements, the Locally Weighted - Partial Least-Squares (LW-PLS) method was applied in this project. For carbohydrate, for example, calibration parameters of $R^2 \sim 0.90$, RMSEC 3.31 g/100 g and RMSECV 4.48 g/100 g were achieved by LW-PLS for a content range of 17.3 – 85.9 g/100 g. Figure 1 A shows 498 NIR spectra of different pasta/sauce formulations measured during three subsequent years after Extended Multiplicative Scatter Correction (EMSC), and truncation to 940 – 1355 nm. The comparative actual/predicted plots of the corresponding PLS and LW-PLS calibrations for carbohydrates are shown in Fig. 1 B and C, respectively.

Using LW-PLS significantly increased the quality of the calibration model compared to PLS using the same number of latent variables. The same procedure has been applied to the other nutritional parameters (energy, fat, protein, sugar, and fibers). These LW-PLS calibrations provide an overview of the realistic prediction accuracy achievable by handheld NIR spectroscopy.

Another aspect is the protection of consumers by classification of parameter contents into three classes (low, medium, high).

Therefore, Partial Least-Squares - Discriminant Analysis (PLS-DA), was applied to obtain classification models. However, before applying PLS-DA we created gaps in the dataset, to distinguish between low content (18 – 28 g/100 g), medium content (35 – 45 g/100 g), and high content (52 – 62 g/100 g). Thereby, we reduced the dataset from 498 to 254 samples. In Fig. 1, the corresponding PLS-DA carbohydrate models are shown in (D) (red); (E) (blue); and (F) (black), respectively. Sensitivity values for low, medium, and high reached 97.1%, 100.0% and 88.6% respectively, and specificity values varied between 93.4%, 90.2%, and 75.0%, respectively.

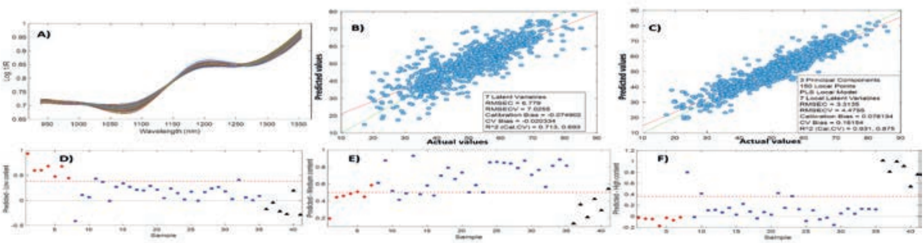


Figure 1. (A) EMSC pre-treated NIR spectra of 498 pasta samples; (B) carbohydrate PLS calibration; (C) Carbohydrate LW-PLS calibration; (D) carbohydrate PLS-DA prediction model for low content class (7 samples - red); (E) medium content class (28 samples - blue); and (F) high content class (6 samples - black).

References:
1. D. G. Neves, M., Poppi R., Siesler H. W., "Rapid Determination of Nutritional Parameters of Pasta/Sauce Blends by Handheld Near-Infrared Spectroscopy", Molecules, 2019, 24 (11), 2029.

001.07

DEVELOPMENT OF NIR CALIBRATION FOR PREDICTION OF AMINO ACIDS IN ANIMAL BY-PRODUCTS

Rocio Dona Garcia[1], Virginie Blanvillain[1], Sanami Tatekura[1], Gilson Gomes[1]

[1]Ab Vista, Marlborough, United Kingdom

Amino acids (AA) are the building blocks of proteins. Feed formulation is now widely based on AA levels rather than on protein, and this allows to optimize animal feed efficiency and growth performance, while reducing feed cost and total nitrogen excretion. Using Near Infra-Red (NIR) technology to determine the AA content of raw materials allows to compare the AA profile to expected levels, or book values, within a relatively quick turnaround, and at a minimal cost. Although NIR calibrations for AA are available for ingredients of vegetable sources, calibrations for ingredients of animal origin are scarce. Animal by-products are from the rendering industry, thus highly variable by design. They also represent a high relative contribution in the total AA content of feed rations, with crude protein level ranging typically between 40% to 70%.

This study aimed to develop AA calibrations for ingredients of animal origin. In total, 408 samples from 12 different animal by-products (e.g., porcine plasma, fish meal, meat and bone meal blends, feather meal) were collected from 18 countries over a 2 years period and analysed by wet chemistry for their total AA profile. The samples were scanned in duplicate using a benchtop NIR monochromator spectrometer (FOSS DS2500F, spectral range 850-2500 nm, 0.5 nm resolution).

The development work was taken using WinISI. The math treatment used, Standard Normal Variation and Detrend, 1st derivative, Gap 8, smooth 8. The outliers were based on spectral comparison (GH, NH) followed by T value. The standard error of calibration (SEC) and the R-square were calculated to evaluate the performance of the calibration. These models were then validated externally by random collection of 18 meat and bone meal blends from North America. The samples were scanned using benchtop NIR (FOSS DS2500F), analysed by wet chemistry in an independent laboratory, and the standard error of prediction (SEP) were calculated.

The Ratio of Prediction to Deviation (RPD) varied between 3.7 and 12.6, indicating very good performance to effectively monitor the quality of the animal by-products (Williams, 2014). Also, the SEP calculated from the external validation confirmed the repeatability of the models, independently of the lab used (HPLC method).

These results allowed to deploy the models on benchtop NIR instruments in different locations, while collecting more samples to increase the diversity of the database, and therefore, the robustness of the calibrations.

Amino acid	Average	Standard Deviation	SEC	R-square	SEP
Aspartic acid	5.07	1.64	0.19	0.987	0.22
Threonine	2.59	0.99	0.09	0.991	0.15
Serine	3.65	2.66	0.19	0.995	0.77
Glutamic acid	7.35	1.41	0.27	0.965	0.40
Glycine	5.94	1.41	0.24	0.972	1.02
Alanine	4.16	1.01	0.17	0.972	0.25
Valine	3.58	1.90	0.16	0.993	0.29
Isoleucine	2.12	1.09	0.11	0.990	0.17
Leucine	4.81	2.51	0.21	0.993	0.25
Tyrosine	1.59	0.67	0.17	0.935	0.10
Phenylalanine	2.77	1.40	0.13	0.992	0.15
Lysine	3.52	1.79	0.17	0.991	0.41
Histidine	1.44	1.34	0.11	0.994	0.17
Arginine	4.01	0.97	0.15	0.977	0.20
Cysteine	1.18	1.37	0.12	0.992	0.32
Methionine	0.96	0.42	0.07	0.972	0.08
Proline	4.43	1.64	0.17	0.990	0.27

001.08

NIR SPECTROSCOPY AND CHEMOMETRICS AGAINST FOOD FRAUD: SPOTTING MECHANICALLY SEPARATED MEAT (MSM) IN PROCESSED MEAT PRODUCTS

Alessandro Giraudo[1], Nicola Cavallini[1], Francesco Pennisi[2], Giovanna Esposito[2], Marzia Pezzolato[2], Francesco Savorani[1]

[1]Polytechnic of Turin, Turin, Italy, [2]Istituto Zooprofilattico Sperimentale del Piemonte, Liguria e Valle d'Aosta, Turin, Italy

Along the meat production chain, mislabelling and meat substitution can take place easily. This is typically the case of sausages in which minced meat can be intentionally replaced with Mechanically Separated Meat (MSM), partially or completely, with no mention in the product label. MSM is obtained by separating from the bone the edible tissues remaining after slaughter, under high pressure conditions. The quality and safety of this product are far lower than minced meat obtained by selected meat cuts, leading not only to health risks and deception for consumers, but also to a considerable economic advantage for producers [1].

In this study, NIR spectroscopy coupled with chemometrics was investigated as a rapid, non-destructive, cheap and green method primarily to discriminate between MSM and non-MSM samples and, as a second step, to quantify the amount of MSM. In order to investigate the suitability of both laboratory and onsite NIR acquisitions, three NIR spectrometers were considered: a benchtop NIR – MPA (Bruker) and two portable ones, i.e., MicroNIR (Viavi) and SCiO (Consumer Physics). The spectra acquired with each instrument were organized as a separated dataset and the same processing steps were operated on each of them.

For the classification step, 70 samples of poultry (chicken and turkey) sausages, including non-MSM and MSM samples, were longitudinally half cut and analysed. After exploratory analysis with PCA (Figure 1a), the spectra were used to build a PLS-DA model for each NIR dataset. For the regression step, sausages with different percentages of MSM were minced and mixed with non-MSM ones to obtain mixture samples with a specific MSM content. One PLS [2] model for each dataset was developed using a training set of 30 samples spanning a MSM content between 0% and 91% (in steps of 10%) and validated with a test set of 27 samples starting from the 5% of MSM content (in steps of 10%, until the 85%).

All the three NIR analytical techniques yielded good prediction performances both in classification, with model accuracies higher than 95%, and in regression (Figure 1b), with R^2 above 0.95 and RMSEP of 3.30%. These promising results suggest a successful applicability of the method, also using cheap portable instruments, to detect food frauds directly in the marketplace.

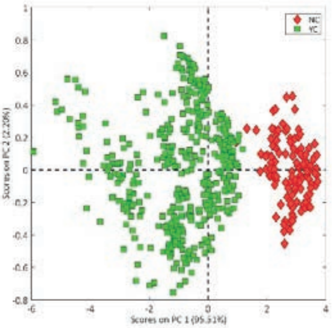


Fig 1a - PCA scores plot on SCiO spectra

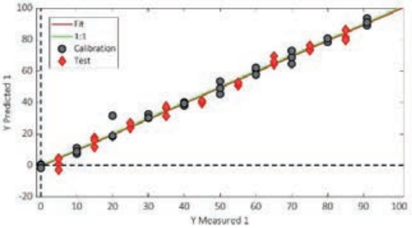


Fig. 1b - PLS results with SCiO spectra

[1] Barrere, V., et al. 2020. Food fraud vulnerability assessment: Towards a global consensus on procedures to manage and mitigate food fraud. Trends in Food Science and Technology, 100, 131-137. https://doi.org/10.1016/j.tifs.2020.04.002
[2] Wold, S., et al. 2001. PLS-regression: a basic tool of chemometrics. Chemometrics and Intelligent Laboratory Systems, 58, 109-130. https://doi.org/10.1016/S0169-7439(01)00155-1

001.09

NIR SPECTROSCOPY TO FIGHT VINEGAR ADULTERATION

Silvia Grassi[1], Cristina Alamprese[1]

[1]Università degli Studi di Milano, Milano, Italy

Vinegar is highly subjected to commercial frauds, such as substitution of value-giving content by inferior substances. Vinegar value chain protection from fraudulent behaviours should guarantee fare trades, and both producers' and consumers' rights. However, the expensive and complex analytical protocols disincentivise routine authenticity assessment.

In this framework, the work aims at determining the adulteration of wine vinegars with spirit vinegars by NIR spectroscopy. For this purpose, five white wine vinegars were adulterated with two different spirit vinegars from 5% to 25% (v/v), at 5% interval. Further twenty-five wine vinegars were considered to enlarge the authentic product variability. Twenty-five vinegars were characterized by 6% acidity, whereas the other five had 7.1% acidity. Samples (60 authentic and 100 adulterated vinegars) were analysed by FT-NIR spectrometer (MPA, Bruker) equipped with a 2 mm pathlength glass cuvette. Moreover, a handheld NIR device (Polispec NIR™, ITPhotonics) was tested on a representative number of samples (41 authentic and 48 adulterated vinegars).

Data exploration by Principal Component Analysis did not reveal sample patterns according to adulteration levels but related to the different vinegar acidity levels (6 or 7.1%). Linear Discriminant Analysis (LDA) models were developed after variable selection, by the SELECT algorithm, and tested in prediction by independent external sets.

The FT-NIR classification model gave very high average correct classification rate in calibration (99.3%), cross-validation (99.1%), and prediction (96.1%). The LDA model based on handheld NIR device data proved to be a valuable tool reaching 100% of correct prediction ability.

The proposed approach demonstrated to be a valid tool in vinegar authentication, suitable to assess the fraudulent behaviours, thus guaranteeing stakeholders' rights. However, a higher number of samples and different operating conditions (e.g., light sources, operators, etc.) should be tested to evaluate possible in-line applications of the handheld device in the food industry.

Acknowledgments

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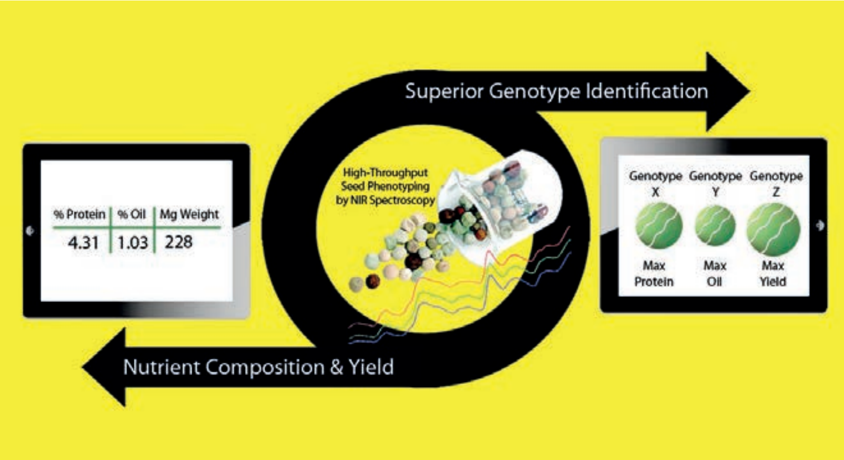
001.10

RAPID NON-DESTRUCTIVE PREDICTION OF OIL, PROTEIN, AND WEIGHT IN SINGLE PEA AND SOYBEAN USING SINGLE KERNEL NEAR INFRARED (SKNIR) SPECTROSCOPY

Gokhan Hacisalihoglu[1], P. Armstrong[2], M. Settles[3], J. Gustin[3]

[1]Florida A & M University, Tallahassee, FL, United States, [2]US Department of Agriculture, Manhattan, KS, United States, [3]University of Florida, Gainesville, FL, United States

Rapid non-destructive measurement methods for seed traits are valuable for plant breeding and global food safety. Single seed near-infrared (SKNIR) spectroscopy predicts multiple crop seed quality traits of moisture, oil, protein, and weight. Partial least-squares (PLS) regression gave accurate predictive models for oil, weight, volume, and protein traits of the seed. Calibrations for oil, protein, and weight were developed in this study. Furthermore, the ability to transfer calibrations between similar single-kernel NIR spectrometers facilitates broader adoption of this high-throughput, non-destructive, seed phenotyping technology. External validation of pea seed PLS models showed high prediction accuracy for protein and weight ($R^2=0.94$ for both). Single seed weight was weakly correlated with protein and oil content in peas. The high accuracy of protein and weight estimation shows that SNIR spectroscopy could be used in the dual selection of high-protein, high-weight peas early in the breeding cycle, allowing for faster genetic advancement toward improved seed nutritional quality. The current status of this project will be presented, including further research results.



001.11

NEW CONCEPTS FOR QUALITY EVALUATION OF FRESH PRODUCE IN JAPAN

Akifumi Ikehata[1]

[1]National Agriculture and Food Research Organization, Tsukuba, Japan

NIR spectroscopy has been widely applied to food and agricultural products. In Japan, nondestructive measurement of sugar content of fruits by NIR is essential for total inspection and grading at fruit sorting plants, and is very familiar to consumers. In recent years, attempts have been made not only to estimate sugar content, but as well as to estimate more diverse items. For example, with the system of "Foods with Function Claims" launch in 2015, interest has focused on rapid analytical methods. Attempts were made to quantify intact lycopene in tomatoes and ß-cryptoxanthin in mandarins. In recent years, there have been many requests for quantification of taste as part of the user experience. While people were forced to stay at home in COVID19, objective quality indices became necessary due to the rapid increase in the online sales market. In this presentation, I will report on recent topics, focusing on the development of a device to estimate the sensory evaluation score of tomatoes by NIR, and a small spectroscopic sensor to quantify the freshness of cabbage.

1. Tomato sensory evaluation score estimation
The sensory evaluation of six tomato varieties was conducted immediately after the non-destructive measurement of VIS and NIR spectra (XDS, Foss). 9-member sensory evaluation panels were selected and trained according to ISO 8586. Nineteen attributes that could be evaluated were selected in advance. We attempted to construct calibration models to estimate the score of each attribute by partial least squares regression (PLS regression) with the sensory scores as the objective variables. Although the estimation of odor was hopeless, several attributes of taste and texture showed the possibility of an unattended sorting. Based on these results, we modified a popular NIR sugar analyzer (Kubota) equipped with a Si detector and constructed a prototype sensory evaluation sensor.

2. Cabbage freshness
In Japan, the freshness of vegetables is the second most important concern after price. To ensure the definition of freshness, we first measured changes in gene expression in cabbage during storage. We defined the freshness of cabbage based on the gene expression changes over time, and trained small VIS and NIR spectroscopic sensors (Hamamatsu). The VIS and NIR sensors were not able to measure gene expression levels but were modeled based on increased leaf yellowing and browning, decreased chlorophyll, and decreased water content, respectively. A data platform to utilize the measurement results for distribution will also be reported.

001.12

HOW MICRO/PORTABLE NEAR INFRA-RED (NIR) SENSORS ACHIEVE LABORATORY-SCALE PERFORMANCE?

Hafiz Muhammad Hussain Khan[1,2], Ultan McCarthy[2], Norah O'Shea[1]

[1]Food Chemistry & Technology Department, Food Research Center, Teagasc Moorepark, Fermoy, Ireland, [2]School of Science and Computing, Department of Land Sciences, South East Technological University, Waterford, Ireland

Broad/overlapping and multiple responses from overtones and combination bands of functional groups are the main characteristics of NIR spectroscopy. Therefore, the response can be captured using micro/handheld NIR sensors. However, some disadvantages are associated with these sensors, such as a lag in the resolution, signal-to-noise ratio (S/N) and spectral range compared to the laboratory scale instruments. The study compared the capabilities of two micro NIR sensors (S-2.0 and S-2.5: Spectral Engines) with a benchtop instrument (MPA, Bruker Optics, Billerica, United States) for the compositional analysis of raw milk in transmission mode. A sample acquisition cell was designed and printed using a 3D printer to hold a flow-through cuvette (170-QS: Hellma Optics, Jena, Germany) and a NIR sensor on either side of the cuvette. The radiation source of the opposite sensor was used to illuminate the sample. The NIR spectrum of the same cuvette, along with the sample, was also acquired using the bench-top instrument. Calibration models were developed for fat and protein using PLS regression on a dataset of 100 samples. The prediction performance of S2.0 (RPD: 12.2 and 5.32 for fat and protein) exceeded the S2.5 (RPD: 2.83 and 2.37 for fat and protein) and laboratory-scale instrument (RPD: 5.5 and 5.15 for fat and protein), which can be explained by two reasons; the curse of dimensionality and the signal strength. The high-resolution laboratory scale devices output a large number of spectral variables, which increase the dimensionality of the data and can impact the prediction performance of calibration models. Moreover, high variability in the water absorption region and the dependence of NIR radiation's scattering and penetration on wavelengths can impact the prediction performance of developed models.

001.13

NIR INFRARED SPECTROSCOPY TO MEASURE THE EXTENT OF THERMAL TREATMENT OF MEAT ANALOGUES: FROM PROTEIN GEL TO HIGH MOISTURE EXTRUDATE

Nienke Köllmann[1], Lu Zhang[1], Atze Jan van der Goot[1]

[1]*Laboratory of Food Process Engineering, Wageningen University, Wageningen, Netherlands*

Meat consumption is linked to environmental, ethical and health issues. The production of meat analogues is one of the possible approaches to facilitate consumers to switch from animal-based to plant-based products. High moisture extrusion is widely used to produce meat analogues. However, the process is still considered a black box system because the effects of process parameters on the processing conditions and their resulting product properties are still unclear. In this study the use of near-infrared (NIR) spectroscopy was explored to quantify the extent of thermal processing during the production of meat analogues. The effect of heat treatment on meat analogue model systems was studied using products made in different ways. First, simple pea protein isolate (PPI) gels heated at different temperatures and for different times were investigated. This resulted in a successful partial least squares regression (PLSR) model for heating temperature (RMSEP (Root mean standard error of prediction) =5.5 oC, R2p(coefficient of determination of the prediction)=0.97) and heating time (RMSEP=1.06 min, R2p=0.99). In addition, an estimation of the combined effect of temperature and time could be made using an Arrhenius like approach (R2p=0.95). Second, high temperature shear cell (HTSC) processing was used to create structured soy protein concentrate (SPC) products. HTSC processing has similar processing steps as high moisture extrusion, but it allows individual control of the processing parameters, such as processing temperature and shear rate. In this study HTSC processing was used to produce fibrous products under controlled conditions. An accurate PLSR calibration curve for the processing temperature of these HTSC products could be created (RMSEP=3.53 oC, R2p=0.98). Finally, the PLSR model created for HTSC products was applied to high moisture extrudates to calculate the equivalent HTSC temperature. The equivalent HTSC temperature calculated for extrudates produced at different barrel temperatures and screw speeds showed a strong correlation with the measured melt temperature during extrusion. This indicates that NIR spectroscopy is a promising technique to quantify the extent of thermal processing during the production of meat analogues.

001.14

NEAR INFRARED SPECTROSCOPY GUIDE APPLE PUREE FORMULATION: AN INNOVATIVE STRATEGY TO DEVELOP ANTICIPATED AND CONSTANT PRODUCTS

Weijie Lan[1], Leiqing Pan[1], Kang Tu[1], Catherine Renard[2,3], Benoit Jaillais[4], Sylvie Bureau[2]

[1]Nanjing Agricultural University, Nanjing, China, [2]UMR^{408} SQPOV, INRAE, Avignon, France, [3]TRANSFORM Division, INRAE, Nantes, France, [4]StatSC, INRAE, Nantes, France

Nowadays, the increasing demands of various anticipated products for current consumers have put much stress on the food processing innovations from industrial manufacturers. It would be highly beneficial to develop innovative food production strategies, which can consider a large variability of raw materials and provide new solutions to reach the anticipated and constant taste and texture of food products.

Puree formulation is one of the most economical and efficient strategies for manufacturers to adjust the texture and taste of final puree products depending on a mixture of different proportions of single apple varieties. However, for fruit manufacturers, they often do not have the good choices to know how to formulate puree products with a large variability of raw apples. The challenge is therefore to develop innovative strategies to provide specific guidance for the formulation of final puree products based on the information of single cultivar puree, in order to reach their anticipated and constant taste and texture.

This report described an innovative concept concerning the feasibility of using visible and near infrared (Vis-NIR) spectroscopy to drive the formulation of apple purees issued from the mix of single-cultivar purees. An innovative chemometric method (multivariate curve resolution-alternative least squares (MCR-ALS) coupled with the spectra of single-cultivar purees) was firstly developed to reconstruct the spectra of formulated purees. As far as we know, this is the first report demonstrating that Vis-NIR spectroscopy has the potential to guide puree formulation: a multiparameter optimization of texture and taste (viscosity, color, sugars and acids) of final apple purees can be obtained using only the spectral data of single-cultivar purees.

For the sample preparation, four apple cultivars: 'Golden Delicious'(GD), 'Granny Smith'(GS), 'Braeburn'(BR) and 'Royal Gala'(GA) were processed into single cultivar purees during three consecutive weeks. A total of 6 formulated puree groups were composed each by two apple cultivars. Each group included 9 samples with different formulated proportions of weight, and divided into two subsets: 6 proportions (10%-90%, 25%-75%, 50%-50%, 75%-25%, 90%-10%, 95%-5%) for the modeling set, while the rest of 3 proportions (80%-20%, 33%-67%, 14%-86%) as the external prediction set. The Vis-NIR spectral data of single and formulated purees was both acquired from 400 to 2500 nm.

A specific exploration of data processing was designed in three steps:

1. Identifying the featured Vis-NIR spectral variables to predict formulated puree quality characterstics

PLS, RF and Cubist regression coupled with all the spectral variables (FULL) or the selected spectral variables based on SPA, CARS and UVE were applied to compare their ability to predict color, rheological and biochemical characteristics of formulated purees. PLS models coupled with three spectral variable selection methods offered the better predictions of puree physical, rheological and biochemical characteristics than using full Vis-NIR spectral variables alone. PLS models coupled with featured Vis-NIR spectral variables (matrix D') has promising to well estimate the a* color parameter (RPD = 5.56), viscosity n50 (RPD = 2.73), TSC (RPD = 3.66), TA (RPD = 3.37), pH (RPD = 2.73) and malic acids (RPD = 3.36) in formulated apple purees. Totally 671 Vis-NIR spectral variables were indetified from the 2722 spectral variables of full wavelengths

2. Spectral reconstruction of formulated purees based on their featured Vis-NIR spetcral vairibles of composed single cultivar purees

The relative contributions given by MCR-ALS were obtained between the featured Vis-NIR spectral information of formulated purees (matrix D') and their corresponding single-cultivar purees (matrix PT) (Figure. 1). The concentration profiles (matrix C) for each single-cultivar spectrum, including Golden Delicious (CGD), Granny Smith (CGS), Braeburn (CBR) and Royal Gala (CGA), were obtained describing the contribution of every single-cultivar purees in reconstructed purees, following Eq (1, 2):

$$D = CP^T + E \qquad (1)$$

$$C = D(P^T)^+ \qquad (2)$$

Afterwards, they were used to reconstruct a new spectroscopic matrix R for monitoring all formulated purees. Each row Ri. (i=1,...n) was made up of a reconstructed spectrum. And each column R.j (j=1,...k) gave the reconstructed spectral intensity at a wavelength of Vis-NIRS based on the corresponding pure puree spectra of Golden Delicious (λGD), Granny Smith (λGS), Braeburn (λBR) and Royal Gala (λGA) (Figure. 1), following Eq (3).

$$R = C_{GD}\lambda_{GD} + C_{GS}\lambda_{GS} + C_{BR}\lambda_{BR} + C_{GA}\lambda_{GA} \qquad (3)$$

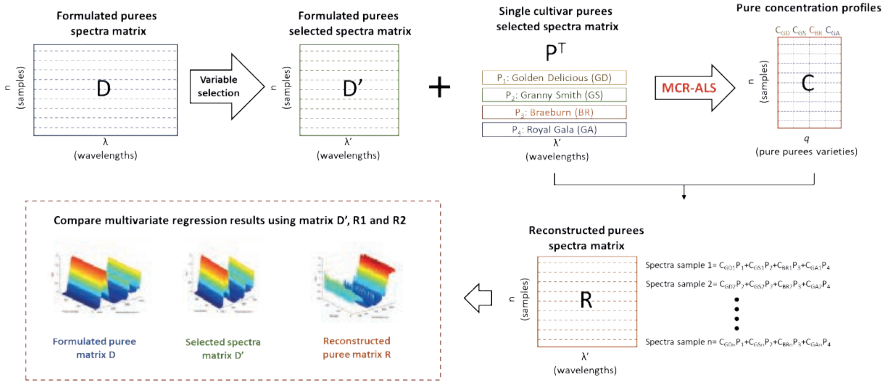


Figure 1

3. Predicting characteristics of formulated purees based on the reconstructed VIS-NIR spectra

PLS models were then developed using these reconstructed Vis-NIR spectra (matrix R) with the reference data characterized on the formulated purees. Overall, the PLS results of these aforementioned quality parameters based on the reconstructed spectra of formulated purees presented the relatively lower prediction accuracy than directly on real puree spectral. Notably, Vis-NIR spectra coupled with the concentration profiles of MCR-ALS showed a potential way to directly estimate the viscosity, a* color parameter (RPD = 3.30), TSC (RPD = 2.64), TA (RPD = 2.55), malic acid (RPD = 2.67), for formulated purees depending only on the CARS selected spectral information of single-cultivar purees.
It's the first time to reconstructed the spectra of formulated products using the concentration profile of MCR-ALS from selected Vis-NIR spectral variable of composed raw materials. Innovatively, this strategy opens the possibility guide the production of constant and anticipated purees by simply scanning the single-cultivar apple purees in apple industry. For instance, after acquiring Vis-NIR spectra of the four single-cultivar purees, our developed PLS models could: i) provide several strategies to formulate purees with defined tastes (e.g. 127.3 ± 5.7 g/kg FW of TSC and 7.8 ± 0.2 meq/kg FW of TA, which might be reached with the formulate solutions as 80% GS-20% GA, 33.3% GD-66.6% GS and 80% GS-20% BR purees), depending the might be used in industry; or ii) simulate and optimize puree formulation for anticipated products development depending on the market, such as 20% GD-80% GS purees (low sweetness, high acidity), 33.3% GD-66.6% GA purees (high sweetness, low acidity) and GS 14%-GA 86% (low sweetness, low acidity).
Further this new chemometric strategy has potential to provide production guidance of other food formulation, such as multifruit juices, blend oils, and even admixed flavoring agent etc, based on the Vis-NIR spectra acquired directly on their composed raw materials.
With TSC: total sugar content; TA: titratable acidity; RPD: residual predictive derivation; FW: fresh weight; CARS: competitive adaptive reweighted sampling; SPA: successive projections algorithm; UVE: uninformative variable elimination; RF: random forest.

Keywords: apples, purees, fruit processing, formulation guidance; food quality control

001.15

OXIDATIVE STABILITY PREDICTION IN OLIVE OIL BY NEAR INFRARED SPECTROSCOPY: APPLICATIONS IN OLIVE BREEDING.

Lorenzo León[1], Hande Yılmaz-Düzyaman[1], Francisco Jesús Gómez-Gálvez[1], Angjelina Belaj[1], Raúl De la Rosa[1], Nieves Núñez-Sánchez[2]

[1]IFAPA Centro Alameda del Obispo, Córdoba, Spain, [2]Departamento de Producción Animal, Universidad de Córdoba, Campus de Rabanales, [14071], Córdoba, Spain

Oxidative Stability Index (OSI) is a key parameter in the characterization of Extra Virgin Olive Oil (EVOO). OSI is related with different chemical compounds and, therefore, is an indirect indicator of commercial, nutritional and sensory properties of EVOO quality. OSI determination by reference Rancimat methodology is time-consuming so there is a need for developing fast and cheap analytical procedures, particularly regarding olive breeding programs where a high number of samples must be tested. The objective of this study was to evaluate the potential use of near-infrared spectroscopy (NIRS) for the analysis of OSI in EVOO. A total of 316 samples coming from different genotypes evaluated in the olive breeding program were scanned by NIRS between 12000 and 4000 $cm^{-1}$by using a Multi-Purpose Analyzer (MPA, Bruker) instrument in transmittance mode. Among the evaluated genotypes, there were a group of cultivars representing an olive core collection and another group of common cultivars evaluated under different environmental conditions. These two groups of samples were used to estimate genetic parameters (genetic and environmental variance and heritability) and the results between reference and NIRS estimated were compared. Partial least squares models developed using the whole dataset of samples showed correlation coefficient of 0.84 between predicted vs. reference OSI, and root mean square error of cross-validation of 3.58. The results obtained from OSI reference and NIRS predicted values showed similar variance components partitioning among sources of variation (genotype, environment, error) and, therefore, similar heritability estimates were obtained by both procedures. Besides, similar rankings of cultivars were obtained by both procedures so that selection for OSI could be accurately achieved from NIRS estimates. In summary, models derived from PLS regression using spectroscopic data showed promising results for determining OSI of EVOO, with particular advantages regarding breeding applications.

001.16

NONDESTRUCTIVE ESTIMATION OF GREEN VEGETABLE FRESHNESS WITH SCIENCE-BASED NIR SPECTROSCOPY

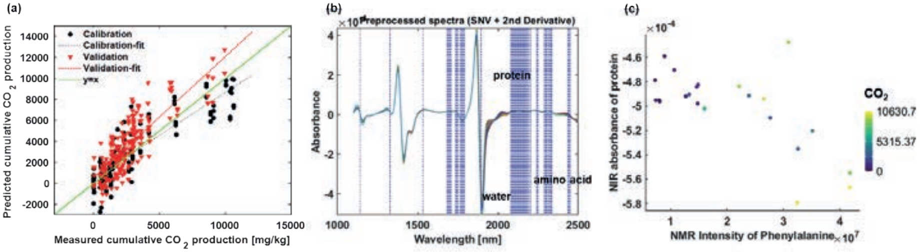
Xinyue Li[1], Mizuki Tsuta[1], Yasuyo Sekiyama[2], Nobutaka Nakamura[1],
Akifumi Ikehata[1]

[1]Institute of Food Research, National Agriculture And Food Research Organization, Tsukuba, Japan, [2]Research Center for Advanced Analysis, National Agriculture And Food Research Organization, Tsukuba, Japan

Visible and near-infrared (Vis-NIR) spectroscopy is often used with chemometric analysis as a "black-box" to build a calibration model for predicting indicators. It is necessary to open this "black-box" and understand its working mechanism before it can be applied reliably on-site. Therefore, this study investigated the ability and mechanism of Vis-NIR spectroscopy in estimating the freshness of vegetables, including komatsuna (Japanese mustard spinach) and broccoli, with the aim of opening the "black-box" of freshness prediction by Vis-NIR spectroscopy.

The monitored cumulative CO2 production of komatsuna and the cumulative temperature of broccoli under different storage conditions were used as indicators of freshness, and the measured Vis-NIR spectra of komatsuna and broccoli were used as predictors. Spectra were recorded using the XDS NIR spectrometry (Foss Analytical, Japan) with ISIscan software in transflectance mode with a wavelength range of 400-2500 nm and a resolution of 0.5 nm. After collecting Vis-NIR spectra, the same samples were used for NMR analysis to identify metabolites. Data analysis was performed by MATLAB with PLS Toolbox.

Based on the informative wavelengths (IWs) selected by the stepwise selectivity ratio (SWSR) method, accurate predictive models of the freshness of komatsuna (R2p=0.678) and broccoli (R2p=0.753) were constructed through PLSR analysis. In the figure, the PLSR analysis results of komatsuna are taken as an example. The blue wavebands marked in Fig. (b) are the IWs in the NIR region selected for building the calibration model (Fig. (a)), which can be assigned to protein, water, and amino acids. Based on the strong correlations between the signals of NIR-IWs and NMR, the key metabolites that could interpret the absorptions of NIR-IWs and are highly correlated with freshness were identified. Ten amino acids in komatsuna and seven amino acids in broccoli increased during storage. Among them, four amino acids such as phenylalanine were found to be common freshness marker metabolites shared by komatsuna and broccoli. Moreover, protein degraded to amino acids (Fig. (c)) because of the senescence that occurred during storage, which could explain changes in the absorbance of NIR spectra chosen to build the model.



In conclusion, Vis-NIR spectroscopy exhibited the ability and reliability to accurately estimate the freshness of vegetables such as komatsuna and broccoli. Protein degradation, detected as one of the important changes in NIR absorption, can be used to interpret the working mechanism of the calibration model constructed for freshness prediction.

001.17

EXPLORATORY STUDY OF THE EVOLUTION OF BÉCHAMEL SAUCES DURING COOKING: USE OF NIRS COUPLED WITH MCR-ALS ANALYSIS

Sonia Nieto Ortega[1], Silvia Mas García[2,3], Ángela Melado Herreros[1], Giuseppe Foti[1], Idoia Olabarrieta[1], Jean Michel Roger[2,3]

[1]AZTI, Derio, Spain, [2]ITAP, INRAE, Institut Agro, University Montpellier, Montpellier, France, [3]ChemHouse Research Group, Montpellier, France

The béchamel sauce is a traditional recipe used in many food preparations in the French cuisine, which has also been adopted by other countries. It is made of milk, flour, fat, salt and spices. The control of the process in the elaboration of the sauce is crucial, since small changes during the cooking will be reflected in the quality of the final product. However, despite the importance of this sauce in gastronomy, few works have been carried out to gain a better understanding of it.

The objective of the work has been to investigate the cooking process of 27 béchamel sauces using a hand-held near infrared spectroscopy (NIRS) device in combination with a multivariate curve resolution-alternating least-squares (MCR-ALS) approach. MCR models have been applied successfully to NIRS data in the past to understand physicochemical changes related with data coming from processes. In this work, MCR-ALS under hybrid hard- and soft-constraints was applied to elucidate the mechanism of the cooking process of evolving béchamel sauces aiming to resolve kinetic profiles and pure spectral signatures of the monitored products.

The MCR-ALS analysis for a particular cooking process showed that the evolving béchamel sauces could be described with a kinetic model of a first-order reaction $(A \underset{\rightarrow}{k} B)$. These two species (A and B) involved on the process were related to changes in light scattering and in the state of the water (Fig. 1).

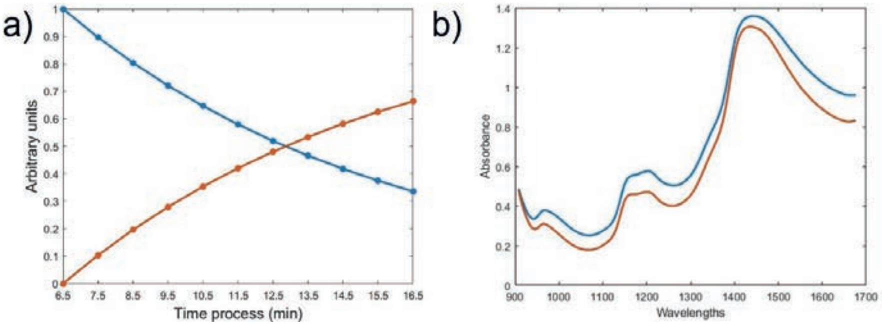


Fig. 1 MCR-ALS results from the elaboration process of the first béchamel sauce with hard-modelling constraints. a) kinetic profiles (C); b) pure spectral signatures (ST). Blue curve represents component A and red curve component B

While analyzing simultaneously the different sauces elaborated in a single multiset structure, the same kinetic model (first order reaction) can be used to describe the evolution of the whole set of sauces, although two additional species (C and D) were needed in order to fully capture the differences between sauces.

The methodology presented in this work offers a new strategy to study the elaboration of béchamel sauces in a nondestructive way, with the aim to give industrial producers a better understanding of their manufacturing process in a rapid and real time manner.

001.18

DEPTH-RESOLVED OPTICAL PROPERTIES OF WET AGED MEAT AND THEIR POTENTIAL FOR QUALITY EVALUATION

David Tjandra Nugraha[1], Annelies Postelmans[1], Wouter Saeys[1]

[1]KU Leuven, Leuven, Belgium

During wet aging of meat, numerous physicochemical quality characteristics evolve due to the enzyme-driven biochemical reactions. One of these parameters is the meat color, which is mostly attributed to the different forms of the myoglobin pigment. The three main forms are deoxymyoglobin (purple), oxymyoglobin (red), and metmyoglobin (brown). As the meat ages, a gradient of pigmentation is formed with different pigment concentration indicating the maturity of the meat. Considering this pigment gradient, diffuse reflectance measurements on the meat surface would not be able to give a complete view on the meat aging maturity due to the limited light penetration into deeper layers of meat, which may contain the pigmentation forms in different ratios/concentrations. To investigate the pigment gradient and the degree of meat aging, reflectance spectra of wet aged bovine longissimus lumborum of the Holstein race were collected using a fiber-based setup that punctures the meat. Spectra were collected at different depths inside the sample (1.5, 3.0, and 4.5 mm) in the 400-1000nm wavelength interval. Bulk optical properties (BOP) of the bovine muscles were also determined to study the effect of aging in the deeper parts of the meat sample. The BOPs were determined from thin tissue slices (0.2 mm) sampled at four depths at 1.5 mm intervals. The bulk absorption coefficient was used to study the pigment dynamics during the aging process, while the reduced scattering coefficient provided information on the structural evolution of the meat at different sampling depths. The two main absorption peaks of oxymyoglobin were found at 562 and 591 nm. The reduced scattering coefficient was found to increase during aging and was higher for the deeper meat layers. The obtained insights contribute to the development and optimization of optical sensors for minimally intrusive meat quality evaluation and monitoring of the meat aging process.

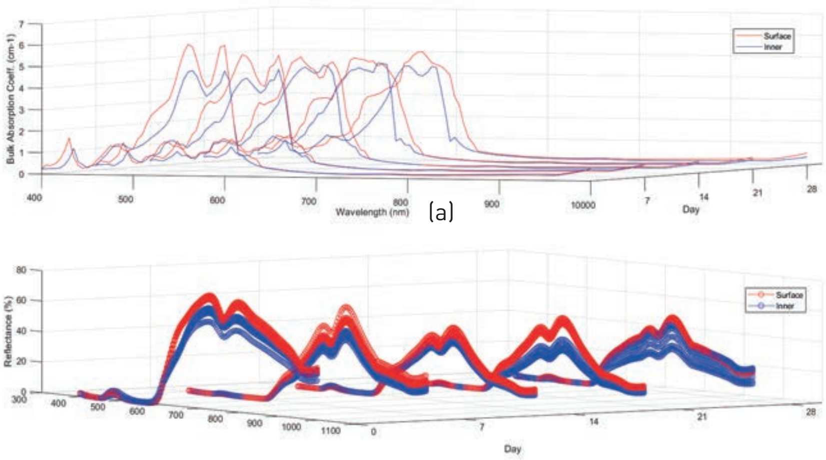


Figure 1. (a) Comparison of bulk absorption coefficient of wet aged Holstein bovine longissimus lumborum from the outer and inner part of the sample; (b) Comparison of reflectance spectra from the same sample.

001.19

ON-FARM NIR SENSOR FOR MILK QUALITY ANALYSIS: BIAS CORRECTION WITH UNSUPERVISED TECHNIQUES AND BULK MILK ANALYSIS

Arnout van Nuenen[1], Valeria Fonseca Diaz[1], Jose Antonio Diaz-Olivares[1], Wouter Saeys[2], Ben Aernouts[1]

[1]Ku Leuven, Department of Biosystems, Division of Animal and Human Health Engineering, Geel, Belgium, [2]KU Leuven, Department of Biosystems, Division Mechatronics, Biostatistics and Sensors, Leuven, Belgium

Regular analysis of the raw milk produced by individual cows can support dairy farmers in improving animal management and milk quality. Previous studies have shown that an on-farm sensor using near-infrared (NIR) spectroscopy can accurately predict the main milk components such as fat, protein and lactose. Unfortunately, studies on NIR applications, including monitoring milk quality, have also reported drift in the prediction performance over time, over different seasons or when environmental conditions vary (Diaz-Olivares et al., 2020). Therefore, calibration maintenance is essential to maintain good accuracy in the long term.

The data for this study was measured by a sensor system that automatically measures the reflectance and transmittance NIR (960 to 1690nm) spectra of milk for each milking performed by an automated milking system. The sensor system collected 20979 spectra between August 2021 and November 2022, for which 2511 also have laboratory fat, protein and lactose analyses available. The first 890 spectral measurements with available laboratory analysis results (August – November 2021) were used to train partial-least-square regression models, which were then used to predict the composition of the later samples. With these models, we achieved a prediction accuracy (root-mean-square error of prediction, RMSEP) of 0.12-0.13% (all % are in weight/weight) for fat, 0.14-0.19% for protein and 0.08-0.27% for lactose on the test data. In these data, we identified bias drift over time, which was also noticed in previous studies. Next, the bias was quantified with the help of an unsupervised bias monitoring method (Fonseca-Diaz et al., 2022), combined with the 2-day average bulk milk composition data, quantified in the context of the dairy payment system. The bias drift was successfully corrected by comparing the bulk milk composition against the weighted average predicted concentrations obtained with our sensor. Using this method, we reduced the RMSEP to 0.09-0.11% for fat, 0.08-0.09% for protein and 0.06-0.08% for lactose. As illustrated in Table 1, this approach shows a significant improvement compared to the non-bias-corrected prediction performance.

RMSEP (weight/weight)		Fat	Protein	Lactose
Transmittance	Raw	0.13 %	0.19 %	0.08 %
	Bias corrected	0.11 %	0.09 %	0.06 %
Reflectance	Raw	0.12 %	0.14 %	0.27 %
	Bias corrected	0.09 %	0.08 %	0.08 %

Table 1. RMSEP before and after correction

Díaz Olivares, J. A. O. (2020). Online milk composition analysis with an on-farm near-infrared sensor.
Fonseca Diaz, V. D. (2022). Cost-efficient strategies for building, transferring, and maintaining multivariate calibration models for spectroscopic sensors.

001.20

APPROACHING AUTHENTICITY ISSUES IN PROCESSED MEAT PRODUCTS WITH HYPERSPECTRAL IMAGING

Paul J Williams[1], Kiah Edwards[1], Marena Manley[1], Louw Hoffman[2]

[1]Stellenbosch University, Stellenbosch, South Africa, [2]Centre for Nutrition and Food Sciences Queensland Alliance for Agriculture and Food Innovation (QAAFI), The University of Queensland, Queensland, Australia

This study demonstrated that NIR-HSI and SVM-C can be used for the classification and authentication of beef patties and for detecting and quantifying adulterants and fat content. In total 800 beef patties (200 per category ['ground patty'; regular 'burger patty'; 'value-burger/patty' and the 'econo-burger'/'budget']) were imaged with a SWIR hyperspectral system in the wavelength range of 950-2500 nm. Various classification algorithms were evaluated, with SVM-C models achieving an accuracy of ≥98.5% in distinguishing between the four categories. We also investigated the detection and quantification of adulteration in the fat content of raw beef patties. The SVM-C models almost perfectly classified fat content classes with accuracies of 98-100%, while SVM regression (SVM-R) achieved an RMSEP of 3.6% for the best quantification results. In addition, we classified and quantified an assortment of adulterants (pork, lamb, ostrich, textured vegetable protein, and mechanically recovered meat) in raw beef patties, achieving accuracies of ≥98% in distinguishing between authentic and adulterated patties. The SVM-R models predicted the adulteration levels with RMSEP values of 5.14%, 3.71%, and 3.37% for pork, lamb, and ostrich substitution, respectively. With SVM-C models, we were capable of predicting total meat and mechanically recovered meat contents with accuracies of 98% and 97%, respectively. Finally, waveband reduction and optimization were conducted for potential development of a multispectral imaging (MSI) system for beef patty classification and authentication. Three waveband selection algorithms were explored: PC loadings (28 wavebands), recursive feature elimination (31 or 33 wavebands), and variable importance in projection (25 wavebands). Overall, SVM-C-RFE allowed for the individual classification of 14 classes and shows promise for extending the implementation of HSI and/or MSI for the on-line authentication of processed meat products at an industrial scale.

001.21

STRAWBERRIES GO HIGH-TECH - NOVEL SAMPLING AND MEASUREMENT NIR SOLUTIONS IN PRACTICE PART 2

Jens Petter Wold[1], Petter Andersen[1], Kjersti Aaby[1], Siv Remberg[2], Anders Hansen[3], Marion O'Farrell[3], Jon Tschudi[3]

[1]Nofima, Ås, Norway, [2]Norwegian University of Life Sciences, Ås, Norway, [3]SINTEF Digital, Oslo, Norway

Autonomous field robots are being developed for fruit picking, where each fruit needs to be individually graded and handled. It is therefore a need for rapid and non-destructive sensing to measure critical fruit quality parameters. In this presentation we will show how total soluble solids (TSS), a measure for total sugar content, can be measured in strawberries in the field by non-contact near-infrared (NIR) interactance spectroscopy. A specially designed prototype NIRS system working in the wavelength range 760-1080 nm has been tested for this purpose. This novel instrument is compared with a commercial handheld NIR reflection instrument working in the range 900-1600 nm. The instruments were calibrated in the lab using data collected from 200 strawberries of two varieties and tested in a strawberry field on 50 berries. Both systems performed well during calibration with root mean square errors of cross validation for TSS around 0.55 % and 0.65 %, for interaction and reflection, respectively. For prediction of TSS in new berries, the interactance system was superior, most likely because it probes deeper into the berries. The results suggest that interaction measurements of average TSS are more robust and would most likely require less calibration maintenance compared to reflection measurements. The non-contact feature is important since it reduces the spread of diseases and physical damage to the berries.

The non-contact interaction system has also been tested for applications such as rapid determination of meat content in the legs of live king crabs, and for water content in dried salted cod. Performance in these cases is also superior to that of reflectance measurements. Examples will be presented and discussed, and the importance of instrument design underlined.

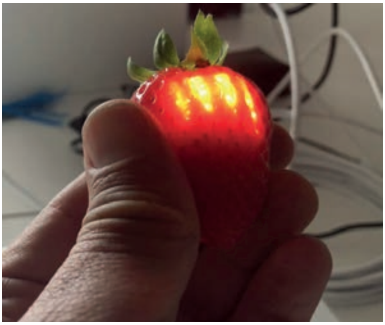


Figure 1. Rapid and non-contact assessment of soluble solids in strawberry

F01.01

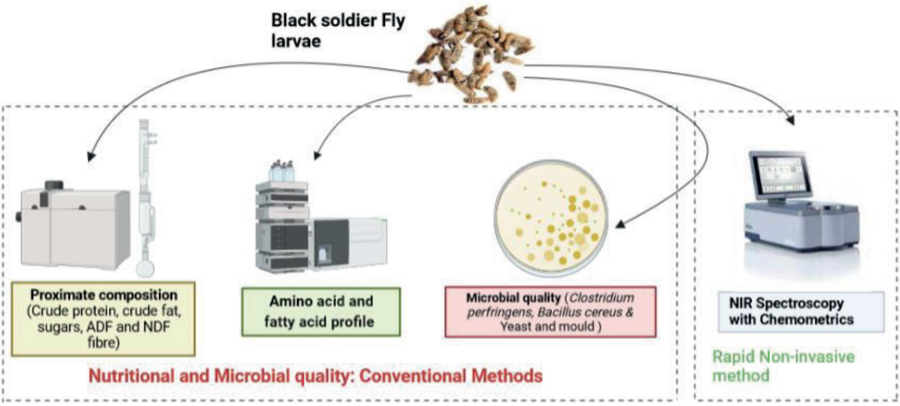
NEAR INFRARED SPECTROSCOPY FOR MONITORING THE NUTRITIONAL AND MICROBIAL QUALITY OF BLACK SOLDIER FLY LARVAE (BSFL)

Shanmugam Alagappan[1,2], Daniel Cozzolino[1], Louwrens Hoffman[1,2,3,]
Deirdre Mikkelsen[4], Peter James[5], Sandra Olarte Mantilla[1], Olympia Yarger[6]

[1]CNAFS, Queensland Alliance for Agriculture and Food Innovation, The University of Queensland, Brisbane, Queensland 4072, Australia, [2]Fight Food Waste Cooperative Research Centre, Wine Innovation Central Building Level 1, Waite Campus, Urrbrae, SA 5064, Australia, [3]Department of Animal Sciences, University of Stellenbosch, Private Bag X1, Matieland, Stellenbosch 7602, South Africa, [4] School of Agriculture and Food Sciences, Faculty of Science, University of Queensland, Brisbane, Queensland 4072, Australia, [5]Centre for Animal Science, Queensland Alliance for Agriculture and Food Innovation, The University of Queensland, Brisbane, Queensland 4072, Australia, [6]Goterra, 14 Arnott Street, Hume, Australian capital Territory 2620, Australia

Global food security is threatened as the world population is projected to increase up to 11 billion people by the end of this century. FAO's recent report suggests that the meat demand is expected to increase up to 14% by the year 2030 with a high demand for poultry meat followed by pork, beef, and goat. However, livestock production with conventional feed ingredients and practices is becoming more challenging and accounts for 18% of the greenhouse gas emissions contributed by the agri-food sector.

The use of edible insects, especially black soldier fly larvae (BSFL) as sustainable feed ingredients has been receiving a lot of attention lately. BSFL can valorise different organic side streams and are rich in nutrients with high levels of crude protein (32-46%), crude fat (15-38%), fibre (20.4-33.5%) and several other micronutrients. The amino acid and fatty acid profile of BSFL is balanced and considered to better than fish and soybean meal making them a suitable feed ingredient. However, the commercialization of BSFL is limited due to lack of knowledge about the safety of BSFL, regulatory framework, and the absence of relevant feed safety management systems.



The use of Near Infrared Spectroscopy (NIRS) in combination with chemometrics to rapidly monitor various quality attributes in food and feed sectors has been increasing over the past years. The aim of this study is to use NIRS in combination with chemometric techniques to rapidly determine the nutritional and microbial quality of BSFL reared on different waste streams. In our studies, BSFL reared on 3 different waste streams were harvested and subjected to two different post-harvest treatments (blanching and drying). The larvae along with the feed and left-over substrate (frass) were subjected to the following nutritional analysis: crude protein, crude fat, acid detergent fire, neutral detergent fibre, sugars, amino acid, and fatty acid profile. The above-mentioned samples were also subjected to detection and enumeration of pathogenic microbes including Clostridium perfringens, Bacillus cereus, yeast & mould counts. The FT-NIR spectra was collected in reflectance for all samples to which partial least squares regression (PLS) and PLS-Discriminant analysis was employed to build calibration models for the different predictor variables. Our findings indicate the possibility of implementing NIR in commercial manufacturing facilities to monitor some of the above-mentioned parameters in BSFL.

F01.02

NIR SPECTROSCOPY TOWARDS GREEN TRANSITION

Tomasz Czaja[1], Søren Balling Engelsen[1]

[1]Department of Food Science, University of Copenhagen, Frederiksberg, Denmark

Sustainable food production and a healthy lifestyle is fast becoming overarching trends, but also a necessity in our fast-paced society where a healthy diet is a crucial element of our well-being. This puts the food industry in a critical and changing period, when the consumer wants to take control of what they want to eat. In recent years, food manufacturers have reacted to growing flexitarian and vegetarian trends. This accelerated green transition requires dramatically increased usage of plant based raw materials and ingredients. Plant proteins need less energy for production and the objective is to broaden the choice of proteins and novel food products. Proteins are used not only for their nutritional value but also for their structuring properties. The latter depend on their functional properties including variability and food implementation contexts: composition, pH, salt concentration, temperature and process. The many meat alternatives emerging on the market rely on plant proteins, which are complex and vary in structural and functional properties such as water-holding ability, gelling and emulsifying ability. This study investigates the potential of NIR spectroscopy in accelerating the transition towards more sustainable and environmentally friendly plant-based foods.

Near Infrared spectra were recorded for plant materials of ten varieties of three different crops (faba bean, pea and oat) cultivated in conventional and organic way. From the crops proteins were extracted as isolates and concentrates. Using a carefully planned experimental design, plant proteins are added to different plant-based food products and their emulsifying performance were studied by NIR and LF-NMR spectroscopy.

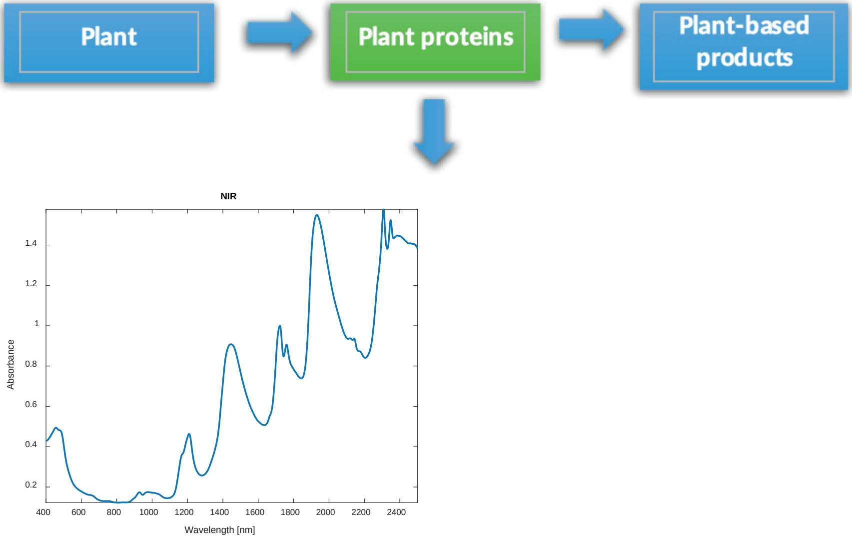


Figure 1. Study design

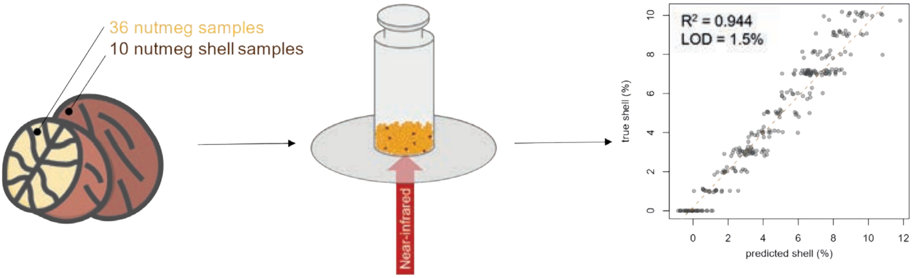
F01.03

RAPID DETERMINATION OF NUTMEG SHELL CONTENT IN GROUND NUTMEG USING FT-NIR SPECTROSCOPY AND MACHINE LEARNING

Alissa Drees[1], Bernadette Bockmayr[2], Michael Bockmayr[3], Markus Fischer[1]

[1]University Of Hamburg, Hamburg, Germany, [2]Husarich GmbH, Hamburg, Germany, [3]University Medical Center Hamburg-Eppendorf, Hamburg, Germany

Nutmeg is a popular spice often used in ground form which makes it highly susceptible to food fraud. Therefore, the aim of the present study was to detect adulteration of ground nutmeg with nutmeg shell via Fourier transform near-infrared (FT-NIR) spectroscopy. For this purpose, 36 authentic nutmeg samples and 10 nutmeg shell samples were analysed pure and in mixtures with up to 50% shell content. The spectra plot as well as a Principal Component Analysis (PCA) showed a clear separation trend as a function of shell content. A Support Vector Machine (SVM) regression used for shell content prediction achieved an $R^2$of 0.944 in the range of 0–10%. The limit of detection of the prediction model was estimated to be 1.5% nutmeg shell. This is a great improvement in comparison to the detection limit of current analysis methods (⩾ 5% shell content). Based on random sub-sampling, the likelihood was found to be 2% that a pure nutmeg sample is predicted with a nutmeg shell content of >1%. The results confirm the suitability of FT-NIR spectroscopy for rapid detection and quantitation of the shell content in ground nutmeg.



F01.04

NEAR-INFRARED SPECTROSCOPY FOR MYCOTOXIN SCREENING IN FOOD CROPS: CURRENT STATE-OF-THE-ART AND CHALLENGES

Stephan Freitag[1], Michael Sulyok[1], Rudolf Krska[1]

[1]University of Natural Resources and Life Sciences, Vienna, Department of Agrobiotechnology (IFA-Tulln), Institute of Bioanalytics and Agro-Metabolomics, Tulln, Austria

Mycotoxins are omnipresent toxic secondary metabolites produced by fungi. Exposure to these contaminants in food and feed can have negative effects on the health of humans and animals. As a result, tens of thousands of analyses are carried out every year by regulatory bodies and the industry to ensure food and feed safety. Liquid chromatography coupled with tandem mass spectrometry and enzyme-linked immunosorbent assays are commonly used techniques for this purpose. However, both techniques require skilled personnel, laboratory equipment such as mills, and heavily rely on consumables.

Near-infrared spectroscopy (NIRS) as a non-destructive technique enables analysis without the need for sample preparation or consumables, making it inherently eco-friendly. NIRS thereby facilitates on-site analysis and high sample throughput, in contrast to the beforementioned methods. While NIRS has been recognized for mycotoxin screening, its routine implementation is still pending since method development is significantly more challenging compared to other techniques as it often requires comprehensive sample collection trials accompanied by proper reference and data analysis. This is linked to the fact that mycotoxin contamination is indirectly determined using NIRS. The concentration obtained by the reference method is correlated to spectral changes induced by the growth of mycotoxigenic fungi using chemometrics.

In this presentation, we provide a summary of the current state-of-the-art of NIRS for the determination of mycotoxins in food crops, including an overview of current research activities conducted by our institute on the usage of NIRS for mycotoxin screening in wheat and maize.

F01.05

APPLICATION OF DEEP LEARNING AND NEAR INFRARED SPECTROSCOPY FOR ASSESSMENT OF QUALITY FOR ASSAM CTC TEA

Ajanto Hazarika[1], Dilip Sing[2], Manisha Hazarika[1], Santanu Sabhapondit[1], Romen Gogoi[1], Sandip Sanyal[1], Rajib Bandyopadhyay[2]

[1]Tocklai Tea Research Institute, Jorhat, India, [2]Jadavpur University, Kolkata, India

Quality of black tea may be assessed by bio-chemists or by tea tasters. Bio-chemical procedures are precise, but are generally destructive, skill requiring and time consuming. This method is more popular among the scientists. On the other hand, quality of tea is traditionally assessed by professional tea tasters to rank teas and to decide its market price. This is done based on the sensory attributes viz., brightness, briskness, strength and overall quality. But their assessment is subjective. Though the professional tea tasters are well trained to be least affected by extraneous factors, the possibility of being influenced by physiological, psychological and environmental factors cannot be completely ruled out. This may lead to inconsistency.

The goal of this study is to develop a chemometric model with deep learning method, using a portable near-infrared (NIR) spectrometer and graphical user-interface for rapid estimation of quality of crush-tear-curl (CTC) black teas. A total of 80 Assam CTC black tea samples were considered for the study.

The absorbance spectra of tea samples were taken in the near infrared (NIR) wavelength range of 900 nm to 1701.1 nm. The spectra were acquired with scan average 16 and integration time of 300 ms. A total of 256 spectral points were produced by the spectrometer for each sample. Fifteen replicates were taken for each sample, so for 80 samples a total of 256 × 80 × 15 spectral data points were obtained. Principal component analysis (PCA) was used to reduce data before Artificial neural network (ANN) analysis. ANN algorithm was constructed by three layers - one input layer, two hidden layer and one output layer. Using backward and forward propagation, the weights and biases, associated with the neurons, are adapted to minimize the error. The activation function “relu” was used for hidden layer. To develop the model, “Adaptive Moment Estimation (adam)” optimizer was used in ANN compilation. The loss (mean square error) of the training set was 0.0042. We applied this deep learning model in our test data set and got average deviation around 6.77 %. This result demonstrated that NIR spectroscopy and deep learning model can be used to assess the quality of CTC black tea.

F01.06

STUDY ON BLACK SPOT DISEASE DETECTION AND VISUALIZATION ON WINTER JUJUBES USING HYPERSPECTRAL IMAGING SYSTEM

Kang Tu[1], Mengwei Jiang[1], Leiqing Pan[1], **Weijie Lan**[1]

[1]*Nanjing Agricultural University, Nanjing, China*

Winter jujube (Ziziphus jujuba Mill. cv. Dongzao) is one of the most important fresh-eating jujube varieties in China. However, they are susceptible to black spot disease caused by Alternaria alternata, can cause fruit decay and quality reduction during storage, resulting in considerable losses to the winter jujube industry. Therefore, it's of great importance to realize the monitoring and detection of the black spot disease on winter jujubes. In this work, the potential of a hyperspectral imaging (HSI) system for the detection of black spot disease on winter jujubes infected by Alternaria alternata during postharvest storage was investigated. The HSI images were acquired using two systems in the visible and near-infrared (Vis-NIR, 400 –1000 nm) and short-wave infrared (SWIR, 1000 –2000 nm) spectral regions. Meanwhile, the change of physical (peel color, weight loss) and chemical parameters (soluble solids content, chlorophyll) and the microstructure of winter jujubes during the pathogenic process were measured. The results showed the spectral reflectance of jujubes in both the Vis-NIR and SWIR wavelength ranges presented an overall downtrend during the infection. Partial least squares discriminant models (PLS-DA) based on the HSI spectra in Vis-NIR and SWIR regions of jujubes both gave satisfactory discrimination accuracy for the disease detection, with classification rates over 92.31% and 91.03%, respectively. Principal component analysis (PCA) was carried out on the HSI images of jujubes to visualize their infected areas during the pathogenic process. The first principal component (PC1) of the HSI spectra in the Vis-NIR region could highlight the diseased areas of the infected samples. Consequently, Vis-NIR HSI and NIR HSI techniques had the potential to detect the black spot disease on winter jujubes during the postharvest storage, and the Vis-NIR HSI spectral information could visualize the diseased areas of jujubes during the pathogenic process.

Keywords: winter jujube; black spot disease; hyperspectral imaging; pathogenic process visualization

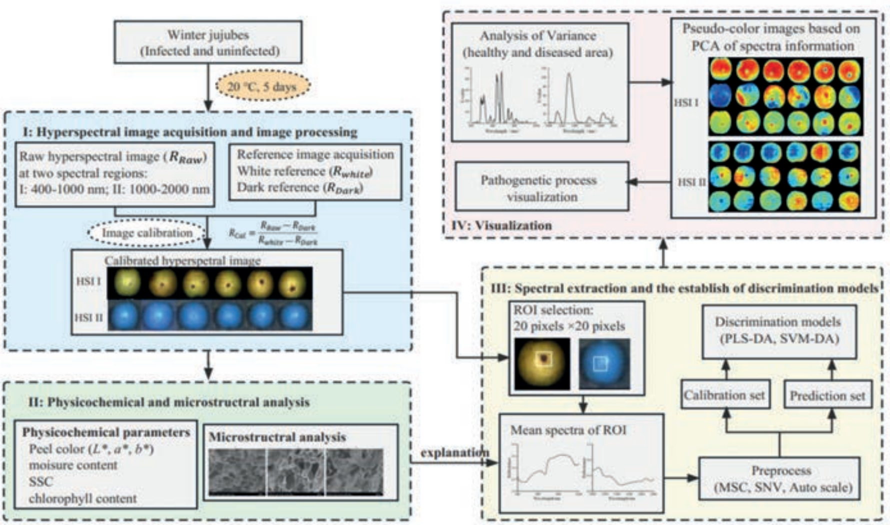


Figure 1. Procedures for black spot disease detection and pathogenetic process monitoring on winter jujube fruit using the HSI system in two spectral regions. (L^*: lightness (0 = dark, 100 = light); a^*: greenness (–)/redness (+); b^*: blueness (–)/ yellowness (+); SSC: soluble solids content; ROI: region of interest; PLS-DA: partial least squares discrimination analysis; SVM-DA: support vector machine discrimination analysis; PCA: principal component analysis).

F01.07

THE USE OF NEAR INFRARED SPECTROSCOPY FOR THE ANALYSIS OF FUMONISIN B1 DISSOLVED IN WATER AND METHANOL

Anja Laubscher[1], **Paul J Williams**[1], Lindy J Rose[2]

[1]*Department of Food Science, Stellenbosch University, Stellenbosch, South Africa,*
[2]*Department of Plant Pathology, Stellenbosch University, Stellenbosch, South Africa*

Fumonisin B1 (FB1) is a mycotoxin produced by Fusarium fungi which has been classified as a group 2B human carcinogen. FB1 regularly contaminates maize, which is an important staple food in South Africa, and therefore, Maximum levels (MLs) for FB1 in raw maize has been established at 4 ppm. To ensure adherence to these levels, efficient detection methods are required. Near infrared (NIR) spectroscopy has gained widespread interest as a rapid and non-destructive mycotoxin analysis method. Various studies used this technology to successfully classify maize kernels according to their FB1 content. However, these studies measured properties which are associated with FB1, such as the fungal contamination and its effect on maize and used it to predict the FB1 content. Due to the focus on these properties, the absorbance bands of FB1 in maize kernels have not yet been determined. Therefore, the purpose of this study was to determine the NIR absorbance bands of FB1. The spectra of 100 ppm FB1 solutions, constituted in methanol, were recorded at 1000 – 2500 nm. The spectra were pre-processed with multiplicative scatter correction and the variable importance in projection scores and selectivity ratio values were used for wavelength selection. A PLS-DA model was computed with 150 wavelengths and 9 latent variables and a 100% classification accuracy was achieved for both the calibration and external validation sets. The regression vector was investigated and important FB1 bands were identified at 1446 nm, 1453 nm, 1891 nm, 2036 nm, 2046 nm, 2148 nm, 2224 nm, 2262 nm and 2273 nm. To confirm that these bands belong to FB1 and that it is possible to detect it at the regulatory levels, FB1 was dissolved in water and a dilution series of 100 ppm, 10 ppm, 4 ppm and 1 ppm was created. A similar data analysis approach was followed and potential FB1 bands were identified up to 1 ppm. These bands include: 1621 – 1631 nm, 1893 – 1903, 2055 – 2070 nm, 2194 – 2220 nm and 2437 – 2458 nm. Although the band positions differed from those of FB1 in methanol, similar molecular vibrations were responsible for the FB1 bands in both solvents. This study therefore proved that FB1 absorbs detectable amounts of NIR energy at the ppm level. The FB1 bands identified are however dependent on the environment in which it is measured and therefore further research must focus on the determination of the FB1 absorbance bands in artificially and naturally contaminated maize kernels.

F01.08

CLASSIFICATION OF TAPIOCA STARCH HYDROLYSIS PRODUCTS BASED ON THEIR BRIX AND DEXTROSE VALUES USING NIRS IN TRANSFLECTANCE MODE

Chayanid Sringarm[1], Saowaluk Rungchang[1], Sudarat Jiamyangyuen[1], Sonthaya Numthuam[1], Sila Kittiwatchana[2], Wouter Saeys[3]

[1]Faculty of Agriculture Natural Resources and Environment,Naresuan University, Muang District, Phitsanulok Province, 65000 , Thailand, [2]Department of Chemistry, Faculty of Science, Chiang Mai University, Chiang Mai, 50200 , Thailand, [3]KU Leuven Department of Biosystems, MeBioS Division, Kasteelpark Arenberg 30, Leuven, 3001, Belgium

The industrial process of starch hydrolysis products involves liquefaction and saccharification steps, resulting in products with different functionality depending on their concentration level in terms of Brix value and their sugar composition in terms of dextrose equivalent. As the current quality control procedures are time-consuming, destructive, and chemically wasteful. The objective of this study was to use Near-Infrared (NIR) spectroscopy to discriminate the various types of industrial tapioca starch hydrolysis products. Samples of seven tapioca starch hydrolysis products (n = 261) were used in this study and randomly divided into sets of 195 calibration samples and 66 prediction samples. To classify the different starch hydrolysis products, support vector machines (SVM) were trained using either the raw spectra or latent variables (LVs) obtained from PLSR models for predicting the average Brix and DE value per type. The best classification accuracy was obtained by the SVM classifier based on the LVs from the PLSR model for Brix prediction, resulting in a 100% correct classification of the samples in the test set. This may be attributed to the larger impact of the Brix values on the syrup spectra, Brix is related to the sugar content whereas DE is related to the molecular structure of the sugars. In summary, the support vector machine classifier based on LVs from the PLSR model for Brix can be applied to classify tapioca starch products in the industry for enhancing the efficiency of quality control.

Graphic: Classification of tapioca starch hydrolysis products based on their Brix and Dextrose values using NIRS in transflectance mode

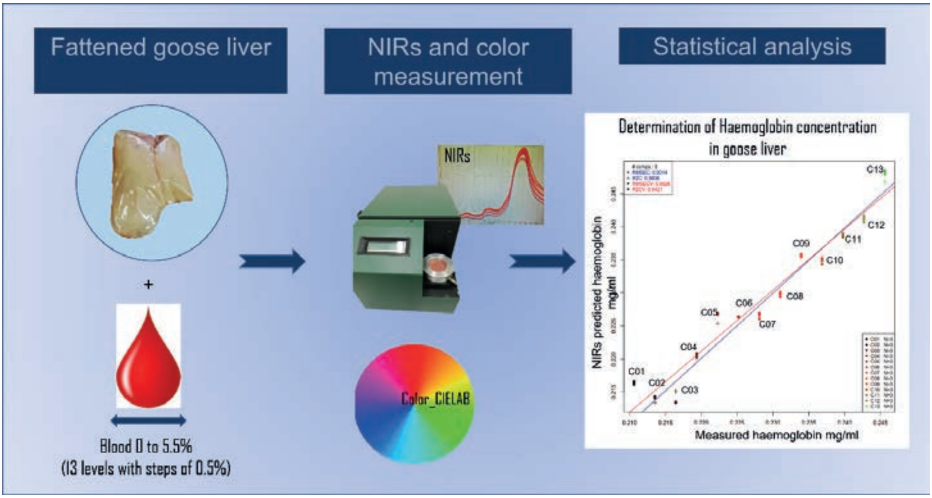
P01.01

DETERMINATION OF THE BLOOD CONTENT IN FATTENED GOOSE LIVER AS AN IMPORTANT QUALITY PARAMETER BY MEANS OF NIR SPECTROSCOPY

Juan Pablo Aguinaga Bósquez[1], Koppány Majzinger[2], Gábor Jónás[2], László Friedrich[2], Zoltan Kovacs[1]

[1]Hungarian University of Agriculture and Life Sciences. Institute of Food Science and Technology. Department of Measurements and Process Control, Budapest, Hungary,
[2]Hungarian University of Agriculture and Life Sciences. Institute of Food Science and Technology. Department of Livestock Product and Food Preservartion Technology, Budapest, Hungary

Fattened goose liver is a premium product which is sold in intact form. Inappropriate breeding and slaughtering conditions can lead to accumulation of blood in the liver. This condition is difficult to determine by simple inspection hence liver must be cut to reveal any anomaly. A non-destructive method that allows to inspect a whole liver can be beneficial for the food industry. Therefore, distinguishing properly a premium liver from a liver with blood accumulation which may be adequately processed prior to market entry. The objective of this study was to determine the applicability of Near Infrared Spectroscopy (NIRS) to evaluate the blood content and hemoglobin concentration in goose liver. Fattened goose liver from four animals was used for the experiments. The liver samples were mixed with NaCl stabilized goose blood at different concentrations between 0% and 5.5% by 0.5% steps resulting in 13 levels of added blood concentration for each of the four livers. The hemoglobin concentration of the samples was determined by Drabkin method. The color of the samples was measured in CIELAB with a Vitalis Colorimeter. NIR spectra of the samples were acquired with a MetriNIR spectrophotometer in diffuse reflectance mode in the range of 900 to 1700 nm. Several spectral pretreatments were tested as part of the optimization of the chemometric models which followed by Principal Component Analysis (PCA) and Partial Least Square Regression (PLSR). Models were built for the four livers together and individually. Additionally, blood concentration level was the variable selected for cross validation in the PLSR models. PCA results presented a clear trend for the hemoglobin concentrations belonging to each liver. PLSR models for single liver analysis revealed high accuracy and low error for the prediction of the hemoglobin concentration. The best model resulted in a determination coefficient for validation (R2CV) of 0.94 with an error RMSECV of 0.003. A general PLSR model, including all the livers in this study, showed a R2CV of 0.63 with an error RMSECV of 0.03. Results from NIRS based PLSR prediction of color coordinate components (L, a, b) from liver samples showed high accuracy of the models specially for a* (red/green coordinate) accounting for R2CV of 0.85 and RMSECV of 0.70. Near Infrared spectroscopy is a promising technique for measuring hemoglobin concentration in goose liver, nevertheless, further complementary research is recommended.


Fattened goose liver
NIRs and color measurement
Statistical analysis
+
Blood 0 to 5.5%
(13 levels with steps of 0.5%)
NIRs
Color_CIELAB
Determination of Haemoglobin concentration in goose liver
NIRs predicted haemoglobin mg/ml
Measured haemoglobin mg/ml
C01
C02
C03
C04
C05
C06
C07
C08
C09
C10
C11
C12
C13
0.210

P01.02

NIR AND MIR MILK SPECTRA TO PREDICT FEED EFFICIENCY OF DAIRY COWS

Silvia Ampuero Kragten[1], Marlyse Raemy[1], Thorsten Haak[2,3], Fredy Schori[2]

[1]Agroscope, Method Development and Analytics, [1725] Posieux, Switzerland, [2]Agroscope, Ruminant Nutrition and Emissions, [1725] Posieux, Switzerland, [3]University Bonn, Institute of Animal Science, [53115] Bonn, Germany

The need to mitigate the environmental footprint of dairy production is strongly fueling the development of effective tools to assess the feed efficiency (FE) of cows. An improved FE (more useful products- milk or meat- with less feed) not only reduces production costs, but also makes the system more sustainable by limiting nitrogen and greenhouse gas emissions. However, its inclusion in breeding programs is strongly hampered by its costly and cumbersome determination, especially in grazing dairy cows. This paper assesses the potential of MIR and NIR milk spectra to predict FE as: feed conversion ratio [FCR = dry matter intake (kg DMI/d)/energy corrected milk yield (kg/d)], nitrogen use efficiency [NUE = milk-N yield (g/d)/feed-N intake (g/d)], residual feed, energy and nitrogen intake [actual - expected values (RFI: kg DMI/d), (REI: MJ NEL/d), (RNI: g N/d) resp.] Expected intake, NEL and N, derived from Swiss feeding recommendations for ruminants. The determination of DMI was performed via the n-alkane marker method and by weighing consumed rations (stall-fed cows only). Milk was collected on days 1, 4 and 7 of a week measurement period from lactating Holstein and Swiss Fleckvieh cows, grazing and stall-fed, primiparous and multiparous, in mid and late lactation. Morning and evening, mass-pooled, milk samples (76x3 for MIR and 132x3 for NIRS) were measured with a MilkoScan RM (Foss) in the mid-infrared region (920-1600 cm^{-1}, 1689-3008 $cm^{-1}$and after freeze-drying with FT-NIR in diffuse reflectance mode, NIRFlex N-500 (Büchi, CH-Flawil), with 3 replicate spectra per sample, 21 scans per replicate, in the range of 4000 cm^{-1} to 10000 cm^{-1}. Predictive models were developed with PLS algorithms and snv (normalization standard normal variate), mf (normalization MSC Full) and db1g2 (1st derivative BCAP Gap 2) with R studio® for MIR or NIRCal software (Büchi, CH-Flawil) for NIRS. Independent calibration (cal) and validation (val) sets were used: ~87 / 13 % for MIR or ~70 / 30 % of available samples resp. for NIRS). The models showed fairly good predictive potential for FE traits confirming their phenotyping potential. R^2(cal/val), NIRS: 0.75/0.76, 0.84/0.84, 0.58/0.58, 0.67/0.65 and 0.88/0.82, and MIR: 0.81/0.69, 0.80/0.77, 0.59/0.31, 0.75/0.27 and 0.90/0.83; residual prediction deviation (SD calibration-set/standard error of prediction), NIRS: 2.2, 2.5, 1.6, 1.9 and 2.8 and MIR: 1.6, 1.6, 1.1, 1.2 and 2.0 for FCR, NUE, RFI, REI and RNI resp. These models should be incremented with samples from diverse origins to improve their robustness and applicability.

P01.03

PROPLANT: A ML-DRIVEN APPROACH FOR PREDICTING PROTEIN IN SPECIES BEYOND THE KNOWN USING GENERALIZED NIR SENSOR-BASED MULTIPLE CEREAL MODEL

Krithika Anbazhagan[1], Jana Kholova[1,2], Sunita Choudhary[1], Jamie Flynn[3], Chris Jones[4], Altyn Taisheva[2]

[1]International Crops Research Institute For The Semi-arid Tropics (icrisat), Hyderabad, India, [2]Czech University of Life Sciences Prague (CZU), Prague, Czech Republic, [3]HoneAg, New South Wales, Australia, [4]International Livestock Research Institute (ILRI), Addis Ababa, Ethiopia

Plant-based proteins are gaining a lot of global attention as a more sustainable and affordable alternative to protein from animal sourced foods. This growing market-pull necessitates timely, inexpensive, rapid, and reliable methods for accelerated assessment of grain quality. This study explores the potential of portable near infrared (NIR) sensor-based instruments as a 'fit-for-purpose tool' for enabling on-field real-time estimation of protein content in cereals. For this, a multiple cereal protein prediction model was built for portable Hone Lab Red instrument using automated HoneCreate software combining spectral data of 373 cereal grain flour samples (finger millet, foxtail millet, maize, pearl millet, sorghum, and wheat) offering a wider range of protein (i.e., 6–22 % [g·100g-1]) compared to that of single species. The calibration model built using a stacked ensemble of machine learning (ML) algorithms (n=373; R2=0.96; RMSE=0.67, RPD=5.0, Fig. 1a) predicted protein in 14 independent wheat samples with a prediction accuracy of R2=0.97; RMSE=0.60; RPD=5.7 (Fig 1b). In order to test the potential of HoneCreate's ML algorithm pipeline, another prediction model was built using the multiple cereal samples excluding the wheat species (n=362 with 6–22 % protein, [g·100g-1]). This model showed $R^2$of 0.96, RMSE of 0.70, and RPD of 4.9 for calibration dataset (Fig. 1c) and $R^2$of 0.88, RMSE of 1.08, and RPD of 2.9 for independent validation set (n=88). Interestingly, this model predicted protein content in unseen wheat species (n=45) with an accuracy of R2=0.86; RMSE=1.3, RPD=2.5 (Fig 1d). This result exemplifies the potential of automated ML algorithms that generalize from the training dataset and make reasonable predictions for unseen samples as well. This portable NIR sensor technology is scalable in making reliable estimates of plant-based protein using advanced data driven methods and delivering need-based insights to multiple stakeholders.

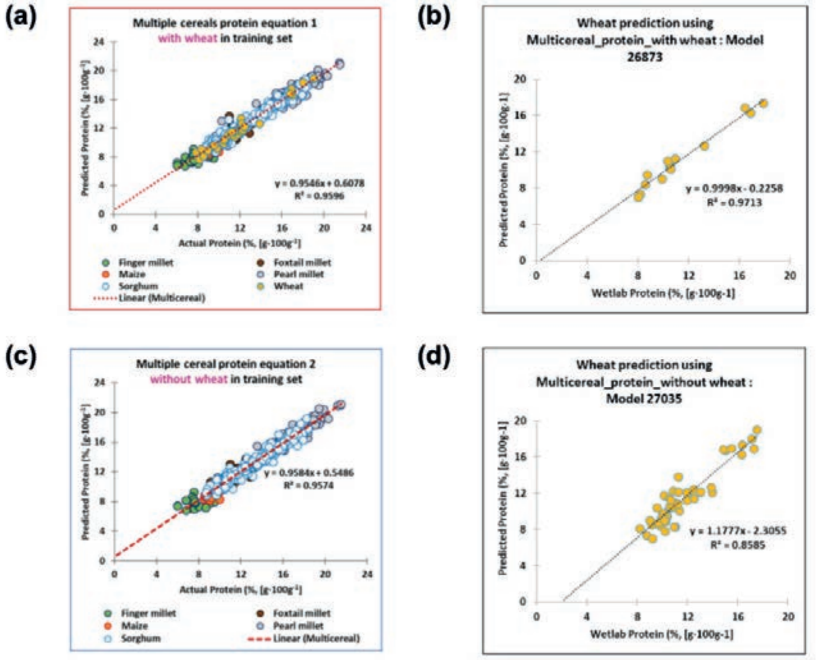


Figure 1. Calibration models built with wheat (a) and without wheat samples (b) for the prediction of protein content in wheat grain flour samples (b, d) using portable Hone Lab Red instrument.

P01.04

DEVELOPMENT OF A PORTABLE, LOW-COST AND NON-INVASIVE SCREENING OF VIRGIN OLIVE OILS QUALITY BY NIR

Alejandra Arroyo-Cerezo[1], Ana M. Jiménez-Carvelo[1], Marina Pellegrino[2,3], Xueping Yang[2], Angela Felicita Savino[3], Paolo Berzaghi[2]

[1]University of Granada, Granada, Spain, [2]University of Padova, Legnaro, Italy, [3]Laboratorio di Perugia –ICQRF-MASAF, Perugia, Italy

Virgin olive oil is one of the food products most affected by food fraud, both economically and consumer health. Current European legislation requires the determination of many physicochemical parameters to verify and control the proper labeling of this product in the highest quality categories (virgin and extra virgin olive oil, VOO and EVOO). Against this background, there is a need to develop rapid analytical methods to detect possible fraud (adulteration, blending with oils of poorer quality, authentication of the variety or geographical origin). In this sense, NIR offers the possibility to develop a unique multi-parametric method to characterize a VOO sample in a few minutes. To do so, it is necessary to perform a calibration suitable for this type of products with the information provided by the spectral data.

The aim of this work was to develop a fast, low-cost and non-destructive analytical method based on NIR and chemometrics for the control of the quality and authenticity of olive oil. For this purpose, two portables miniaturized NIR equipment and a set of 200 oil samples were used. The analyses of physicochemical parameters of interest performed by the official ICQRF laboratory were used as reference data for the development of calibrations for 7 parameters (acidity, peroxides, K232 and fatty acids) using PLS regression method. All calibrations were developed under Python 3.9, using the NumPy package, obtaining good predictive quality metrics such as root mean square errors (RMSE) less than 1% in all cases and coefficients of determination (R^2)around 0.9 in the calibration set and between 0.82-0.92 in the validation set for the fatty acids prediction models (palmitic, oleic and linoleic). In addition, the NIR measurements and calibrations for the oil parameters of interest were also performed with a benchtop NIR spectrometer. As expected, most of the results were better (given the higher spectral resolution). However, in the calibrations for acidity, palmitic and oleic acid, slightly higher RMSE values were obtained than in the calibrations performed with one of the two portable instruments.

Also, through the calibrations developed it was possible to detect and identify vegetable oils (non-olive oils) and differentiate them from genuine oils (VOO and EVOO), as well as to detect refined olive oils as outliers. This detection was carried out using the Mahalanobis distance by means of the GH parameter, as well as attending to the scores obtained from the development of PCA with the predicted chemical data.

These results demonstrated the potential of NIR to be used in the detection and screening of suspicious non-EVOO, and to be implemented as a routine method in control laboratories directly at the production site, thus improving the efficiency of quality control of this product.

P01.05

INFLUENCE OF MILK FAT ON MILK COAGULATION PROCESS: AN AQUAPHOTOMICS APPROACH

Stefania Barzaghi[1], Nicolò Pricca[1], Giovanni Cabassi[1], Laura Marinoni[2]

[1]Consiglio per la ricerca e l'analisi dell'economia agraria, Lodi, Italy, [2]Consiglio per la ricerca e l'analisi dell'economia agraria, Milano, Italy

Several papers show that NIR instruments are valuable auxiliary tools to aid the operator through the cheese-making process management: this possibility relies mainly on the variations of the light scattering induced by the formation of the clot after the rennet addition to the milk.

Light scattering and spectral baseline are also affected by fat globules that can interfere with the scope of the measurements: the aim of this study was to verify the fat influence on the milk coagulation signals using an aquaphotomics approach. This study was conducted through an experimental design, in which different levels of fat and amounts of rennet were considered: low heat skim milk powder was reconstituted at 3,4% of protein content, and cream was added to obtain three levels of fat content 0%, 1,5% and 2,7% respectively. Three different doses of calf rennet were added to 200 mL of milk previously acidified with natural starter whey. The NIR spectra were collected in reflectance mode, every 5s during the coagulation, with a MicroNIR spectrometer, (VIAVI) at constant temperature (36.4°C). The experiment was replicated three times.

Evaluating the linear model calculated from the full factorial experimental design for predicting the clotting time, obtained from the absorbances time-profile at 1300nm, it was verified that the fat content and the interaction "fat x calf rennet content" were not significant.

Aquagrams were developed considering the absorbance values of wavelengths, in the first overtone of the O–H stretching band, which resulted significative in the exploratory PCA of the spectra.

Linear models with interaction were calculated at every two minutes of the coagulation process, using the normalized absorbances of the most significative wavelengths as responses.

Until 12 minutes, the models coefficients showed significance only for the factor "fat" for 1342nm absorbance (asymmetric stretching of the first overtone of water), 1490nm (water molecules with 4 hydrogen bonds) and 1509nm (bending of the first overtone of water) Conversely, the factor "calf rennet" was significant for the absorbance at 1441 nm (water molecules with 1 hydrogen bond) from 4 to 28 minutes.

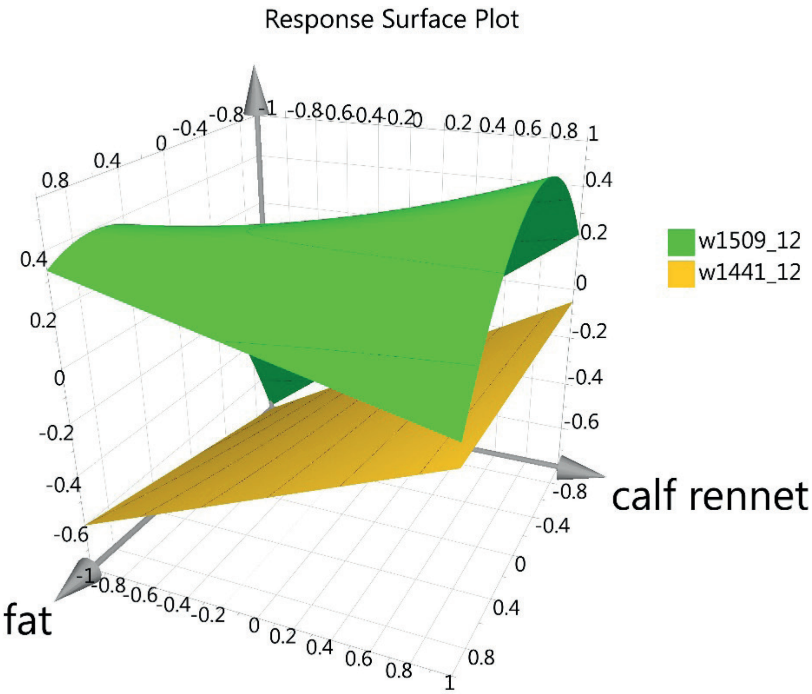


Figure 1: Response Surface Plot of the complete model with interaction of the absorbance at 12 minutes of the 1509nm and 1441nm wavelengths.

P01.06

ENVIRONMENTAL PERFORMANCE COMPARISON AMONG A VIS/NIR BENCHTOP DEVICE, A COST-EFFECTIVE VIS/NIR PORTABLE DEVICE, AND WET-CHEM ANALYSES FOR THE ASSESSMENT OF GRAPE (VITIS VINIFERA L.) QUALITY PARAMETERS

Roberto Beghi[1], Andrea Casson[1], Alessia Pampuri[1], Alessio Tugnolo[1], Sara Vignati[1], Riccardo Guidetti[1], Valentina Giovenzana[1]

[1]Università degli Studi di Milano - DiSAA, MILANO, Italy

Grape quality composition at harvest is one of the most important factors that determine the future quality of the wine. By measuring grape quality parameters, it's possible to determine the optimum harvest timing, ensuring the production of high-quality wines. These parameters are usually obtained through traditional analytical methods, the wet-chem analyses, which require samples to be sent to geographically distant laboratories and waiting a long time before achieving results. All these procedures imply the use of chemicals. As a solution to destructiveness and slowness problems, optical methods can be suitable alternatives to monitor the technological maturation of grapes.

The aim of the work is to evaluate and compare the environmental impact of three different types of analyses carried out to evaluate technological parameters (total soluble solids content, pH, and total acidity) using three different approaches: the wet-chem method, an optical method using a benchtop vis/NIR device, and a second optical method using an innovative and cost-effective vis/NIR prototype. The Life Cycle Assessment (LCA) methodology was used to identify the most sustainable solution and propose actions to reduce the impact along the wine supply chain in a "from-cradle-to-grave" approach. The functional unit was identified by the execution of the analyses necessary to obtain the three technological parameters.

Results demonstrate how, first, the wet-chem analyses should be carried out in triplicate to obtain technological parameters, while for the vis/NIR benchtop and the innovative prototype, the three quality parameters can be obtained with only one single analysis. Moreover, the optical analysis carried out with the cost-effective prototype results in a solution with a lower impact and is 3.2 times more sustainable than the wet-chem analysis. If an innovative optical prototype can be identified as the best solution, its performance may not be so reliable in obtaining precise and trustworthy results. For this reason, a performance factor was included, and results have been normalized; nevertheless, results confirmed what was obtained in the previous scenario.

In conclusion, the study demonstrates how innovations in agriculture and the development of smart optical solutions could provide advantages both in monitoring the quality of agri-food products in an industry 4.0 approach and in proposing green solutions.

P01.07

THE FUTURE OF FISH AGE ESTIMATION: DEEP MACHINE LEARNING COUPLED WITH FOURIER TRANSFORM NEAR INFRARED SPECTROSCOPY OF OTOLITHS

Irina Benson[1], Thomas Helser[1], Beverly Barnett[2]

[1]Resource Ecology and Fisheries Management Division, Alaska Fisheries Science Center, National Marine Fisheries Service, NOAA, Seattle, United States, [2]Fisheries Assessment, Technology, and Engineering Support Division, Biology and Life History Branch, Southeast Fisheries Science Center, Panama City Facility, National Marine Fisheries Service, NOAA, Panama City, United States

Rapid and reliable age estimation is crucial for managing fish stocks, which amplifies the economic value of fisheries. We present a novel method of fish ageing based on Fourier transform near infrared (FT-NIR) spectroscopy of otoliths and convolutional neural networks (CNN). The proposed model is capable of accepting and learning information from multiple data feature types, extracting important spectral features automatically, and yielding comparable precision but significantly greater efficiency than traditional methods. A CNN was trained to extract information out of diffuse reflectance spectral data before integrating with scaled data block that includes corresponding fish length, latitude, gear depth, and gear temperature for nearly 9,000 walleye pollock (Gadus chalcogrammus) specimens. To compare CNN performance with traditional chemometrics approach, two PLS models were developed using either all spectral wavenumbers or selected wavenumbers. For the optimal CNN model, an RMSE of 0.83 for training data and an RMSE of 0.91 for test data indicated that at least 67% of estimated ages were predicted within ± one year of age compared to traditional microscopic ages. The overall performance was good with R^2 of 0.93 for training data and an R^2 of 0.92 for test data (Figure 1A-B). The performance of both PLS models was worse than CNN results. For the two PLS models built on all spectral wavenumbers and on selected wavenumbers, the RMSE was 0.99 and 0.97, respectively, for training data and 1.14 and 1.12, respectively, for test data; the R^2 was 0.89 and 0.90, respectively, for training data and 0.87 for both models for test data. Based on the factor importance plot (Figure 1C), otolith spectra in the 4,000s to 7,000s cm^{-1} wavenumber regions had the highest influence on the model. When the accuracy between FT-NIR spectroscopic ages generated by the CNN model and traditional microscopic age estimates was evaluated, we detected insignificant difference between the two methods. (Figure 1D-F). Our findings represent a paradigm shift in a century-old method of fish age estimation from otoliths and holds the promise of a fish ageing process that rises to the challenge of ever-increasing demands for this crucial data input for fish stock assessments.

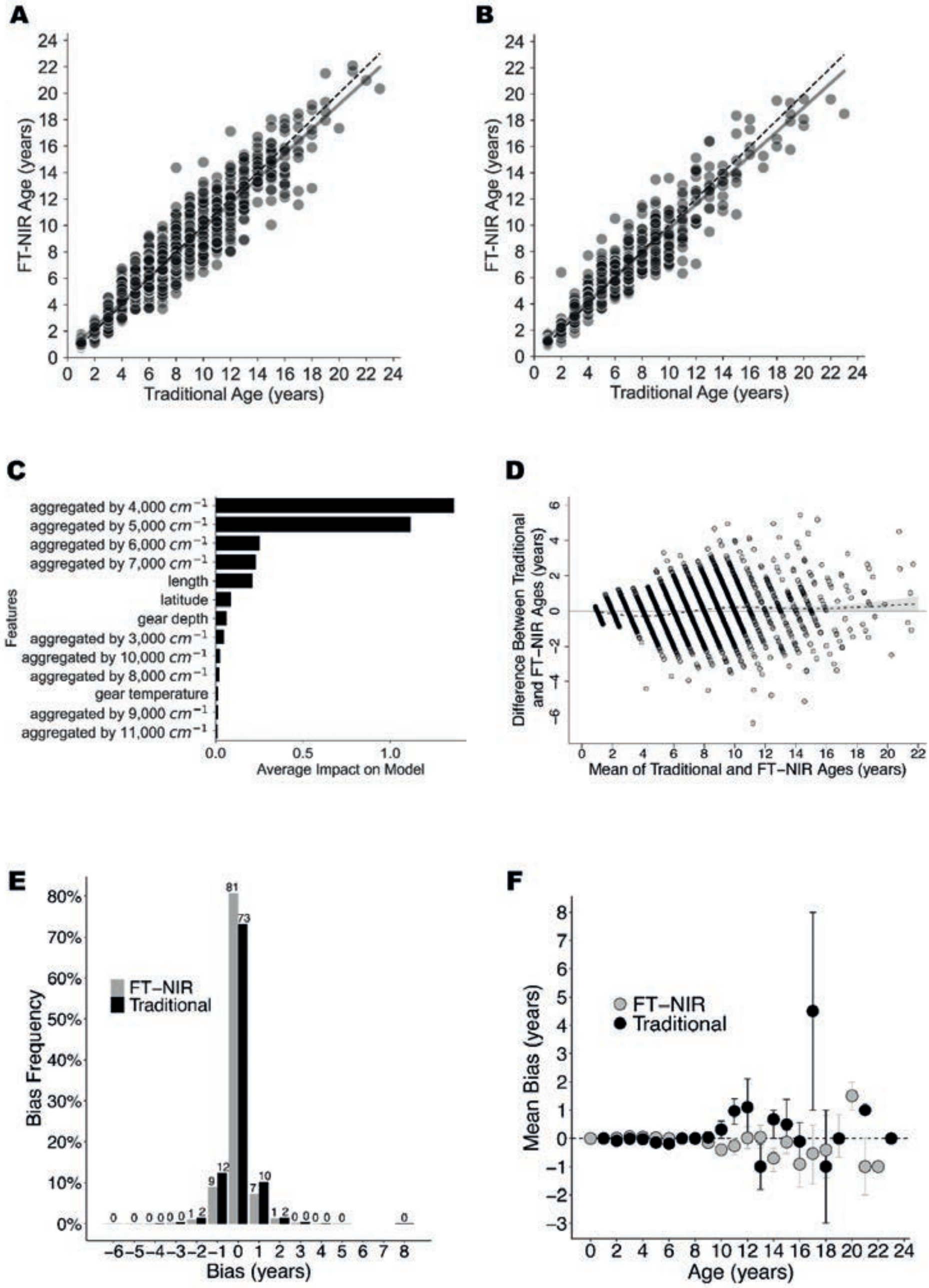


Figure 1. Performance analysis for multi-input convolutional neural network (CNN)

P01.08

STANDARDIZED PROCESSES FOR THE CONTINUOUS IMPROVEMENT OF NEAR-INFRARED SPECTROSCOPY IN-FIELD APPLICATION

Virginie Blanvillain[1], Craig Wyatt[1]

[1]AB Vista, Plantation, United States

Near-InfraRed Spectroscopy (NIRS) is used to assess raw materials and animal feed composition. Benchtop and portable NIRS instruments are now deployed beyond laboratories and into feed mills and farms to analyze forages, feed ingredients, feed rations, milk, manure, etc., and obtain results instantaneously. From sample collection to preparation and scanning, analyses are performed on many different product types and under variable conditions. Thus, calibration robustness has become critical for successful in-field applications, and to ensure results are reliable and repeatable.To keep calibrations up to date, or to implement new calibrations, a standard process may be implemented following 3 sequential steps, which are presented here with practical examples. Firstly, scope definition is a means to define the practical application of a calibration. It allows one to define both the exploration range and the expected level of accuracy of the product types to be tested. For example, the accuracy of Lysine in soybean meal is more important than the accuracy of Proline because Lysine is a key driver of feed cost and animal performance. Secondly, external validation may be performed to evaluate the accuracy and robustness of the predictions, under the pre-defined scope. Samples may be analyzed by both NIRS and reference method, and one should include extreme values to verify robustness. As an example, starch digestibility can be calculated from fecal starch content, with samples being collected to reflect the feasible range of starch levels (i.e., high and low values) to allow subsequent decision-making by the end-user. Thirdly, external validation allows one to build end-user confidence as to the reliability of the calibrations, while acknowledging the limitations of the applicability, and identifying the gaps for further improvement. For instance, phytate-P content in complete feed is used in feed mills to verify that minimum expected levels are met. Under this scope of application, a calibration must detect low content, and the external validation should focus on collecting feed samples with different ingredient profiles, and formulated at low phytate-P levels, to verify the calibration is accurate enough to detect these levels. By accompanying end users through a standard process, continuous improvement plans may be defined by calibration developers, as well as subsequent sampling plans to fill the gaps and broaden NIRS applicability.

P01.09

HYPERSPECTRAL IMAGING AND AQUAPHOTOMICS FOR DISEASE DETECTION IN PLANTS

Sahameh Shafiee[1], Daniel Stebekk Leirdal[1], Svein Kristoffersen[1], **Ingunn Burud**[1]

[1]*Norwegian University of Life Sciences, Aas, Norway*

Hyperspectral imaging in NIR/SWIR wavelengths, in combination with machine learning methods and aquaphotomics have been explored as a tool to classify diseases in wheat leaves and to detect fusarium infected wheat grains.

In the first study 44 leaf samples including healthy, rust-infected, leaf blotch-infected, and samples infected by both diseases were collected and scanned. In the second study 400 healthy and 400 fusarium infected grains were investigated. All the samples were from research fields located in Ås, Norway. Hyperspectral images were collected using a SWIR camera (900-2500nm) in laboratory environment. In both studies, machine learning algorithms yielded high accuracy for classification of disease. For the leaf disease discrimination, applying the whole spectral range provided the best accuracy. For the fusarium detection on wheat grains, it was found that high resolution RGB image with deep learning analysis yielded similar accuracy as the hyperspectral images.
In addition to the standard machine learning approach the first overtone of water (1300-1600 nm) was selected for an aquaphotomics model. Aquaphotomics is an approach using the light-water interaction to extract information about the structures of water and its inherent interactions with other matters of the aqueous system. Absorbance in the NIR wavelength region has proven a good tool for obtaining information about the water molecule structure. The aquagram is a radar chart that displays the normalized absorbance at selected water absorbance bands. For the first overtone of water, the axes of the aquagram are usually based on the 12 water vibrational frequency bands (labelled ci, i=1,12) called water matric coordinates (WAMACS). For the study on wheat leaves, large differences in absorption between healthy and diseased leaves were detected using aquaphotomics. It was concluded that water-related bands possibly can show the differences in early stages of the disease developments. In the aquagram for the wheat grains both the infected and healthy grains show a similar pattern, the healthy grains exhibit considerably higher absorbance values, particularly in the C6 to C12 range (hydrogen-bonded molecules and hydration shell), which are responsible for the bending and symmetric stretching modes observed in this range, compared to the infected grains.

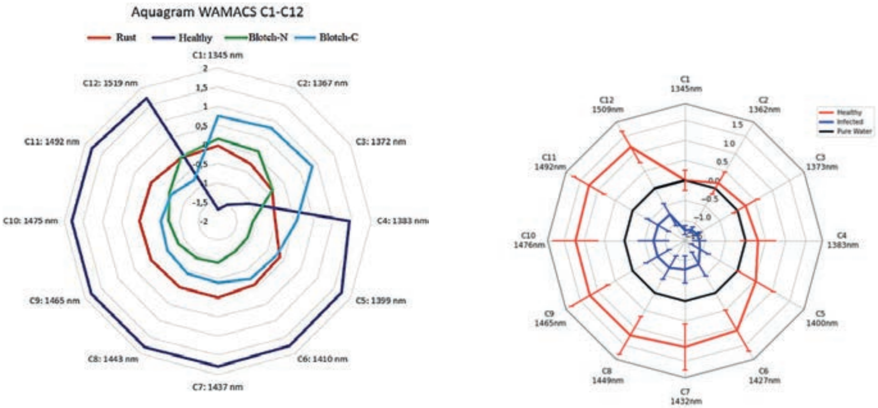


Figure 1. Left: Aquagram for wheat leaves. Right: Aquagram for wheat grains.

P01.10

MONITORING VEGETABLE DEHYDRATION PROCESS BY AQUAPHOTOMICS FROM LAB SCALE TO FARM

Tiziana Maria Piera Cattaneo[1], Laura Marinoni[1]

[1]CREA, Milan, Italy

Solar dehydration processes, implemented with NIR sensors, have been studied. The research plan has been divided into phases to achieve specific objectives. Phase 1: laboratory tests on micro plants; phase 2: scale transposition tests on small-sized plants - pilot scale; phase 3: tests on commercial systems already in use by Italian SMEs. The realisation of the scheduled activities started with the design, programming, and positioning of NIR sensor for data collection and configuration optimization. NIR spectra were collected in reflectance mode (900-1700 nm) using the MicroNIR1700™ or the MicroNIR On-site W™ (VIAVI Solutions Italia S.r.l., Monza, Italy) portable spectrometers with the MicroNIR Pro ES 1700 software. Spectra were acquired automatically throughout the process by placing the NIR probe over a sample slice, positioned on the intermediate shelf. The probe was thermally insulated to avoid temperature variations. Before the analysis, the instrument underwent calibration for the black, in the air, and for the white on the supplied standard tile. The spectra were first transformed by converting reflectance to absorbance; then the second derivative Savitzky–Golay filter (second order polynomial fit and 21 points) and multiplicative scatter correction were applied to remove potential scatter effects, according to Tsenkova et al. [1]. Aquagrams were calculated from the spectral data. The experiments were carried out with a micro-drying system (45x45x45 cm), and a pilot scale plant available at CREA.IT (Milan). Then, the transfer of the selected configuration was realised using a commercial plant already active in a farm of Pavia area. Different food matrices were tested (pineapple, apple, melon, eggplant, onion). NIR data processing by multivariate analysis was made to prove the reliability of the Aquaphotomics approach in detecting the actual end of the drying process. The evaluation of the potential development of dedicated App, easy to consult, to be made available through integration on virtual platform has been finally computed.

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P01.11

HOMEBREWING AND PORTABLE NIR INSTRUMENTS: MONITORING THE FERMENTATION OF READYMADE MALT EXTRACT USING THE SCIO SENSOR

Nicola Cavallini[1], Eugenio Cavallini, Francesco Savorani[1]

[1]Polytechnic Of Turin, Torino, Italy

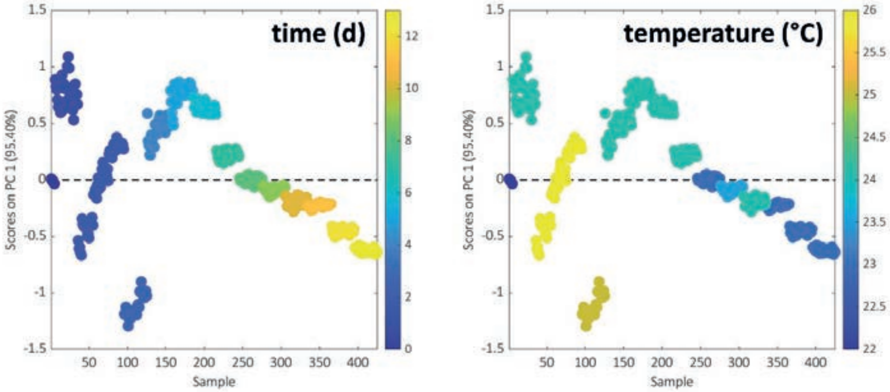
What do miniaturized NIR spectrometers and homebrewing have in common? From a conceptual point of view, the do it yourself motto. Let's break this claim down.

New miniaturized spectroscopic instruments [1,2] are launched on the market to try to satisfy the need of both researchers and consumers for quick ways for analysing stuff, especially in the food field: these small instruments are frequently operated via smartphone apps, which also often include pre-trained models to quantify specific properties (e.g., fat or protein content). The user can therefore, to different extents, use the instruments to perform measurements and rather easily build her/his own calibration models.

Homebrewing has become a very widespread hobby [3], with a wide range of equipment available on the market, allowing people to simply ferment readymade malt extracts, but also to brew more complex recipes. Standard recipes provided by the malt producers are generally "optimized": few actions must be taken, and few parameters must be controlled to obtain a fairly decent beer.

In this study, we investigated the overlap between these two conceputally-connected worlds. The fermentation of a commercial readymade malt extract was followed for 14 days by means of NIR spectroscopy, using the portable handheld SCiO sensor (Consumer Physics) equipped with its accessory for liquid measurements. A small amount of brewing liquid was taken once a day from the fermentation tank, and 30 spectra were acquired with the SCiO instrument.

The exploratory analysis done by Principal Component Analysis (PCA) revealed interesting trends that could be related to the evolution in time of the homebrewing process and to the recorded temperature of the brewing liquid, both of which can be related to the different stages of maturation of the beer during production.



[1] K. Beć, J. Grabska, C.W. Huck, Miniaturized NIR Spectroscopy in Food Analysis and Quality Control: Promises, Challenges, and Perspectives, Foods (2022), 11(10), 1465; https://doi.org/10.3390/foods11101465

[2] G. Gorla, P. Taborelli, H.J. Ahmed, C. Alamprese, S. Grassi, R. Boqué, J. Riu, B. Giussani, Miniaturized NIR Spectrometers in a Nutshell: Shining Light over Sources of Variance, Chemosensors (2023), 11(3), 182; https://doi.org/10.3390/chemosensors11030182

[3] M. Wolf, W. Ritz, S. McQuitty, Prosumers who home brew: a study of motivations and outcomes, Journal of Marketing Theory and Practice (2020), 28(4), 541-552; https://doi.org/10.1080/10696679.2020.1801321

P01.12

A PRELIMINARY INVESTIGATION OF GEOGRAPHICAL ORIGIN CLASSIFICATION OF DURIAN (CV. MON THONG) USING NEAR-INFRARED DIFFUSE REFLECTANCE SPECTROSCOPY COUPLED WITH K-NEAREST NEIGHBOR AND RANDOM FORESTS METHODS

Kingdow Chanachot[1], Wanphut Saechua1, Panmanas Sirisomboon1

[1]King mongkut's institute of technology ladkrabang, Bangkok, Thailand

Geographical origin is one indicator for flavours, texture, and quality of durian, which are responses to the purchase decision making of the customer. The main propose of this study is to develop a reliable, efficient, and accurate method to discriminate geographical origin of durian (CV. Monthong) in Thailand, grown in 5 provinces including: Eastern Thailand including Chanthaburi (C) of 15 fruits, Rayong (Ra) of 15 fruits and Trat (T) of 15 fruits, upper Southern Thailand which was Prachuap khiri khan (P) of 15 fruits where the durian obtained the geographical identification (GI), and North-Eastern Thailand which was Sri sa ket (S) where the durian obtained the geographical identification (GI) of 15 fruits. NIR spectral data were acquired in the wavenumber range of 12,500-4,000 cm^{-1} in diffuse reflectance mode by the Fourier transform (FT) near infrared (NIR) or FT-NIR spectrometer (Multi-Purpose Analyzer, MPA, Bruker optics, Germany). Pre-processing of spectra consisted of Detrending (Detrending), Mean Normalization (Mean Nor) Multiple scatter correction (MSC), 1st Derivertive (FD), 2nd Derivertive (SD) and Standard Normal Variate (SNV) was applied. NIR diffuse reflection spectroscopy in full wavenumber coupled with K-Nearest Neighbours (K-NN) and Random Forests (RF) algorithms was applied where the grid search method was used to obtain optimal values of model hyperparameters. All pre-processing of spectral data and model development and analysis were performed by Python 3.8 using machine leaning package and scikit-learn and with sample of training set. NIR spectra test set was used to evaluate the %Accuracy, %Precision, %Sensitivity and F1 score, which are the common criteria used to evaluate the performance of classification models. Equations 1-5 present the evaluation parameter formulas:
$\%Accuracy = [(TP + TN) / (TP + TN + FP + FN)] \times 100$ (Equation 1)
$\%Sensitivity = [TP/(TP+FN)] \times 100$ (Equation 2)
$\%Specificity = [TN/(TN+FP)] \times 100$ (Equation 3)
$\%Precision = [TP/(TP+FP)] \times 100$ (Equation 4)
$F1\ score = (2 \times Precision \times Sensitivity) / (Precision + Sensitivity)$ (Equation 5)
TP is the number of true-positive samples, which are samples that are correctly classified as model samples; TN is the number of true-negative samples, which are samples that are correctly classified as other-group samples; FP is the number of false-positive samples, which are samples from another group that have been classified as model samples; and FN is the number of false-negative samples, which are model samples that have been classified as other-group samples. To find an overall effective classification model, the highest %Accuracy model is chosen. The fairly classification model developed by MSC pretreated spectra using K-NN algorithm achieved 75% and 52% accuracy

in training and testing sets, respectively. RF algorithm achieved 100% and 61% accuracy in training and testing set, respectively. These indicated the overfitting models. However, %Specificity indicating the correct classification of other group to be other group was the highest value among evaluation indicators while the models by MSC with K-NN by grid search provided 97, 94, 94 and 91% for C, R, P and S, respectively but T of 87% for training set and 92, 80, 83 and 89 for test set, respectively but for T of 69%. The model could differentiate other group not to be the member of model itself accurately especially for C but fairly accurate for T indicated T sample have more similar characteristic to other province durian. There was less accuracy in identify sample of model itself to be member of its province (%Sensitivity). The differences of durians from different geographic origins might be caused by due to seasons, climates, soil minerals, drip irrigation, fertilizer, and planting method, and other factors. All these led to changes in the types and contents of the constituents in durian fruit of different geographic origins. Additional, in future experiments, the more robust model should be developed by using more samples, motivates the use of the variable selection method and test accuracy of model by using unknown durian samples.

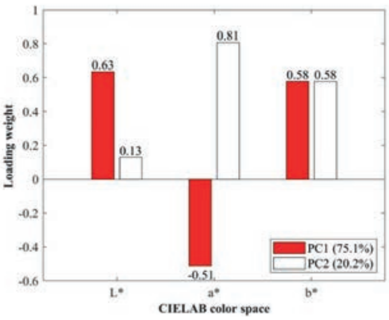
P01.13

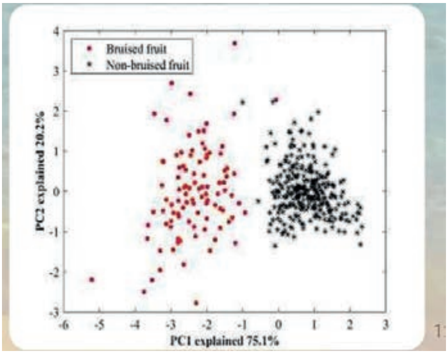
CLASSIFICATION OF BRUISED AND NON-BRUISED LONGAN FRUITS BY COLOR MEASUREMENT COUPLED WITH MACHINE LEARNING

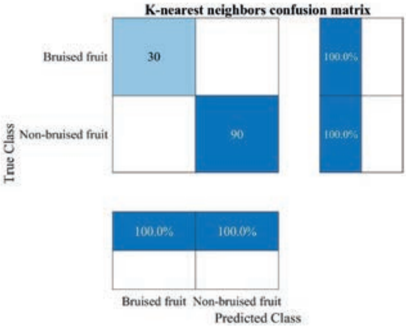
Kingdow Chanachot[1], Kittisak Phetpan[2], Panmanas Sirisomboon[1]

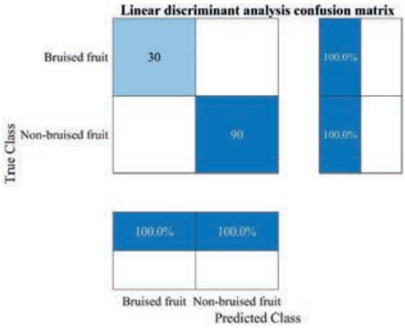
[1]King Mongkut's Institute of Technology Ladkrabang, Bangkok, Thailand, [2]King Mongkut's Institute of Technology Ladkrabang Province of Chumphon Campus, Chumphon, Thailand

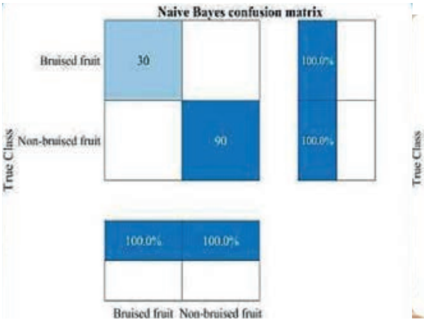
Longan fruit (Dimocarpus longan Lour.) bruises happen during transportation and storage by the impact and compression forces which reduced the quality of the fruits. This can cause short shelf life time and can make the customers to refuse buying. The cheapest non-destructive technology for classifying the bruised and non-bruised fruits and vegetable is color detector. Before using the machine learning to classify, the group of 300 non-bruised and 100 bruised fruits were manually selected and the L*a*b* color scale of each fruit was measured by spectrophotometer (MiniScan XE Plus 45/0 LAV, Hunter Associates Laboratory Inc., Reston, USA) using reflectance mode. A D65 illuminant with 10° observer angle was used. The L*a*b* of non-bruised fruit (LD) and bruised of longan fruits (LG) were subjected to K-nearest neighbors (KNN), Decision tree (Tree), Naïve Bayes (NB), Linear discriminant analysis (LDA) and Support vector machine (SVM). By principal component analysis (PCA), the data sets could clearly be separated and PC1 and PC2 covered the variance of the data of 75.1% and 20.2%, respectively. The KNN, NB and SVM provided 100% classification accuracy whereas Tree missed 1 fruit sample in each class.

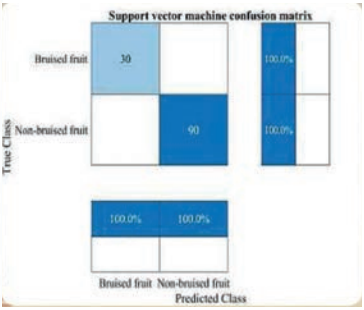


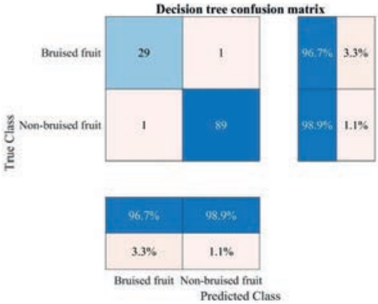



K-nearest neighbors confusion matrix
Bruised fruit
Non-bruised fruit
True Class
30
90
100.0%
100.0%
100.0%
100.0%
Bruised fruit
Non-bruised fruit
Predicted Class


Linear discriminant analysis confusion matrix
Bruised fruit
Non-bruised fruit
True Class
30
90
100.0%
100.0%
100.0%
100.0%
Bruised fruit
Non-bruised fruit
Predicted Class


Naive Bayes confusion matrix
Bruised fruit
Non-bruised fruit
True Class
30
90
100.0%
100.0%
100.0%
100.0%
Bruised fruit
Non-bruised fruit


Support vector machine confusion matrix
Bruised fruit
Non-bruised fruit
True Class
30
90
100.0%
100.0%
100.0%
100.0%
Bruised fruit
Non-bruised fruit
Predicted Class


Decision tree confusion matrix
Bruised fruit
Non-bruised fruit
True Class
29
1
1
89
96.7%
3.3%
98.9%
1.1%
96.7%
98.9%
3.3%
1.1%
Bruised fruit
Non-bruised fruit
Predicted Class

P01.14

USE OF PORTABLE NIR SPECTROMETERS IN TANDEM WITH PCA, DD-SIMCA AND PLS-DA TO CLASSIFY AND DISTINGUISH OLEOGELS

Marina De Gea Neves[1], Ingrid Moraes[2], Douglas Barbin[2], Heinz W. Siesler[1]

[1]University Duisburg-Essen, Essen, Germany, [2]University of Campinas, Campinas, Brazil

Oleogel represents a promising alternative as a conventional fat replacement in various health foods. The selection of oil type is a crucial factor in determining the technological properties and applications of oleogels due to their distinct fatty acid composition, atomic weight, and thermal characteristics, which impact the gelation capacity. Thus, monitoring these parameters is important for their production, however, many traditional methods of analysis for oleogels are laborious and involve the use of some chemical reagents that are toxic to humans and the environment. During the last two decades the miniaturization of NIR spectrometers has made large progress and the instruments have become increasingly portable and even handheld. NIR spectrometers with weights down to <100g are available and are targeted for integration into mobile phones. Due to a significant reduction in price compared to benchtop instruments, handheld NIR instruments have also reached tremendous potential and popularity for quality control in industry. Thus, the present study aims to classify oleogels rapidly and reliably, without the use of chemicals, by employing two different handheld NIR spectrometers. Three types of oleogels were prepared, containing 95% oil (sunflower, soy, olive) and 5% beeswax as a structuring agent, melted at 90 °C. The portable NIR spectrometers, NIR-S-G-1 (Inno-Spectra Corp., Taiwan) in the wavelength range 900 – 1700 nm, and Development Kit FT-NIR Scanner (Si-Ware Systems, Cairo, Egypt), in the wavelength range 1350 – 2540 nm, were used in this work. A total of 246 spectra were obtained for each NIR spectrometer. After spectra pre-treatment, 1st derivative (window 7, polynomial 2) + SNV (Standard Normal Variate) + mean center, a Principal Component Analysis (PCA) was performed, and it was observed that the samples clustered according to the type of oil in the PC1, PC2 and PC3 scores. Classification models were then constructed using the complete dataset, with samples split into calibration set (70%, 172 spectra) and validation set (30%, 74 spectra). The best DD-SIMCA (Data Driven - Soft Independent Modeling of Class Analogy) and PLS-DA (Partial Least Squares-Discriminant Analysis) models demonstrated 100% accuracy. Figure 1 shows the portable NIR scanners (A) and (B), the spectra dataset acquired by them (C) and (D), the DD-SIMCA model developed for oleogel made from olive oil (E), and the PCA model (F) using the whole dataset with 246 spectra for the Innospectra scanner. These results indicate that a portable NIR spectrometers in conjunction with chemometrics can be a valuable tool for classifying different types of oleogels. Photos of investigated oleogels are shown in (G).

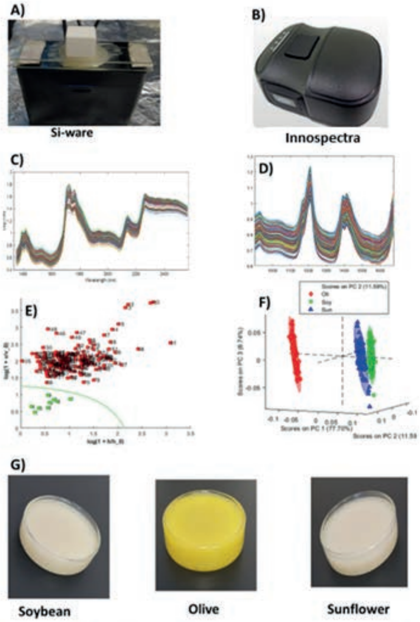


Fig 1. Portable NIR spectrometers used to classify oleogels: (A) Si-ware, (B) Innospectra. 246 NIR raw spectra of oleogels acquired by (C) Si-ware, (D) Innospectra. (E) DD-SIMCA model developed to classify oleogel made from olive oil using Innospectra scanner. (F) PCA model developed using the whole dataset (red: olive oleogel; blue: sunflower oleogel; green: soybean oleogel) using Innospectra scanner. (G) Photos of investigated oleogels.

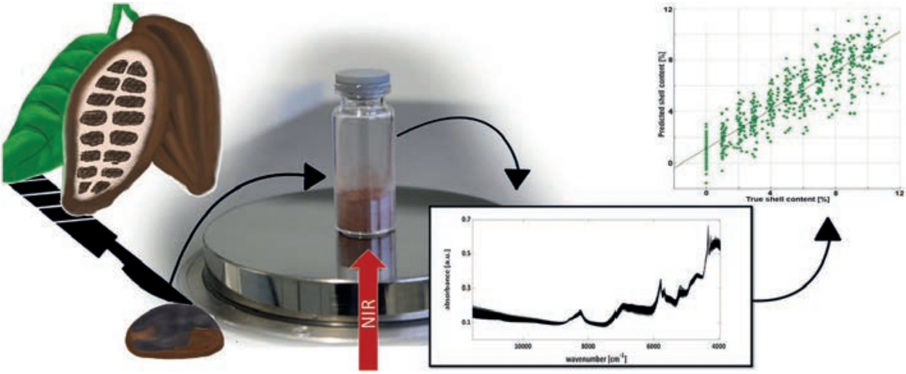
P01.15

RAPID DETERMINATION OF THE SHELL CONTENT IN COCOA PRODUCTS USING FT-NIR SPECTROSCOPY AND CHEMOMETRICS

Alissa Drees[1], Johannes Brockelt[1], Lina Cvancar[1], Markus Fischer[1]

[1]University of Hamburg, Hamburg, Germany

The determination of the cocoa shell content is of interest because a high shell content causes a reduction in the quality of cocoa products. Consequently, the aim of the present study was the development of a routinely applicable method for the quantitation of shell material in cocoa nibs. For this, 51 fermented cocoa samples of different varieties from 14 cocoa growing countries covering the crop years 2012–2017 were acquired. Admixtures of cocoa nibs with shell material were prepared in a range of 0–20% cocoa shell and subsequently analysed by Fourier transform near-infrared spectroscopy (FT-NIRS). Support vector machine regression models were created, which enabled the prediction of the cocoa shell content in a mixing ratio range of 0–20% with an RMSE of 2.05% and a R^2 of 0.88 and in a range of 0–10% with an RMSE of 1.70% and a R^2 of 0.72. This predictive capability suggests that the presented method is suitable for rapid determination of cocoa shell content in cocoa nibs. In addition, it was demonstrated that the method is applicable to other relevant cocoa matrices, as the prediction of the shell content of several industrial cocoa masses by the FT-NIRS-based model showed good consistency with the prediction by liquid chromatography–mass spectrometry. This emphasizes that FT-NIRS combined with chemometrics has great potential for the determination of cocoa shell content in cocoa nibs and cocoa masses in routine analysis, such as incoming inspection.



P01.16

RIDING A ROAD ROLLER IN A FORMULA 1 RUN; NIR VS HPLC IN GLP REGUATED ENVIRONMENT

János Elek[1], Máté Csontos[1]

[1]Science Port Ltd., Debrecen, Hungary

Our company is a service provider laboratory, and a big part of our activities is supporting analysis (concentration, homogeneity and stability) in toxicology studies, mainly rodent feeding. The analysis of UVCB materials (chemical of unknown or variable composition) and novel food products – such as protein, algae and other biomass products – is demanding and most of the times impossible by traditional chromatographic techniques. The application of NIR spectroscopy would be an obvious choice, but the golden standard at the partner laboratories, registration offices is the performance of the HPLC. Most of the regulation systems – such as GLP - can hardly handle the issues around variable selection, and slight modifications due to the changes between the test diet batches. This presentation is the problem definition and kind of foreword to the presentation of Mr. Máté Csontos and focuses on our efforts to successfully fulfill the regulatory requirements and successfully compete on a filed overruled by HPLC analytics.

P01.17

REAL-TIME SPECTRAL MEASUREMENTS OF QUALITY IN A BREWERY SETTING

Glen Fox[1], William Palmer[2]

[1]University of California, Davis, United States, [2]Hone, 22 Lambton Rd/Broadmeadow/NSW 2992, Australia

Capturing more accurate data on the brewing process and beer quality is important for all breweries. However, not all breweries can have a quality assurance laboratory or trained staff to be conducting regular and expensive testing. However, one alternative is the use of infrared technology which has been shown to be capable to quantify beer traits such as color, gravity, bitterness and alcohol. Most infrared instruments are bulky and require a temperature-controlled space for more accurate operation. More recently, portable infrared devices offer the flexibility to have the instrument at a workstation in the brew-house. The portable device used in this study provides a spectral range from UV to NIR. It only requires 4 ml of liquid sample. The spectral data capture is transferred to a cloud-based software where predictions are carried out and then predicted results returned to the PC. We have built calibrations for gravity, alcohol and bitterness in a portable near infrared device. The calibrations have been built on samples from fresh wort pre-fermentation, during fermentation, pre- and post-dry hopping and terminal gravity. The sample range includes non-alcoholic beers to double IPAs as well as alcoholic hopped water. The time to get a results takes less than 4 minutes when working with a degassed sample. The opportunity to be able to get more data in the brewery will allow brewers to have more control of the process.

P01.18

NEAR INFRARED SPECTROSCOPY AS A RELIABLE TOOL FOR THE CONTROL ANALYSIS OF CANNABIS SATIVA L.

María Teresa García-Valverde[1], María del Carmen Díaz-Liñán[1],
Carlos Ferreiro-Vera[1]

[1]Phytoplant Research SLU, Córdoba, Spain

Cannabis sativa L. is a multi-use and chemically complex plant that has been the subject of controversy for many years due to the negative connotation associated to its recreational use. However, the interest in the plant has been renewed due their demonstrated therapeutic effects, such as pain alleviation or the treatment of epilepsy, among others. Cannabis contains a unique group of terpenophenolic secondary metabolites, i.e. cannabinoids, which are the main biologically active compounds of the plant. For this reason, the quality control of the plant and their active pharmaceutical ingredients (APIs) is mandatory to guarantee the safety and effectiveness of the pharmaceutical product. In this sense, different analytical approaches have been proposed for the analysis of Cannabis, including liquid (HPLC) and gas chromatography (GC), among others. Despite the great advantages offered by these techniques, one of the main drawbacks is the high consumption of organic solvents, in addition to time of analysis and the need for specialized personnel for their use. The introduction of NIRS technology in the quality control of Cannabis has been a breakthrough as these disadvantages are totally avoided. Due to its great versatility, this technique allows the determination of several parameters related to the production of the plant, such as nitrogen content, or directly to the APIs, like cannabinoids, terpenes and/or moisture percentage. Moreover, qualitative analysis may be also performed, such as cultivation or variety type. On the other hand, the extensive array of equipment available on the market facilitates the monitorization of the plant during its growth stage, without the need of harvesting.

The present work describes the use of different NIRS equipment, including Fourier transform (FT) and dispersive NIR spectrometer for the analysis of diverse parameters in Cannabis samples. In addition, a portable unit has been used for in-situ monitorization of the plant. All the samples were dried and milled to facilitate the comparison among modalities along with the wet analysis by the different analytical techniques (GC-FID and HPLC-DAD for cannabinoids, Kjeldahl method for nitrogen content and Karl Fischer titration for moisture determination). Multivariate analysis was used to develop prediction equations of cannabinoids, nitrogen and water content with SEP values close to the errors obtained in the laboratory.

P01.19

OPTIMIZATION AND EVALUATION OF DIFFERENT PORTABLE NIRS INSTRUMENTS FOR ON-SITE INSPECTIONS OF VIRGIN OLIVE OILS (VOOS)

Mar Garrido-Cuevas[1], Ana Garrido-Varo[1], Jose A. Entrenas[1], María Teresa Sánchez[2], Dolores Pérez-Marín[1]

[1]Department of Animal Production, Faculty of Agriculture & Forestry Engineering, University of Cordoba, Cordoba, Spain, [2]Department of Bromatology and Food Technology, Faculty of Agriculture & Forestry Engineering, University of Cordoba, Cordoba, Spain

Despite the efforts invested in research on physico-chemical and sensory methods (Panel Test) to determine the quality, purity and authenticity of virgin olive oils (VOOs) and the existence of international standards used in official inspections, the fraudulent labelling of VOOs is still a major international problem. The implementation of NIRS technology in the olive oil sector makes it necessary to search for instruments accessible to the main actors of the olive oil chain (producers, inspection bodies, and consumers). In the last ten years, the progress in reducing the size of NIRS instruments has been very significant. The available portable instruments were in principle not designed for the analysis of oils. Therefore, the decision on which instrument to buy is not easy and requires a great deal of experience and scientific knowledge of NIRS and chemometrics. The present work will provide scientific evidence of the potential of portable NIRS instruments (MicroNIR Pro 1700 (908-1676 nm), AlbaNir (950-1650 nm) and AlbaNir+ (1350-2150 nm)), for the on-site analysis of VOOs. Spectral repeatability was evaluated using the Root Mean Square (RMS) statistic, as well as the predictive capacity of these sensors for determining Free Acidity (FA). A total of 300 olive oils were analysed using different modes of analysis. In the MicroNIR Pro, transmission and transflectance were studied while with the AlbaNir and AlbaNir+ oils were analysed in transflectance. MPLS regression was used to predict FA. Results show that the transmission mode offers a higher spectral repeatability. The lowest RMS value was obtained with the MicroNIR Pro working in transmission (2,424 µlog (1/R)), while those obtained by the AlbaNir and Albanir+ sensors were 6,313 and 5,493 µlog (1/R), respectively. The MicroNIR Pro working in transflectance had a higher RMS value (12,189 µlog (1/R)), although transflectance mode has some advantages, such as ease of sample preparation or cleaning of the cup. The MPLS models developed to predict FA using the MicroNIR Pro and the AlbaNir instruments, both working in transmission mode, showed R^2 values higher than 0.9 and SECV values below 0.1 (% oleic acid), which are in the same range that those obtained with bench-top at-line instruments ($R^2 = 0.962$; SECV = 0.055%). Therefore, the models developed to predict FA highlight the potential of the new portable NIR sensors for the quality control during production and storage in the olive oil industry.

P01.20

NIR MONITORING OF HEMP OIL SHELF LIFE STORED IN DIFFERENT MATERIALS AND AT TWO TEMPERATURES

Andrea Gasparini[1], Francesca Bonazza[1], Lucia Monti[1], Valeria Pellizzola[1], Milena Povolo[1], Stefania Barzaghi[1], Giovanni Cabassi[1]

[1]Crea-za Lodi, Lodi, Italy

Recently there has been a renewed interest in hempseed oil (Cannabis sativa L.) in the food industry due to the beneficial properties provided by hemp seeds and oil as well. Hemp oil is extremely rich in linoleic (18:2 w6) and a-linolenic (18:3 w3) essential fatty acids. Moreover, it is rich in antioxidants, such as tocopherols and phenolic compounds. The presence of high concentrations of unsaturated fatty acids in hemp oil determines its high sensitivity to oxidative and photo-oxidative degradation.These processes generate peroxides, dienes, and trienes that are at the basis of the phenomenon of rancidity.

A batch of hemp oil was obtained by cold-pressing Futura 75 seeds. After a production, the oil was left to settle in its can for 14 days to allow sediment to separate at the bottom. The oil was then transferred and subdivided into 20 ml vials made of polypropylene, clear glass, amber glass, and amber glass coated with aluminium foil. The samples were stored for 169 days under ambient (25°C) and refrigerated (10°C) temperatures. Samples were analysed every 14 days for the first two months and then every 30 days for the remaining storage period.

To monitor the stability of hemp seed oil, peroxide value determination and conjugated diene and triene production were evaluated. The peroxide value (expressed as meq O2/kg) was determined using the iodometric technique. Spectrophotometer analysis was used to measure the conjugated dienes and trienes of fatty acids.
NIR spectra were measured in transmission. The spectral dataset was analyzed using ASCA in order to identify the sources of spectral variation in the dataset and separate effective factors from noise factors. The factors tested were Temperature, Time and Storage Material. The Effect Table of the ASCA model is shown in Table 1.

Model Effect	Effect (%)	Num. PCs	P value
Temperature	6.90%	1	<0.05
Time	15.40%	6	<0.05
Storage Material	9.10%	3	<0.05
Temperature x Time	10.20%	6	<0.05
Temperature x Storage Material	5.90%	3	<0.05
Storage Material x Time	14.10%	18	<0.05
Residuals	38.30%	2	

Table 1 Effect Table of the ASCA model

From the table, it can be inferred that the main effect is due to the storage time, as expected, while among the interactions, the one between storage time and material has a greater effect than the others. All effects, both factors and interactions, were tested using 3000 random permutations of samples and showed a significance level greater than 5%. ASCA analysis showed that samples in amber glass coated with aluminum foil exhibited the greatest stability over time at both storage temperatures.

The spectral dataset was also used to test the predictive ability of NIR analysis of the Hemp oil with respect to parameters that measure its degradation, highlighting the ability of the NIR technique to discriminate the early rancidity of oil with satisfactory performances for the PLS models developed for the number of peroxides, K232 and K268. The prediction errors were 2.82, 0.15, and 0.06, respectively, corresponding to an RPD (Ratio of Performance to Deviation) of 2.3 for the first two variables and 1.8 for K268.

P01.21

FARM TO FORK' QUALITY CONTROL DURING THE PRODUCTION OF A VALUE-ADDED DAIRY PRODUCT

Zoltan Gillay[1,2], Tamas Tóth[1,3], Viktória Éles[1], Róbert Roszkos[1], Hajnalka Hingyi[1], Éva Csavajda[1], Zsolt Svraka[4], Norbert Zelei[4], Miklós Tóth[5], George Bazar[1]

[1]Adexgo Kft., Balatonfüred, Hungary, [2]Hungarian University of Agriculture and Life Sciences, Institute of Food Science and Technology, Budapest, Hungary, Budapest, Hungary, [3]Széchenyi István University, Agricultural and Food Research Center, Győr, Hungary, Győr, Hungary, [4]Pálhalmai Agrospeciál Kft., Dunaújváros, Hungary, [5]University of Pécs, Faculty of Health Science, Pécs, Hungary

With the growing awareness of consumers, the demand for health-promoting foods rich in bioactive ingredients is increasing. It has long been reported that the nutritional quality of dairy products can be improved through the feeding of cows. The main challenges associated with the traditional methods used in analyzing the nutritional properties of feed or food are cost and time of analysis. To address this issue, cost-effective rapid correlative methods have been developed to complement or replace the conventional methods. In this study, a handheld NIR spectrometer was used to acquire spectra of total mixed rations (TMRs) containing control feed supplementations (rich in saturated fatty acids) and experimental feed supplementations (rich in polyunsaturated fatty acids). Dairy cows (n=70 per group) fed the investigated TMRs over 6-week trials, while the collected milk of the groups was used for cheese production. The freshly cut surface of the cheese samples were scanned with a handheld NIR spectrometer. Additionally, the cheese samples were subject to chemical analysis to determine dry matter, fat, protein, ash, and sugar content, and fatty acid profile. NIRS with multivariate classification methods was found to be efficient in differentiating the TMRs of the control and experimental groups, thus, a monitoring protocol was set up for future quality control of feeds at farm level. NIRS was also tested to assess the quality influence of improved feeding on cheese. Satisfactory multivariate calibrations were fitted on the general chemical composition and ratio of major fatty acids of the cheese samples. The study demonstrates the ability of handheld NIR technology in testing feeds of dairy cows, and in the evaluation of the produced cheese to affirm the significantly increased level of n-3 polyunsaturated fatty acids caused by the fed diet.

P01.22

DETERMINING MOISTURE CONTENT OF BASIL USING HANDHELD NIR SPECTROSCOPY

Reyhaneh Gorji[1], Jan Skvaril[1]

[1]Future Energy Center, School of Business, Society and Engineering, Mälardalen University, Västerås, Sweden

Accurate and rapid determination of moisture content is essential in crop production and decision-making for irrigation. Near-infrared (NIR) spectroscopy has been shown to be a promising method for determining moisture content in various agricultural products, including herbs and vegetables. The objective of this study was to develop a calibration model for predicting the moisture content of basil (Ocimum basilicum L.).
The spectral data were obtained from a total of 120 basil leaf samples over a period of 6 days. This included freshly harvested and detached leaves, as well as those left in ambient air for 1-6 days. Five spectra were taken from each leaf using a handheld NIR spectrophotometer, which covers the first and second overtone of the NIR spectral region: 950 – 1650 nm. After the spectral acquisition, the leaves were weighed for fresh weight and then put in an oven for 72 hours at 80°C to determine the dry weight and calculate the reference moisture content. The calibration model was developed using multivariate analysis in Aspen Unscrambler, including pre-processing and regression modeling. The data obtained from 70% of the samples were used for model training, 30% for validation. Preliminary findings indicate that the calibration model for predicting moisture content in basil leaves using NIR spectroscopy has a promising accuracy. The practical implications of the findings could benefit the controlled environment farming industry, particularly in terms of irrigation management and crop production. The data analysis is currently ongoing, and the model validation results will be presented during the conference.

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P01.23

SAFFRON VALORISATION BY NIR SPECTROSCOPY

Irene Locatelli[1], Davide Pedrali[1], **Silvia Grassi**[1]

[1]*Università degli Studi di Milano, Milano, Italy*

Saffron commercial quality is determined by test methods reported by the ISO 3632 establishing the specifications for dried saffron obtained from the pistils of Crocus sativus L. flowers. The ISO testing methods are not particularly complex but require the use of precious grams of products. Beyond trading standards, professionals in saffron value chain, especially top quality and PDO producers, are looking for a non-destructive method to assess the quality characteristics saving the precious product, together with the definition of stricter and rewarding quality assignment.

In the contest of continuous updating of analytical tools, the potential of FT-NIR spectroscopy for saffron quality assessment is explore in this work.

To the aim 115 samples of Italian saffron (1.5 - 2 g of dry stigmas) were collected along two harvesting years (2021 and 2022) and analysed for content of water and the three main metabolites responsible for saffron colour, flavour, and aroma (crocin, picrocrocin and safranal) according to ISO 3632. The same samples were analysed by FT-NIR spectrometer (MPA, Bruker) by mean of an integration sphere.

Both chemical and spectroscopic data were explored by Principal Component Analysis to uncover patterns linked to (1) geographical area of production (North, Centre of South Italy), and altitude (valley, hill, mountain), (2) harvesting year, (3) quality and sub-quality categories. No relevant distribution was highlighted according to origin and harvesting year, however some insights related to crocin and safranal content were uncovered. Thus, regression models were developed by PLS. Promising results were obtained in calibration and cross-validation (Table 1).

Property	N_{tot}	***Range***	**Treatment**	**LVs**	R^2_{cal}	**RMSEC**	r^2_{CV}	**RMSECV**
Colouring strength $A_1^{1\%}$ cm 440 nm	115	187.29-290.97	SNV - 1der	5	0.80	10.0	0.75	11.1
Flavour strength $A_1^{1\%}$ cm 257 nm	115	76.26-116.39	SNV - 1der	5	0.80	3.67	0.76	4.06

Table 1. PLS – Regression Statistics

However, the robustness of the prediction capability must be improved and still lacks of an independent validation phase programmed with samples to be analysed after 2023 harvesting.

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P01.24

USES OF PORTABLE FT-NIR TO DETERMINE CANNABINOIDS AND TERPENES IN DRY CURED CANNABIS FLOWERS

Adham Hamed[1], Ruben Valenzuela[2]

[1]Si-ware Inc., Menlo Park, United States, [2]Valenveras, Sant Andreu de Llavaneres, Spain

Introduction
Cannabis industry is a growing exponentially worldwide. The crop can engage old and new farmers to adopt it as a novel crop. In that sense, there is a need for a fast, on-site, accurate technology to provide to the growers, distributions, and producers a tool to manage the quality control of their site and improve crop optimization. The objective of this work was to develop models for cannabinols and terpenes on a portable and affordable FT-NIR systems that democratize the uses of the technology in this sector. 400l of 400 samples were used to calibrate the cannabinols, and 250 samples to calibrate the total terpenes. The reference analyses were done using ISO certified HPLC-PDA method for cannabinols and GC-FID for the total terpenes. Partial Least Square regression (PLSR) was used to correlate the spectra obtained from NeoSpectra Scanners (17 scanners (Si-Ware Inc., Menlo Park, CA, USA)) from 1350 – 2550 nm with the reference analysis. Moreover, to avoid the spectral changes that the flower suffers during the collection of the spectra, a General Least Square Weighted filter was used to compensate for the temperature shifts and moisture loss.
Models showed a good performance predicting THC, CBD, CBG, Total Terpenes, THC acid, and CBD acid with low error of predictions (Table 1).

	Low Concentration 0-3%				High Concentration 3-30%			
	RMSECV	R^2_{cv}	RMSEP	R^2_p	RMSECV	R^2_{cv}	RMSEP	R^2_p
CBD Total	0.19	0.80	0.16	0.91	1.60	0.89	1.70	0.91
THC Total	0.15	0.85	0.10	0.93	2.10	0.91	2.10	0.90
CBG Total	0.15	0.71	0.11	0.72				
Total Terpenes	0.20	0.70	0.30	0.65				
THC acid	0.16	0.84	0.11	0.91	2.20	0.90	2.20	0.89
CBD acid	0.20	0.79	0.18	0.90	1.65	0.88	1.75	0.90

The current technological solution coupled with PLSR could be used as an alternative to the classical HPLC and GC analysis for in-situ analysis of the cannabis flowers. Moreover, besides the prediction of the cannabinols, total terpenes also can be predicted, giving the final user the tools to discriminate between high and low content phenotypes.

P01.25

FEASIBILITY OF USING PORTABLE NIRS TO DISCRIMINATE IBERIAN HAM ACCORDING TO BREED

Miriam Hernández-Jiménez[1], Ana M. Vivar-Quintana[1], Isabel Revilla[1], Krzysztof B. Bec[2], Justyna Grabska[2], Christian W. Huck[2]

[1]Universidad de Salamanca, Zamora, Spain, [2]University of Innsbruck, Innsbruck, Austria

For decades, the Iberian pig sector has been operating with NIRS technology aimed at detecting fraud in raw materials and final products. The portable NIRs has a great potential in this sector, however, miniaturized devices implement different technology solutions which can affect the analytical performance and feasibility studies are relatively scarce in this area. In this context, the aim of this work was to study the feasibility of discrimination of Iberian ham samples according to racial purity (100% Iberian vs 50% crossbreed) using different portable NIR devices.

A total of 60 pieces of controlled breed Iberian ham were analysed: 24 pieces from the 100% Iberian and 36 pieces from the 50% Iberian ham. The analytical performance in this application was systematically investigated for four portable instruments; microPHAZIR (Thermo Fisher Scientific), MicroNIR Pro ES 1700 (VIAVI), SCiO (Consumer Physics) and Enterprise Sensor (TellSpec; NIR-S-G1). The diffuse reflectance spectra were collected from the surface of ham slice from two distinct domains: muscle and fat. The spectra were subjected to different mathematical pre-treatments, prior the discrimination by means of the RMS-X residual method employed via the WinISI 4.10 software. In order to validate the classification performance, the samples were split into calibration (80% of samples) and prediction (20% of samples) sets.

The table below shows the results of the predictive performance obtained for each of the devices using the best spectral pretreatment based on the percentage of correctly classified samples. While well-fit models were achieved in the calibration for all four instruments, the test-set validation indicated lower predictive performance for TellSpec and MicroNIR. In the case of fat domain measurement, the performance was lower and less disproportionate between the compared instruments. However, the SCiO and microPHAZIR tended to perform better in that case as well, which was reflected by both the performance in the calibration and prediction. In conclusion, miniaturized NIR spectrometers can be successfully used to discriminate Iberian ham according to breed, preferably with spectral measurements of the muscle domain.

	Muscle			Fat		
	Pre-treatment	CAL	VAL	Pre-treatment	CAL	VAL
microPHAZIR	SNV Detrend (2,10,10,1)	89%	81%	SNV Detrend (1,4,4,1)	97%	75%
ScIO	None (1,4,4,1)	97%	83%	Detrend (2,4,4,1)	97%	69%
TellSpec	None (2,8,6,1)	85%	61%	None (1,4,4,1)	91%	64%
MicroNIR 1700	Detrend (28,6,1)	78%	61%	SNV Detrend (1,4,4,1)	76%	61%

P01.26

PREDICTION OF THE TEXTURE OF IBERIAN HAM AND SHOULDERS USING NIRS TECHNOLOGY

Miriam Hernández-Jiménez[1], Ana Vivar-Quintana[1], Isabel Revilla[1]

[1]*Universidad de Salamanca, Zamora, Spain*

Iberian shoulders and hams are very valuable meat products from Spain and, especially hams from acorn-fed finished pigs, reach the highest prices on the market. Among the quality parameters, texture is an important criterion for consumer acceptance. Ham texture depends on proteolysis and the drying process. In fact, some texture defects are related to these processes, such as crust development, which appears when the water content of the product reaches a critical value, and pastiness, which is related to high proteolysis. Traditionally, texture has been assessed by destructive sensory and instrumental measurements, then the feasibility of using NIR technology for texture prediction would be of great interest. However, the prediction of instrumental texture parameters has not yet been carried out and is therefore the objective of the present work.

366 samples of Iberian ham and shoulders produced in the traditional way (Guijuelo, Salamanca) and matured for different periods of time (12, 24 and 36 months) were analysed. One slice (3 cm thick) was cut perpendicular to the bone at the centre of the piece. NIR spectra were recorded using a Foss NIRSystem 500 (Hillerod, Denmark) coupled with a fibre-optic probe (1.5 m 210/210, Ref. n° R6539-A) and a 5 cm x 5 cm window quartz was directly applied on the slice surface at different locations: two points for shoulders and three points for ham. For textural profile analysis (TPA) four samples of each location were cut into cubes of 1 cm × 1 cm × 1 cm. The cubes were axially compressed twice at 1 mm/s until reaching each time 50% of its initial height. A TX-T2iplus equipped with a 5 cm cylindrical probe (Stable Micro Systems, Surrey, England) The following textural parameters were measured from force–deformation curves: hardness, adhesiveness, springiness, cohesiveness and chewiness. The modified partial least squares (MPLS) regression method was used to obtain the NIR equations for all the parameters studied using Winlsi 4.10 Software

The RSQ values obtained in the calibration step (80% of samples) were 0.801 for hardness, 0.787 for gumminess, 0.744 for chewiness and 0.552 for springiness while adhesiveness showed RSQ values < 0.5 then the internal and external validation were not carried out. The calibrated parameters showed RPD values higher than 1.5 and similar values of SEC and SECV. However, in the external validation (20% of samples not used in calibration) when the predicted and reference values of a new set of data were compared by means of t de Student, springiness and chewiness showed p values lower than 0.05. Therefore, significant differences between observed and predicted values were observed for these parameters. On the other hand, results showed that it was possible to predict hardness and gumminess by NIR in Iberian ham and should at different ripening times.

P01.27

CALIBRATING FOR FATTY ACIDS IN CAMELINA BY NIR USING MULTIPLE OILSEED VARIETIES

David Honigs[1], Emily Moore[1]

[1]PerkinElmer, Shelton, CT, United States

When making NIR calibrations for oilseeds, it is important that a wide variability of concentrations is included. Unfortunately, it is not always easy to find this in smaller breeding sets or even in a single variety. In this work we demonstrate how 4 different brassica varieties can be pooled together using Honigs Regression into a single calibration for each of the important fatty acids. This gives more variability for individual seeds, such as camelina, by being able to incorporate more commercially available data from canola and rapeseed.

There are several important oilseeds in the Brassica genus including camelina, rapeseed, canola, and pennycress.

Breeding these small oilseeds requires knowing the ratios of several important fatty acids. One needs to know not only the common commercially important oils like oleic, linoleic, and linolenic, but also the less desirable oils like erucic acid which is considered unhealthy by some as it is sometimes linked to the fatty degeneration of the heart. This is one reason canola is preferred over the older high erucic acid varieties of rapeseed.

Figure 1 shows the PCA of the NIR spectra of the 4 different seed types in this study.

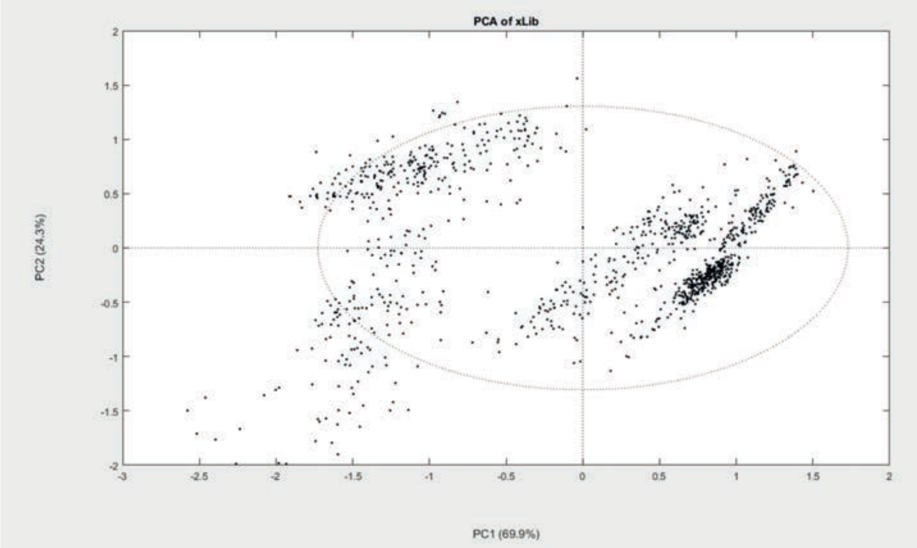

Figure 1. Calibration results will be supplied for fatty acids including 22:1 erucic acid which spans 0 to 40% as a fraction of the total oil in this population, and the fraction of variability coming from the different seeds will be shown emphasizing camelina. Without adding other oilseeds to the NIR dataset, it would be challenging

to measure erucic acid in camelina as it is generally low and doesn't vary much which makes for poor calibration on its own.

Even though the different oilseeds have different spectra and different oil profiles, they can be combined into a single calibration using Honigs Regression. This allows for inclusion of a much broader variability for calibration purposes.

P01.28

STUDY ON THE DETECTION OF RICE SEED VIGOR BY NEAR-INFRARED HYPERSPECTRAL IMAGING TECHNOLOGY

Haiyan Ji[1]

[1]China Agricultural University, Beijing, China

Rice seeds in the drying process due to improper control of process and temperature often cause heat damage, due to low temperature environment and high moisture content of rice seeds during harvest often cause frost damage, all these affect seed vigor. How to quickly and non-destructively identify seeds vigor that are heat-damaged or frost-damaged plays a key role in agricultural production.

The hyperspectral images of rice seeds with different degrees of heat damage and frost damage were obtained by using a hyperspectral imaging system of 866.4–1701.0 nm. After the acquisition of hyperspectral images, a number of rice seeds were randomly selected from each group and tested for germination according to the rules of the International Seed Testing Association (ISTA). Germination force (GF) and germination rate (GR) are one of the main indicators reflecting the quality of seeds. In general, seeds with high GR and GF have high vigor, while seeds with high GR and low GF are also likely to have low vigor.

Preprocessing methods (Savitzky-Golay first derivative(SG1), standard normal variate(SNV), and multivariate scatter correction (MSC)) were used to process the original spectral data, feature extraction algorithms (second derivative, principal component analysis (PCA), successive projections algorithm(SPA), and neighborhood component analysis (NCA)) were used to extract the feature wavelengths.

For heat-damaged situation, three classifier models (k-nearest neighbor(KNN), support vector machine(SVM), and naive Bayes(NB)) were used for modeling analysis. After multivariate data analysis, the MSC-NCA-NB model performed best and was selected as the best model. Finally, the hyperspectral images of the verification set were visualized based on the object-wise method to show the intuitive classification effect.

For frost-damaged situation, deep forests (DF) model and three traditional machine learning models (decision tree (DT), KNN and SVM) were built based on different numbers of sample sets. After multivariate data analysis, it showed that the treatment effect of MSC was the most excellent, and the characteristic wavelength extracted by NCA algorithm was the most useful. In addition, the performance of DF model was better than these three traditional classifier models, and it still performed well in small-scale sample set data. Therefore, DF model was chosen as the best classification model.

In summary,near infrared hyperspectral imaging technology is an effective tool for quickly identifying the vigor of rice seeds that have been damaged by heat and frostbite.

P01.29

RAPID QUANTIFICATION OF POLYPHENOLS USING DISPERSIVE SPE ASSISTED NIRS

Christoph Kappacher[1], Markus Neurauter[1], Matthias Rainer[1], Günther Bonn[1,2], Christian W. Huck[1]

[1]Leopold-Franzens University, Innsbruck, Austria, [2]ADSI - Austrian Drug Screening Institute GmbH, Innsbruck, Austria

Recently polyphenols attracted great interest in the fields of food, nutrition, pharmaceutical and cosmetic industry. This is mainly caused by their positive health effects through their antioxidative behavior in the human body. Due to the large number of compounds that are characterized as antioxidants and the high structural diversity, quantification turns out to be a highly complex task. A wide variety of analytical methods exist to determine the total polyphenolic content, or antioxidative potential. However, they all lack in different tasks such as limit of detection and quantification, repeatability, accuracy and specificity. Therefore, a novel approach combining the existing advantages of solid phase purification, near infrared analysis and multivariate data analysis was investigated for the prediction of the total polyphenolic content within varying sample matrices. Dispersive solid phase extraction was optimized using polyvinylpyrrolidone as sorbent to selectively bind polyphenols. Subsequently, near-infrared spectroscopy was performed and analyzed using multivariate data analysis. Additionally, the method was in-house validated, examining selectivity, repeatability and accuracy, working range, as well as multivariate limit of detection and limit of quantification. In a last step, the performance of the novel developed method was compared to two routinely used methods namely Folin-Ciocalteu photometric assay and Löwenthal titration. The methods were further compared for the prediction of the polyphenolic content in real-life samples, such as tea and wine samples.

P01.30

NIR IMAGING SYSTEM COUPLED WITH A RGB IMAGER FOR WEATHER STRESS EVALUATION OF SWEET POTATO

Subeen Cho[1], Jiwon Choi[1], Euna Cho[1], **Geonwoo Kim**[1,2]

[1]Department of Bio-industrial Machinery Engineering, Gyeongsang National University, Jinju, Republic of Korea, [2]Institute of Agricultural and Life Science, Gyeongsang National University, Jinju, Republic of Korea

Recently, the climate change has led to a decrease in both production and quality of field crop in the Republic of Korea. Consequently, there is a pressing need for technology development that can quantitatively evaluate field crops. Among various field crops, the quality characteristics (such as quantity, meat color, skin color, moisture, protein, and crude fat) of sweet potatoes vary depending on the growing environment. Hence, even if they are of the same variety, the quality of sweet potatoes produced in different regions can differ significantly. Therefore, in the current study, a NIR imaging system integrated with RGB imager was designed to quantify the weather stress of sweet potatoes cultivated in field. The main components of the proposed system are thermal and RGB cameras, control software, battery, and transfer unit. The thermal/RGB imagers were used to evaluate the climate damage and growth monitoring of sweet potato. Consequently, the detailed specifications of the thermal/RGB cameras, control software, and design parameters were derived to quantify the weather stress damage of sweet potato. The developed imaging system demonstrate the capability of collecting real time and multi-imaging data for weather stress evaluation in the sweet potato cultivated in field.

Keywords: Agriculture, environmental analysis, imaging, instrumentation

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P01.31

NONDESTRUCTIVE DETECTION OF MAJOR DEFECTS ON TOMATO USING VISIBLE AND NEAR-INFRARED (VIS-NIR) HYPERSPECTRAL IMAGING.

Haeun Kim[1], Hangi Kim[1], Ye-na Kim[1], Byoung-Kwan Cho[1]

[1]Non-destructive Biosensing Lab/ Department Of Biosensing Machinery Engineering/ Chungnam National University, Daejeon, South Korea

Tomatoes are one of the most widely consumed vegetables in the world and their quality is highly important to both consumers and producers. Tomatoes are exceptionally susceptible to defects such as scratches, compression defects, shape abnormalities, and color abnormalities due to soft and vulnerable pulp. These major defects in tomatoes can affect their overall quality and marketability. However, the process that classify products and defective products relies heavily on human perception and is prone to error when it comes to detecting mixed or ambiguous defects. Therefore, an objective, quantitative, and automated method for screening tomato defects is essential in the agriculture packing facilities. This study proposes to use visible and near-infrared (VIS-NIR) hyperspectral imaging to detect and visualize major defects (scratches, bruises, shape and color abnormalities) in tomatoes using machine learning techniques. To collect spectral data, more than 200 tomatoes with different defects were imaged at wavelengths of 400-1000 nm to obtain 500 images. The data were analyzed using multivariate analysis methods to determine whether the tomato is defective with spectral information extracted from VIS-NIR image data. The proposed method can be used to detect major defects on tomatoes automatically without human labor. The result showed the potential of VIS-NIR hyperspectral imaging for the quality control and grading of tomatoes in the agricultural industry, leading to increased market value and customer satisfaction.

P01.32

PERFORMANCE OF NIR SPECTROSCOPY TO PREDICT AMINO ACIDS CONTENT IN FORAGES

Vincent Larat[1], Lahlou Bahloul[1], Patrick Riche[1], Brian Sloan[2]

[1]Adisseo, Commentry, France, [2]Adisseo, Alpharetta, United States

Near Infrared Spectroscopy (NIRS) has been used as precise and cost-effective tool to predict chemical composition and nutritive values of ingredients. Nutrient-based feeding systems are required in ruminants to formulate more efficient, lower cost and sustainable diets. Progress towards this goal is possible with better characterization of ingredients for amino acids (AA) especially forages that are usually not analyzed in feed analysis. The objective of this work was to develop NIRS calibrations to demonstrate the interest to predict individual amino acids profile in grass silages. 135 grass silages samples were collected between November 2021 and July 2022 from west and center regions of France. The samples were dried at 70°C for 48h before being ground with a 1mm sieve and further analyzed for proximate parameters as well as AA. Spectra of these samples were collected from both undried and dried forms, using a DS 2500 NIR instrument from Foss, over the range 400 – 2500 nm. The set of spectra was split into a calibration set of 119 samples and a test set of 16 samples. Lysine (Lys) and methionine (Met) are of utmost interest as they are usually considered as the first limiting AA for milk production, health and reproduction. The database presents large variation range (CP: 13.7±3.19, Met: 0.145±0.05 , Lys 0.39±0.15 as $g.100g^{-1}$).The NIR calibrations performed on the 1mm ground samples using the Partial Least Squares (PLS) regression reached good predictive precision for both Lys (R^2 = 0.93, Standard Error of Prediction (SEP) = 0.047 $g.100g^{-1}$, Ratio of percent deviation (RPD) = 2.7) and Met (R^2 = 0.91, SEP = 0.013 $g.100g^{-1}$, RPD = 2.8), as well as crude protein (CP) (R^2 = 0.95, SEP = 0.74 $g.100g^{-1}$, RPD = 4.5). Interestingly, the SEP obtained with NIR calibrations surpass in precision the standard error obtained if the AA concentration is estimated from CP wet chemical analyses or NIR predictions (respectively 0.098 and 0.099 $g.100g^{-1}$ for Lys; 0.021 and 0.023 $g.100g^{-1}$ for Met). In conclusion, the results show the ability of NIR spectroscopy to accurately estimate AA in grass forages and its interest to move forward precise formulation of dairy rations.

P01.33

POTENTIAL OF A PORTABLE NIR SPECTROMETER TO CLASSIFY BEEF CARCASSES IN THE ABATTOIR ACCORDING TO THE DIET SUPPLIED

Sara León-Ecay[1], Irantzu Goenaga[1,2], Silvia Arazuri[3],
Ainara López-Maestresalas[3], Joana De Las Heras[1], José Antonio Mendizabal[1],
Kizkitza Insausti[1]

[1]Department of Agricultural Engineering, Biotechnology and Food, ETSIAB-ISFOOD, Universidad Pública de Navarra, Campus de Arrosadia, Pamplona, Spain, [2]Tratamiento Subproductos Agroalimentarios, S.L. (TRASA), Camino San Juan s/n,, Milagro, Spain, [3]Department of Engineering, ETSIAB-ISFOOD, Universidad Pública de Navarra, Campus de Arrosadia, Pamplona, Spain

Booster sustainability of livestock farming is crucial to keep the sector alive. This requires the introduction of novel feeds based on circular economy techniques to reduce the food/feed competition. To ensure the authentication of this local and sustainable beef product with a higher added value, it is necessary to implement assessment and quality control strategies. The objective of the study was to classify beef carcasses in the abattoir without meat sampling according to the finishing feed provided, either vegetable by-products-based (VBPR) or grain and straw-based diet (GS), using a portable near infrared (NIR) spectrophotometer in the range 1200-2000 nm.

11 purebred Pirenaica young calves were supplied a VBPR-based ration, composed by 53.08% concentrated feedstuff, 37.4% of VBPR, 5.45% beet pulp and 4.07% straw whereas other 12 calves were fed with 90% concentrate feed and 10% straw. 24h post-mortem, spectra were collected in the abattoir, before each carcass side was divided into quarters, with a Luminar 5030 NIR spectrophotometer. 20 reflectance spectra were collected in total on the surface of the left carcass Latissimus dorsi muscle. 10 data between the 5th and 6th ribs, and afterwards another 10 entries by the 12th and 13th ribs, were recorded. For a given scan, 50 spectra at 2nm interval were recorded and the mean is provided by the equipment. The spectral data were later imported into the software PLS_Toolbox 9.0. under MATLAB R2020b for analysis. To classify the carcasses, the Radial Basis Function-Support Vector Machine (RBF-SVM) method was utilized. Different pretreatments were tested, although the best results were achieved thanks to the application of Multivariate Scatter Correction (MSC).

The overall accuracy of the RBF-SVM model ranged from 90.91% for Calibration (Cal) to 82.20% for Cross Validation (CV), with a cost of 10 and gamma 1. In Cal, the % of Correctly Classified carcasses started from 81.82% to VBPR calves up to a 100% for GS-finished animals. In CV, the % were slightly lowered, with a 72.73% for VBPR and 91.67% for the GS group.

Hence, the potential of portable NIR spectrometers as an at-line system in a commercial abattoir environment was demonstrated to authenticate beef carcasses according to the finishing feed provided to fattening calves.

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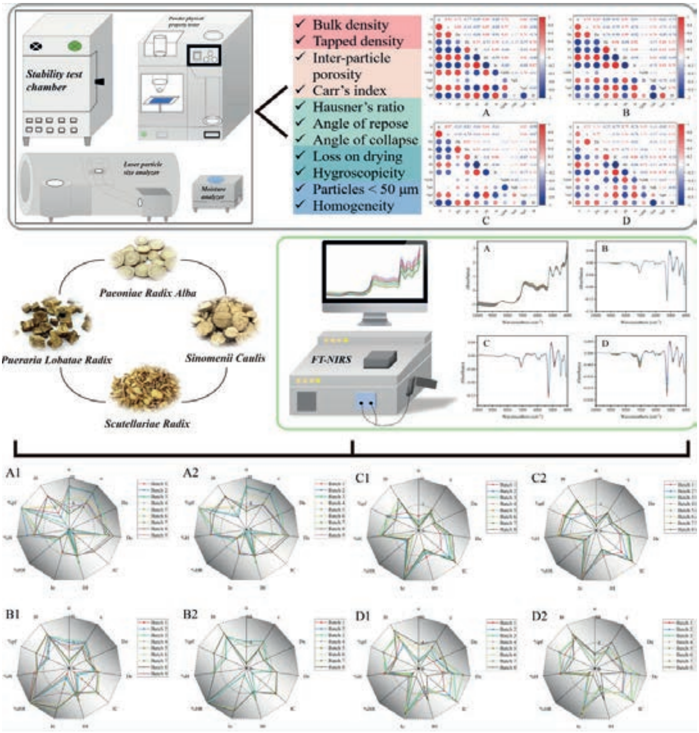
P01.34

PHYSICAL FINGERPRINT TRANSFORMATION OF HERBAL MEDICINES POWDERS USING NEAR-INFRARED SPECTROSCOPY

Sijun Wu[1], **Wenlong Li**[1]

[1] Tianjin University of Traditional Chinese Medicine, Tianjin, China

The physical properties of herbal medicines powders are of upmost important in the pharmaceutical manufacturing. Before blending, direct compression or wet granulation, it is critical to obtain the knowledge and subsequently control the physical properties. However, the determination of physical parameters requires specific and expensive equipment with time-consuming analysis. In the research, near-infrared (NIR) spectroscopy was utilized to collect the NIR spectra of Scutellariae Radix, Pueraria Lobatae Radix, Paeoniae Radix Alba and Sinomenii Caulis. The physical parameters of these medicines were used to establish the physical fingerprints. Direct standardization, partial least squares regression and generalized regression neural network (GRNN) were used to relate the NIR spectra and physical fingerprints, respectively. The result demonstrated that GRNN could perfectly achieve the physical fingerprint transformation. The cosine similarity and residual predictive deviation of two physical fingerprint of each sample was larger than 0.99 and 2.0, respectively.


Stability test chamber
Powder physical property tester
Laser particle size analyzer
Moisture analyzer
✓ Bulk density
✓ Tapped density
✓ Inter-particle porosity
✓ Carr's index
✓ Hausner's ratio
✓ Angle of repose
✓ Angle of collapse
✓ Loss on drying
✓ Hygroscopicity
✓ Particles < 50 μm
✓ Homogeneity
A
B
C
D
Paeoniae Radix Alba
Pueraria Lobatae Radix
Sinomenii Caulis
Scutellariae Radix
FT-NIRS
A
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A1
A2
C1
C2
B1
B2
D1
D2

P01.35

DEVELOPMENT OF NIR SPECTROSCOPY METHODS FOR THE QUANTITATIVE EVALUATION OF FRUIT EXTRACTS AND FORTIFIED FRUIT JUICE

Matyas Lukacs[1], Flora Vitalis[1], Zoltan Gillay[1], Zoltan Kovacs[1]

[1]Hungarian University Of Agriculture And Life Sciences, Institute of Food Science and Technology, Budapest, Hungary

Nowadays more and more consumers are following selective diets often including added-value products for physical and/or mental health. Grape seed extract (GSE) is one of the best-selling dietary supplement-constituents in the World, due to its numerous associated health benefits and the vigorous marketing communication of vendors. With a high economic value, GSE is a frequent target of intentional adulteration using cheaper substitute compounds with similar chemical profiles, often carrying health-related risks (allergens). These frauds can remain undetected throughout the supply chain due to the frequent use of unspecific analytical evaluation methods for quality assessment. Since the health-preserving impact of these products is often debatable due to insufficient or contradictory scientific research, the risk of consumer deception is already significant. For these reasons, it is important to develop rapid and highly selective analytical methods for the quality assessment of these products. Present research aims to develop NIRS methods for the quantitative evaluation of GSE powder when adulterated with chemically similar compounds, and when used as an additive for fruit juice fortification. Adulteration was modelled by mixing GSE with peanut skin extract, pine bark extract and green tea extract on various levels (0-13%) and in different combinations. Fruit juice samples were prepared by using GSE and other high-value fruit extracts (pomegranate extract, cranberry extract) for fortification in different concentrations (0-12%) and mixtures. All measurements were performed on a desktop instrument, a handheld and a self-built functional NIRS (fNIRS) device for comparison with future developments in mind. Following spectral pretreatment, linear discriminant analysis was performed to classify samples of different extract type, while quantitative models were built to predict extract concentrations and proanthocyanidin content using partial least squares regression and support vector machine regression. Multivariate limit of detection and limit of quantification were calculated for each extract type. Classification accuracy on higher concentration levels reached a 100% for most samples, while quantitative models were capable to accurately predict extract concentrations both in powder mixtures and fruit juice matrices. Present research proves the applicability of NIRS coupled with chemometrics for the rapid and accurate quality evaluation of fruit extracts.

P01.36

MONITORING THE RIPENING OF CHEESE AT VARIOUS TEMPERATURES BY MEANS OF NEAR INFRARED SPECTROSCOPY

Mariem Majadi[1], Zoltan Kovacs[1], Zoltan Gillay[1,2], Tamás Tóth[2,3], George Bazar[2]

[1]Hungarian University of Agriculture and Life Sciences, Institute of Food Science and Technology, Budapest, Hungary, [2]ADEXGO Kft, Balatonfüred, Hungary, [3]Széchenyi István University, Agricultural and Food Research Center, Győr, Hungary

The dairy market is significantly evolving as people are becoming more aware of their nutritional needs. Recent studies suggest the modification of the animal feed instead of the dairy product itself to increase its nutritional value and essentially to meet the market's demands in terms of the quality of the product. Consequently, there is a need to evaluate these new products by not only its physicochemical aspects but also by its sensory and general composition. In the present study, we aim to evaluate the potential of near infrared spectroscopy (NIRS) in monitoring the ripening of Trappist style cheese at different temperatures, and to detect differences in the ripening process caused by the diet fed to the cows (control: supplemented with hydrogenated palm oil; experimental: supplemented with algae extract and linseed). Cheese samples were stored in triplicates at different temperatures (16 °C, 10 °C, 4 °C) and kept in storage for 0, 4, 8, and 12 weeks. For repeatability purposes, the experiment was performed twice. Hence, producing 120 samples ([fresh samples + 3 temperatures x 3 storage times] x 2 diet types x 3 repeats x 2 experiments). The NIR spectra were acquired using a Metrohm XDS rapid content analyzer in the wavelength range of 400 - 2500 nm. Three consecutive scans were recorded for each sample in diffuse reflection mode at room temperature using a circular glass cuvette. Prior to any measurements, the cheese was carefully cut into pieces with a sample thickness of 1 cm and the freshly cut surface was scanned. The chemometric evaluation of the NIRS data was carried out with principal component analysis (PCA) and PCA-based linear discriminant analysis (PCA-LDA). The PCA-LDA models were cross-validated by 'leave one repeat out' method. PCA showed a clear tendency of separation between the cheese samples according to the type of diet. Further analysis with PCA-LDA revealed a good discrimination between samples at different storage time (Figure 1. Discrimination of control and experimental Trappist cheese samples stored at different temperature for 4, 8 and 12 weeks in two independent experiments using NIRS.) and temperature. NIRS technique has many advantages compared to other traditional methods. It is rapid, highly accurate, versatile and multi-analytical. It was proven in this study that NIRS coupled with chemometrics is an efficient technique for monitoring cheese quality variations during ripening and to detect alterations related to the type of cow feeding. The technology may be applied (1) in quality control to confirm the declared storage temperature and ripening duration, and (2) in product development to monitor the differences in the ripening process caused by the altered composition of milk.

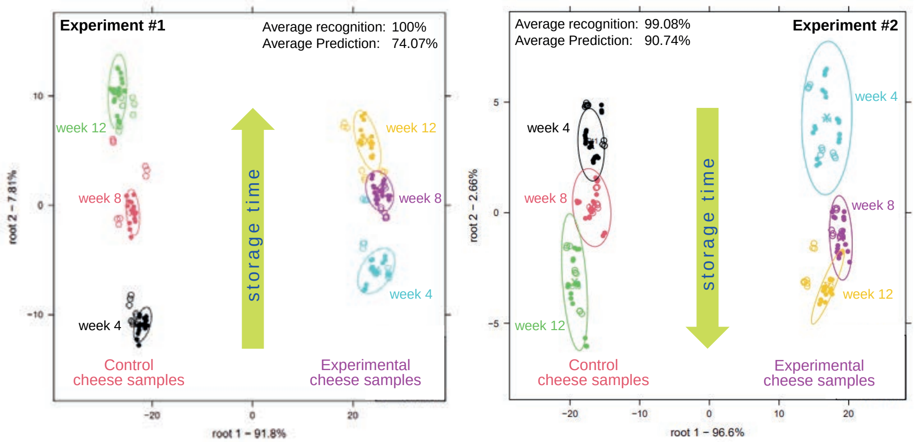


Figure 1. Discrimination of control and experimental Trappist cheese samples stored at different temperature for 4, 8 and 12 weeks in two independent experiments using NIRS

P01.37

NEAR INFRARED SPECTROSCOPY AS A TOOL FOR THE MONITORING IN THE COMMERCIALIZATION OF THE MUSSEL PERNA PERNA (LINNAEUS, 1758)

Amanda Oliveira[1], Vanessa Valladares[1], Claudia Santos[1], **Clélia Mello-Silva**[1]

[1]Oswaldo Cruz Foundation, Rio de Janeiro, Brazil

The Perna perna (Linnaeus, 1758) mussel is the most commercialized mollusk species in Brazil, but the lack of control over the size of extracted mussels is a recurrent and worrisome issue. According to the current Brazilian legislation, mussels with a shell length of 50 mm or larger are adults and suitable for commercialization and human consumption. However, those animals are usually sold precooked and without shells, which prevents accurate identification of juvenile and adult individuals. Based on this, Near Infrared Spectroscopy (NIRS) was analyzed here as a tool to differentiate juvenile mussels from adults and sample provenance. A total of 176 mussels were collected on the Jurujuba and Vermelha beaches, which are in opposite sides of Guanabara Bay, Rio de Janeiro State, Brazil. Spectra were obtained from the muscle group and a specific part of the shell. The discrimination of specimens < 50 mm and > 50 mm was of 66.03% for the muscle group, and 78% for the shells. The two size classes were also distinguished efficiently when the two beaches were evaluated individually. It was observed that the environment influences the metabolism of the adult mussels, with a 85.71% hit rate. These results indicate NIRS as a tool to control the legal extraction of P. perna for commercialization, thus favoring the maintenance of natural mussel stocks.

P01.38

NIR SPECTROSCOPY MODELS FOR PHENOTYPING WOOD PROPERTIES OF CORYMBIA TORELLIANA X CORYMBIA CITRIODORA

Leticia Miranda[1], Fernanda Maria Guedes Ramalho[1], Regiane Abjaud Estopa[1], Flaviana Reis Milagres[1], Bruno Afonso Magro[1], Silvana Meister Sommer[1], Carlos Augusto Soares do Amaral Santos[1]

[1]Klabin S/A, Telêmaco Borba, Brazil

The selection of individuals in a breeding program must always be done according to the characteristics of interest in the production process. For the production of cellulosic pulp, the physical and chemical wood characteristics must be considered in the genotypes selection for commercial plantations, as they affect the industrial process directly, both economically and in the quality of the final product. In this study, wood from trees previously selected in hybrid progenies tests of Corymbia torelliana x Corymbia citriodora (CTOxCCT) between 4 and 5.5 years was evaluated. Predictive models were built using near-infrared (NIR) spectroscopy for the properties of wood basic density of chip, extractive content, total lignin content, syringyl/guaiacyl ratio (S/G) and pulp yield. Sawdust samples from 208 CTOxCCT individuals genetically selected were collected in Telêmaco Borba, Paraná state in Brazil and were classified and prepared for NIR spectra readings. After the readings, 25 samples of CTOxCCT were selected by principal component analysis (PCA) and Kennard-Stone algorithm for the composition of the models. The selected trees were cutted down and sectioned into short logs, peeled, chopped and their chips selected, and then sent for determination of chemical properties through kraft pulping. Based on the reference data and spectra in NIR, prediction models for each of the properties were developed. The NIR spectroscopy predictive models showed a satisfactory ability to estimate the chemical properties of wood. The prediction models of basic chip density, extractives contents, total lignin contents, S/G ratio and pulp yield showed R^2cv of 0,60; 0,37; 0,56; 0,63 and 0,52; with RMSEcv of 23,13 Kg.cm-3, 0,81%; 0,67%; 0,24 and 2,94% respectively. The RPDcv (Standard Deviation Performance Ratio) values for these characteristics varied between 1,2 and 1,6. The model showed potential for ranking CTOxCCT trees in genetics selection for all parameters studied highlighting basic chip density and S/G ratio. NIR spectroscopy can potentially be applied in breeding programs, allowing the non-destructive selection of trees with physical and chemical properties suitable for the production of cellulose. In addition to the study having innovation in the development of models for Corymbia.

P01.39

ADAPTIVE NIR PREDICTION MODELS FOR THE FACILE INCLUSION OF NEW DATA

Emily Moore[1], David Honigs[1], Zhongyang Wan[2], James House[2],
Thomas Warkentin[3]

[1]PerkinElmer Scientific Canada ULC, Woodbridge, ON, Canada, [2]Department of Food and Human Nutritional Sciences, University of Manitoba, Winnipeg, MB, Canada, [3]Crop Development Centre, University of Saskatchewan, Saskatoon, SK, Canada

A learning regression approach uses new spectra and reference values to evolve an existing NIR prediction model, rather than reconstructing one from scratch. In this work, protein content in peas was modelled by 1) partial least squares (PLS) regression or 2) Honigs regression (HR); The latter being a "learning regression", which utilizes PLS in conjunction with the K-nearest neighbours algorithm. The original database (N=550) was supplemented with a new dataset (N=379) and, initially, each calibration model was reconstructed. Comparison of SEP values (on a validation set of N=100) showed improvement in predictive accuracy from 1.70 to 0.67 for the PLS method, and 1.82 to 0.61 for the HR method. The HR method was then used to add the new dataset directly to the previous model's library, resulting in a comparable SEP (0.66) to the conventional reconstruction approach. This evolutionary HR approach offers a faster and less cumbersome solution to updating prediction models, without needing to access the original database.

P01.40

A CLASSIFICATION MODEL FOR VARIOUS MILK POWDER BRANDS AND TYPES USING NEAR-INFRARED (NIR) SPECTROSCOPY AND PARTIAL LEAST SQUARES-DISCRIMINANT ANALYSIS (PLS-DA)

Áine Ní Fhuaráin[1], Aoife Gowen[2]

[1]University College Dublin, Dublin, Ireland, [2]University College Dublin, Dublin, Ireland

Milk powder is important as it increases the shelf-life of milk, which in turn increases export opportunities from Ireland (Bista et al., 2021). Milk powder has uses in various foods such as yoghurts (Khan et al., 2021). In this study, 120 spectra of six classes of milk spectra are analysed using NIR spectroscopy and chemometric methods. The milk powders have varied compositions and a wide range of protein and fat contents. The powders consist of two brands of WMP (Whole Milk Powder), two brands of SMP (Skim Milk Powder), one brand of PP (Protein Powder) and one brand of IF (Infant Formula). An MPA II (Bruker, Germany) with OPUS software was used to take the NIR spectra in the range of 3,950 – 11,550 cm^{-1}. The integrating sphere accessory was used which has a measurement method of diffuse reflection. The instrument contains a TE-InGaAs [Sphere] detector. Half the spectra made up the calibration set taken one week, and the other half of the spectra made up the validation set taken the week after, rendering the validation set independent (10 reps each were used for the calibration set and validation set). MATLAB R2022a was used to perform the data analysis. Principal Component Analysis (PCA) was performed and was able to separate six clusters, however, the two brands of SMP are overlapping in places, as in Figure 1. Next, a milk powder brand and type prediction model is created using PLS-DA, with seven pretreatments evaluated. The model has 100% accuracy when Multiplicative Scatter Correction (MSC) pretreatment is used with one latent variable. Therefore, NIR spectroscopy is suitable when distinguishing between different types of milk powder, even within different brands of the same milk powder type.

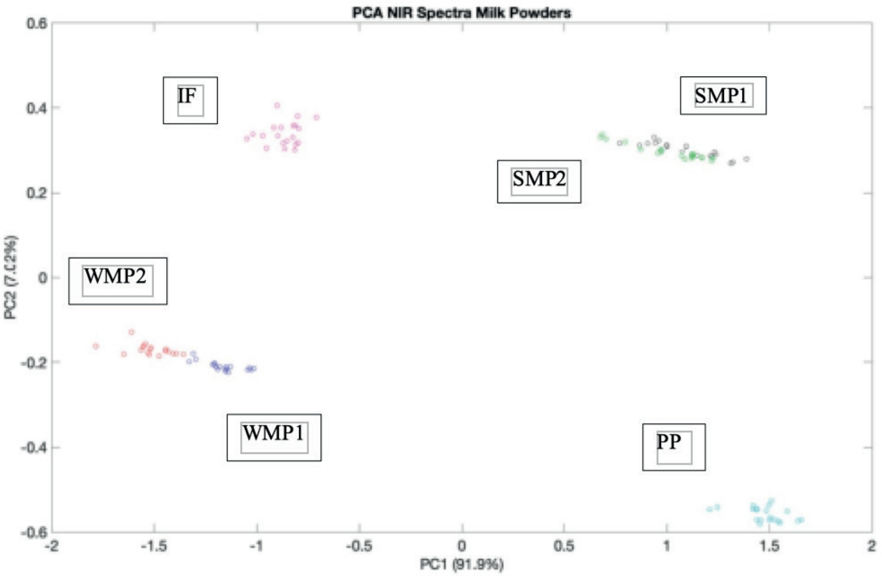


Figure 1. Plot showing PCA of the NIR spectra of the calibration and validation sets (n=120) of six classes of milk powder.

Reference List

Bista, A. et al. (2021) 'Key parameters and strategies to control milk concentrate viscosity in milk powder manufacture', International Dairy Journal, 121, p. 105120.

Khan, A. et al. (2021) 'Near-infrared spectroscopy and data analysis for predicting milk powder quality attributes', International Journal of Dairy Technology, 74(1), pp. 235–245.

P01.41

AUTHENTICATION OF THE IBERIAN PIG' FEEDING REGIME AND THE CORRESPONDING COMMERCIAL CATEGORY VIA NIRS ANALYSIS OF FAECES AND CHEMOMETRICS

Pablo Rodríguez-Hernández[1], Vicente Rodríguez-Estévez[1],
Cristina Burguillo-Martín[1], **Nieves Núñez-Sánchez**[1]

[1]Department of Animal Production, University of Cordoba, Córdoba, Spain

The Iberian pig is an autochthonous breed traditionally reared in the Iberian Peninsula (Spain and Portugal). Its products hold international reputation not only due to its superior quality but also because of the added value from its traditional free-range finishing system, foraging acorns in an agroforestry system called dehesa. Although attempted in the past, nowadays there is no official analytical methodology standardised and employed for the authentication of the pigs' feeding regime. However, consumers demand food authenticity and the development of accurate analytical techniques to authenticate productive systems and to support field inspections. Near Infrared Reflectance Spectroscopy (NIRS) constitutes a technique that has been increasingly implemented in the agri-food industry because this method is non-destructive, cheap and rapid, and allows to obtain a great repeatability and reproducibility. The objective of the present study was to evaluate the potential of NIRS analysis to discriminate and authenticate the feeding regime and the corresponding commercial category of the Iberian pig by means of faeces analysis, considering their easy collection, especially in extensive systems where pigs' handling is difficult.

166 individual faecal samples were collected in 12 farms, differentiating three groups in terms of feeding regime: free-range acorn-fed or bellota (B), outdoors feed-fed or cebo de campo (CC) and feed-fed or cebo (C). NIRS analysis was carried out with a FOSS-NIR-Systems 6500, using the spectral information through chemometrics. A discriminant model based on partial least squares-discriminant analysis (PLS-DA) was performed with an 80% of the initial sample set as training set, and the remaining 20% as blind samples to validate the model (Figure 1). Only two samples belonging to B and CC categories were misclassified, reaching a general classification success of 94.17% in the external validation. All the samples from the C group were correctly predicted. The classification error of the discriminant model could be explained considering the diet of the animals: pigs from CC farms may have sporadic access to natural resources just like animals from B farms (completely based on grazing natural resources), so the faeces profile and thus the resulting spectral information could become similar in some cases. Hence, the inclusion of more samples and more farms in future studies are factors of paramount importance to compile more variability and to improve the discriminant models' robustness. Nevertheless, the results obtained are considered very satisfactory and NIRS analysis of faeces has demonstrated to be a promising approach for the authentication of the Iberian pigs' diet during field inspections.

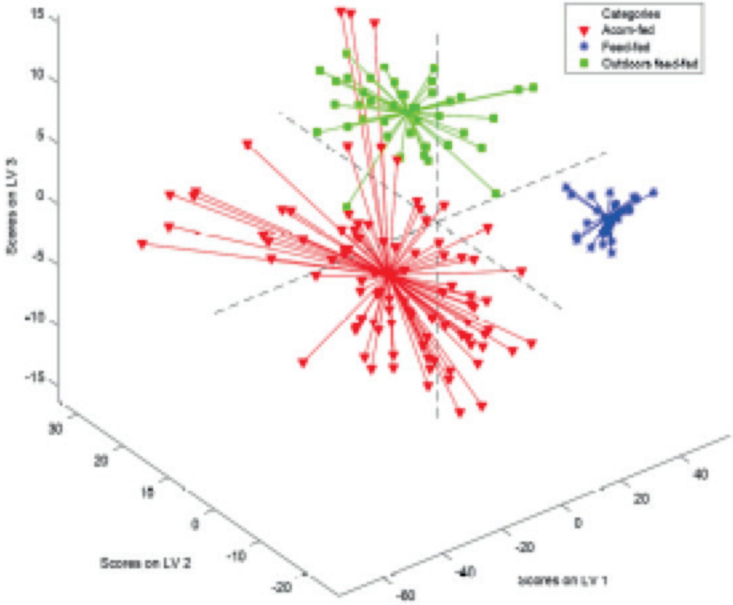


Figure 1. Score plot of PLS-DA model obtained after NIRS analysis of Iberian pigs' faeces.

P01.42

MEASUREMENT OF QUALITATIVE PARAMETERS OF GRAPES DURING THE FINAL STAGES OF RIPENING USING VIS/NIR SPECTROSCOPY: APPLICATION IN REAL SCALE CONDITIONS IN WINERIES

Alessia Pampuri[1], Alessio Tugnolo[1], Sara Vignati[1], Marco Menegon[1], Andrea Casson[1], Roberto Beghi[1], Riccardo Guidetti[1], Valentina Giovenzana[1]

[1]Università Degli Studi Di Milano, Milano, Italy

The present work had the aim of investigating the application of vis/NIR spectroscopy for the rapid measurement of grape main ripening parameters and of the polyphenol content during the final stages of the maturation process and at the winery consignment. This is the second experimental campaign carried out to validate the previously obtained results and the predictive models with further samples. The first campaign involved analyzes conducted in lab-scale conditions in the spectral range between 400 and 1650 nanometers on samples of grapes (Vitis Vinifera L.) variety "Ancellotta" at a distance of 150 mm between the sensor and the sample. During this second campaign, using the same instrument and respecting the conditions of the previous year, the acquisitions were conducted both in the laboratory and directly in the winery to verify the real feasibility and applicability of the technique. Partial Least Square (PLS) regression analysis was applied on grape spectra in order to predict different parameters such as Babo degrees and anthocyanins and polyphenol content. The results from the PLS models gave coefficients of determination and root mean square errors in calibration and in prediction, respectively, equal to 0.81 and 0.69, and 0.67 (mg/l) and 0.94 (mg/l) for Babo degrees, equal to 0.79 and 0.79, and 118.9 (mg/l) and 119.9 (mg/l) for anthocyanins, equal to 0.55 and 0.45, and 266.3 (mg/l) and 287.6 (mg/l) for polyphenols. Regarding anthocyanins and Babo degrees estimation, classification models (PLS-DA) have also been created, which are also very useful for evaluating the grape samples upon consignment. Vis/NIR technology has long been recognized as a green method that allows for considerable time savings: applying it in a sector with high added value, such as the wine one, to streamline and automate quality controls in cellars could help this industry to the leap in quality from an industry 4.0 point of view.

P01.43

THE USEFULNESS OF NIRS CALIBRATIONS BASED ON FEED AND FECES SPECTRA TO PREDICT NUTRIENT CONTENT, DIGESTIBILITY AND NET ENERGY OF PIG FEEDS

Louis Paternostre[1], Vincent Baeten[1], Sam Millet[2], Johan De Boever[2]

[1]Cra-w, Gembloux, Belgium, [2]ILVO, Melle, Belgium

Feces are mainly composed of the indigestible feed residues that are resistant to the digestion process. Because feces composition varies among animals and growing conditions, it thus reflects feed-animal interactions. In recent years, studies have shown the interest of near infrared spectroscopy (NIRS) on feces to predict different parameters related to the animal or its diet. In a study with pigs, we investigated the usefulness and accuracy of NIRS based on feed and feces spectra to predict nutrient composition, digestibility and the net energy (NE) of feeds. For this purpose, 62 feeds and 310 freeze-dried feces samples (5 per feed) from 3 in vivo digestibility experiments were used. First, calibrations based on either feed spectra or feces spectra only were developed to predict their chemical composition. Then, calibrations based on combined feed and feces spectra were developed. The calibrations were evaluated by cross-validation either by leave-one-feed-out or by splitting the dataset in 4 random groups. Most of the chemical parameters of the feed and the feces could be predicted accurately (residual prediction deviation, RPD ~ 3, $R^2 > 0.8$), but predictions for feed were somewhat less accurate for crude protein (RPD = 2.1), non-starch polysaccharides (RPD = 2.0) and sugar (RPD = 1.0); and for feces, crude fiber (RPD = 2.1), organic matter (RPD = 2.2) and gross energy (RPD = 1.8). Net energy was better estimated using feed spectra than feces spectra, with a standard error of cross validation (SECV) of 0.33 and 0.46 MJ/kg, respectively. The digestibility of nutrients was poorly estimated from both calibrations based on feed or feces spectra alone (RPD ~ 1.5). The combination of spectra resulted in an overall better estimation of the digestibility and an SECV of 0.26 MJ/kg for the NE. For all calibrations based on feces spectra, either singular or combined, the leave-one-feed-out cross-validation resulted in higher SECV than the validation with 4 random groups. However, the latter cross-validation method may lead to over-optimistic results as the group to be validated may contain spectra which are not independent.

P01.44

NEW INSIGHTS INTO THE DISCRIMINATION OF SUGARCANE CLONES IN THE FIELD USING NEAR-INFRARED SPECTRA

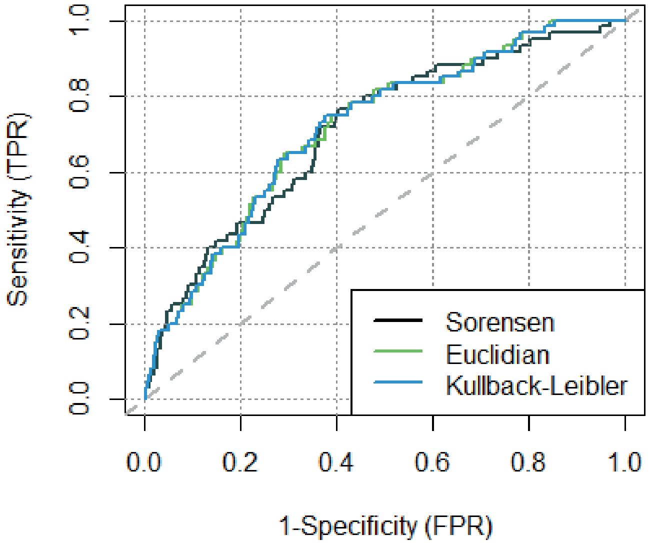
Luiz Alexandre Peternelli[1,2], Andréa Carla Bastos Andrade[1]

[1]Universidade Federal de Viçosa UFV. Department of Statistics, Viçosa, MG, Brazil,
[2]Sugarcane Breeding Program, UFV, Viçosa, MG, Brazil

Sugarcane is an essential crop for sugar and bioenergy needs. Breeding programs are essential to meet world demand since they increase sugarcane yield without augmenting croplands. These programs work on efforts to find the best clones in a long-lasting selection process. The initial phase (T1) of sugarcane breeding can be considered the most important in a breeding program. In this phase occurs the first selections of plants or families. The visual selection performed in T1 is fundamental in identifying the best genotypes within the best families, which is crucial for the breeding program's success. Currently, some programs have considered dense experiments in the T1 phase, which can lead to problems in the selection since, due to the typical tillering of the plant, stalks belonging to a unique individual might be considered different and mistakenly taken to the later evaluation phases. Near-infrared (NIR) spectra could identify which samples belong to the same individual, enabling more accurate early selection in these dense experiments. Recent work under controlled conditions has indicated the potential of using NIR for this purpose.

The present work aimed to (i) use NIR data obtained at the field level, that is, without controlling environmental conditions, in samples of clones as a way of discriminating stalks from the exact clone (clump) within families; (ii) evaluate the possibility of defining a NIR-based protocol for the early differentiation of genotypes, mainly in dense experiments, with a view to the selection of sugarcane families.

Data were obtained from leaf samples of clones from family selection experiments carried out at CECA-UFV. Raw and pretreated spectra were considered when taking distance measures like Euclidian, Sorensen, and Kullback-Leibler distances to compare pairs of samples. ROC curves were used to evaluate the potential of using such a simple procedure to infer the similarity of the samples and, in the end, to classify samples into distinct genetic groups.



The use of NIR showed robust results in conditions without environmental control. The distance measures showed comparable results for indicating similar samples. As expected, the results are pretreatment dependent and need further evaluation. Thanks to: CNPq, CAPES, RIDESA, and FINEP.

P01.47

COMBINATION OF NIR AND RAMAN SPECTROSCOPY FOR THE ASSESSMENT OF THE BOTANICAL ORIGIN OF MONOFLORAL HONEY USING THE LASSO METHOD

Marco Bragolusi[1], Andrea Massaro[1], Carmela Zacometti[1], Alessandra Tata[1],
Roberto Piro[1]

[1]Istituto Zooprofilattico Sperimentale Delle Venezie, Laboratorio di Chimica Sperimentale, Vicenza, Italy

Monofloral honey is usually certified by melissopalynological analysis and sensory assessments. The technique is laborious, expensive and needs highly qualified operators. The goal of this study is the development and validation of a spectroscopic method based on the combination of near infrared and Raman spectroscopy for the rapid and accurate authentication of the botanical origin of monofloral honey.

We analyzed 87 samples from 9 different botanical origins (10 acacia, 10 citrus, 10 chestnut, 10 eucalyptus, 10 sunflower, 10 taraxacum, 10 forest honeydew, 7 fir honeydew and 10 linden). The Italian National Observatory previously verified the botanical origin of each sample.

After warming up at 40°C for 60 min in a thermostat, the liquefied honey were subsequently analyzed on a Bruker MPA™ (Bruker Optik GmbH, Ettlingen, Germany) equipped with a quartz beam splitter, an integrated Rocksolid™ interferometer, a rotating sample wheel and two detectors (a PbS and a TE-InGaAs detector) combined with OPUS 7.0 (Bruker Optik GmbH) software. Absorption spectra were collected by transflectance mode. The resolution was 8 cm−1. The scanner speed was 10 kHz and each spectrum had 6 scans averaged. Each sample was placed into a glass Petri dish with a thickness of 0.3 mm, and then analyzed four times. The data were acquired between 7600 cm−1 and 4100 cm−1 and then smoothed by Savitzky Golay filter. Finally, the spectra were averaged and normalized by multiplicative scatter correction (MSC).

Honey samples were transferred into a four milliliters-glass vial, heated at 40°C and analyzed by Raman spectroscopy. Raman fingerprints were acquired by using a handheld BRAVO spectrometer (Bruker Optik GmbH), equipped with a DUO LASERTM excitation system and a fluorescence mitigation system, called Sequentially Shifted Excitation (SSE), based on controlled variations of the laser temperature. The data were acquired between 1800 cm−1 and 300 cm−1 wavenumber. Each aliquot was analysed in duplicate and then normalized by standard normal variate (SNV). The spectra were averaged. The pre-processing of NIR and Raman data was performed by R (version 4.0.2) and the R package caret was used for model build-up.

Both the Raman and NIR pre-processed datasets were separately submitted to least absolute shrinkage and selection operator (LASSO) and the classifiers were validated by 10-repeated 5-fold cross-validation with decent performances.

In order to enhance the classification performances of the classifiers, the data were merged by data fusion and one unique LASSO model was generated. The classifier was validated by 10-repeated 5-fold cross-validation. The resultant overall accuracy, sensitivity and specificity were excellent. The performances of the combined LASSO model are being further evaluated by validation with an independent batch of samples.

P01.48

IDENTIFICATION OF LENTIL VARIETY IN WHEAT FLOURS FORTIFIED WITH LENTIL FLOUR USING NIR TECHNOLOGY

Isabel Revilla[1], Miriam Hernández-Jiménez[1], Iván Martínez-Martín[1],
Ana M. Vivar-Quintana[1]

[1]Universidad de Salamanca, Zamora, Spain

The consumption of legume flours, including lentil flour, is becoming increasingly important as it is used as a fortifier due to its high iron content. Spain is an important producer of lentils, with the brown varieties being the best adapted to the climatic conditions. Therefore, the three most produced varieties in Spain are Guareña, Pardina and Castellana. Part of the production of the first two is distinguished by the Protected Geographical Indications 'Lenteja de la Armuña' and 'Lenteja de Tierra de Campos' respectively, while the third is the most produced in Spain. Although there are some studies that address the characterisation and differentiation of some of these varieties, all of them have a yellow endosperm, which can make it difficult to identify them when they are made into flour. Therefore, the feasibility of discriminating the lentil variety in wheat samples fortified with lentil flour by means of NIRS were studied.

A total of 153 samples were analysed, corresponding to 3 wheat samples, 20 samples of each lentil variety: Guareña, Pardina and Castellana (60 samples of 100%) and fortified flour samples prepared by mixing 10 samples of each lentil variety with wheat flour at 25%, 50% and 75% (90 samples). The flours were analysed with a Foss NIR System 5000. A 1.5 m 210/210 remote reflectance fibre probe (Ref. No. R6539-A) with a 5x5 quartz window area was used and applied directly to 10 g of flour. The spectral range used was between 1100-2000 nm at 2 nm intervals. The RMS-X residual discriminant method (WinIsi 4.10) was performed after applying different mathematical, derivative and smoothing treatments to the spectra. For this purpose, the total number of samples was divided into 80% for calibration and 20% for the prediction groups.

As mentioned above, in all cases flour from brown varieties with yellow cotyledons was used. However, NIR technology can discriminate the lentil variety very effectively. After the optimisation process, the discrimination ability reached 100% in the samples used for calibration and 96.67% in the data used for prediction. Thus, the SNV (2,4,4,1) and SNV-Detrend (2,4,4,1) combinations correctly classified 100% of the samples of the three lentil varieties in the calibration group, and 100% of the samples of the Armuña and Pardina varieties and 90% of the Castellana variety in the prediction group. The Pardina variety showed in general the highest percentages of correct classification.

P01.49

THE USE OF NIR SPECTROMETRY FOR THE PREDICTION OF FE AND MG IN MUFFINS MADE WITH WHEAT-LENTIL FORTIFIED FLOURS

Isabel Revilla[1], Miriam Hernández-Jiménez[1], Iván Martínez-Martín[1],
Ana M. Vivar-Quintana[1]

[1]Universidad de Salamanca, Zamora, Spain

The World Health Organisation estimates that iron deficiency is one of the most prevalent micronutrient deficiency, with children and women in developing countries being the most vulnerable population. In addition, some studies point to an increased risk of magnesium deficiency in developed countries, due to a significant decrease in Mg concentration in cereals. Legume flours, such as lentil flour, could be an alternative for use as a fortifier, as it is an important source of Fe and Mg and it can be used to make different products, such as muffins, which have been successfully used to include fortified flours. The determination of metal composition is usually performed by ICP-mass analysis, which is a complex and time-consuming method. This work proposes a rapid method to determine the composition of Mg and Fe in muffins by near infrared (NIR) spectroscopy. A total of 116 muffin samples were made using wheat flour, lentil flour of three brown varieties (Guareña, Pardina and Castellana) and mixtures of wheat and lentil flours at the 25, 50 and 75% levels. NIR spectra were recorded with a Foss NIR System 5000 coupled to a 1.5 m 210/210 remote reflectance fibre probe which has a 5x5 quartz window. Muffins were milled in a hammer mill and the quartz window was applied directly on 10 g of the ample. The spectral range used was between 1100-2000 nm at 2 nm intervals. Fe and Mg in the muffin samples were measured by ICP-mass analysis after nitric digestion. The modified partial least squares (MPLS) regression method was used to develop the NIR calibration model.

The results of the calibration models shown in the table were very satisfactory, with RSQ values >0.9 for both metals and a very low difference between SEC and SECV values for Mg. To check the predictive models, an external validation was performed on 20% of the samples. The results showed that there were no significant differences between the predicted and reference values (p-values >0.05) and that the RSME values were low. These results indicate that it was possible to predict Fe and Mg by NIR with a fibre optic probe, with a range of applicability of the obtained calibration equations comparable to that obtained by the reference technique.

Table 1. Statistical descriptors for the best calibration equations

Mineral	Math treatment	N	Mean	SD	Min Est.	Max Est.	SEC	SECV	RSQ
Fe	Detrend-SNV (1,4,41)	88	30.0086	12.2460	0.0000	66.7464	2.4890	3.2734	0.958
Mg	Detrend (2,4,4,1)	89	0.0357	0.0119	0.0002	0.0713	0.0012	0.0017	0.990

P01.50

NIR-SPECTROSCOPY OF FORAGES – SAMPLING ASPECTS FOR DRIED POWDERS AND CONSEQUENCES FOR MULTIVARIATE CALIBRATIONS

Robert Schulenburg[1], Sergej Anisimov[1], Herbert Michael Heise[1]

[1]South-Westphalia University of Applied Sciences, Iserlohn, Germany

For many decades now, NIR spectroscopy has been used for rapid and non-destructive quantitative multicomponent analyses of organic materials and especially of forages. A powerful measurement technique is diffuse reflection spectroscopy, which can be applied to powders, requiring little or even no sample preparation. Important aspects for routine work are multivariate calibrations with chemometric tools such as partial least squares, which require a population of samples that are representative for the forages to be analyzed. For reference analysis often laborious analytical methods are applied that can handle larger forage specimens for making sure to yield representative analytical results for samples also taken at random.

Whole plant rye chaff samples were dried and milled to powders with the major part of fibrous consistency of sub-millimeter size. From these powderous samples, diffuse reflection spectra were recorded by using a special ellipsoidal mirror-based accessory attached to a Bruker Vector FT-NIR spectrometer equipped with a Peltier-cooled InGaAs detector [1, 2]. Samples were repeatedly loaded within a special cup for presenting the powder against the semi-spherical lens of the accessory, leading to a five-fold spectral data set for each forage sample with an average relative spectral standard deviation of less than 1 % after baseline offset correction at 10500 cm-1. These five-fold spectral measurements of individual forage samples were averaged providing another calibration data set leading to a smaller cardinality of the calibration population, but expecting to be better representative for the studied samples. Besides the use of raw spectra, also spectral pretreatments such as multiplicative scatter correction (MSC) or spectral derivation based on Savitzky-Golay polynomial fitting were applied. For calibration optimization different numbers of PLS factors were obtained either using all spectra or mean spectra of the five-fold measurements. The consequences for optimal rank selection are described and discussed for several parameters such as hemi-cellulose, raw protein and others based also on different spectral ranges.

1. R. Marbach and H.M. Heise, Applied Optics 34 (1995) 610–621.
2. H.M. Heise and R. Schulenburg, Near-infrared spectroscopy for medical, food and forage applications, in: Molecular and Laser Spectroscopy: Advances and Applications, V.P. Gupta (ed.), Vol. III, Elsevier, Singapore, 2022, Chapter 6.

P01.51

DEVELOPMENT OF REFERENCE MATERIALS FOR SPECTROMETER STANDARDIZATION WITH FOCUS ON NIR-SPECTROMETRIC FORAGE MOISTURE ASSAYS USING DIFFUSE REFLECTION MEASUREMENTS

Robert Schulenburg[1], Christian Reichler[1], Herbert Michael Heise[1]

[1]South-Westphalia University of Applied Sciences, Iserlohn, Germany

For several decades, NIR spectroscopy has been established for the analysis of feed and food. Especially in the field of agricultural products, the determination of moisture content, constituents such as crude protein, fiber content and other parameters plays an important role using calibrations with NIR reflectance spectra. Here, chemometric aspects and the use of reference materials are fundamentally important, especially when it comes to quality assurance and especially the performance testing of NIR spectrometers. The main focus was on long-term stable reference materials with defined water content without decomposition of the organic material or evaporation of the water leading to changes in the spectrum of the reference materials.

Many of the development hurdles of the last 25 years, such as enormous spectral differences between previously suggested substitute materials as reference standards and forage samples, the volatility of moisture present in harvested material, and the decomposition of moist biomass by bacterial processes if complete sterility cannot be guaranteed, were either completely eliminated or greatly attenuated by an innovative combination of mixing water-binding silica gel and dry feed. Preliminary tests were carried out with precisely water-loaded silica gel in combination with whole-plant chaff preserved by drying. It was found that blends of water-loaded silica gel of type SGR50B (with maximum water holding capacity of about 40 %) and dried grass powder showed tremendous spectral similarity compared to wet grass with defined moisture. The silica content in the blended samples could be easily increased without causing any significant spectral deviations especially in the intervals of 5500 to 4500 cm^{-1} as well as of 7700 to 6000 cm^{-1} when compared to the spectra, e.g., of wet grass specimens showing significant water absorption bands. To verify the suitability of such mixtures as standard reference materials, as well as the possibility of setting defined moisture contents, standards were prepared and analyzed for their water concentration using a pre-developed PLS method based on reflectance spectra of whole-plant chaff with defined moisture content. It was found that the prediction results of SGR50B blends differed only minimally from the actual moisture values from gravimetry. In addition, the prepared reflectance standards could be sealed in an appropriate housing with a sapphire disk to achieve long-term stability.

P01.52

ANOVA-SIMULTANEOUS COMPONENT ANALYSIS (ASCA) TO CHARACTERISE EFFECT OF TEMPERATURE ON FT-NIR SPECTRAL DATA OF ROASTED WHEAT

Mia Schutte[1], Federico Marini[1,2], Stefan Hayward[1], Marena Manley[1]

[1]Stellenbosch University, Stellenbosch, South Africa, [2]University of Rome, Rome, Italy

The antioxidant properties of whole wheat can be enhanced by exposing the grains to dry thermal treatment, providing the required temperatures and moisture levels for non-enzymatic browning, which contributes to increased antioxidant activity, have been used. Near infrared (NIR) spectroscopy can be used to investigate the physicochemical changes without the need for lengthy and expensive chemical analysis techniques. Analysis of variance (ANOVA) simultaneous component analysis (ASCA) can be applied to indicate the statistical significance of the variation caused in multivariate data by different factors of an experimental design. ASCA was used to investigate the effect of roasting temperature on standard normal variate (SNV) preprocessed Fourier transform (FT) NIR spectra (1000-2500 nm) of whole wheat (four batches of each sample). A single factor multilevel experimental design consisted of 10 roasting temperatures, including an unroasted sample. Simultaneous component analysis indicated that roasting temperature had a significant effect (p0.05) on the wheat sample spectra with a trend from unroasted and low (108°C) to the highest temperature (232°C) in the direction of component (SC) 1. The high temperatures (170–232°C) were associated with changes in starch and amino acids while the low temperatures (108–150°C) were associated with changes in starch and moisture. SC2 explained the variation, mainly associated with moisture for the samples roasted at 232°C. An investigation into non-enzymatic browning of these samples, indicates that the free amino nitrogen (FAN) content decreases with increase in roasting temperature and formation of 5-hydroxymethyl furfural (HMF, indicator of the Maillard reaction) was observed at 195°C. This suggests that the reducing sugars originating from the starch and the amino acids have been involved in the occurrence of non-enzymatic browning, accounting for the changes in the starch and protein structures observed in the direction of SC1 and the loading line plot. ASCA applied to FT-NIR spectral data of whole wheat effectively illustrated the significant effect of roasting on moisture content as well as the starch and protein structures as roasting temperatures increase.

P01.53

ASSESSMENT OF THE FEASIBILITY AND RELEVANCE OF HANDHELD NEAR-INFRARED SPECTROSCOPY FOR DETERMINING DAIRY MANURE COMPOSITION ON FARM

Mohy-El-Din Sherif[1], Virginie Blanvillain[1]

[1]AB Vista, Marlborough, United Kingdom

As dairy producers strive to feed a growing global population, they must also ensure the sustainability of their operations in a highly competitive market. Characterising forage and grain nutrient profiles optimises feed formulation, while analysing dairy manure composition monitors nutrient utilisation efficiency. For example, faecal starch analysis is a common technique to estimate total tract starch digestibility (Fredin et al., 2014). Similarly, estimating Neutral Detergent Fibre (NDF) in manure can be used to evaluate fibre degradation efficiency.

Besides wet chemistry, Near-infrared Spectroscopy (NIRS) is commonly used to analyse manure composition in laboratories. However, this method does not remove the constraint of safely collecting and shipping excreta samples to a laboratory. Scanning fresh manure on farm appears to be an alternative to efficiently determine manure composition and adjust feed ration formulation in real time.

This study involved NIRS calibration development to predict Dry Matter (DM), Crude Protein (CP), NDF and starch in manure, by defining a standard sampling and scanning protocol. Using a handheld NIRS (HHNIR) device (MicroNIR Gen2, 900-1650 nm wavelength, 6.2nm resolution, Viavi Solutions), 200 fresh manure samples were scanned on farm, with composite samples collected in a container from at least 4 pats per cow group. After thorough mixing, borosilicate petri dishes (Duroplan) were placed on the samples and the spectrometer was placed on the petri dishes to collect 5 scans of each sample. Within 48 hours of collection, the samples were sent for wet chemistry analysis to an external laboratory.

The standard error (SE) of calibration were acceptable at 1.01% for DM, 0.60% for starch, 3.55% for NDF, and 1.85% for CP. An external validation was subsequently performed by collecting fresh dairy manure from herds at different stages of lactation and fed different types of forages and concentrates. Over 60 samples were collected in the United Kingdom in a 3-months period. The SE of prediction averaged 1.28% for DM, 2.30% for starch, 4.25% for NDF and 1.78% for CP.

The results allowed to verify the feasibility and applicability of using HHNIR to determine manure composition on farm. Dairy and beef manure samples are now being scanned in global locations to increase the diversity of the database, before further deploying the calibrations to bovine producers and provide real-time information for fast decision-making.

P01.54

HYPERSPECTRAL IMAGING COUPLED WITH SUPPORT VECTOR MACHINE FOR CLASSIFICATION OF ANTHRACNOSE INFECTION ON MANGO FRUIT

Ubonrat Siripatrawan[1], Yoshio Makino[2,3]

[1]Department of Food Technology, Faculty of Science, Chulalongkorn University, Bangkok, Thailand, [2]Department of Biological and Environmental Engineering, Graduate School of Agricultural and Life Science, The University of Tokyo, Tokyo, Japan, [3]Department of Life and Culture, Kagawa Junior College, Kagawa, Japan

Mango is one of the most important economic fruit in Thailand and exported worldwide. However, mango is susceptible to anthracnose disease causing quality loss and reduced market value. This research developed a rapid, nondestructive and chemical-free method based on hyperspectral imaging spectroscopy (HIS) coupled with chemometrics for early detection of anthracnose caused by Colletotrichum gloeosporioides on mango. Nam Dok Mai mango fruit (Mangifera indica L.) was wounded using a sterile syringe and inoculated with 10 mL of the conidial suspension of C. gloeosporioides (106 spores mL–1). Each fruit was analyzed after storage for 0, 2, 4, 6, 8, and 10 days (marked as 0d, 2d, 4d, 6d, 8d, and 10d) at 20 °C and 90 % RH. The HIS system covering the wavelength range of 400-1000 nm was used to acquire the full contiguous spectral and spatial data of the samples. The hypercube data of the region of interest obtained from one sample scanned comprised spatial data of 100*50 pixels and reflectance spectral of 121 distinct wavelengths. The reflectance spectra of each sample were mathematically pretreated using mean normalization to compensate the intensity variations from path length differences. The data were then analyzed using chemometrics including principal component analysis (PCA) and linear discriminant factor analysis (DFA).

From PCA, the 121 correlated wavelength variables were linearly transformed into a relatively small set of 3 uncorrelated variables (PCs) which represented as high as 98 % of the total variance of the data. The PCA score plot did not show clear clustering among 6 groups. Therefore, DFA was used to classify samples with different levels of anthracnose symptom as a result of storage time. From the DFA, the data were correctly classified into 6 groups. The HIS coupled with chemometrics was able to separate the healthy samples (0d) from the 2d samples, which was in the quiescence stage and showed no sign of anthracnose. The technique can detect the C. gloeosporiodes infection on mango fruit even before the onset of disease symptoms and can classify different levels of symptom severity.

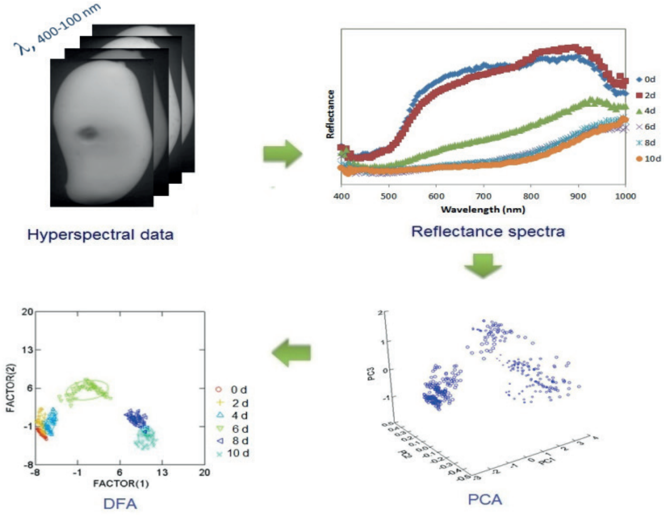


Figure 1. Detection of anthracnose on mango fruit using HIS and chemometrics

P01.55

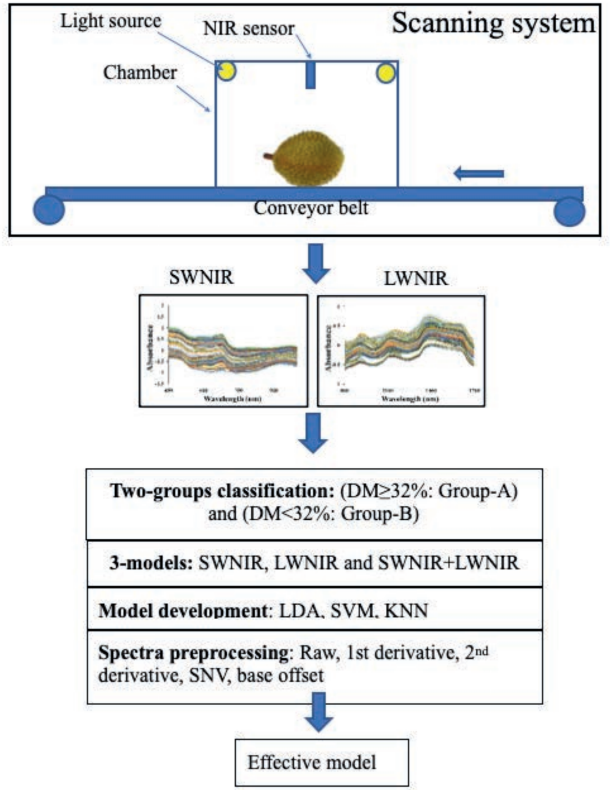
FEASIBILITY ONLINE CLASSIFICATION FOR DURIAN FRUIT MATURITY USING NIR SPECTROSCOPY

Panmanas Sirisomboon[1], Sirirak Ditcharoen[2], Jetsada Posom[2],
Chayuttapong Malai[2], Chirawan Saenphon[2]

[1]King Mongkut's Institute Of Technology Ladkrabang, Bangkok, Thailand, [2]Khon Kaen University, Khon Kaen, Thailand

Maturity of durian is very important for trading and indexed by dry matter (DM) level. The criterial for trading, DM of pulp of durian fruit must have more than or equal to 32%. At present, the DM of durian fruits were randomly checked using laboratory method, drying oven. However, it is required a long time and cannot confirm for all fruit. Non-destructive technique is requested to measure on intact fruit for the DM. This study aims to classify maturity of durian using near infrared spectroscopy. The durian fruit was scanned on the conveyor belt with velocity of 0.2 m/s using both NIR spectrometers: the visible and short wavelength NIR (VIS-SWNIR) ranging from 450 to 1000 nm and long wavelength NIR (LWNIR) ranging from 860 to 1750 nm. The class of durian divided into two group i.e. group A ($DM \geq 32\%$ meaning accepted) and group B ($DM < 32\%$ meaning rejected). The classification models were performed using three supervised machine learning algorithms that is linear discriminant analysis (LDA), support vector machine (SVM), and K-Nearest neighbours (KNN). The model created from raw, 1st derivative, 2nd derivative, standard normal variate (SNV) and baseline offset were compared. The 3-model developed using VIS-SWNIR, LWNIR and combined VIS-SWNIR+LWNIR were compared. Therefore, 45 treatments (5-spectra preprocessing x 3-models x 3-algorithms) were studied. The maximum accuracy was LWNIR-baseline spectra-KNN algorithm, provided overall accuracy of 98.61%. However, the result shows that overall accuracy of VIS-SWNIR and LWNIR was the similar. While the combination between VIS-SWNIR+LWNIR do not increase accuracy. Author suggest the VIS-SWNIR was recommended because low cost.

Graphical Abstract



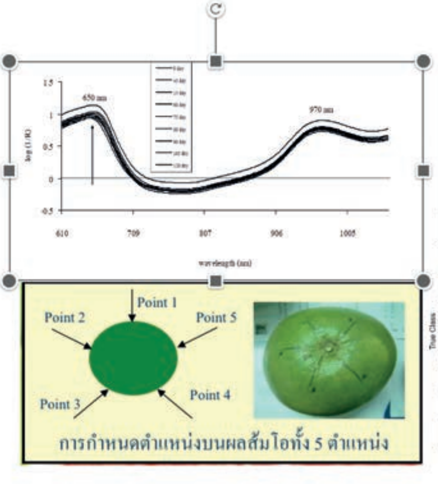
P01.56

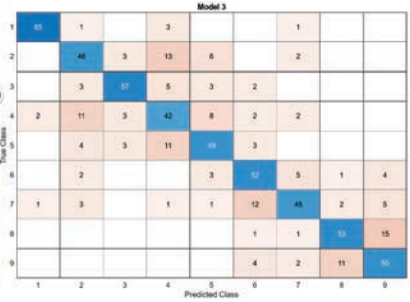
CLASSIFICATION OF STORAGE DURATIONS OF INTACT POMELO BY NARROW NEURAL NETWORK

Panmanas Sirisomboon[1], Kingdow Chanachot[1], Ravipat Lapcharoensuk[1]

[1]King Mongkut's Institute Of Technology Ladkrabang, Bangkok, Thailand

Artificial Neural Network (narrow neural network) algorithm was used for classification of 9 storage period (0, 15, 30, 45, 60, 75, 90, 105 and 120 days after harvest) of pomelo (Citrus Maxima Burm.f. cv Khao Nam Pueng) which was 4 months in total in a storage room of 10 °C and 70% relative humidity using VISNIR spectra of the intact pomelo obtained by FQA NIR Gun (Fantec, Japan) in the wavelength range of 600-1100 nm with 2 nm resolution and Teflon is reference material. The pomelo was harvested on a commercial harvesting date of 210 days after flower blooming. The scanning was done on 2 positions 144° apart on each pomelo (point1 and point3). There were 25, 25, 25, 25, 25, 20, 25, 25 and 20 fruits which was 86 fruits in total (100, 100, 100, 100, 100, 95, 100, 100, 95 spectra) in the different storage durations, respectively. The data were separated into training set and test set of 70:30 which was 624:266 spectra. The result shows the classification accuracy of training set was 73.6 % and test set was 70.7%




Model 3

True Class \ Predicted Class	1	2	3	4	5	6	7	8	9
1	65	1		3			1		
2		46	3	13	6		2		
3		3	57	5	3	2			
4	2	11	3	42	8	2	2		
5		4	3	11	49	3			
6		2			3	52	5	1	4
7	1	3		1	1	12	45	2	5
8						1	1	53	15
9						4	2	11	50

P01.57

APPLICATIONS OF POINT NEAR-INFRARED SPECTROSCOPY AND HYPERSPECTRAL IMAGING FOR REAL-TIME QUALITY MONITORING OF HYDROPONICALLY GROWN LEAFY GREENS

Jan Skvaril[1], Reyhaneh Gorji[1], Monica Odlare[1]

[1]Future Energy Center, Schools of Business, Society and Engineering, Mälardalen University, Västerås, Sweden

The rapidly growing population, especially in urban areas, challenges the agricultural sector to effectively improve local food availability and achieve circular and sustainable production. The Indoor Urban Vertical Farming (IUVF) concept, which involves growing plants hydroponically in multiple layers in a controlled environment, offers many opportunities to meet the demands compared to conventional agricultural production. The IUVF concept aims to grow high-quality crops with greater water and energy use efficiency and help reduce CO2 emissions and environmental footprint by producing food hyper-locally. To achieve the ambitious goals and ensure the sustainability of the concept, IUVF operations must be further optimized by integrating automation and control solutions and strategies for indoor precision agriculture. Important components of the solutions include sensor systems that enable non-destructive monitoring of plant growth and product quality. Near-infrared (NIR) spectroscopy combined with a chemometric approach offers excellent potential for the development of such sensors. The objective of the present work is to evaluate the potential of monitoring hydroponically grown leafy vegetables using point and imaging NIR sensors.
For the evaluation, NIR sensors for a contact measurement and a hyperspectral imaging camera for close-range remote measurements were employed together with qualitative and quantitative chemometric approaches. Algorithms of different complexities were employed for image and spectral pre-processing and model development. The models were validated using standard chemometric validation procedures. Preliminary results are promising and demonstrate the great potential of NIR spectroscopy for non-destructive monitoring of crop growth and product quality for the IUVF setting.

Acknowledgment: This work is supported by Richertska stiftelsen/SWECO under project No. 2021-00742 Aqua2Farm - Boosting sustainable urban farming by near-infrared spectroscopy with aquaphotomics.

P01.58

FUSION OF NIR AND RAMAN SPECTRA FOR THE CLASSIFICATION OF PARMIGIANO REGGIANO MOUNTAIN PRODUCT

Lorenzo Strani[1], Caterina Durante[1], Juan Antonio Fernandez Pierna[2], Sara Michielini[3], Valentina Pizzamiglio[3], Marina Cocchi[1]

[1]Department of Chemical and Geological Sciences, University Of Modena And Reggio Emilia, Modena, Italy, [2]Walloon Agricultural Research Centre (CRA-W), Quality of Agricultural Products Department, Gembloux, Belgium, [3]Consorzio del Formaggio Parmigiano Reggiano, Reggio Emilia, Italy

In order to support food producers in mountain areas, where the production conditions are challenging, European Community has defined a quality denomination called "Mountain Product" in one of its regulations [1]. Nonetheless, this denomination is still not enough well-known among consumers, even if it is present in labels of very popular Italian food products, such as Parmigiano Reggiano cheese, a PDO (Protected Designation of Origin) product. Therefore, food producers want to both promote their products and safeguard their authenticity.
In this context, the objective of the present work is to assess that the product "Prodotto di Montagna - Progetto Territorio Consorzio" ("Quality Project - Mountain Product", QP-MP) represents a quality denomination for Parmigiano Reggiano PDO (Protected Designation of Origin) cheese, which in addition to the requisites of mountain denomination EU label must comply with additional rules, as established by the producer consortium [2].
To this aim, NIR and Raman spectroscopies were employed to analyse both MP-CQ and conventional-PDO Parmigiano Reggiano samples collected from certified producers. A data fusion approach was used to exploit the different features of the two techniques, and several chemometric strategies were tested, including class modelling/discrimination methods for the classification of QP-MP samples and unconventional variable selection techniques, such as Response-Oriented Sequential Alternation (ROSA) [3]. The present study is part of a research project, i.e. MOUNTAIN-ID (https://www.mountainid.unimore.it), which aims at developing analytical methodologies to conjugate identity, sustainability, and value of Mountain Products. The ambitious objective of this part of the project is to demonstrate that spectroscopic techniques can be considered for a fast and cost-effective assessment of Parmigiano Reggiano QP-MP authenticity.

[1] regulation (EU) No 1151/2012
[2] The biodiversity - Parmigiano Reggiano, (n.d.). https://www.parmigianoreggiano.com/product-biodiversity (accessed April 11, 2023).
[3] Liland, K. H., Næs, T., & Indahl, U. G. (2016). ROSA—a fast extension of partial least squares regression for multiblock data analysis. Journal of Chemometrics, 30(11), 651-662.

P01.59

SOURDOUGH FT-NIR SPECTRA: EFFECT OF DOUGH YIELD (DY) AND FLOUR TYPE USING ANOVA-SIMULTANEOUS COMPONENT ANALYSIS (ASCA)

Mari Van Wyk[1], Federico Marini[1,2], Marena Manley[1]

[1]Stellenbosch University, Stellenbosch, South Africa, [2]University of Rome "La Sapienza", Rome, Italy

Sourdough consumption has increased dramatically due to an increase in consumer demand for clean-label and artisan products. This study evaluated the impact of stoneground (coarse and sifted) and roller-milled flour, obtained from the same wheat, on the FT- NIR spectra of Type I (dough yield 160) and Type II (dough yield 220) sourdough over a period of 28 days. Type II sourdough was produced using Type I as inoculum. Both types of sourdoughs were refreshed every fourth day for 28 days with the three types of flour and a control (commercial sifted stoneground flour) and FT-NIR (1000–2500 nm) spectra collected. ASCA was performed for each sourdough type to investigate the effect of type of flour and time. A full factorial multilevel experimental design consisted of flour (four levels) and time (eight levels). For the Type I dataset, both factors and the interaction were shown to have a significant effect (p0.05) on the spectra. SCA demonstrated a separation between roller-milled and stoneground flour in the direction of SC1. Additionally, plotting the scores of the SCA model of the sum of the main effect of time and the interaction as a function of time showed a gradual increase from day 10 until reaching a plateau. In contrast, SCA of the effect of flour type showed a clear separation of the three types and the control. Plotting the scores over time resulted in scores increasing and decreasing in a repeating pattern over time without following a smooth monotonic trend. For the Type II dataset, flour type and the interaction term were statistically significant (p0.05), while the time factor was marginally non-significant, with a p-value slightly greater than 0.05. The SC1 and SC2 results followed a similar trend as for the Type I dataset but were less pronounced. To further explore the effect of time, score plots were generated with projected residuals on SC1 to SC3 as a function of time along with corresponding loadings and confidence intervals (based on 1000 bootstrap runs). A gradual increase was seen from day 10 until reaching a plateau from day 16. SC3 showed a more monotonic trend. These observations highlight the importance of multivariate analysis in identifying subtle variations in complex NIR spectral datasets and elucidating underlying patterns and trends. The findings provided valuable insights into the impact of different types of flour on sourdough characteristics and can be used to guide the selection of flours for sourdough maintenance.

P01.60

QUALITY AND SAFETY ASSESSMENT OF IN-SHELL AND SHELLED ALMONDS USING AN NIR PORTABLE DEVICE

Miguel Vega-Castellote[1], María-Teresa Sanchez[1], Jose-Antonio Entrenas[2], Irina Torres-Rodriguez[2], Dolores Perez-Marin[2]

[1]Department of Bromatology and Food Technology, University of Cordoba, Rabanales Campus, Cordoba, Spain, [2]Department of Animal Production University of Cordoba, Rabanales Campus, Cordoba, Spain

The determination of the nutritional profile of almonds is of paramount importance for the processing industry in order to satisfy consumer demand for healthy foods. In this sense, a key quality index is the fatty acid profile given the high content of unsaturated lipids in these fruits and their potential benefits for human health, and that it is also related with the shelf-life of the product. Additionally, the almond processing industry seeks to eradicate the presence of bitter almonds from the sweet almond batches given their typical unpleasant taste and the toxicity of the compounds present in this type of kernels. Thus, the aim of this study was to determine the fatty acid profile in intact in-shell and shelled almonds and to discriminate sweet from bitter in-shell and shelled almond batches using a new generation near infrared spectroscopy (NIR) sensors, suitable for the in situ analysis of almonds. The partial least squares (PLS) regression method was used for the prediction of the fatty acid profile, and the classification of the sweet and bitter almonds was carried out by means of the partial least squares discriminant analysis (PLS-DA) using in both cases different combinations of signal pre-treatments. Good prediction results were obtained for the main fatty acids (oleic and linoleic acids) in almonds, showing those models developed using almonds without shell a greater prediction accuracy when compared to the ones using in-shell almonds, as it was expected. Furthermore, excellent classification performance was obtained both for the in-shell and shelled almonds when using the PLS-DA method to discriminate between sweet and bitter almonds, and to detect sweet almond batches adulterated with bitter almond kernels.

P01.61

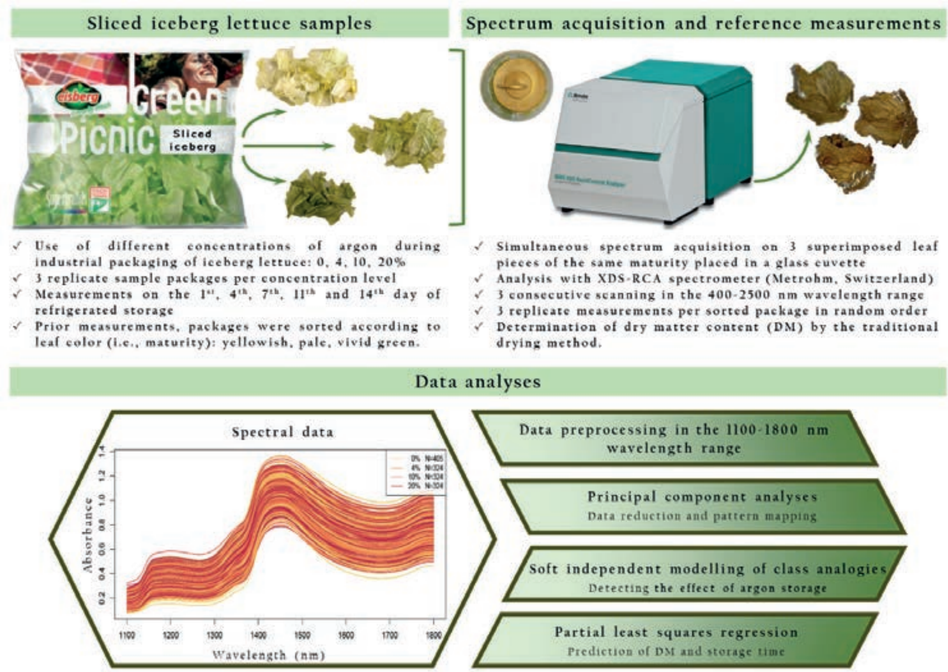
PINPOINTING THE EFFECT OF ARGON PACKAGING AND STORAGE ON THE NEAR INFRARED SPECTRA OF SLICED ICEBERG LETTUCE

Flora Vitalis[1], Monika Gob[2], Lueji Regatieri Santos[1],
Zsuzsanna Horvath-Mezofi[2], Geza Hitka[2], Jelena Muncan[3], Zoltan Kovacs[1]

[1]Department of Food Measurement and Process Control, Institute of Food Science and Technology, Hungarian University of Agriculture and Life Sciences, Budapest, Hungary, [2]Department of Postharvest, Commerce, Supply Chain and Sensory Science, Institute of Food Science and Technology, Hungarian University of Agriculture and Life Sciences, Budapest, Hungary, [3]Aquaphotomics Research Department, Graduate School of Agricultural Science, Kobe University, Kobe, Japan

Lettuce is one of the highly sought-after leafy vegetables, and widely produced crops. It is a key raw material in the minimally processed food market. In commercial ready-to-eat salads, iceberg lettuce is one of the most common ingredients, due to its crunchy texture. Leaves show varying composition and fragile tissue structure depending on environmental exposure (e.g., growth location) and postharvest technologies, influencing the metabolic activity. Lettuce leaves are characterised by shelf life of about 7 days, even under controlled storage conditions. High demand exacerbates these challenges, making it essential to develop post-harvest technologies and non-destructive quality control. This research investigated the feasibility of argon gas storage as a possible alternative to conventional modified atmosphere storage (MAP) of iceberg lettuce, also to non-invasively monitor the effects of storage condition. On the 1st, 4th, 7th, 11th and 14th day of cold storage, three replicate lettuce packages of different argon concentration (0, 4, 10, 20% V/V) were sorted according to leaf maturity (yellowish, pale, vivid green). The reflectance spectra were recorded simultaneously on 3 superimposed leaf pieces of the same maturity in a glass cuvette with the RCA module of XDS spectrometer (Metrohm, Switzerland). This was repeated three times per package and leaf maturity. Subsequently, the dry matter content (DM) of the leaves was determined by the traditional drying method. The spectra were evaluated in the 1100-1800 nm range. Principal

component analysis (PCA) was performed for preliminary pattern recognition; soft independent modelling of class analogies (SIMCA) to detect the effect of argon storage on leaves of different maturity; partial least squares regression (PLSR) to predict DM and storage time. The preliminary evaluations testified high spectral variance, depending on storage condition and leaf maturity. SIMCA classification accuracies varied between 0.54–0.63 and 0.44–0.53 during validation when discriminating argon concentration and storage time. When PLSR-predicting the storage-dependent change in dry matter, the coefficients of determination and root mean square errors were between 0.66–0.86 and 0.57–0.88% m/m during validation, respectively. As a synthesis of all modelling results, 20 wavelengths that best described storage processes were identified and considered as a fingerprint-like biomarker for depicting the state of sliced iceberg lettuce.



- ✓ Use of different concentrations of argon during industrial packaging of iceberg lettuce: 0, 4, 10, 20%
- ✓ 3 replicate sample packages per concentration level
- ✓ Measurements on the 1st, 4th, 7th, 11th and 14th day of refrigerated storage
- ✓ Prior measurements, packages were sorted according to leaf color (i.e., maturity): yellowish, pale, vivid green.

- ✓ Simultaneous spectrum acquisition on 3 superimposed leaf pieces of the same maturity placed in a glass cuvette
- ✓ Analysis with XDS-RCA spectrometer (Metrohm, Switzerland)
- ✓ 3 consecutive scanning in the 400-2500 nm wavelength range
- ✓ 3 replicate measurements per sorted package in random order
- ✓ Determination of dry matter content (DM) by the traditional drying method.

P01.62

QUALITY CONTROL ON BIOCHAR ON FEEDSTOCK AND PYROLYSIS CONDITION USING NIRS

Richa Neupane[1], Nicholas Anderson[1], **Kerry Walsh**[1]

[1]CQUniversity, North Rockhampton, Australia

Pyrolysis involves thermal treatment of organic matter under anoxic conditions. The number of functional groups in the product is impacted by the feedstock used, e.g., degree of lignification, and negatively impacted by pyrolysis severity, e.g., temperature. Biochar can thus be 'designed' for specific applications. NIRS has potential for use in characterisation /quality control of product.

Spectra were collected of biochar produced from 3 feedstocks varying in lignification (macadamia nut shell, eucalypt wood chip and hemp straw) and pyrolyzed at 3 temps (350, 450 and 600 oC) at each of 3 durations (5, 10 and 20 mins), with three replicate runs for each of the 27 treatments. Samples were ground before spectra (to 2500 nm) were collected using a Thermo Antares FTNIR and a FOSS 6500, operated in diffuse reflectance mode and using a spinning cup accessory in both cases.

A PCA was undertaken using the R software package (version 4.1.1), based on raw absorbance spectra. The first 2 PCs explained 99% of spectral variance. A support vector machine (discriminant) model was then developed based on absorbance spectra. Two replicates of each of the 27 treatments were used in model modelling and the third replicates used in class prediction.

As expected, peaks associated with water and functional groups of cellulose, hemicellulose and lignin with decreased with increasing pyrolysis temperature. Biochars were well discriminated by temperature but not time of pyrolysis, with clear separation of macadamia nut shell from other feedstocks, but some misclassification of hemp straw as eucalypt. Results will be contrast to those based on FTIR-ATR spectra.

The SVM model for the scanning grating instrument performed slightly better than that for the fourier transform instrument in classification of feedstock and process temperature (Table 1). Discrimination on process time was very poor (data not shown).

Table 1. Prediction of feedstock and pyrolysis time and temperature (oC) using a SVM model for each of a scanning grating and a Fourier transform instrument.

Feedstock prediction	Eucalyptus		Hemp		Macadamia	
	FTNIR	Grating	FTNIR	Grating	FTNIR	Grating
Eucalyptus	9	8	6	2	6	0
Hemp	0	0	3	6	0	0
Macadamia	0	1	0	1	3	9

Temperature prediction	350		450		600	
350	8	8	0	0	0	0
450	1	1	9	9	1	0

600	0	0	0	0	8	9

Results are consistent with completion of reactions within 5 minutes, i.e., that fluidised bed operation was efficient. Discrimination between feedstock (differing in lignification) and process temperature was achieved, supporting the concept of use of NIRS in quality control of ‘designer’ biochars.

P01.63

EARLY DETECTION OF MYCOTOXIGENIC FUNGI ON MAIZE USING SPECTRAL IMAGING: CONSIDERATIONS

Paul J Williams[1], Kim Mannix[1], Lindy Rose[2]

[1]Department of Food Science, Stellenbosch University, Stellenbosch, South Africa, [2]Department of Plant Pathology, Stellenbosch University, Stellenbosch, South Africa

Maize is a critical crop for both human and animal nutrition globally, but mycotoxigenic fungi can cause significant economic and yield losses, as well as pose a substantial food safety risk. Spectral imaging has shown promise in detecting mycotoxigenic fungi in maize, but most detection studies only consider spore inoculum as the infection source, overlooking the potential for infection via mycelia. This study aimed to determine if there is a spectral difference between spore and mycelial inoculum and to investigate how inoculum concentration affects the ability to discriminate between infected and control maize.

Maize kernels were artificially inoculated with Fusarium verticillioides: either consisting of mycelial or spore suspension, while kernels inoculated with distilled water served as the control. The kernels were imaged daily for four days post-inoculation, and multiclass and binary classification models were developed using five different classification algorithms. Multiclass classification evaluated control, mycelia, and spores, while binary classification evaluated control and infected, merging mycelia and spores into one infection class.

PCA-LDA, PLS-DA, and SVM models generally performed the best, with binary classification performing better than multiclass classification. Classification accuracy ranged from 73.3% to 96.7% from day 1 to day 4 for binary classification. A confirmation study on day 1 showed improved classification accuracy for both multiclass (74.7%) and binary (86.7%) classification with the addition of more samples, although a high degree of variation between cross-validation folds was noted.

Varying concentrations of mycelial inoculum were compared to spore and control classes, with high and mid concentrations producing comparable results, while low concentrations showed a decrease in classification accuracy. Large variation was noted in the cross-validation results at all concentrations.

In conclusion, this study suggests that there is no difference between inoculum sources after day 3 post-inoculation; however, the ability to distinguish between inoculum sources improves with the addition of more samples during the initial period of infection (day 1), albeit with increased variation. The study highlights some critical considerations for early detection of mycotoxigenic fungi in maize using spectral imaging, including the potential for mycelial inoculum and the importance of considering inoculum concentration.

P01.64

ASSESSMENT OF THE OPTICAL PROPERTIES WITH CELL WALL POLYSACCHARIDES OF ‘KORLA’ PEAR FLESH DURING ALTERNARIA ALTERNATA-INDUCED DISEASE DEVELOPMENT

Sicong You[1], Weijie Lan[1], Kang Tu[1], Leiqing Pan[1]

[1]*Nanjing Agricultural University, Nanjing, China*

‘Korla’ pear is particularly susceptible to pathogen infection during harvest, transportation and storage periods. The cell wall of pears is a dynamic matrix and can be regarded as an important barrier to pathogen infection. The fruit cell wall contains a large variability of polysaccharide fractions, especially pectin, cellulose and hemicellulose. In addition, cell wall degrading enzymes, such as polygalacturonase, pectinesterase, ß-galactosidase, and cellulase can catalyze the depolymerization and solubilization of cell wall polysaccharides. Moreover, pathogen infection would produce a lot of extracellular enzymes which could disassemble the cell wall structure, allowing fungi to spread through the fruit tissue. Therefore, it will be of great importance to provide deep insights into the changes of cell wall polysaccharides in infected ‘Korla’ pear during postharvest. Light interaction with tissues are characterized by two optical parameters: absorption coefficient (μa) and the reduced scattering coefficient (μs’). It is generally accepted that the two optical properties are related to the specific chemical compounds and physical structure information about fruit tissues, and they change with the tissue’s physiological and biochemical properties. Hence, the optical properties measurement could provide the opportunity to explore the relationship between the optical parameters of fruit and their cell structural and chemical changes.

This report described the relationship of optical properties with cell wall polysaccharides of ‘Korla’ pear flesh during Alternaria alternata-induced disease development (Figure. 1). The A. alternata inoculation promoted the degradation of covalent-soluble pectin (CSP), cellulose and hemicellulose, but facilitated the water-soluble pectin (WSP) and ionic-soluble pectin (ISP) content accumulation in pear flesh. Two absorption peaks at 675 nm and 980 nm were found in the spectral range of 600–1000 nm. The μa values of infected pear tissues decreased during storage time, while the μs’ increased obviously. Correlation analysis and path-coefficient analysis demonstrated that ISP was the main reason responsible for the change of μs’ in A. alternata infected ‘Korla’ pears, while the μa were associated with firmness and CSP. The study demonstrated the potential of using optical properties to provide specific insights into the cell wall polysaccharides of ‘Korla’ pear flesh while the process of disease infection.

Keywords: ‘Korla’ pear, Cell wall polysaccharide, Optical properties

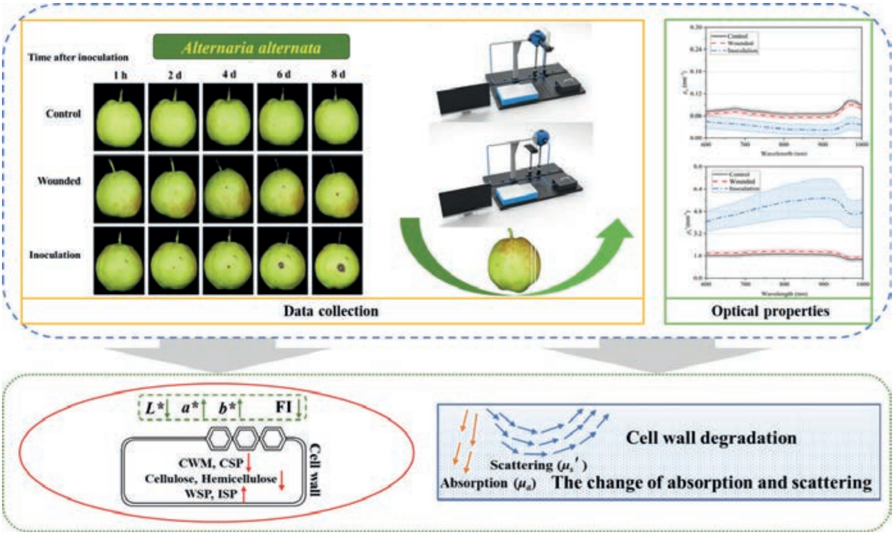

Alternaria alternata
Time after inoculation
1 h
2 d
4 d
6 d
8 d
Control
Wounded
Inoculation
Data collection
Control
Wounded
Inoculation
Wavelength (nm)
Optical properties
L*
a*
b*
FI
CWM, CSP
Cellulose, Hemicellulose
WSP, ISP
Cell wall
Cell wall degradation
Scattering (μ_s')
Absorption (μ_a)
The change of absorption and scattering

Figure 1

P01.65

ANALYSIS OF FOREIGN MATTER MISIDENTIFICATION CASES FOUND IN ALCOHOLIC BEVERAGES USING FT-IR

Yeon Cheol Yu[1], Jong Hyun Choi[1], Kyung Wook Kim[1], Hei-Seung Kang[1], Hyungsoo Kim[1], Jae Eun Moon[1]

[1]Ministry of Food and Drug Safety of South Korea, [187], Osongsaengmyeong [2]-ro, Osong-eup, Heungdeok-gu, Cheongju-si, Chungcheongbuk-do, South Korea

Foreign matters are recognized as a measure of food safety and hygiene. In South Korea, strong penalties such as production suspension and sales prohibition are imposed depending on the degree of potential harm to consumers when foreign matters are found in food. Therefore, it is important to quickly and accurately identify the type of reported foreign matters. With the globalization of online markets, the occurrence of foreign matters in alcoholic beverages has increased as it has become easier to consume alcoholic beverages from various countries. However, there are often cases where crystals generated during food storage, such as glass powder, are mistakenly reported as foreign matters. The purpose of this study is to compare and analyze crystals and glass powder in alcoholic beverages (beer, whiskey, white wine, red wine) to quickly and accurately identify them. In this study, crystals in alcoholic beverages were measured morphologically using stereoscopic microscopes, optical microscopes, and scanning electron microscope(SEM). Organic functional groups were detected using Fourier transform infrared spectroscopy(FT-IR), and inorganic components were analyzed using X-ray fluorescence(XRF). The results of the analysis showed that the crystals found in beer and whiskey were calcium oxalate, which had a hexagonal shape morphologically and did not dissolve well in water, floating in alcoholic beverages like glass powder. FT-IR analysis also showed peaks corresponding to calcium oxalate, and the inorganic component was 100% calcium. In the case of white wine and red wine, tartaric acid was found as crystals, but there were no specific morphological characteristics. However, FT-IR analysis showed characteristic peaks of tartaric acid, and the inorganic component was 100% potassium. Glass powder was not analyzable by FT-IR, and the main inorganic component was silicon(Si). Therefore, the rapid and accurate identification of crystals and glass powder in alcoholic beverages was possible through the peaks of Fourier transform infrared spectroscopy(FT-IR), and further research on reducing crystallization in alcoholic beverages is considered necessary.

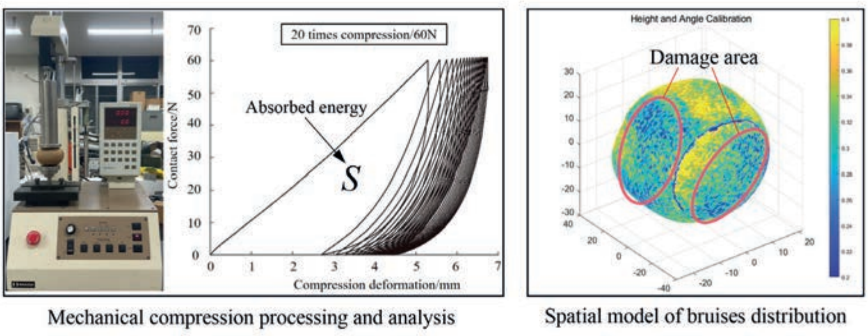
P01.66

MULTIPLE DAMAGE MECHANISM AND DAMAGE PREDICTION MODEL OF KIWIFRUIT BASED ON NEAR INFRARED HYPERSPECTRAL

Jiacheng Yuan[1,2], Te Ma[2], Xingyu Wan[1], Bin Li[2], Leshang Bai[2], Qingxi Liao[1], Satoru Tsuchikawa[2]

[1]Huazhong Agricultural University, Wuhan, China, [2]Nagoya University, Nagoya, Japan

The harvesting, packaging and transportation of kiwifruit result in a significant rate of loss due to contact damage. During these processes, kiwifruit is frequently subject to multiple mechanical contacts, which can cause microscopic damage to the internal tissue, especially when contact is repeated. Therefore, clarifying the mechanism of multiple damage and identifying the microscopic damage within kiwifruit, particularly tissue and cell micro-damage resulting from multiple mechanical contact, poses a major challenge for the kiwifruit production industry. This study uses near-infrared (NIR) hyperspectral imaging (HSI) technology and High precision laser scanner to construct a spatial model of bruises distribution in kiwifruit tissue, and the change of bruises area in kiwifruit tissue under multiple compression was analyzed, which clarified the changes in tissue under multiple contact damage. Combined with the changes in the spatial distribution of buries in kiwifruit, a viscoelastic model of kiwifruit and the evolution equation of compression damage was established. Additionally, the accumulation method of compression damage was clarified. The results indicated that near-infrared (NIR) hyperspectral imaging (HSI) could accurately identify the buries area of kiwifruit under mechanical compression. When the mechanical compression deformation is less than 2.34%~3.44% of the height in the compression direction, the damage to kiwifruit can be ignored. The frequency and force of mechanical compression have a significant impact on the damage of kiwifruit. Furthermore, the proposed damage accumulation method is consistent with the changes in buries area of kiwifruit under multiple mechanical compression. This study can serve as a basis for exploring the damage mechanism and predicting the damage of kiwifruit.



Mechanical compression processing and analysis

Spatial model of bruises distribution

Figure 1. Mechanical compression and near-infrared hyperspectral analysis of kiwifruit

P01.68

PRELIMINARY EVALUATION OF NEAR INFRARED SPECTROSCOPY TO PREDICT KEY COMPOSITIONAL PARAMETERS OF IRISH RAW MILK INDUSTRY SAMPLES OVER A FULL SEASON

Kexin Zhang[1], Jeremiah Sheehan[1], Colm O'Donnell[2], Norah O'Shea[1]

[1]Food Chemistry and Technology Department, Teagasc Food Research Centre, Moorepark, Co. Cork, Ireland, [2]UCD School of Biosystems and Food Engineering, University College Dublin, Belfield, Dublin, Ireland

The commercial value of milk has increased steadily in the past decades due to a growing consumer demand. The compositional content of raw milk is one of the most critical parameters used as part of the decision-making process regarding the segregation and processability of milk into cheese, butter, powder and cream production. For instance, the compositional content (e.g. total solids, protein, fat, and calcium ions) of raw milk is an influential factor in milk coagulation, an important process step part of the cheese-making process, whereby liquid milk is converted into a solid mass with desired viscoelastic properties. The rheological characteristics of raw milk during coagulation directly contribute to the final quality and yield of cheese. In Ireland, the compositional parameters in milk vary depending on seasonal changes and stages of lactation. To reduce the impacts of these changes, there is a requirement to develop an at-line rapid methodology for monitoring compositional parameters in raw milk prior to processing. The rapid methodology will allow for more efficient decision-making of milk segregation and processability of incoming milk.

This study investigated the feasibility of NIR spectroscopy combined with chemometrics to predict the key compositional parameters (e.g. total solids, total protein, fat, lactose and calcium ions content) in raw milk. The milk samples (n=555) used in this study were collected weekly from 8 Irish dairy processors' sites over a full season (from April 2022 to April 2023). Reference chemical analysis of key milk compositional properties and spectral data acquisition over the wavelength range of 800-2500 nm were carried out. The samples were divided into calibration (70%) and validation (30%) sets using Kennard-Stone algorithm. NIR spectroscopy was evaluated as an at-line methodology to rapidly identify the compositional parameters of raw milk over the period studied. Principal component analysis (PCA) and partial least analysis regression (PLSR) were used to establish the prediction models for milk coagulation properties. The performance of the preliminary prediction models were evaluated using coefficient of determination for cross validation (R^2_{cv}), root mean square error (RMSE), ratio of standard error of prediction to sample standard deviation (RPD) and the range error ratio (RER). The NIR prediction was acceptable for fat ($R^2_{cv} \geq 0.91$, RMSEcv$\leq$ 0.11, $RER_{cv} \geq 14.53$, RPDcv$\geq$3.45), total protein ($R^2_{cv} \geq 0.81$, $RMSE_{cv} \leq 0.11$ RERcv$\geq$9.95, $RPD_{cv} \geq 2.31$), and limited for total solid, lactose and Ca^{2+} ($R^2_{cv} \leq 0.59$, $RMSE_{cv} \geq 0.10$, RERcv$\leq$ 7.17, $RPD_{cv} \leq 1.54$). This study demonstrated the feasibility of using NIR as a rapid and reliable analytical method for the key compositional parameters in Irish raw milk industry samples over a full season.

P01.69

DIFFERENT WAYS TO ASSESS THE VITREOUSNESS OF DURUM WHEAT KERNELS USING NIR SPECTROSCOPY

Juan Antonio Fernández Pierna[1], Corentin Demoitié[1], Pierre Yves Werrie[1], Philippe Vermeulen[1], Sébastien Gofflot[1], Bruno Godin[1], Vincent Baeten[1]

[1]Walloon Agricultural Research Centre, Gembloux, Belgium

Kernels vitreousness is a major factor in assessing the technological quality of durum wheat intended for the food industry due to its impact on the yield of semolina. The standard method used to grade a batch consists of a visual inspection of sliced kernels which tends to be tedious. In order to automate the analysis, different ways to assess vitreousness using various instruments based on NIR spectroscopy and a wide range of samples have been explored. Hyperspectral imaging (HSI) was previously proved to be a promising alternative to the conventional technique [1,2]. Therefore, the performances of two NIR HSI systems (FX17 and SWIR XEVA, SPECIM, Finland) covering a combined spectral range of 900 to 2400 nm were studied.

In a first approach, vitreousness was assessed at kernel-level by measuring groups of 50 separated kernels, similar to the principle of the reference method. Individual kernel predictions provided good classification results but this process was still deemed time-consuming. Measurements were conducted in a second phase on bulk samples displayed in a single layer. While this approach was not as precise as the first one, the predictions results still showed a good correlation with the reference values and allowed for a faster analysis.

Afterwards, vitreousness prediction using conventional NIR benchtop spectrometer (XDS, FOSS, Denmark) was considered. If applicable, this parameter could be added to the others already predicted through this method such as protein content and humidity.

Finally, a sorting machine (QSorter EXPLORER 2.0, QualySense AG, Switzerland) based on NIR inspection (900-1700 nm) was tested to improve the quality of a batch by removing kernels with the undesirable trait. This system allows for the measurement of individual kernels and their classification into two distinct fractions. To conclude, this study showed the potential of NIR spectroscopy to efficiently grade and possibly sort a batch of durum wheat kernels.

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M. Shahin and S. Symons, "Detection of hard vitreous and starchy kernels in amber durum wheat samples using hyperspectral imaging (GRL Number M306)", NIR news 19(5), 16–18 (2008)

Acknowledgements
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P01.70

PHYTATE-BOUND PHOSPHORUS AND NIR SPECTRA OF WHEAT GRAINS

Roger Meder[1], Graeme Batten[2], Lindsay Campbell[2], Robert Caldwell[2], Richard Trethowan[2]

[1]Meder Consulting, Bracken Ridge, Australia, [2]University of Sydney, Sydney, Australia

Near infrared (NIR) spectroscopy has been used to determine the concentrations of total phosphorus (TP) and phytic acid in cereal grains, cereal-based foods and animal feeds. In current cereal grains, phytic acid is present in the form of myo-inositol hexa-phosphate with ionically held mineral cations (hence phytate). Over 50% of the TP is found as phytate-bound-P (PP) and this possibly influences NIR prediction models.

The NIR spectra of whole wheat grains were collected from genotypes generated by crossing two parents: IRS 812.9 (grain PP concentration of 0.228%) and IDO 637 (grain PP concentration of 0.121% due to the lpa-1 gene). The TP in each parent line was 0.35% P. Spectra were recorded using a Bruker MPA operating in diffuse reflectance mode between 12493 and 3599 cm^{-1} at a resolution of 8 cm^{-1}. Second derivative (2der) spectra were calculated using a Savitzky-Golay transform (window size 19 and 2nd order polynomial fit).

For this study 227 progeny were sorted according to their PP/TP ratio and 15 samples with a high PP/TP and 15 samples with a low PP/TP ratio were selected. The average spectrum of the high PP/TP and the average spectrum of the low PP/TP samples were calculated. Figure 1 shows the 2der difference spectrum between the spectra of the high and low samples.

Peaks in the 2der difference spectrum were assigned according to Workman and Weyer (2008). It was apparent that spectra of samples with the lpa-1 gene from IDO 637 were different primarily due to P-OH (5245 cm^{-1}) and O-H (7124, 5311 cm^{-1}) bonds: with weaker changes due to C-H, CHO, and N-H bonds. These changes reveal the action of the lpa-1 gene that reduces the synthesis of phytic acid from glucose-6-phosphate at one or more biochemical steps (Raboy 2009).

NIR regression calibrations for TP in cereals are likely to rely, at least partly, on the positive inter-correlation between TP and PP.

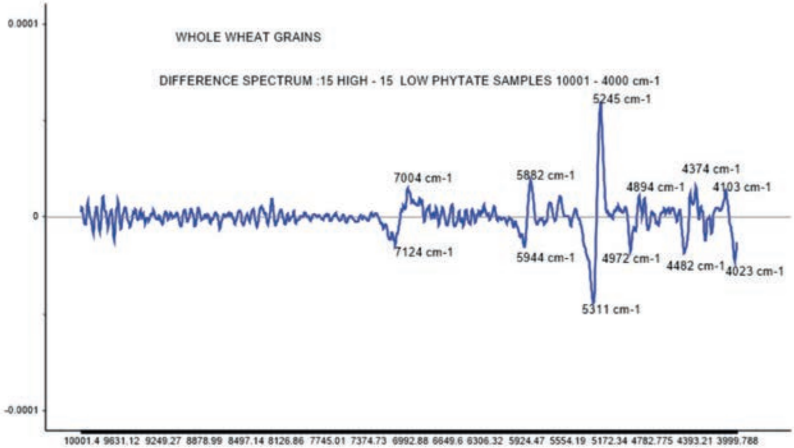


Figure 1. Difference between 2der spectra of wheat grains with low and high PP%TP ratios

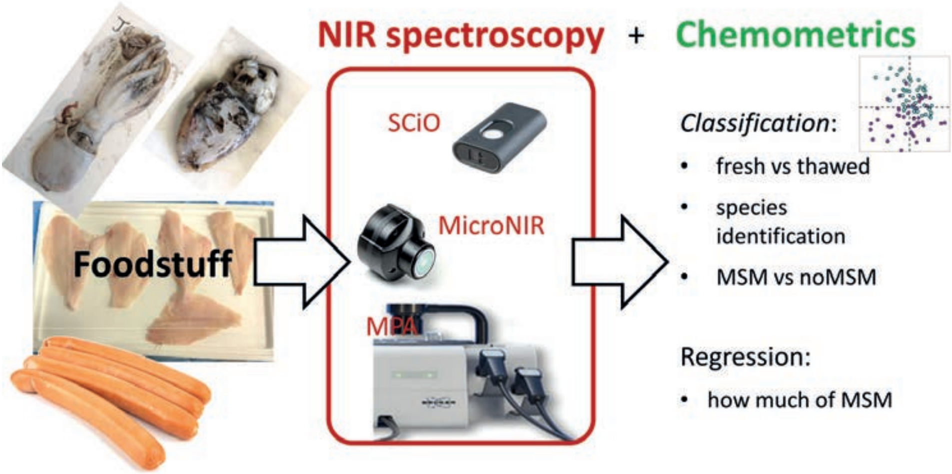
Workman J. Jr. and Weyer, L (2008) “Practical guide to interpretive near-infrared spectroscopy” CRC Press, Boca Raton
Raboy, V. (2009) Plant Science 177, 281-296

P01.71

FIGHTING FOOD FRAUDS WITH NIR SPECTROSCOPY AND CHEMOMETRICS: A STORY OF SUCCESS

Francesco Savorani[1], Nicola Cavallini[1], Gentian Gavoci[1], Alessandro Giraudo[1], Marzia Pezzolato[2], Elena Bozzetta[2], Francesco Geobaldo[1]

[1]Politecnico Di Torino, Torino, Italy, [2]Istituto Zooprofilattico Sperimentale del Piemonte, Liguria e Valle D'Aosta, Torino, Italy



Food fraud is a widespread problem with potentially heavy and wide impacts on consumers' safety and health, as it affects almost any type of food commodity sold on the market. Products may be mislabelled or counterfeit, making it challenging to detect these illegal practices. Therefore, efficient and reliable methods are necessary to tackle this issue. NIR spectroscopy coupled with chemometrics is particularly suitable for this purpose due to its ease of use and promptness.

In collaboration with Istituto Zooprofilattico Sperimentale del Piemonte Liguria e Valle d'Aosta, our research group at Polytechnic of Turin carried out a project about different types of food fraud. We investigated three food commodities, aiming at specific frauds: 1) the sale of frozen-thawed cephalopods mislabelled as fresh; 2) the sale of mislabelled fish fillets which can be substituted with cheaper ones; 3) the adulteration of meat sausage products with the addition of undeclared mechanically separated meat (MSM).

All research lines were based on the same analytical approach. The experimental data were acquired using three different NIR instruments: one portable low-cost instrument (SCiO by Consumer Physics), one portable medium-high-cost instrument (MicroNIR by Viavi) and one benchtop instrument (MPA by Bruker). The data were then inspected and processed using chemometric methods, which allowed to fully exploit the information content of all acquired datasets. More specifically, we aimed at building classification models using partial least squares-discriminant analysis (PLS-DA) to perform quick and automatic detection of food frauds. Regarding the research line about meat, we also dwelved into the possibility of quantifying the amount of MSM in the sausages, building PLS regression models.

Two papers from the cephalopods and fish research lines were published [1,2], and the work regarding the meat research line (identification and quantification of MSM) is currently under advanced development, close to submission.

The aim of this poster is to provide a representation of the research project and its results, showing how valuable the collaboration among different research groups is, and how powerful the combination of NIR spectroscopy and chemometrics can be. This project fully demonstrates that food fraud can sometimes be efficiently tackled even with simple instruments and methods.

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[2] N. Cavallini, F. Pennisi, A. Giraudo, M. Pezzolato, G. Esposito, G. Gavoci, L. Magnani, A. Pianezzola, F. Geobaldo, F. Savorani, E. Bozzetta, Chemometric Differentiation of Sole and Plaice Fish Fillets Using Three Near-Infrared Instruments, Foods (2022), 11(11), 1643; https://doi.org/10.3390/foods11111643

P01.72

A RAPID QUANTIFICATION NIR MODEL TO DETECT MELAMINE ADULTERATION IN SPORT NUTRITION SUPPLEMENTS

Kristina Shutevska[1], **Zoran Zhivikj**[1], Ana Marija Bajatovska[1],
Liljana Anastasova[2], Tanja Petreska Ivanovska[1], Nikola Geskovski[3]

[1]Institute of Applied Biochemistry, Faculty of Pharmacy, Ss. Cyril And Methodius University In Skopje, Skopje, Republic of North Macedonia, [2]Institute of Applied Chemistry and Pharmaceutical Analysis, Faculty of Pharmacy, Ss. Cyril And Methodius University In Skopje, Skopje, Republic of North Macedonia, [3]Institute of Pharmaceutical Technology, Faculty of Pharmacy, Ss. Cyril And Methodius University In Skopje, Skopje, Republic of North Macedonia

The growing popularity of sports nutrition supplements has made them a target for adulteration with inexpensive substances like melamine. This substance has been demonstrated to induce nephrotoxic effects, posing a health risk to consumers. Hence, rapid analytical methods are needed for detection and quantification of such adulterants in sport nutrition supplements. NIR spectroscopy is a non-invasive, rapid analytical method that can be used for on-site field examination of a variety of sample types without the need for sample preparation.

The aim of this study was to establish a fast quantification model using NIR spectroscopy and multivariate data analysis for the detection of adulteration of melamine in various sport nutrition supplements. Three types of sport nutrition supplements (proteins, amino acids and creatine) were spiked with melamine in a concentration range of 0.5-20% (w/w). A total of 6 different matrices within the given melamine concentration range were prepared to obtain a calibration set of 60 samples. An OPLS model was constructed using SNV processed NIR spectra as X variables and the concentration of melamine as Y variable. The obtained results (R2Y= 0.946, Q2=0.94) showed satisfactory correlation and prediction coefficients for the model, and both RMSEE and RMSEEcv (1.6 and 1.63%) demonstrated the model's ability to reasonably predict the melamine concentration, which is expected to be present as an adulterant. The VIP plot (Fig. 1) revealed that the unique NIR spectral features of melamine were extracted by the model and employed for successful quantification in different matrices. Furthermore, the scatter plot depicting the relationship between t1 and t1o (Fig 1b) highlights two key capabilities of the OPLS model: the effectiveness of the first predictive component in determining the melamine concentration, and the orthogonal component's ability to differentiate between the different types of matrices employed in the model.

NIR in combination with chemometric algorithms could be applied as a valuable tool that enables rapid determination and quantification of melamine adulteration in different types of sport nutrition supplements. Such approach can be utilized for surveillance of food supplements, ensuring the safety of end-consumers.

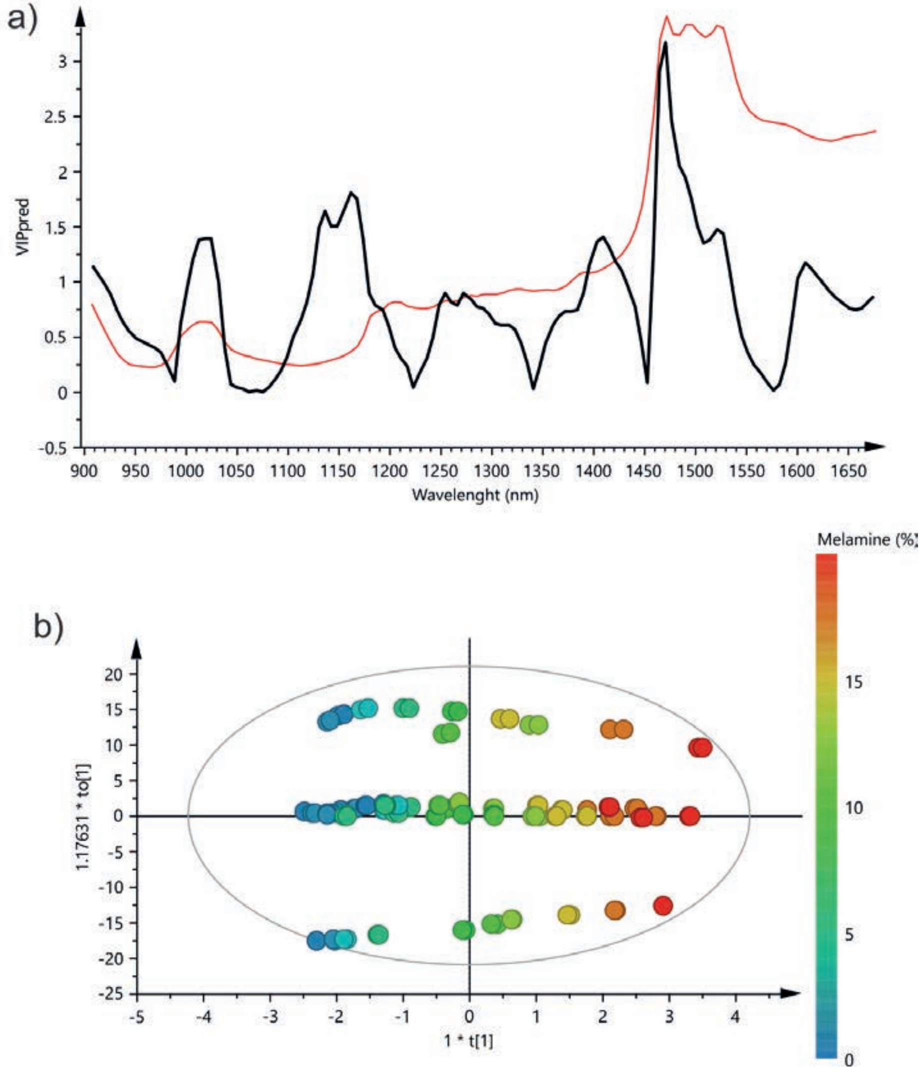

Figure 1. a) VIP plot of the model (black line) and NIR spectrum of melamine (red line) b) t1 vs t1o score scatter plot of the OPLS model

P01.73

NIR SPECTROSCOPY FOR MONITORING THE DECARBOXYLATION PATHWAY OF THE MAIN PHYTOCANNABINOIDS IN CANNABIS SATIVA

Olga Gigopulu[1], Nikola Geskovski[1], Gjoshe Stefkov[1], Christian W. Huck[2], Krzysztof B. Beć[2], Justyna Grabska[2], Petre Makreski[3]

[1]Ss. Cyril and Methodius University in Skopje, Faculty of Pharmacy, Skopje, Macedonia, [2]Institute of Analytical Chemistry and Radiochemistry, CCB – Center for Chemistry and Biomedicine, Innsbruck, Austria, [3]Ss. Cyril and Methodius University in Skopje, Faculty of Natural Sciences and Mathematics, Institute of Chemistry, Skopje, Macedonia

The medical cannabis industry has experienced substantial growth due to potential therapeutic benefits, increased research, expanded legalization, and market demand. More than 460 compounds have been identified in the plant of Cannabis sativa L., of which more than 140 are phytocannabinoids, genuine synthesized in acidic form, where Δ9-tetrahydrocannabinolic acid (THCA) and cannabidiolic acid (CBDA) predominate (Vergne et al., 2023). These acids can be decarboxylated into their pharmacologically active neutral forms, Δ9-tetrahydrocannabinol (THC) and cannabidiol (CBD). In recent years, employment of near-infrared spectroscopy (NIRS) for cannabinoid analysis and quantification has gained interest, mainly due to its advantages, such as being rapid, non-destructive and with minimal sample preparation. The objective of this study was to monitor the CBDA and THCA decarboxylation reactions using NIRS over a wide temperature range, to evaluate the reaction rate as a function of temperature and to determine the key spectral characteristics of the CBDA and THCA decarboxylation process, identifying the optimal temperature range by employment of multivariate analysis. The decarboxylation protocol was first applied to THCA and CBDA standards by exposing them to a temperature range of 25–160°C and performing scans at a rate of 2°C/min (t-NIR scans) with handheld NIR spectrometer. Similar set of experiments were performed on two samples of CBDA-dominant and THCA-dominant cannabis flowers. The collected spectra were preprocessed with standard normal variate (SNV) and first order derivative and were subjected to principal component analysis (PCA) in order to identify the NIR spectral changes due to decarboxylation of THCA and CBDA. The obtained results demonstrate that NIRS can be used to effectively monitor the decarboxylation reaction of CBDA and THCA in the solid state for both standards and cannabis flowers. Multivariate statistical analysis depicted the spectral regions with highest importance for the decarboxylation reaction. The PCA score plots (as a function of temperature) of the models were used to monitor the decarboxylation reaction. The results demonstrate initiation of decarboxylation in the plant material at lower temperatures compared to the standards. This represents a step forward in the development and optimization of a simple, effective method for monitoring the decarboxylation reaction that could be used as process analytical technology in the rapidly developing medicinal cannabis industry.

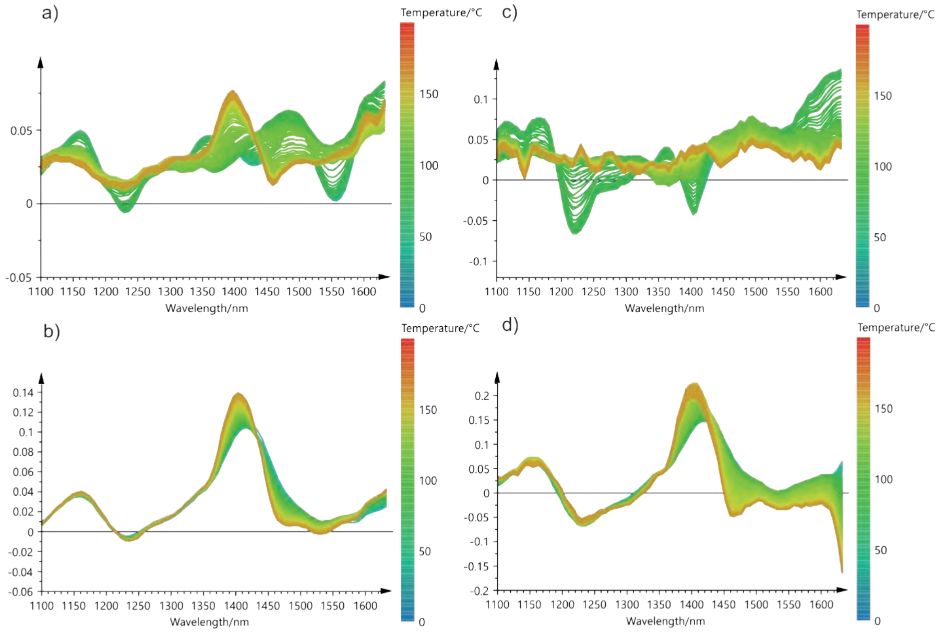

Figure 1. Overlaid SNV transformed, first derivative spectra of the 25–160°C decarboxylation experiments, colored by temperature: a) CBDA standard; b) CBDA-dominant flower; c)THCA standard; d) THCA-dominant flower.

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P01.74

SAMPLING STRATEGIES IN THE USE OF PORTABLE NIR INSTRUMENTS FOR QUALITY CONTROL OF HUMAN MILK

Candela Melendreras García[1], M. Luisa Fernández-Sánchez[1],
Belén Fernández Colomer[2], Marta Valledor[3], Francisco Ferrero[3], J.C. Campo[3],
J.M. Costa-Fernández[1], Federico Marini[4], Ana Soldado[1]

[1]Department of Physical and Analytical Chemistry, University Of Oviedo, Oviedo, Spain, [2]Department of Pediatrics, Hospital Universitario Central de Asturias, Oviedo, Spain, [3]Department of Electrical Engineering, University of Oviedo, Oviedo, Spain, [4]Department of Chemistry, University of Rome "La Sapienza", Rome, Italy

The initial source of nutrition for humans is breast milk. It has a lot of nutrients and bioactive elements that are important for the growth of newborns. It is commonly known that a mother's own milk provides the best nutrients, however, sometimes this is not possible. These reasons support the inclusion of milk banks as a specialist service inside hospital neonatology departments. One major issue with providing breast milk from hospital banks to neonates is that the milk's contents should be sufficient to meet their nutritional needs. Pre-calibrated milk analyzers devices are being used in laboratories to test milk quality, these instruments are expensive and require expert technicians to be used. An alternative for on-site and real-time monitoring milk quality control is the use of portable Near Infrared devices, for real-time monitoring milk quality [1].

Previous work has demonstrated portable NIRS systems' applicability for quantifying nutrients in pasteurised donated human milk [2]. However, these milk samples, often preserved frozen, are necessary to defrost before analysis. The aim of this work is to evaluate different sampling procedures and NIR instruments, to facilitate the work in milk banks, therefore samples of pasteurised donated human milk in a liquid state and tempered at 37ºC (optimum state of the milk) and frozen, both in their containers and in an alternative storage method, plastic bags, have been analysed. The data obtained are included in a multi-block dataset, which has been the basis for chemometric procedure.

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P01.75

ASSESSING THE POTENTIAL OF HYPERSPECTRAL IMAGING AND FOURIER TRANSFORM-NEAR INFRARED SPECTROSCOPY FOR PREDICTION OF INTERNAL CONSTITUENT OF TOMATOES

Maria Luisa Amodio[1], Hassan Fazayeli[1], Danial Fatchurrahman[1],
Aysha Saleem[1], Giancarlo Colelli[1]

[1]Università Di Foggia, Foggia, Italy

This study aimed to investigate the potential of optical nondestructive techniques for evaluating the chemical components, including pH, total soluble solid content (TSS), titratable acidity (TA), Ascorbic Acid (AA), and Vitamin C (Vit-C), in intact soilless cultivated tomatoes, given their great significance in the fresh fruit and vegetable sector and daily consumption. Two distinct tomato varieties were used: a cherry tomato variety, 'Carminio' (cv 'Carminio'), grown in autumn-winter, and an intermediate tomato variety, 'Mose' (cv 'Mose'), grown in spring-summer. Hyperspectral imaging (HSI) in the visible-near infrared (Vis-NIR) (400-1000 nm) and near infrared (NIR) (900-1700 nm), along with Fourier Transform-Near Infrared (FT-NIR) spectroscopy (800-2777 nm), were employed to capture spectral characteristics of fully ripe tomatoes. The specrtal scanner (version 1.4, DV srl, Padova, Italy) had a spatial resolution of 1000 × 2000 pixels (Vis-NIR) and 600 × 320 pixels (NIR), both with a 5 nm spectral resolution, while FT-NIR (MPA Multi-Purpose FT-NIR Analyzer, Bruker Optics, Ettlingen, Germany), had a sphere macrosample resolution of 1.71 nm (64 scans per sample). Mean spectra were extracted from the HSI (after image segmentation) and FT-NIR spectra, and multivariate calibration models were developed using partial least squares (PLS) regression. Various preprocessing techniques wereapplied to the spectra to enhance prediction accuracy. Although HSI yielded good results, FT-NIR spectroscopy outperformed in predicting the chemical components. The spectra obtained from FT-NIR resulted in good predictions of TSS content ($R^2 = 0.83$; NRMSEPr = 3.92%), pH ($R^2 = 0.89$; NRMSEPr = 1.3%), TA ($R^2 = 0.84$; NRMSEPr P = 3.61%), AA ($R^2 = 0.73$; NRMSEPr = 6.48%), and Vit-C ($R^2 = 0.76$; NRMSEPr =6.1%). These findings demonstrate the potential of optical nondestructive techniques, including HSI and FT-NIR spectroscopy, in predicting chemical components in intact tomatoes. Notably, our results for Vitamin C content exceeded reported literature values, while other components, such as TSS, showed similar or better trends, considering experimental conditions and inherent variability. These advanced technologies offer potential benefits in quality assessment and grading processes, leading to enhanced product selection and reduced waste.

P01.76

MONITORING OF PROTEIN HYDROLYSATION BY NEAR-INFRARED SPECTROSCOPY

Yoshitomo Amo[1]

[1]Morinaga Milk Industry Co.,Ltd., Tokyo, Japan

Protein hydrolysates are often used as flavor enhancers, and serve as functional ingredients to modify foaming, emulsifying, and gelation properties of various foods. These are also applied to infant formula to reduce allergenicity. The bioactive peptides in hydrolysates can exert physiological effects, including antihypertensive and antimicrobial activities, and are, thus, considered lead compounds for nutraceutical or functional food development. In food protein digest manufacturing, these functional properties depend on their hydrolysation degree. Therefore, monitoring the number of peptide bonds cleaved during a proteolytic reaction to obtain the desirable activities and production efficiency is important. Several monitoring techniques, including pH-stat, osmometry, and the o-phthaldialdehyde (OPA)/ trinitro-benzene-sulfonic acid method, have been developed. In this study, we evaluated the discrimination ability of peptide bond cleavage and investigated the usefulness of reaction monitoring of protein hydrolysation using near infrared spectroscopy.

Model peptide NIR spectra of crystalline powders or aqueous solutions was measured by diffuse reflection or transflection mode. The prepared model peptides mimicked the degradation process from pentaglycine to glycine. Milk protein hydrolysation was performed with pancreatin, papain, and subtilisin individually under optimal conditions using casein or whey protein as substrates. Spectral data of these enzymatic reactions were continuously collected using a FT-NIR spectrometer equipped with fiber optics and transflection probe. The degree of hydrolysis (DH) of the reaction samples was quantified using the OPA colorimetric method. DH prediction models were developed from pretreated NIR data using partial least squares regression.

The model peptides crystalline NIR spectra were markedly different, showing clear differences in the 2nd derivative spectra of their solutions. Comparing these treated spectra, peak changes associated with a decreased number of peptide bonds were observed. In each prediction model, the predicted values correlated well with the corresponding theoretical number of hydrolytic cleavage or OPA method values for each peptide solution. In contrast, in the milk protein hydrolysis process, especially in casein, spectral changes occurred in the high wavenumber region, which were likely due to the scattering caused by aggregates generated as a result of the enzymatic reaction. However, the prediction models for each reaction could capture the kinetics of enzymatic proteolysis for various substrate-enzyme combinations. In conclusion, this technology can be used for DH prediction during proteolysis reactions, thereby detecting abnormal runs and enabling improved control of the protein hydrolysis process.

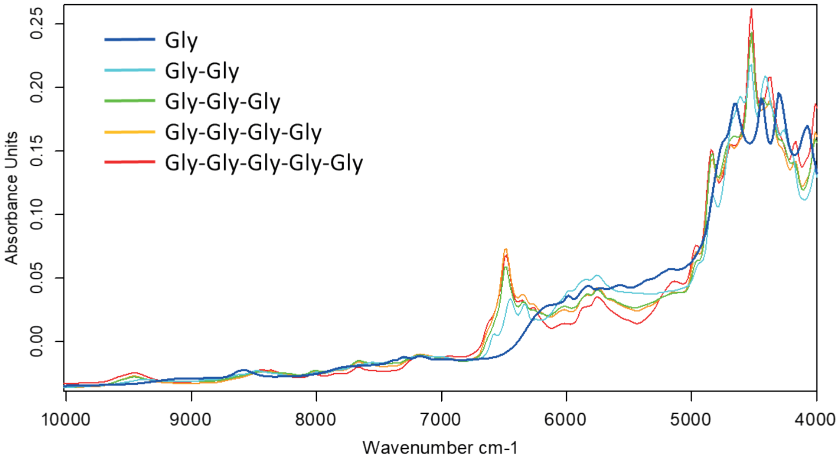
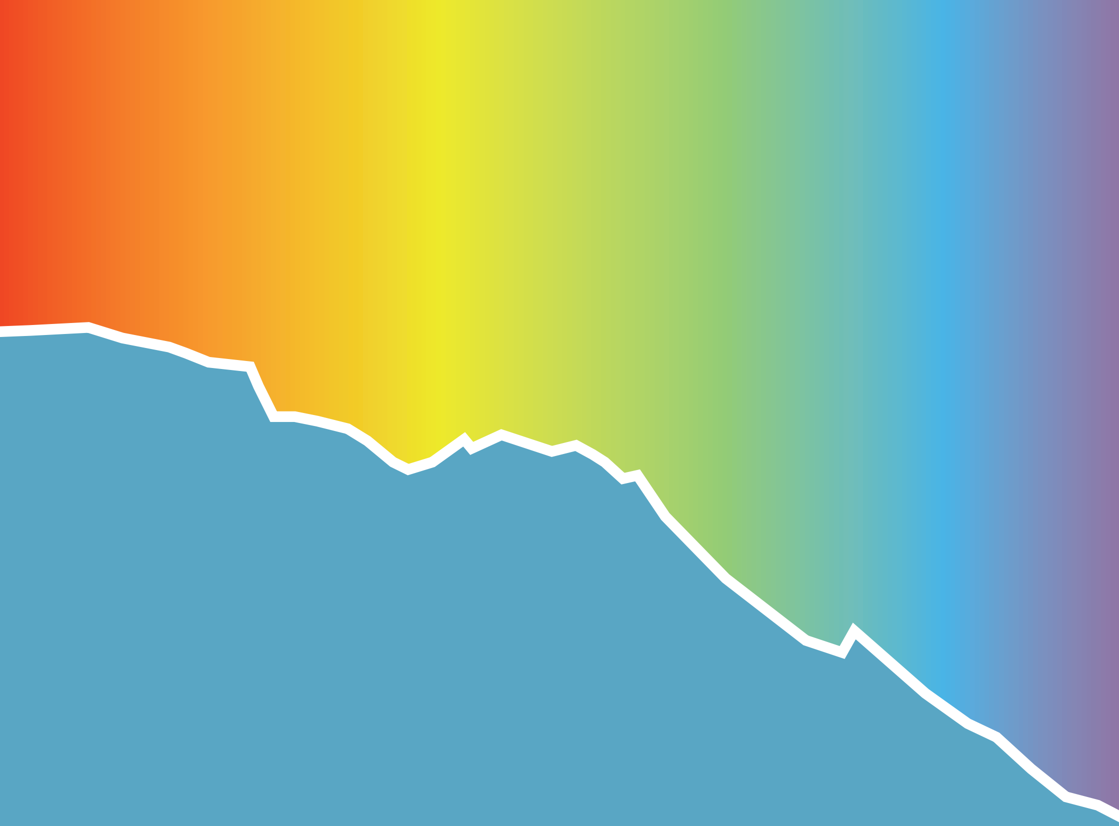


Figure 1: NIR spectra of model peptides crystalline (treated with SNV)



THEME 02

ALLERGENICS/TRADITIONAL MEDICINE

POSTER PRESENTATION: P02.01

P02.01

RAPID GRADATION OF PIPER NIGRUM SEEDS USING A PORTABLE NIR SPECTROMETER AND MACHINE LEARNING METHOD

Rajib Bandyopadhyay[1], Dilip Sing[1,2], Subhadip Banerjee[2], Kalyan Majumder[1], Pulok Kumar Mukherjee[3]

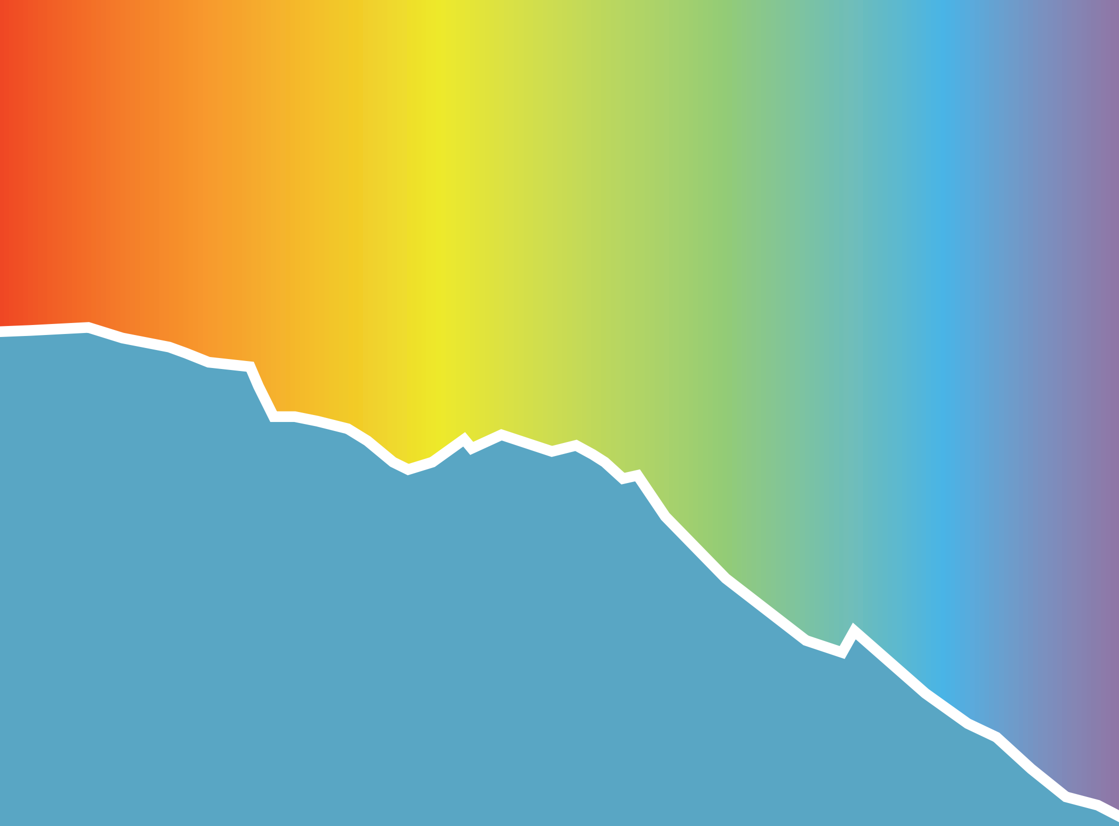
[1]Department of Instrumentation and Electronics Engineering, Jadavpur University, Salt Lake Campus, Kolkata, India, [2]MetaspeQ Division, Ayudyog Private limited, Kolkata, India, [3]Institute of Bioresources and Sustainable Development, An autonomous Institute under Department of Biotechnology, Govt of India, Imphal, India

Black pepper (Piper nigrum) is abundantly farmed throughout India's tropical regions. Piperine (2-9%) is the primary active ingredient. Recent pharmacological research have demonstrated that piperine has antioxidant, cytoprotective, anticonvulsant, anti-ulcer, anti-depressant, and anti-inflammatory properties. Additionally, it is employed in conventional medicine. Black and white pepper's primary bioactive ingredient is piperine.

The genotype and agroecology of black pepper determine the nature and quantity of the active molecules likepiperine and hence the pharmacological activities. It is important to estimate the most important active molecules, the piperine and classify black pepper plants with respect to this group of marker molecule. The active molecules are not at all taken into account in conventional classifications that are based solely on visual examination of physical features. As a result, analyses using techniques like high performance liquid chromatography (HPLC), high performance thin layer chromatography (HPTLC), and LC-MS/MS are mostly used to estimate active compounds for pharmacognosy and physicochemical research. These methods are labor-intensive, expensive, time-consuming, and sophisticated. They also generate a considerable amount of trash. Any field-level quality control system should be able to process a lot of samples quickly and reasonably quickly.

Black pepper samples were divided into three categories: good quality, medium quality, and low quality based on their piperine content. Grade I was assigned to plants with a high piperine content (>5%), and grade III was given to those with a low piperine content (2%). 2% and 5% of the intermediate types were assigned to grade II.

The objective of this work was to create a quick and non-destructive gradation method for measuring the amount of piperine in Piper nigrum. The spectral data was gathered in diffusion reflectance mode over the wavelength range of 900 nm to 1700 nm. Twenty samples of Piper nigrum were gathered from various parts of India. A model based on the support vector machine (SVM) on NIR spectra was constructed to classify the data into three groups. Using standard normal variate (SNV) preprocessing, the model's outputs for tenfold classification had an accuracy of 80%. The preliminary findings show that the portable NIR approach developed is effective for grading Piper nigrum seed.



THEME 03

CHEMOMETRICS

ORAL PRESENTATION: O03.01 – O03.14
FLASH TALK: F03.01 – F03.07
POSTER PRESENTATION: P03.01 – P03.18

003.01

ADVANCE DENOISIONG METHODS FOR SPECTRAL ANALYSIS BASED ON DECOMPOSITION ALGORITHMS

Xihui Bian[1], Yue Hao[1], Mengxuan Ling[1], Zitong Shi[1], Peng Liu[1]

[1]College of Chemical Engineering and Technology, Tiangong University, Tianjin, China,
[2]NMPA Key Laboratory for Technology Research and Evaluation of Drug Products, Shandong University, Jinan, China

Due to the influence of temperature, humidity and instrument vibration, the spectral signal usually contains noise, which obscures useful information and reduces the resolution of the signal. The denoising methods mainly include smoothing-based and decomposition-based methods. Savitzky-Golay (SG) smoothing[1] is one of the most commonly used methods. However, the denoising effect of SG for the signal with sharp peaks is not good. As a decomposition-based denoising method, WT decomposes the original signal into a series of details and approximations and then the high frequency details are deleted. The denoised signal is reconstructed by inverse wavelet transform. However, it is difficult to select the optimal parameters for WT because there are many candidates for wavelet functions and de-composition scales[2]. Thus, three new developed decomposition-based methods, including empirical mode decomposition (EMD)[2-6], local mean decomposition (LMD)[7] and variational mode decomposition (VMD)[8] are introduced for denoising of different spectral signals.

A denoising method based on HHT combined with F-test is proposed firstly. EMD combined t-test is developed for X-ray diffraction (XRD) spectral denoising of Ag/Ag2S@C and Rh/C catalysts. For solving the end effect and mode mixing of EMD, VMD is introduced for Raman spectral denoising of MnCo-ISAs/CN and Fe-NCNT catalysts[6]. Result shows that the Raman spectrum denoised by VMD is more excellent than that of EMD and SG smoothing in terms of visualization and SNR. Finally, a piecewise mirror extension local mean decomposition method[7] is proposed for near-infrared spectral denoising with uneven noise. The feasibility of the proposed method is validated by visualized, signal-to-noise ratio and modeling results. Results show that the proposed denoising methods are superior to SG smoothing and WT for NIR spectra of Chinese medicine and beer samples.

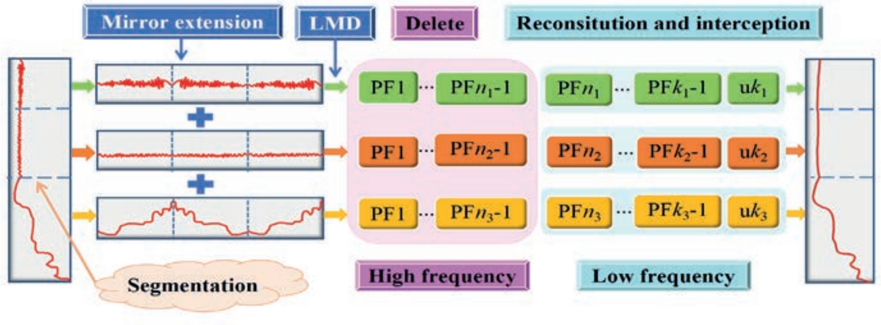


Figure 1. The flowchart of piecewise mirror extension local mean decomposition method for denoising.

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003.02

NEEDLE IN A HAYSTACK – NEAR-INFRARED METHOD DEVELOPMENT AND VALIDATION FOR QUANTITATION OF A BIOLOGICAL NOVEL FOOD IN RODENT DIET

Máté Csontos[1,2], János Elek[1]

[1]Science Port Ltd., Karcag, Hungary, [2]University of Debrecen, Department of Physical Chemistry, Debrecen, Hungary

The registration and toxicological testing of the next generation of the food additives and dietary supplements – so called novel foods - bring new challenges from the analytical point of view as well. A large number of these materials belong to the group of UVCB materials: Unknown or Variable Composition, Complex Reaction product or Biological Origin1. These multi-component test items may contain a variety of proteins, fibres, fat compounds, and many other types of constituents. Not only the separation of these components can be strenuous with the classical analytical methods, but also the selection and targeted analysis of the main components. In this presentation I will demonstrate the development and validation of a near-infrared method combined with multivariate data analysis. The method was validated for the quantitation of a multicomponent biological novel food in a quite complex matrix: semi-synthetic rodent diet.

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003.03

EVOLUTIONARY SEARCH IN NIR SPECTRAL LIBRARIES

Laura Michelle Diaz Villagomez[1], Leonardo Ramirez-Lopez[2],
Claudio Orellano[2], Zefang Zen[3], Raphael Viscarra Rossel[3]

[1]Eth Zurich, Zurich, Switzerland, [2]BUCHI Labortechnik, Flawil, Switzerland, [3]Curtin University, Perth, Australia

Due to the large sample diversity usually found in spectral libraries, some of these samples might be non-informative to build an accurate model for a given target population. In this regard, more appropriate and accurate custom models can be built from a subset of selected samples (from the spectral library) which are highly informative in the context of the target population. Therefore, effective sample search methods are paramount to guarantee the usefulness/operability of spectral libraries for quantitative applications.
Despite sample search methods are key to make large and diverse spectral libraries operational, research in this matter has been largely overlooked. Thus, in this work we introduce a novel genetic algorithm, which extracts relevant observations to build custom predictive models for a given target population. Our algorithm ensures that the samples found can be used to build NIR models that provide sound predictive and descriptive power.
In our algorithm, a spectral library is treated as a pool of genes where each gene represents a sample. In the evolution process, the algorithm iteratively removes irrelevant or weak genes/samples from the library until a predefined subset size is met. At each iteration a large number of random subsets is generated and each of them is used to fit a partial least squares (PLS) model. This model is used to project into the PLS space the samples of the target population, then, the samples are back-transformed into the spectral space and finally the back-transformation error is computed. For every subset, its samples are labeled with the error returned by it. For every sample, its labeled errors (across the multiple subsets in which it was present) are aggregated into a mean error. Finally, the samples with the largest errors are identified as weak samples and removed for the next iteration. These weak samples can be seen as samples that consistently increase the explanatory error of a PLS model when they are present in a subset.
Apart form the spectral back-transformation error, our algorithm also incorporates distance measures to further guide the search. This evolutionary search is then entirely guided by figures of merit that are obtained from the target population.
In this presentation we provide the basic mathematical concepts behind the genetic algorithm we have designed and how they ensure that the samples found provide accurate models that are simple to interpret and update.

003.04

SO-PLS NEEDS PROSAC TO BOOST ITS CONFIDENCE IN HANDLING MULTIBLOCK SPECTRAL DATA

Jose Antonio Diaz-Olivares[1], Ryad Bendoula[2], Wouter Saeys[3], Maxime Ryckewaert[2,4], Jean-Michel Roger[4], Ben Aernouts[1]

[1]KU Leuven, Department of Biosystems, Division of Animal and Human Health Engineering, Livestock Technology Laboratory, Geel, Belgium, [2]ITAP, Univ. Montpellier, INRAE, Institute Agro, Montpellier, France, [3]KU Leuven, Department of Biosystems, Division of Animal and Human Health Engineering, MeBioS, Leuven, Belgium, [4]ChemHouse Research Group, Montpellier, France

Multiblock spectral data are commonly used to construct multi-sensor fusion chemometric models to analyze sample composition. However, such spectra are affected by extraneous sources of variability, unrelated to the variables of interest. Preprocessing is typically applied to mitigate this issue, but the selection and combination of pretreatment methods, as well as the identification of the most informative data blocks, can be a time-consuming task.

To address this challenge, the presented method employs preprocessing ensembles with response-oriented sequential alternation calibration (PROSAC) to determine the preprocessed blocks and their order of implementation for a targeted application of sequentially-orthogonalized partial-least squares (SO-PLS). The method prioritizes preprocessed blocks that yield a minimal residual in a step-wise forward selection using the rapid Gram-Schmidt process, enabling the computation of numerous and complex blocks.

The effectiveness of the method is demonstrated in two empirical case studies. The first involves predicting raw milk composition (fat, protein, and lactose) using long-wave near-infrared (LW-NIR) reflectance measurements at thirty different illumination-to-detection distances. The second case study involves estimating the crude protein and total sugar content of sugarcane using combined visible and NIR spectral measurements from six different spectrometers. The results obtained from the methodology are compared with partial-least-squares regressions on the individual preprocessing of the data sets and with only applying the PROSAC step of the methodology.

The combined PROSAC and SO-PLS method achieved prediction errors significantly lower than their individual PLS and only-PROSAC counterparts, except for fat in the raw milk dataset and total sugar in the sugarcane dataset. The proposed approach is a versatile technique that can be employed for ensemble preprocessing in NIR data modeling, with the advantages of applying SO-PLS without concern of preprocessing sequence or block order, and has demonstrated efficacy in handling a high number of blocks (>5).

003.05

DOW POLYOLS NIR CHEMOMETRIC MODEL REDUCTION BASED ON CLUSTERING –REDUCE >30 GLOBAL HYDROXYL MODELS TO < 5

Wendy Flory[1], Kazimierz Czarnecki[2], Matthijs Mercy[3], Mark Joswiak[4], Mary Beth Seasholtz[1], **Frederic Despagne**[5]

[1]Dow, Inc., Midland, MI, United States [2]Dow, Inc., Lake Jackson, TX, United States, [3]Dow, Inc., Terneuzen, Netherlands, [4]Dow, Inc., Houston, TX, United States, [5]ABB, Pessac, France

Polyurethane Materials are present in a wide range of industrial segments such as Furniture, Building & Construction, Composites, Automotive, Electronics, and more. Dow is one of the leaders for the manufacture of the two main raw materials, Isocyanates and Polyols used to produce polyurethane products. Dow is also a key player for the manufacture of Polyurethane Systems/Formulations designed for targeted applications.
In ~1990, the first analytical chemometric models were developed and deployed for use in the QC labs of the polyols business for the quantification of hydroxyl, water, cloud point, and viscosity. Over the years many models have been added; there are now hundreds of models for quantification and hundreds for product identification, too many to be reasonable for support. There are more than 30 global models alone for the quantification of hydroxyl across 70+ products at many sites. An attempt was made to consolidate these into a single model. While the consolidated model showed good statistics across the entire range of hydroxyl, several products had a bias by ASTM E1655 with individual product validation.
This work will show the strategy for global model updates for hydroxyl, to reduce the number of models for quantification from over 30 to less than 5 using chemometric methods. In order to gain an understanding of the best product groupings, we identify clusters by reducing spectra to a few dimensions via Principal Component Analysis (PCA) and Uniform Manifold Approximation and Projection (UMAP). Results from these cluster analyses suggest that 2-3 models are ideal for reduction of the number of hydroxyl models used in the Dow Polyols business.

003.06

A COMPLETE SCHEME FOR UNSUPERVISED MULTIVARIATE CALIBRATION MONITORING

Valeria Fonseca Diaz[1,2], Arnout Van Nuenen[1], Jose A. Diaz-Olivares[1], Ben Aernouts[1]

[1]KU Leuven Department of Biosystems, Division of Animal and Human Health Engineering Geel Campus, Geel, Belgium, [2]Software Competence Center Hagenberg, Hagenberg, Austria

Multivariate calibration models have been the predominant analytical tool supporting quality control processes for chemical inspection that use non-destructive and cost-efficient technology such as spectral sensors. For several decades, the knowledge about the use of multivariate calibration for chemical quantification has solidified the methods and the practical requirements to build models that provide accurate predictions based on spectral signals. One of the key aspects in the entire pipeline of quality processes that use these models is to equip them with monitoring approaches so that it is possible to detect degradation of prediction performance. This aspect enables the correct use of multivariate calibration as models can be easily challenged by unknown interferents making them invalid in the short or long term. While the relevance of model monitoring is increasing nowadays with the evolution of the technology and its capacity to measure vast majorities of spectral data in time, the methodological approaches for an efficient and accurate calibration monitoring scheme remain limited with most of the solutions designed based on the use of new reference values [1]classification and fault detection models are ubiquitous in QbD (Quality by Design. In this work, a complete scheme for multivariate calibration monitoring has been developed to monitor the drift of the spectral variation and the drift of the quality of the quantitative predictions. The former has been designed as a consolidation of the state-of-the-art approaches combining outlier detection with a metric to quantify spectral variation drift [2], [3]. The latter corresponds to an innovative proposal to measure bias drift in the predictions using only new spectral measurements. This calibration monitoring scheme has been applied to a large dataset consisting of more than thirty thousand near-infrared milk samples with about 10% of reference values available. The data has been collected from a milking robot setup over 1.5 years for the quantification of fat, lactose and protein content at experimental dairy farm “Hooibeekhoeve” in Belgium. We present the performance of the designed scheme based on models built at the beginning of the sample collection period together with insights and recommendations for the practical scope of this methodological solution.

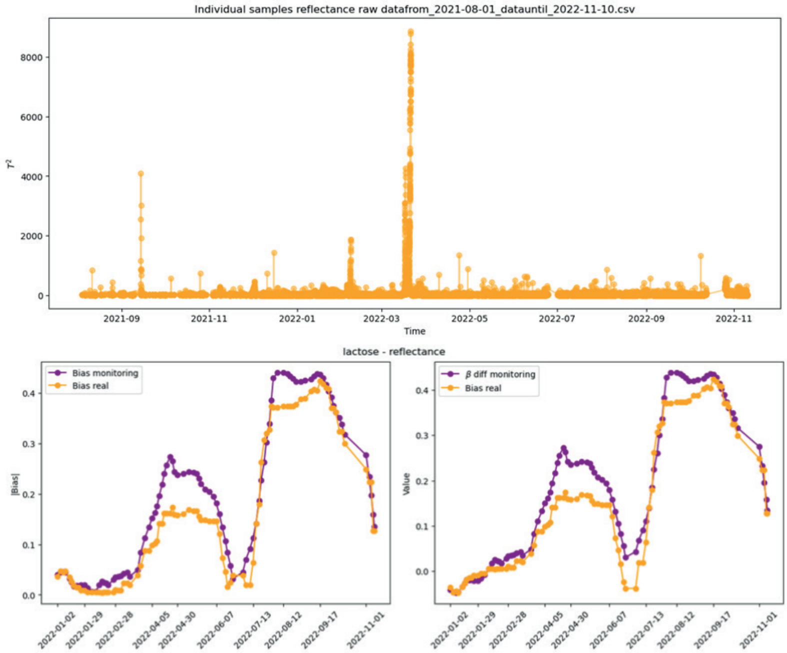

Figure 1. Unsupervised monitoring scheme for milk samples in reflectance mode to quantify lactose content.

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003.07

EXTENDED MULTIPLICATIVE SIGNAL CORRECTION WITH A PRIORI KNOWLEDGE TO IMPROVE PLS REGRESSION

Arnoud Jochemsen[1], Gry Alfredsen[2], Ingunn Burud[1]

[1]Norwegian University of Life Sciences, Ås, Norway, [2]Norwegian Institute of Bioeconomy Research, Ås, Norway

NIR measurements are affected by unwanted spectral variations from sources such as measurement noise, light scattering, changes in density, particle size and optical path length. Many of these unwanted physical variations can -to a certain degree- be corrected for by mathematical modelling, such that any chemical variations in the samples can be measured more accurately.

To filter out the interference from physical variations and measure the chemical variations, a preprocessing method such as Multiplicative Signal Correction (MSC) or Extended Multiplicative Signal Correction (EMSC) can be applied. The MSC estimates an offset and a scaling factor for each of the spectra, which aims to correct for variations in effective optical path length. EMSC extends this two-parameter model by adding wavelength dependent parameters and any known interference spectra. In addition, EMSC can estimate the relative presence of any known chemical constituent.

This study shows the benefits of adding such a-priori knowledge to modelling using the EMSC pre-processing method before using a Partial Least Squares Regression (PLSR) algorithm.

The spectroscopic data in this study describes the decay of Scots pine sapwood with either a brown rot fungus or a white rot fungus and was recorded with a short-wave infrared (SWIR) hyperspectral camera. The mass loss due to fungal decay is the response variable of the regression.

EMSC modelling with the library spectra of cellulose and lignin can estimate variations in these main wood polymers as the decay progresses and it shows the clear difference in decay due to brown rot and white rot fungi.

The addition of knowledge on these to chemical constituents to the preprocessing improves the prediction of wood massloss from spectra, but it mostly improves the amount of variance in the spectra explained, which leads to a better understanding of the PLS loading components (see Fig. 1).

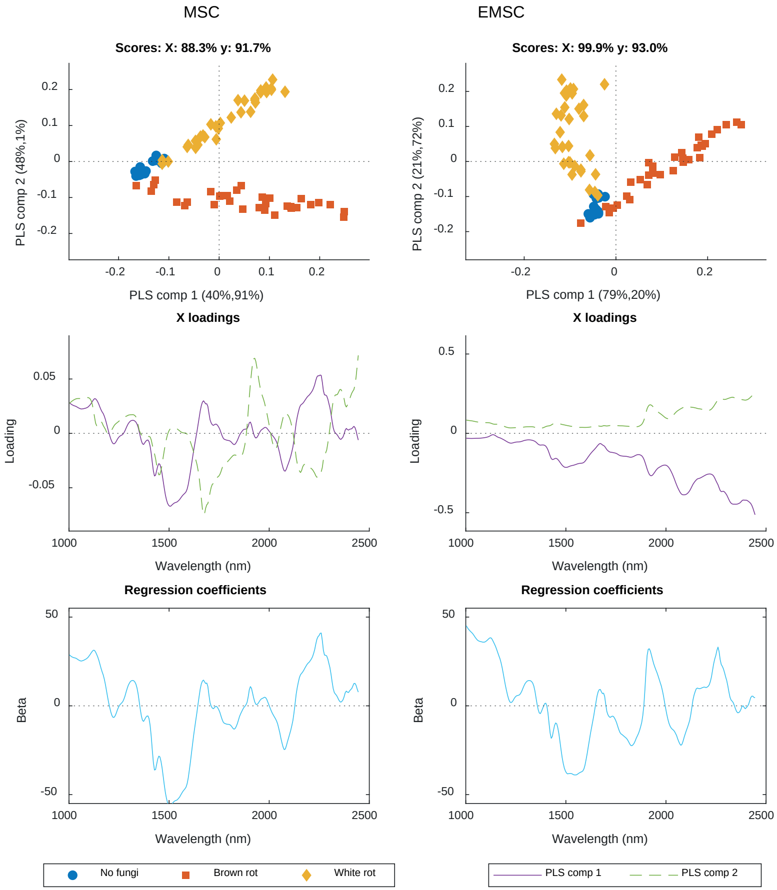

Figure 1. Score plots, X-loading plots and regression coefficients of a 2-component PLS model of the wood decay dataset, with massloss as the response variable. The wood decay dataset consists of 80 spectra, 32 with brown rot decay, 32 with white rot decay and 16 with no fungi. The left column plots are preprocessed with MSC and the right column plots are preprocessed with EMSC. The percentage pairs in the score plot labels are the variances in X and y explained by the PLS components.

003.08

DEVELOPMENT OF A PORTABLE RAPID AND ROBUST SPECTROSCOPIC METHOD TO DETERMINE UNKNOWN MARINE PLASTICS AND THEIR CHEMICAL ADDITIVES USING NIR SPECTROSCOPY.

Adam Kolobaric[1], Rebecca Orrell-Trigg[1], Stephanie Owen[1], Julia Jaegar[3], Andy Ball[1], Daniel Cozzolino[2], James Chapman[1]

[1]Rmit University, Melbourne, Australia, [2]QAAFI, University of Queensland, Brisbane, Australia, [3]Eurofins MGT, Melbourne, Australia

Plastic pollution is ubiquitous in the marine environment. It is estimated that 9-14 million tonnes of plastic enter our oceans annually[1-5], negatively affecting hundreds of marine species at all trophic levels. Furthermore, as plastic pollution weathers, secondary micro- and nano- plastics are generated increasing their uptake by living organisms and their mobility in the environment[6]. This weathering also facilitates the leaching of chemical additives present in plastic polymers. There are more than 10,000 chemical additives in the plastic industry's chemical "cabinet" [7-10], and they can make up to 10-70% of plastic by weight[9]. The estimated 1-3 million + tonnes of these chemical additives entering our oceans are almost completely unaccounted for [9, 11].
Classical analytical approaches for characterising plastic particulates (visual/microscopic counts) and their chemical additives (Thermogravimetric analysis, Differential Scanning Calorimetry, pyrolysis GC/MS) are time-consuming and expensive. However, rapid spectroscopic methods such as Fourier-Transformed Infrared FTIR, Raman, and Near Infrared spectroscopy (NIRS) have been gaining popularity.
This study used NIRS to determine the polymer type of marine plastics in the field using a portable NIR system (Micro-NIR). This miniaturised form of NIRS makes this a valuable tool for new approaches in plastic contamination and environmental monitoring.
Beach plastics (n=60) were collected from beaches along the South Coast of New South Wales, Australia. 9 reference/standard polymers were selected as the spectral training set (polypropylene (PP); polystyrene (PS); polyethylene (PE); polyamide (PA); polycarbonate (PC) Polycapralactome (PCL) and polyvinylchloride (PVC), polystyrene (PS), polysulfone (PSU), polyethylene-terephthalate (PET).

Figure 1 c) shows the factor 1 loadings in blue relating to the aliphatic polymers (PP, PE, PCL, PA) were based on the CH/CH2/CH3 2nd overtone and CH 1st overtone. While for aromatic polymers (PC, PSU, PS, PET) by ArCH intensity and discriminated by the CH/CH2/CH3 2nd overtone and combinations as shown in the factor loadings 1 c). The PLS was selected based on the optimal number of latent variables using the RMSECV. The PLS-DA model produced an overall rate of correct classification of 94% using NIRS method.

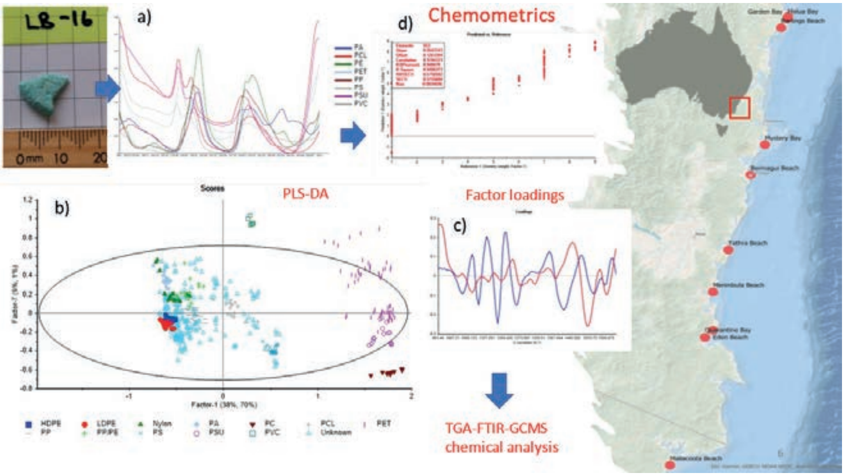

Figure 1. Figure 1 shows the AVG spectra of 9 polymer references (a) the PLS-DA score plot showing Factor 1 & 7 (b) and the factor loadings (c). The validation statistics of the model showing R² and RMSECV (d).

The same weathering of the beach plastics that has hindered the classic library match approach in vibrational spectroscopy of plastics [12, 13] made discrimination between some of the aliphatic polymers difficult. This aligns with the findings of other vibrational spectroscopy studies of plastics and suggests the need for non-virgin polymer standards in developing future models.

Future work will include Subsequent analysis via FTIR and Evolved Gas Analysis (EGA) via a thermogravimetric analysis system coupled with gas chamber FTIR and GC/MS analysis (TGA-FTIR-GC/MS) allowing for exploratory data analysis into the presence of additives and their ability to be discriminated via NIR.

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003.09

DETERMINATION OF THE OLIVE LEAVES SUITABILITY FOR BIOREFINERY PROCESSING ASSISTED WITH PORTABLE NIR SPECTROMETERS

Albert Kravos[1,2], Jakub Sandak[1,2], Anna Sandak[1,2], Balázs Dávid[1,2], Miklós Krész[1,2], Helena Sandak[1], Enya Kvarantan[1], Emilija Ivanova[3], Alba Ramos[3], Jose Maria Pinilla[3], José Carlos Quintela[3], Daniel Claudio[3], Ibai Funcia Muguerza[4], Irantzu Alegría[4]

[1]University of Primorska, Koper, Slovenia, [2]InnoRenew CoE, Izola, Slovenia, [3]Natac Group, Madrid, Spain, [4]National Renewable Energy Centre (CENER), Sarriguren, Spain

Currently, world-wide, more than 4.5 million tons of olive leaves are produced by the olive oil industry. This recalcitrant biomass contains high-added-value bioactive chemical components. Olive leaf residuals are underexploited; they are burnt in the fields, given to cattle, or in some cases, incinerated to produce energy. Circular bioeconomy and bio-based product development/production are becoming essential in the indispensable transition from a fossil-based economy towards future sustainable and environmentally friendly alternatives. From this perspective, the possibility for valorisation of underexploited bio-residuals is a great advantage. The Oleaf4Value project will address this challenge by developing alternative solutions for the high value-added transformation of olive leaf residuals. The chemical composition of the leaves varies depending on the tree species, local horticultural practices, and/or seasonal climate variations, among other factors. Similarly, the requirements of downstream biorefineries for specific biomass quality aspects depend on the characteristics of the final products and the specific production process available. Consequently, the proper biomass for proper use paradigm is a key for the profitable biorefinery industry.

The goal of this research is to develop a complete solution for reliable, low-cost, and rapid determination of the chemical composition of olive leaves. Different near-infrared-based sensors, including prototype solutions, were tested for their suitability for accurate prediction of reference quantifiers determined by wet-chemistry methods. The solutions rely on portable instruments which are suitable for implementation at the early stage of the value chain, including direct measurement in the field and/or at the biorefinery gate. Customised chemometric models were developed and validated for the prediction of biomass moisture content as well as oleuropein concentration. It confirms the high suitability of near-infrared spectroscopy for routine application.

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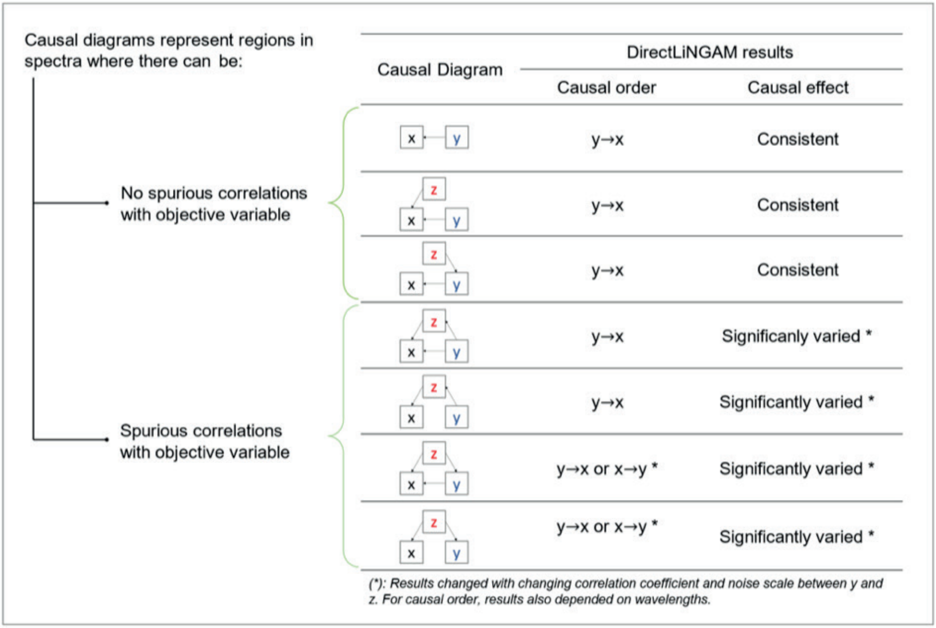
003.10

CAN HIDDEN SPURIOUS CORRELATIONS IN PREDICTION MODELS FOR SPECTRAL DATA BE TACKLED BY CAUSAL DISCOVERY?

Quan Nguyen Minh[1], Mizuki Tsuta[2], Mito Kokawa[3], Yukata Kitamura[3]

[1]Graduate School of Science and Technology, University of Tsukuba, 1-1-1 Tennodai, Tsukuba, Ibaraki 305-8572, Japan, [2]Institute of Food Research, National Agriculture and Food Research Organization, 2-1-2 Kannondai, Tsukuba, Ibaraki 305–8642, Japan, [3]Faculty of Life and Environmental Sciences, University of Tsukuba, 1-1-1 Tennodai, Tsukuba, Ibaraki 305-8572, Japan

The use of various prediction models built from spectral data assumes the correlation between explanatory variables (X) and response variables (Y) in the field of chemometrics. Spurious correlations, however, can reduce model robustness and predictive power, undermining informative wavelength selection. On the other hand, causation, which has been materialized with mathematical and statistical language in recent decades, can exclude unwanted factors and reveal the true relationship between X and Y. Using only observational data, Direct method of Linear Non-Gaussian Acyclic Model (DirectLiNGAM) can perform causal discovery in the form of causal order (an indicator of whether there is a causal relationship for any pair of variables and if yes, which of the two is the cause) and causal effect (an indicator of the connection strength between causal-related variables). This study applied DirectLiNGAM to seven simulated data sets (n = 10000) representing seven possible causal structures between X, Y, and Z (unobserved variables). The generality of seven simulated data sets was reinforced by the background knowledge that X is always caused by others and that there is a possibility of Z's presence - the source of spurious correlation with Y. For each data set, regarding the relationship between Y and Z, correlation coefficients and noise scales of the descendant were varied from 0.1 to 10 in seven levels to observe their effects on DirectLiNGAM's performance. Three out of seven causal structures had Y as the direct cause of X without having its effect mediated by Z. These represented reliable regions in the spectra for building prediction models with high robustness. With the input of X and Y from the three respective data sets, DirectLiNGAM models showed consistent causal effects between the explanatory variable and the response while yielding accurate causal directions from Y to X. Consistent causal effects despite the changing correlation and noise scale were the good indicator to solidify the advantage of causal models. The results also suggested a promising approach using causal discovery to find informative, reliable regions that might be obscure due to spurious correlation.
Graphic



Causal diagrams represent regions in spectra where there can be:	Causal Diagram	DirectLiNGAM results	
		Causal order	Causal effect
No spurious correlations with objective variable		y→x	Consistent
		y→x	Consistent
		y→x	Consistent
Spurious correlations with objective variable		y→x	Significanly varied *
		y→x	Significantly varied *
		y→x or x→y *	Significantly varied *
		y→x or x→y *	Significantly varied *

(*): Results changed with changing correlation coefficient and noise scale between y and z. For causal order, results also depended on wavelengths.

003.11

PARTIAL LEAST SQUARES WITH MULTIPLE DOMAINS

Ramin Nikzad-Langerodi[1]

[1]Software Competence Center Hagenberg, Hagenberg, Austria

Domain adaptation (DA), the branch of machine learning devoted to learning from different domains, has recently gained increasing attention from the chemometrics community, and provided important impulses for research on calibration transfer and model maintenance. Domain-invariant representation learning, that tackles distribution shift between some source and target domain by identifying latent variable-spaces where the corresponding distributions are well aligned, has recently been integrated into partial least squares modelling. However, previous methodology could not handle data situations with more than two domains. To this end we recently proposed multiple domain-invariant partial least squares (mdi-PLS) regression that allows handling multi-domain setups in multivariate calibration. In this contribution, results from the application of mdi-PLS for NIR-based dry matter content prediction of mango across cultivars, harvesting periods and ripeness levels will be presented. Our experiments show the benefit of mdi-PLS for deriving models with superior in-domain and out-of-domain generalization capability using partially labeled data, when compared against global PLS on fully labeled or di-PLS on pooled target domain data.

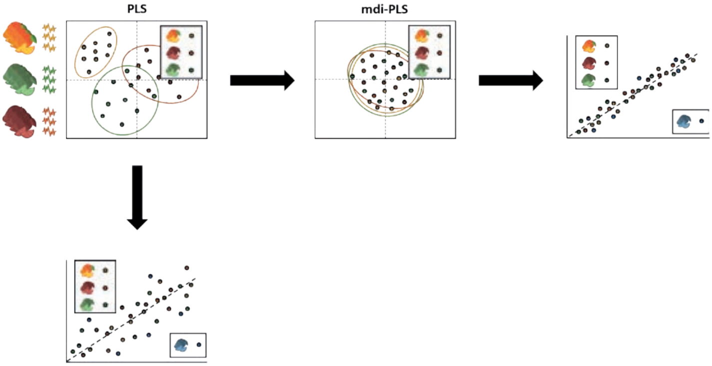


Figure 1. Multi-domain-invariant partial least squares (mdi-PLS) regression.

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003.12

WATER AS A NEAR-INFRARED SPECTRAL PROBE FOR CHEMICAL PROCESSES

Xueguang Shao[1], Wensheng Cai[1]

[1]*Research Center for Analytical Sciences, College of Chemistry, Nankai University, Tianjin, China*

Due to the flexibility and the complexity of the hydrogen bonding, the structure of water changes adaptively in different systems and during chemical or biological processes. The hydrogen bonding of water may change under perturbations, such as the variation of composition, temperature, pH and concentration. Therefore, the change of water structures can be a reflection of the composition and properties of the analyzing system. Near–infrared (NIR) spectroscopy has been employed as a powerful tool for the study of water structures. The spectral features of water structures can be distinguished by analyzing the NIR spectra of water, and the spectral changes of water under perturbations can be used to reveal the structural changes and the interactions in aqueous systems. In our works, chemometric methods were developed to enhance the resolution of the spectrum or to extract the spectral features from the NIR spectra of water. At first, a model to describe the complex water structures by the number of hydrogen bond on oxygen (the proton acceptor) and hydrogen (the proton donor) was proposed, and then Gaussian fitting was employed to investigate the spectral components in the spectral peak around 6900 cm^{-1}. Continuous wavelet transform (CWT) was used to enhance the resolution of the NIR spectrum. With the help of the method, the resolution of NIR spectrum can be significantly improved. Combining CWT with different chemometric methods, such as principal component analysis (PCA), independent component analysis (ICA), the spectral features of water structures with different hydrogen bonds were obtained. Discrete wavelet transform (DWT) and wavelet packet transform (WPT) were also employed for extracting high resolution spectral component from the NIR spectrum of water. The spectrum with very high resolution was obtained, and the spectral information of different hydrogen-bonded water can be a probe for chemical or bio-processes.

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003.13

REVOLUTIONIZING FOOD QUALITY ASSESSMENT AND TRACEABILITY WITH AI-POWERED MULTIMODE SPECTROSCOPY HANDHELD SCANNER

Fartash Vasefi[1]

[1]Safetyspect Inc., Grand Forks, United States

Seafood is often mislabeled and almost 50% of harvested fish is wasted due to the difficulty in accurately evaluating its freshness. In order to address this issue, we have developed a fast, non-destructive, and user-friendly handheld multimode spectroscopic system for fish species identification and quality assessment. This innovative system combines visible near-infrared (VIS-NIR), short wave infrared (SWIR) reflectance, and fluorescence (FL) spectroscopy to achieve superior results.

Our device has four data modes, including fluorescence with 365 nm and 395 nm excitations, diffuse reflectance in the visible range of approximately 450-950 nm, and SWIR reflectance covering around 900-1700 nm with a tungsten bulb illumination source. It is compact, battery-operated, and takes all spectral data in a fixed sequence that only takes seconds. This technology provides the ability to classify fish along four separate axes, including species identification, freshness assessment, farmed versus wild-caught, and frozen-then-thawed versus fresh.

For species identification, DNA sequencing is used as the ground truth, while the number of days since the fish was caught is used to assess its freshness. We have utilized large datasets as inputs in AI learning prediction and developed a submodel technique to identify the species of fish fillets. This model has been applied to 44 fish types, with highly accurate results.

We have also been able to predict fish freshness within +/-1 day with over 95% accuracy for four fish species. Furthermore, our observations suggest that there are promising features that can distinguish farmed from wild salmon. The results indicate that multimode spectroscopy significantly improves the accuracies of stand-alone FL, VIS-NIR, and SWIR single-mode spectroscopies across all four axes.

The AI models we have developed can be run on the device with edge computing after being trained on a cloud processor. Overall, our handheld multimode spectroscopic system has the potential to revolutionize fish quality assessment and species identification, with implications for both the seafood industry and consumers.

003.14

GRAY CLASSICAL LEAST SQUARES: HOW FAR CAN IT BE PUSHED?

Barry M Wise[1], Donal O'sullivan[1], Rasmus Bro[2]

[1]Eigenvector Research, Inc., Manson, United States, [2]University of Copenhagen, Copenhagen, Denmark

There is some resurgence in the use of Classical Least Squares (CLS) models primarily due to their interpretability. When used with spectroscopic systems that follow the Lambert-Beer law CLS models follow naturally from first principles. Unfortunately, CLS models typically do not have the predictive ability of inverse least squares (ILS) models such as Partial Least Squares (PLS) regression: the prediction error of CLS models is usually higher, and often notably so. This is largely due to non-idealities in the data of interest along with the presence of unaccounted for minor components, e.g. scatter and baseline variations. PLS models handle these situations by adding components to the model that keep the resulting regression vector orthogonal to the variations introduced by the non-idealities. Previously, we have proposed a method for developing CLS models with predictive properties competitive with ILS formulations. This is done by creating the CLS model "half-residuals" and then using these to develop pre-filters with Generalized Least Squares Weighting (GLSW) or External Parameter Orthogonalization (EPO). We refer to these as Gray CLS models. It is shown for the EPO case that these models are equivalent to Extended Least Squares (ELS). These formulations retain the opportunity to learn from the analyte factors (i.e. pure component spectra) and from the GLSW or EPO model parameters. In this work we investigate how far the Gray CLS formation might be pushed in terms of accounting for missing information, e.g. unaccounted for components and background and scatter issues.

FLASH TALK

F03.01

SYNERGY: FTNIR AND CHEMOMETRICS MULTIVARIATE FOR SMART GASOLINE BLENDING SYSTEM

Hemadevi Balasubramaniam[1], **Abdul Rahim Othman**[1]

[1]Petroliam Nasional Berhad (PETRONAS), Kuala Lumpur, Malaysia

PETRONAS has developed a uniquely fast and highly precise automated gasoline blending system which not only accelerates the release of quality certification but also achieves the perfect gasoline product blend for a Malaysian Refinery.

Blending is a critical production process, in which up to eight (8) different hydrocarbon streams from a variety of feed stocks required to be precisely blended to produce petroleum products such as Unleaded Gasoline RON 95. The ratio between flow velocity and quality of different blending component streams is of great importance, to ultimately achieve the target quality of the product. Generally, conventional blending at the refinery resulted in around 25% of re-blend and RON giveaway approximately 0.2 Octane Number which has resulted in significant amount of value leakage.

This advance, fully automated and integrated system was mooted to control and optimize gasoline blending process to achieve higher accuracy and reduce product quality giveaway. Synergy between Fourier Transform Near Infra-Red (FTNIR) analyzer and chemometrics multivariate methodology, together with blending control system paves path to incorporate predictive and prescriptive analytics. The FTNIR analyser coupled with chemometrics multivariate models is installed at site to perform online quality measurement and monitor the gasoline product quality continuously throughout the blending process, and subsequently feed the quality measurements to Blend Property Control (BPC) and Blend Ratio Controller (BRC) system to control and optimize the overall blending process. The aim is to maximize low-cost blending components and minimize high-cost blending components without jeopardizing the blended product quality to meet product specification. The use of chemometrics multivariate calibration models enables the FTNIR analyser to produce result for all the quality specifications (research octane number (RON), vapour pressure, density, olefin, benzene, oxygen, aromatics, oxygenate, initial, intermediate, and final boiling points) simultaneously in a single analysis and analyser, every 8 minutes' cycle time. The fully automated and integrated online gasoline blending system is a non-human intervention set-up, which only requires initiation from Distributed Control System (DCS) by operator.

The underlying concept of this innovation is based on the strategy of gasoline on-line blending with an intelligent and fast FTNIR/chemometrics multivariate data analytics to make necessary adjustment in the ratio of the blending components to meet the product quality. This innovation has resulted in a smarter way of gasoline blending with zero re-blend and minimal quality give away which translates to substantial value creation of millions dollar per annum.

F03.02

QUALITY PREDICTION AND MONITORING OF GASOLINE USING FTIR SPECTROSCOPY DURING BLENDING PROCESS

Melike Duvanoğlu[1], Selahattin Şentürk[1], Ayten Ekin Meşe Sezen[1],
Durmuş Özdemir[2]

[1]Turkish Petroleum Refineries Corporation, Kocaeli, Turkey, [2]İzmir Institute of Technology, İzmir, Turkey

Gasoline blending units play a crucial role in the production process of refineries, complex blending procedures that combine various light streams and additives to provide the appropriate qualities result in high-quality commercial fuels. Gasoline mixtures may include over 600 or 700 distinct molecules. Most of these molecules fall into the category of light hydrocarbons with a carbon number between 4 and 12, while there is a great deal of variation in their precise chemical composition.

In gasoline blending processes, different refinery products obtained from different hydrocarbon streams are platformate, isomarate, reformate, fluidized catalytic cracking (FCC) gasoline used in different ratios. Blending process and gasoline composition can be influenced by several variables, including the refinery's strategy for the blending, the properties of the crude oil, and the regional and seasonal specifications for the finished products. The blending process may be affected by the change in quality in the hydrocarbon production process, but it is also expected to meet certain specifications of the finished gasoline. The quality of blended gasoline is mostly determined in refinery laboratories after blending is completed and final product is obtained in product tanks. In any case of out-of-spec production, the re-blending procedure is applied, which is a waste of time and money. Hence, online quality control of gasoline blending is necessary for refineries. Standard quality controls of refinery products are performed in quality control laboratories based on conventional analysis which are time consuming and should be performed by trained personnel. As an alternative, spectroscopic techniques that allow rapid and on-site analysis are used in refinery sites to obtain product parameters.

The main purpose of this study is to determine the chemical and physical properties of gasoline throughout the blending processes using Fourier Transform Infrared Spectroscopy with multivariate calibration. Approximately 600 samples were collected over three years to include compositional variations based on the two seasonal grade changes and diverse origins of crude oils. The absorbance spectra of the petroleum samples were obtained in the range from 6000 cm^{-1} to 1000 cm^{-1} and will measure an extensive set of properties for a variety of petroleum derivative fluids. employing a quartz transmittance flow through cell with a 0.1 mm light path and a resolution of 4 cm^{-1}. Prediction results were compared with the results of traditional methods. Successful prediction performance of proposed models in this work shows FTIR spectroscopy combined with multivariate calibration techniques can be used easily and reliably to monitor quality parameters of gasoline in online blending process.

F03.03

DEVELOPING NEW MICROBIOLOGICAL TESTING APPROACHES TO EXPEDITE ANTIMICROBIAL PRODUCT DEVELOPMENT – A PROOF-OF PRINCIPLE STUDY USING A HANDHELD NIR INSTRUMENT

Rebecca Orrell-Trigg[1], Adam Kolobaric[1], Stephanie Owen[1], Daniel Cozzolino[2], James Chapman[1]

[1]School of Science, RMIT University, Melbourne, Australia, [2]Centre for Nutrition and Food Sciences, Queensland Alliance for Agriculture and Food Innovation (QAAFI), The University of Queensland, Brisbane, Australia

A rapid analytical technique to evaluate the effects of different antifungal and antibacterial is required. The aim of this study was to evaluate the ability of a handheld near infrared (NIR) instrument (Micro-NIR, VIAVI, USA) combined with chemometric methods to monitor a range of agents and nanoparticles on mixtures of E. coli (bacteria) and C. albicans (fungi). Two well established and prescribed antimicrobial agents: amoxicillin and fluconazole, and two doped ZnO nanoparticles (Fe and Co) were tested against the microorganisms in a range of microbial mixtures. The microbial mixtures of 1:1, 1:3, and 3:1 ratios E. coli : C. albicans were incubated for 24 h with the antimicrobial agents, where NIR measurements were taken at 0, 3, and 24 hr time periods.

In this study NIR spectroscopy was able to distinguish between different levels of microbial growth at specific time points. An example of this using amoxicillin and fluconazole is shown in Fig 1. This growth was monitored by changes at 1150 nm and 1400 nm that are associated with the second and third overtones of C-H stretching and O-H (1). These wavelengths are associated mainly with water (changes in dry matter associated with growth) as well as lipids. Principal component analysis (PCA) was used to interpret the trends in the data associated with the growth and antimicrobial effect. The first two principal components (PCs) explained 63% and 29% of the variance in the spectra (after second derivative and Savitzky-Golay smoothing pre-processing). The highest loadings in the PCA were observed at the same wavelengths described above. Using the handheld NIR instrument, the doped ZnO nanoparticles were confirmed to possess antimicrobial activity against the mixtures of bacteria and fungi as there was less cell growth than untreated samples. These samples presented different operational challenges to the amoxicillin and fluconazole due to the particles' tendency to adsorb the contents of the nutrient broth to their surfaces, demonstrated through UV-Visible spectroscopy. More studies will be conducted to further investigate the feasibility of this handheld NIR instrument in microbiological settings.

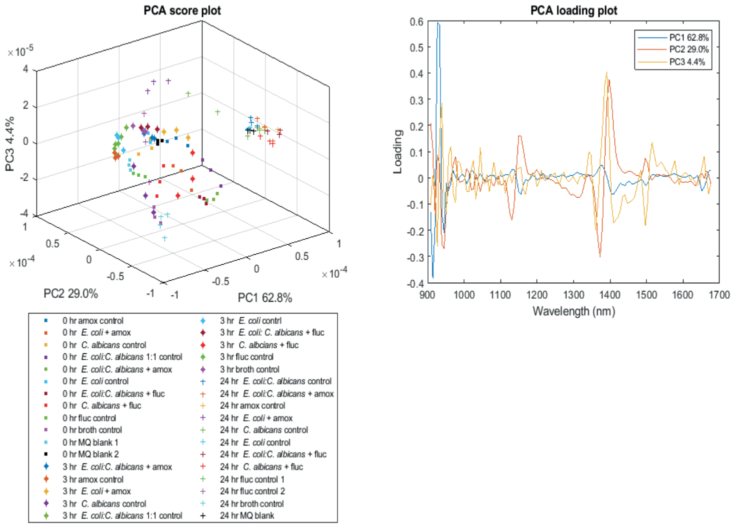

Figure 1. PCA score and loadings plots for E. coli + C. albicans 1:1 mixture with amoxicillin and fluconazole showing 0 hr, 3 hr, and 24 hr sample times.

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F03.04

INCREASING UNDERSTANDING AND ROBUSTNESS OF NIR MODELS BY DIAGNOSING THE CAGE OF COVARIANCE

Erik Tengstrand[1], Ingrid Måge[1], Lars Erik Solberg[1], Nils Kristian Afseth[1], Jens Petter Wold[1]

[1]Nofima, Ås, Norway

When NIR spectroscopy is used for quantification purposes, multivariate analysis is often used to extract information from covariances between the spectra and any given reference values. In complex samples, there is a high risk that the constituents covary with each other. In such scenarios many methods may confuse the analytes and use signal from several analytes, rather than just the analyte of interest. While this allows the method to use more signal, and thus have a better effective signal-to-noise ratio, it also makes them less robust to changes to the chemical composition in the samples. This effect has been termed the cage of covariance.

The cage of covariance limits the model robustness for new samples. By diagnosing the cage of covariance, we can understand the limitations of the model. Once we understand the limitations we can decide if they are acceptable or if the model needs to be improved. Knowing the limitations also help us understand when the model will fail, and which samples that need to be added to resolve a particular issue.
This study aims to illustrate the cage of covariance, as well as to provide methods for diagnosing it. The diagnosis methods include different ways of inspecting and comparing the covariances in the model predictions and the references, both overall in the dataset, and for the individual analytes, as well as assessing the impact on the model predictions.

F03.05

EVALUATION OF 1D CONVOLUTIONAL NEURAL NETWORK IN ESTIMATION OF MANGO DRY MATTER CONTENT

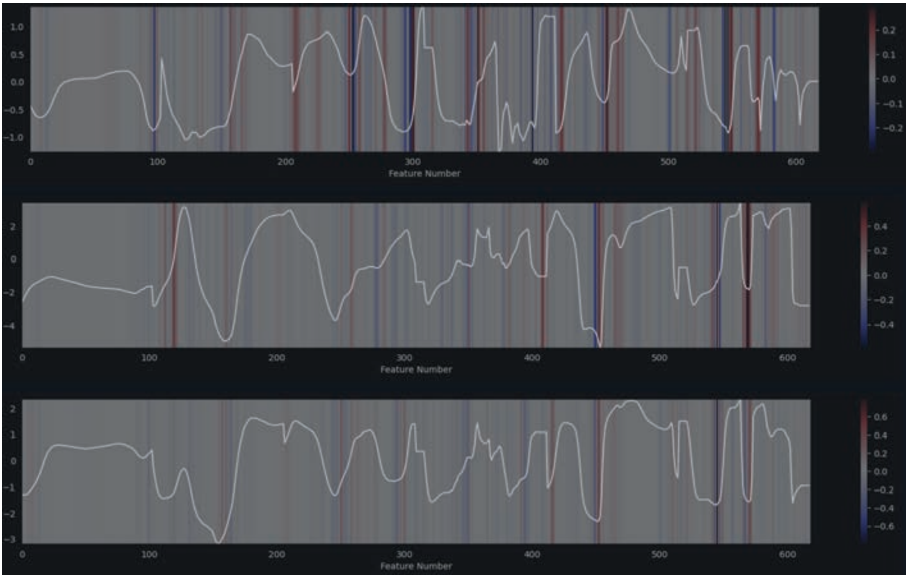
Jeremy Walsh[1]

[1]CQUniversity, Rockhampton, Australia

A large dataset of mango spectra and associated dry matter content reference values (n = 11,834) from 112 unique populations and four growing seasons was made publicly available by CQUniversity in 2020. This data set has been subsequently utilised by several research groups. With use of the suggested training and test set, direct comparison of results between studies is possible. A recent paper in JNIRS, "Review: The evolution of chemometrics coupled with near infrared spectroscopy for fruit quality evaluation. II. The rise of Convolutional Neural Networks", has compared the results of published studies, with the best results achieved using a 1D Convolutional Neural Network (CNN) model by Mishra & Passos (with RMSEP of 0.79 %FW on the independent season test set). However, these studies have also implemented other changes, including different outlier removal techniques, wavelength range and spectra pre-treatment, thus confounding model comparisons. This study aimed to 'level the playing field' in evaluation of 1D CNN modelling for this application.

Mishra and Passos developed a 1D CNN model using augmented data input (an ensemble of different pre-treatments) and identification and removal of outliers using a manual technique. The 1D CNN model was recreated, but the model was trained using the original training dataset to allow direct comparison with the results of other studies. This resulted in an increase in RMSEP to 0.823 %FW, although this result was still superior to results reported for other linear and non-linear model techniques (best result - 0.84 %FW).

To address 'black box' interpretation concerns, a Gradient-weighted Class Activation Mapping (GradCAM) technique was applied to the final layer of the 1D CNN. The augmented input data for this model consisted of raw spectra (features 1-103), standard normal variant (SNV) (104-206), Savitzky-Golay first derivative (207-309, second derivative (310-412), SNV + first derivative (413-515) and SNV + second derivative (516-618). The GradCAM output is specific to each test sample, as shown for three randomly selected spectra from the independent test below. The GradCAM result indicates that the CNN model utilised all pre-treatments, although the raw absorbance spectra appear to be the least used of the ensemble data set.



The CNN model is being further evaluated on new populations of mango fruit with spectra acquired using the same instrument as the training set and multiple new instruments.

F03.06

SPECTROSCOPIC TRACKING OF MOLECULAR CHANGES DURING WHOLE FLOUR STORAGE

Verena Wiedemair[1], Akifumi Ikehata[1]

[1]National Agricultural Research Organization (NARO), Tsukuba, Japan

Background: Whole flours are known to be rich in nutrients and fibre. Their global availability, however, is limited due to rapid rancidification, which is a complex process that includes water loss, oxidation of fats and fatty acids as well as hydrolysis of fats. The deterioration can be influenced by other molecules within the flours, and only little is known about the interactions between various molecular classes during rancidification. Therefore, a multi-method approach using spectroscopic as well as traditional chemical methods was used to further investigate whole flour rancidification.

Methods: Various whole flours of rice and millet were stored at room temperature over 8 weeks and NIR and Raman spectra as well as traditional chemicals parameters, such as e.g. protein content, fat content and antioxidant capacity, were recorded. 2D-COS was then used to uncover latent interactions between molecular classes and PCA, SVM-C and PLS-R were used to qualitatively and quantitively determine the state of rancidity of the samples.

Results: Overall, during storage the spectral intensity increased for C-H stretching vibrations, whereas it decreased for O-H stretching vibration. The fat content slightly decreased over the storage period, whereas the peroxide value and fat acidity increased. This was to be expected as fats are hydrolysed to fatty acids and glycerol during deterioration. 2D-COS analysis revealed that the OH-str. & OH def. vibration at 5180 cm^{-1} shows the strongest decrease over time and the decrease also more often than not occurs on a later stage of the deterioration process. This decrease is most likely linked to a loss of water during storage as well as the decomposition of compounds. Using SVM-C it was possible to correctly assign the stage of decomposition. Interestingly, fat content could be correlated to antioxidant capacity, which might be an indicator that both decrease during deterioration.

Lastly, PLS-R models showed good correlation for fat content and antioxidant capacity (R^2 = 0.8926 and 0.8982, respectively) and decent correlation for protein content and water content (R^2 = 0.7397 and 0.7082, respectively). Other parameters did not show good correlation in PLS-R, which is most likely due to the overall range of the parameters being quite low. It is however possible, that with more measurements a better accuracy can be achieved.

F03.07

IMPROVED PRINCIPAL COMPONENT ANALYSIS (IPCA): A NOVEL METHOD FOR QUANTITATIVE CALIBRATION TRANSFER BETWEEN DIFFERENT NEAR-INFRARED SPECTROMETERS

Hui Zhang, Lian Li, Hengchang Zang

[1]*Shandong University, Jinan, China*

Given the labor-consuming nature of model establishment, model transfer has become a considerable topic in the study of near-infrared (NIR) spectroscopy. Recently, many new algorithms have been proposed for the model transfer of spectra collected by the same types of instruments under different situations. However, in a practical scenario, we need to deal with model transfer between different types of instruments. To expand model applicability, we must develop a method that could transfer spectra acquired from different types of NIR spectrometers with different wavenumbers or absorbance. Therefore, in our study, we propose a new methodology based on improved principal component analysis (IPCA) for calibration transfer between different types of spectrometers. We adopted three datasets for method evaluation, including public pharmaceutical tablets (dataset 1), corn data (dataset 2), and the spectra of eight batches of samples acquired from the plasma ethanol precipitation process collected by FT-NIR and MicroNIR spectrometers (dataset 3). In the calibration transfer for public datasets, IPCA displayed comparable results with the classical calibration transfer method using piecewise direct standardization (PDS), indicating its obvious ability to transfer spectra collected from the same types of instruments. However, in the calibration transfer for dataset 3, our proposed IPCA method achieved a successful bi-transfer between the spectra acquired from the benchtop and micro-instruments with/without wavelength region selection. Furthermore, our proposed method enabled improvements in prediction ability rather than the degradation of the models built with original micro spectra. Therefore, our proposed method has no limitations on the spectrum for model transfer between different types of NIR instruments, thus allowing a wide application range, which could provide a supporting technology for the practical application of NIR spectroscopy.

Key words: near-infrared spectroscopy; calibration transfer; piecewise direct standardization; improved principal component analysis

P03.01

FT-NIR CHEMOMETRIC MODELS OPTIMIZATION USING BAYESIAN SEARCH

Yasser Aboelmagd[1], Ali Khater[1], Omar Khater[1], Mai Said[1], Bassem Mortada[1]

[1] *Si-Ware Systems, Cairo, Egypt*

Fourier transform near-infrared (FT-NIR) spectroscopy is a fast and non-destructive analytical technique widely applied in various sectors, such as food, pharmaceutical, and agriculture. However, the cost and complexity of traditional FT-NIR instruments limit their usability in many industries. To address this, portable and affordable FT-NIR systems like NeoSpectra scanners have been developed to make the technology more accessible. While Partial Least Squares Regression (PLSR) is a commonly used algorithm in the industry due to its simplicity and explainability, many business problems require custom PLSR models that allow for hyperparameter tuning beyond traditional options like the number of Latent Variables. The challenge lies in finding the optimal model settings in a repeatable and time-efficient manner, as more customization options are added for cross-validation, outlier removal, feature selection, spectra aggregation, and other factors. On the other hand, Bayesian search is a popular machine learning tuning algorithm that builds a regression problem on hyperparameters space for faster convergence to improved results. It efficiently explores hyperparameters space to find the optimal model settings for a given objective function, eliminating the need to try all possible combinations. It also allows for early stopping when no expected performance gain is observed. In this study, data obtained from 20 NeoSpectra scanners were used for training, while data from 7 scanners were used for testing. The data was collected in the wavelength range of 1350-2550 nm. The performance of conventional model settings optimization with grid search optimization and Bayesian search optimization was compared for several models, considering both performance and time aspects. The study findings indicate that Bayesian search optimization produced better results in terms of performance and modeling time compared to grid search optimization, with an improvement of 11.7% in RMSEP and 12.7% in RMSECV in 60% less time on average. By effectively exploring hyper parameters space and identifying near-optimal model settings, Bayesian search optimization outperformed exhaustive grid search optimization.

	Grid search				Bayesian search			
	RMSECV	R^2_{cv}	RMSEP	R^2_p	RMSECV	R^2_{cv}	RMSEP	R^2_p
Dataset 1	2.26	0.64	2.13	0.68	1.77	0.68	1.87	0.80
Dataset 2	0.79	0.70	0.88	0.83	0.67	0.78	0.73	0.84
Dataset 3	2.16	0.88	1.71	0.94	2.02	0.90	1.60	0.97
Dataset 4	1.73	0.91	2.11	0.87	1.67	0.91	1.79	0.90

P03.02

SPECTRAL VARIABLE SELECTION METHOD BASED ON SWARM INTELLIGENCE OPTIMIZATION ALGORITHMS

Xihui Bian[1], Ruoxin Wang[1], Rongling Zhang[1], Zizhen Zhao[1], Peng Liu[1]

[1]*Tiangong University, Tianjin, China*

Due to their advantages of simplicity, rapidity, and non-destructivity, spectroscopic analysis techniques combined with chemometrics have been widely used for qualitative and quantitative analysis in food, medicine, petrochemical, etc. Whereas, the spectra often contain hundreds or thousands of variables, some of which are not related to the target, which reduces the accuracy of the model predictions. Therefore, it is important to select related variables before modeling. Many variable selection methods have been proposed[1], such as competitive adaptive reweighted sampling (CARS), Monte Carlo uninformed variable elimination (MC-UVE), and randomization test (RT). However, the variable selection is a typical combinatorial optimization (NP) problem, new method is still needed to promote the calculation efficiency. Because of their powerful search ability, simple structure, and efficiency, swarm intelligence (SI) algorithms have gradually attracted attention in the spectral variable selection.

We summarized 40 SI algorithms and classifies them into five categories based on their principles, as shown in Fig. 1. Among these 40 SI algorithms, 17 of them have been applied for variable selection. These SI variable selection algorithms all contain four parts: initialization, fitting function, discretization and iteration. In the initialization phase, they all include population size, dimension, and iteration number. However, each algorithm differs in the parameters, fitting functions, and discretization functions. Sigmoid functions, V-functions, Linear functions, Trigonometric functions, Logistic functions and Arc-tangent functions have been used as discretization functions. Further exploration of more optimization algorithms for variable selection is still needed. Our group introduced firefly algorithm[2], grey wolf optimizer[3] and whale optimization algorithm[4] for spectral variable selection of complex samples.

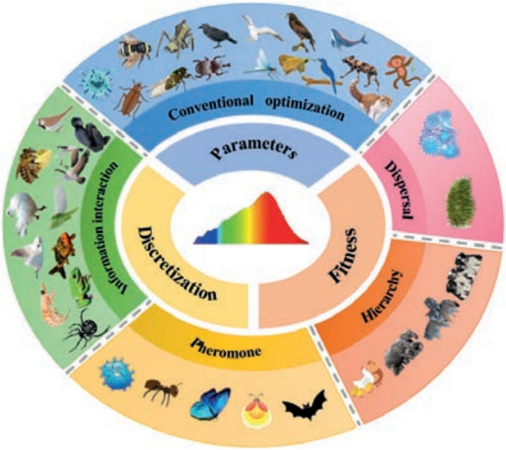


Figure. 1 The summarized 40 swarm intelligence algorithms.

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P03.03

BINDING MECHANISM OF POLYPROLINE TO ICE REVEALED BY MOLECULAR SIMULATIONS

Wensheng Cai[1], Li Han[1], Haipeng Wang[1], Xueguang Shao[1]

[1]Research Center for Analytical Sciences, College of Chemistry, Nankai University, Tianjin, China

Polyproline (PPro) can be considered as a minimal antifreeze glycoprotein (AFGP) mimic, with excellent ice recrystallization inhibition activity. Compared with AFGP, PPro has the advantages of easy access and cell safety, and is becoming a potential cell cryoprotectant. However, the mechanism by which PPro inhibits ice growth has not been reported. To this end, we investigate the structural change of water during ice growth in PPro solutions using near-infrared spectroscopy, which is highly sensitive to water. With the help of chemometric methods, high resolution spectra are obtained, from which the spectral feature of the interfacial water between ice crystal and PPro can be observed. Molecular dynamics (MD) simulations were performed for the ice-water system with different PPro concentrations. It is evident that ice growth is PPro exhibits effective antifreeze activity only at high concentration, consistent with the NIR experiment. Moreover, the analysis of the MD results suggests that the antifreeze activity shown by PPro at high concentration is associated with the stable presence of the interfacial water in the contact area between PPro and ice. This interfacial water is further revealed to have an ice-like structure through which the hydrophobic groups of PPro are adsorbed to the ice. Most importantly, the results of our MD simulations provide clear evidence that the stable presence of this interfacial water is essential for the antifreeze activity of PPro, rationalizing the NIR spectra of ice growth in PPro solutions. We proposed that during the ice growth stage the liquid water molecules in the contact area between PPro and ice are constantly replaced by the ice-like molecules due to the de-solvation of the methylene groups of PPro, leading to the binding of the methylene groups to the ice-growth surface and thus inhibiting the ice growth. The new insight into the antifreeze mechanism of PPro may provide guidance for the design of new antifreeze materials.

P03.04

EFFECTS OF SAMPLE GRINDING IN NIR SPECTROSCOPY

Máté Csontos[1,2], János Elek[1], Zalán Attila Vincze[2]

[1]Science Port Ltd., Karcag, Hungary, [2]University of Debrecen, Department of Physical Chemistry, Debrecen, Hungary

The spectroscopic analysis of solid samples often requires physical sample preparation steps: one of these is grinding. Several demands shall be met for the successful outcome of the process, the particle size of the ground sample should be small enough and homogenously distributed, the grinding should be reproducible even between laboratories. These properties are crucial in order to minimize the undesired physical thus spectral differences between samples caused by the light scattering. Moreover the particle size also affects the homogeneity obtained in the various sample pre-treatment steps; for instance mixing and drying.
A common type of these instruments is the blade grinder, where the only controlled parameter is the process length: the longer grinding - in theory - the smaller the particle size. In this study the effect of the grinding time was investigated of two commercial blade grinders in order to develop a standardized grinding method for pelleted diets. The NIR spectra of the same type but differently ground diet samples were collected and analysed using PCA in order to investigate how the grinding time affects the particle size and homogeneity.

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P03.05

OPTIMIZING FEED QUALITY PREDICTION WITH LOCAL PARTIAL LEAST SQUARES REGRESSION

Antoine Deryck[1], Vincent Baeten[1], Philippe Vermeulen[1],
Juan Antonio Fernández Pierna[1]

[1]Walloon Agricultural Research Centre, Gembloux, Belgium

Feed characterization is critical for optimal growth and health of production animals, but the variation in nutrient concentrations in feed products poses a significant challenge. Near-infrared spectroscopy (NIRS) combined with chemometrics appears to be a promising, rapid, and non-destructive technique for the reliable prediction of forage composition. Our goal is to develop more accurate and efficient NIRS-based predictive models for key parameters describing the quality of fresh green fodders and silages.

To achieve this goal, NIR spectra of four agricultural feed products (fresh green fodders, pre-wilted grass silage, direct-cut grass silage, and whole maize plant silage) were measured with the Aurora NIR spectrometer. Classically global partial least squares (PLS) techniques are used for the creation of predictive models for such products. But because they fail to account for local variability, they may be limited in accuracy and reliability. We thus investigate published local PLS methods to address this issue and improve feed characterization with NIRS.

Predictive models were developed for eight parameters characterizing the feed products' quality. In addition to a global PLS, four local PLS methods were tested: the local PLS regression, the local PLS regression on global PLS scores, the locally weighted PLS regression, and the locally weighted PLS regression on global PLS scores. This study explores the performances of those PLS techniques for feed characterization.

P03.06

IMPROVED NIR CALIBRATIONS ROBUSTNESS USING STACKED MODELING

Adham Hamed[1], Ashar Seif[2], Omar Khater[2], Mai Said[2], Bassem Mortada[2], Yasser Sabry[2]

[1]Si-ware Inc., Menlo Park, United States, [2]Si-Ware Systems, Cairo, Egypt

Near infrared (NIR) spectroscopy subjected to chemometric methods has been recognized as a powerful analytical technique to investigate and elucidate the compositional characteristics of organic products. Partial Least Squares (PLS) is a commonly used regression method in Chemometrics to predict the components (response targets y) of the chemical composition of vegetal materials. This method can be optimized using various preprocessing techniques and settings, such as the number of latent variables (LVs), to achieve the best performance. One difficulty is to determine the optimal settings of the PLS model that works with different substances or samples. Further, some modeling choices are improving some metrics while worsens others. To address these challenges, a stacked machine learning model is proposed to increase the model robustness. The stacked model averages the predictions of multiple PLS models with different settings (e.g., cross validation, outlier removal algorithms, and preprocessing methods). This approach reduces the manual effort needed for model selection, improves prediction accuracy, and increases the model immunity against both data and concept drifts. The effectiveness of the proposed stacking algorithm is demonstrated on 10 NIR feed materials datasets collected using 48 NeoSpectra scanners at different conditions and by measuring many substances.

The results in Figure 1, show the improvement of predictions based on averaging PLS technique compared to selecting the best PLS model for each individual dataset, where a significant reduction is remarked in predictions RMSE by 21% on average. In addition, an average improvement in Residual Prediction Deviation RPD, mean relative error and R-squared is achieved across the 10 datasets by 26%, 20% and 33% respectively. Therefore, we can conclude that PLS stacked model offers a reliable Chemometrics tool for analyzing NIR spectroscopy data.

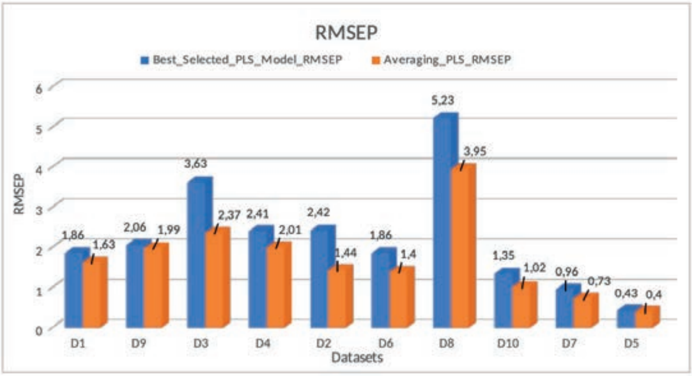

Figure 1. Predictions RMSE for the built models of different datasets.

P03.07

ASSESSING HISTORICAL PAPERS WITH NIR SPECTROSCOPY AND CHEMOMETRICS

Hajar Khaliliyan[1], Florian Bausch[1], Justine Lin[1], Otgontuul Tsetsgee[1], Manuela Mancini[2], Åsmund Rinnan[3], Antje Potthast[1], Stefan Böhmdorfer[1]

[1]University of Natural Resources and Life Sciences, Vienna, Tulln, Austria, [2]Department of Agricultural, Food and Environmental Sciences, Università Politecnica delle Marche, Ancona, Italy, [3]Department of Food Science, Faculty of Science, University of Copenhagen, Frederiksberg C, Denmark

Introduction
Historical papers and papyri were used for documentation and communicating for centuries. A non-destructive analysis of these contributes to a better understanding of them, in several ways. Papyrus characterisation and classification can facilitate the assignment of fragments with unknown background. Rag papers were produced from used clothes that contained various fibre types. A classification of these all papers could be performed by tedious wet chemical analysis, or more rapidly by using spectroscopy and appropriate mathematical modelling. Furthermore, there is a need to estimate the date of the document and assess what variables that can affect the measurement itself.
Methods
NIR spectra of the papyri, rag, and book samples were recorded with Bruker MPA. Matlab with PLS Toolbox were used for data processing and modelling of partial least squares discriminant analysis (PLS-DA) and PLS. A total of 89 papyrus samples were collected with the aim to develop a classification model. In addition, we investigated the hemicellulose composition of a collection of 165 rag papers with methanolysis. Regarding dating historical paper, we recorded NIR spectra of 3 series of journals from different countries, dating from 1874 to 1963 (234 books).
Results
Subsets of papyri could be identified by PLS-DA using six latent variables and 1st derivative pre-processing (Figure 1, left). For rags, methanolysis was used to determine the fraction of monomer hemicellulose. Subsequently, PLS models were developed from NIR spectra to assess this hemicellulose content non-destructively (Figure 1, center). To predict the age of more recent papers, a nested-ASCA method over a period of 78 years was built to investigate the main sources of variability of the samples. The dominant parameter was found to be the country of origin (Figure 1, right).
Conclusions
In summary, the combination of NIR with chemometrics is beneficial for quickly studying many different types of historical papers. In the papyri context, qualitative analysis like PLS-DA can provide a quick and objective way to confirm the identity of a sample. For rags, the hemicellulose composition can be assessed non-destructively. And finally, it is possible to date printed matter across almost one century with NIR spectroscopy and PLS.

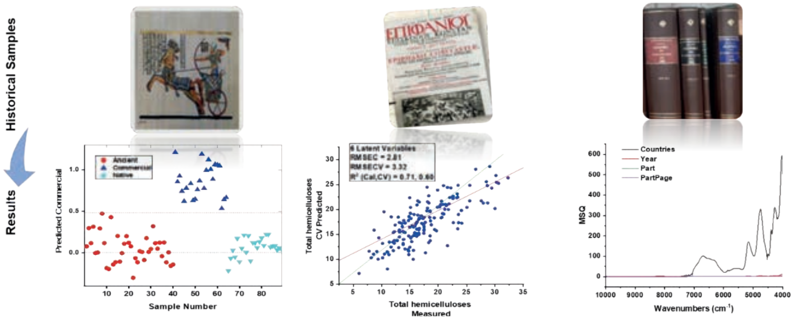

Figure 1. Historical papers Characterization by NIR and Chemometrics

P03.08

FIVE DIFFERENT SPECTRA PER SAMPLE; MULTISPECTRAL ANALYSIS OF A COLLECTION OF SOLID GEOLOGICAL SPECIMENS. CAN DIFFERENT SPECTRA HELP EACH OTHER AND HOW?

Johan Linderholm[1], Paul Geladi[2], Beatriz Galindo-Prieto[3]

[1]Umeå University, Umeå, Sweden, [2]Swedish University of Agricultural sciences, Umeå, Sweden, [3]([1]) Environmental Research Group, School of Public Health, Faculty of Medicine, Imperial College London ([2]) MRC Centre for Environment and Health, School of Public Health, Faculty of Medicine, Imperial College London, London, United Kingdom

Aims and Questions

The aim of this study is twofold:

1. Can vibrational spectroscopic methods analyse inorganic matter?
2. In what way can complementary spectroscopic methods help each other with interpretations?

Material

The analysed material consists of a collection of 76 Scandinavian bedrocks specimens with relevance for geology and archaeology. Magmatic, sedimentary and metamorphic samples have been included, reflecting various base minerals.

Instrumentation

All instrumentations are field adapted. The NIR spectrometers used, both with contact probes and internal illumination.

a) ASD LabSpec4 (350:1:2500 nm).
b) MicroNIR (908:6:1676 nm).
c) Bruker BRAVO FT Raman (dual laser 785 and 852 nm, 300:2:3200 cm^{-1}).
d) FT iRaman (1064 nm, 98:4:2507 cm^{-1}).
e) Thermo Scientific Niton XL5 Plus EDXRF.

Sampling aspects

All five probes have different diameters. Five replicate measurements were taken for obtaining a better average spectrum. The average of the replicates was used as a reproducible spectrum for each specimen.

Statistics/chemometrics

The data obtained consists of five matrices (from a-e instrumentations) with 76 objects and 2151 (a) 125 (b), 1451 (c) 466 (d) and 42(e) variables. This allows multiple ways of analysing the data, each block by itself or blocks combined in pairs or as multiblock approach.

Results

The X blocks (two NIR and two Raman) were preprocessed by SNV and mean-centering, the Y block (XRF) was pareto scaled and mean-centered, and three outliers were removed. An unsupervised multiblock PLS regression model [1, 2] was generated using a full design matrix to explore all possible relationships between the datasets (figure 1). A HPCA model, as well as several statistics and visualizations, were used to evaluate the results, infer the versatility of the experimental spectral techniques, and classify the geological samples according to different categorical variables such as base mineral or basic geology.

Conclusion

Vibrational spectroscopy can be made useful for inorganic materials. Proper sampling is essential as heterogeneity is a complicated matter compared to easily homogenized or extracted organic materials that are usually measured by NIR. For field measurements, illumination, avoiding stray light and field of view are all aspects that must be considered. The preliminary results of the multiblock model show that the spectroscopic methods complement each other to give a complete view of the existent relationships and improved sample classification.

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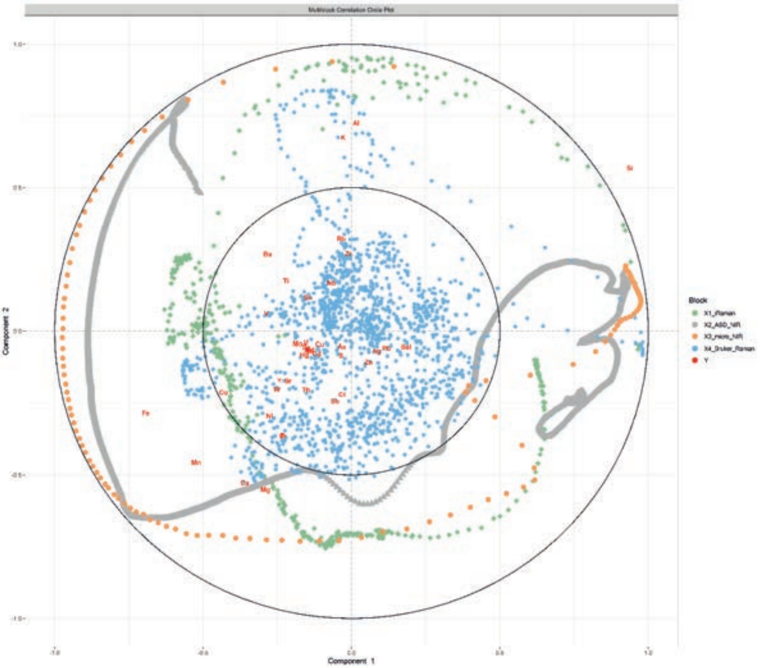

Figure 1. A correlation circle plot of the multiblock model.

P03.09

NIR-SPECTROSCOPY APPLIED TO CONTEMPORARY ART ANALYSIS

Michal Oravec[1], Katarína Kučíková[2], Lukáš Gál[2], Veronika Gabčová[1],
Katarína Vizárová[2]

[1]Slovak National Gallery, Bratislava, Slovak Republic, [2]Faculty of Chemical and Food Technology, Slovak University of Technology in Bratislava, Bratislava, Slovak Republic

The NIR spectroscopy is relatively fast available and nondestructive method used in the variety of research and industrial applications. Due to its benefits, this technique is also getting used in the field of cultural heritage for material identification.

Interesting challenge in this field is the scientific examination of the contemporary art (CA). CA are today's masterpieces, created in the second half of the 20th or the 21st century. The 20th century was typical for increasing the number of heritage objects in galleries, museums, and archive collections containing plastics e.g., PS, PMMA, ABS, PVC, PA, PP, PE, etc. [1 – 3]. A ubiquitous situation characteristic encountered by restaurateurs, conservators or curators is the evident degradation of visual and mechanical properties CA objects. The evaluation of their condition is necessary for the proper decision making to apply right prevention. Therefore, it is crucial to study galleries collections leading to their material identification for better understanding of chemical changes. This can be realized by means of microinvasive or noninvasive approaches, which can accurately describe potential behavior, CA objects state, and life expectancy [1 – 5].

NIR techniques is very perspective in combination of multivariate statistical methods as Principal Component Analysis (PCA) method [5]. As the consequence, this approach is also often used in the plastic material identification.

In this work, more than twenty selected CA objects of the Slovak National Gallery collections were examined. For this analysis, we used portable NIR, UV-Vis NIR, Raman and ATR-FTIR spectroscopy devices. Raman with 532, 785 and 1064 nm and ATR-FTIR were primarily used to identify unknown plastic materials. The NIR and UV-Vis-NIR spectra were embedded into software The Unscrambler X to apply pre-processing methods and to build PCA models. Using this combination, the best identified seem to be materials as PMMA, and PVC.

Reference:

[1] Shashoua, Y. Conservation of plastics: materials science, degradation and preservation, Butterworth Heinemann, Oxford, 2008.
[2] Rabek, J. F. Photodegradation of polymers: Physical characteristics and applications, Springer, London, 1996.
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[4] Klempová, S., Oravec, M., Vizárová, K., Analysis of thermally and UV–Vis aged plasticized PVC using UV–Vis, ATR-FTIR and Raman spectroscopy, Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, Volume 294, 2023, 122541, ISSN 1386-1425, https://doi.org/10.1016/j.saa.2023.122541.

[5] Cséfalvayová, L., Strlič, M., Karjalainen, H., Quantitative NIR chemical imaging in heritage science. Anal. Chem. 83, 5101 (2011).

Acknowledgment:
This work was supported by the Slovak Research and Development Agency under the Contract no. APVV-20-0410.

P03.10

CHANGEABLE MOVING WINDOW-STANDARD NORMAL VARIABLE METHOD FOR VIS-NIR SPECTROSCOPY

Jiarui Lin[1], Kunping Chi[1], Min Chen[1], Junjie Chen[1], Yiming Chen[1], **Tao Pan**[1]

[1]Jinan University, Guangzhou, China

Standard normal variable (SNV) is a common spectral preprocessing method. However, for a wide range of spectral region, the assumption of SNV about global linear correlation between the measured and ideal spectra is difficult to satisfy, and the modeling effect of the global SNV is not ideal. The literature (Anal Chimica Acta, 909: 30-40, 2016) proposed the method of equal segmentation SNV (ES-SNV) based on piecewise linear, which achieved obviously better prediction effect than the global SNV. Due to algorithm limitations, ES-SNV spectra exhibited artificial discontinuities that did not match the continuous distribution rule of molecular absorption. Bases on the point-by-point local linear, changeable moving window-standard normal variate (CMW-SNV) was proposed, which maintained the spectral continuous features. CMW-SNV used the SNV factors of the spectral window of 2p+1 wavelengths to correct the absorbance of the central wavelength; the full wavelengths' correction was realized by moving-window; and the window size p was optimized based on modeling effect. Three Vis-NIR analysis examples were used to validate the CMW-SNV method, including quantification of soil organic matter and corn flour moisture, and discrimination of rice seeds.

Vis-NIR quantification of soil organic matter: SNV-PLS, ES-SNV-PLS, and CMW-SNV-PLS models were established; compared with SNV-PLS, CMW-SNV-PLS has a 26% decrease in SEP, and slightly better than ES-SNV-PLS. NIR quantification of corn meal moisture: compared with SNV-PLS, CMW-SNV-PLS has a 6.5% decrease in SEP, and slightly better than ES-SNV-PLS. Vis-NIR discriminant of rice seeds identification: the models of SNV-PLS-DA, ES-SNV-PLS-DA, CMW-SNV-PLS-DA were established; CMW-SNV-PLS has a 2.3% improvement in total recognition accuracy rate (RARTotal) compared with SNV-PLS, and slightly better than ES-SNV-PLS-DA.

This paper proposed the CMW-SNV method based on point-wise local linear, which maintained spectral continuity. In three examples of quantitative and qualitative NIR analysis, the CMW-SNV achieved significantly better than global SNV and slightly better than ES-SNV, which is an effective improvement of traditional SNV.

P03.12

CALIBRATION TRANSFER BETWEEN DIFFERENT SPECTROMETERS USING WAVELENGTH CORRESPONDENCE

Erik Tengstrand[1], Tiril Lintvedt[1], Lars Erik Solberg[1], Katinka Dankel[1],
Nils Kristian Afseth[1], Jens Petter Wold[1]

[1]*Nofima, Ås, Norway*

There is an increasing number of datasets available for download on the internet. Combined with calibration transfer, this opens a new possibility in NIR spectroscopy: to calibrate a spectrometer without creating a calibration set. This removes one of the largest disadvantages of NIR spectroscopy, the costly and time-consuming calibration. A few samples are still required for tuning and validation, but that number is much smaller than what is usually required for a full calibration. Most traditional calibration transfer methods require samples that have been measured on both spectrometers, and that the spectrometers are similar. To make full use of the advantage of shared dataset, our calibration transfer methods will need to work without using samples with spectra acquired on both spectrometers and be able to handle very different spectrometers. Here we present a calibration transfer method able to do this, as well as a few examples where it has been applied.

The calibration transfer method works in three steps: first, the wavelength correspondence is matched between the spectrometers. In the examples used here, the wavelengths supplied by the spectrometer manufacturer have been used. Other methods are available if those are not sufficiently accurate. Second, a PLS model is trained using data from the source spectrometer using a few spectra from the target spectrometer to select the number of components. Third, the predictions in the target spectrometer are slope-and-bias corrected. A few samples and references are required for selecting the number of PLS components and doing the slope and offset correction, but much fewer samples than required for a full calibration.

This kind of calibration transfer is an important step in sharing data: it enables user to benefit from shared data. Ideally, any field with the potential to share data should have methods for using that shared data. Those methods should also be simple and robust so to make the data sharing easy on both sides, both the sender and receiver should be able to participate in the sharing easily, regardless of instrumentation and scenario. Any data available should be sharable because some user may benefit from it, even if it is incomplete.

P03.13

CRAWLSPEC: CENTRALISED REFERENCE ANALYTICAL WEB-ACCESSIBLE LIBRARY OF SPECTRA

Philippe Vermeulen[1], Maxime Joissains[1], Audrey Pissard[1],
Juan Antonio Fernandez Pierna[1], Vincent Baeten[1]

[1]Cra-w, Gembloux, Belgium

In order to centralise and facilitate access to spectral data derived from vibrational spectroscopy devices and to the corresponding metadata, the CRA-W is developing an original solution called CRAWLSPEC (Centralised Reference Analytical Web-accessible Library of SPECtra).

CRAWLSPEC brings together the tools necessary for the supply and use of the database by focusing on 4 main features: centralisation, integration, extraction and exploitation of the data. The initial step is the introduction of semantics to identify the different elements included in the database: data identifiers, product categories, and qualitative and quantitative analytical parameters. Using an MS Excel file structure, qualitative and quantitative reference data generated by different laboratories and experimental units, both internal and external to CRA-W, can be centralised on a project basis.

All spectral data files acquired in the laboratory or on site are stored in a proprietary format, in a tree diagram established according to instrument type, product category and project. On this basis, a local software (CRAWLSPEC Manager) integrates the information into a centralised and secure database and confirms its integrity.

Another functionality offers a web application (CRAWLSPEC Explorer), designed to send queries to the database. By accessing this simple interface, users can extract spectral data and corresponding metadata. This can be done following standardised queries relating to a product category, a project or a batch of samples. The results are exported in different file formats according to the user's specifications.

CRAWLSPEC allows to extend the exploitation of data for the development of calibration and discrimination models. This spectral data management tool will support the agricultural and food sectors in the implementation of Industry 4.0. It will also help to share expertise in the field of optical sensors and modelling.

ACKNOWLEDGEMENTS

The CRAWLSPEC tool has received funding from the Walloon Region – Mobilab and ValCerWal projects. The authors would also like to thank the CRA-W teams of the Research Unit "Quality and authentication of agricultural products" for having contributed to the development of this application.

P03.14

CONSIDERATIONS AND TOOLS FOR FINAL MODEL SELECTION

Barry M Wise[1], Lyle W Lawrence[1], Robert T Roginski[1], Manuel A Palacios[1]

[1]*Eigenvector Research, Inc., Manson, United States*

Final model selection can be an overwhelming endeavor. The combination of model types, meta-parameters, preprocessing methods, and variable selection lead to a large number of models to consider for deployment. It is useful to consider both the predictive performance of the model and the degree of overfitting. One possibility is to plot $R^2 - Q^2$ versus Q^2 (where Q^2 is the cross-validation equivalent of the calibration R^2). One problem with this plot is that, unlike the root-mean square error of calibration and cross-validation metrics (RMSEC and RMSECV), it is not in the units of the variable to be predicted, thus it is sometimes hard to assess whether the model is fit for purpose. Furthermore, it has a non-linear relationship with these metrics. Here we present an alternative plot which can aid in model selection, which is to plot the ratio of RMSECV/RMSEC versus RMSECV. This plot makes it easy to find models that are not overfit and still have a small error of cross-validation. We also consider adding contours to the plot to aid in finding the model which is “closest” to the perfect model, which would be a model with RMSECV equal to the reference error and no overfitting, i.e. RMSECV/RMSEC = 1. Additional plots to assess model robustness are also considered. Models can be tested to shifts in the wavelength axis and to synthetic interferents. This shows how much the model is sensitive to an unstable instrument or to new minor components in the test samples. Finally, the possibility of using an ensemble of models with similar metrics is considered.

P03.15

APPLICATION OF NIRS IN QUALITATIVE DISCRIMINATION OF DIFFERENT ORIGINS OF TIBETAN YINCHEN

Mengqi Zhang[1], Hengchang Zang[1]

[1]Shandong University, Shandong, China

Tibetan Yinchen (TYC) has a miraculous effect on the treatment of hepatitis diseases because of its special high altitude growing environment. Quality and efficacy are highly correlated with its origin, altitude and other environments. However, traditional Chinese medicine (TCM) quality control tools are difficult to be applied to determine the quality, and herb sellers can usually only judge the origin by experience. NIRS (Near Infrared Spectroscopy) technique is mostly used for quality analysis and control of food, TCM and tobacco in recent years because of its fast, non-destructive and green.

In this study, a rapid discrimination model was established to distinguish the origin from Qinghai Province and Tibet Autonomous Region. Firstly, three NIRS characteristic peaks at 5160 cm^{-1}, 4720 cm^{-1}, and 4270 cm^{-1} were identified to describe the changes of molecular structure information under the TYC origins influence. The difference in molecular structure information was consistent with the result in the concentration of active components. Secondly, the model established by mixing multi-factor variables was more suitable for TCM market to achieve the rapid identification of TYC of different origins. Finally, different modeling strategies, such as PCA, LDA, PLS-DA, SVM, etc., were compared to select the most suitable modeling strategy. The calculation method of CRR (Correct Recognition Rate) was optimized by setting threshold (Td, absolute value of difference between predicted and true values) and applied in the TYC discriminant model. The method could reflect the accuracy of the model prediction results, in other words, the model accuracy was higher when Td was lower. The results showed that the model of PLS-DA was the most effective with Td in 0.4, which could quickly identify TYC in Qinghai Province and Tibet Autonomous Region. In conclusion, these results demonstrated that it was feasible to understand the influence of origin on the quality of TYC through concentration of active components and characteristic groups. These could also lay the foundation for quickly distinction of TYC in Qinghai Province and Tibet Autonomous Region.

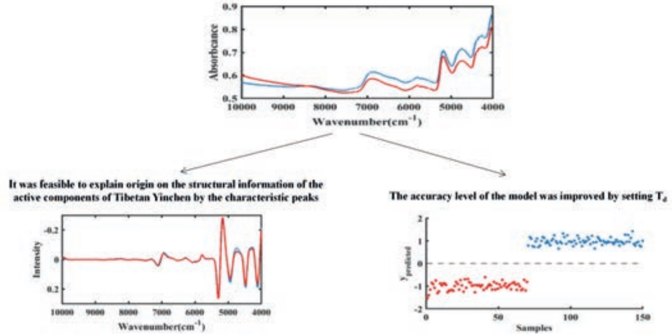


Figure 1. Graphical abstract

P03.16

COMPARISON OF SPECTROSCOPIC METHODS TO DETERMINE PHYSICAL PROPERTIES OF LIGHT DIESEL SAMPLES IN REFINERY PRODUCTION USING MULTIVARIATE CALIBRATION METHODS

Ayten Ekin Meşe Sezen[1,2], Durmuş Özdemir[2]

[1]R&D Department, Turkish Petroleum Refineries Corporation, 41780, Körfez, Kocaeli, Turkey, [2]İzmir Institute of Technology, Faculty of Science, Department of Chemistry, Gulbahce Campus, 35430, Urla, İzmir, Turkey

Crude distillation units are the first processing units of crude oil. It works as a fractional distillation column and under atmospheric condition petroleum fractions are obtained according to boiling point differences. Each product is then sent to their respective units and undergoes several other refining processes to obtain the final product that can be used in engines. Production is carried out according to certain specifications and quick respond should be made in case of any malfunction or out of control processes. The quality of obtained petroleum fractions are strictly observed in refinery laboratories according to their respective American Society for Testing and Materials (ASTM) or standards. Standard analyses are mainly based on classical analytical methods, and they are time consuming, costly, and mostly require training personnel.

In this study, three different spectroscopic methods which are FTIR-ATR, FT-NIR and UV-Vis spectroscopy were performed and compared. Obtained spectral data were treated with different data preprocessing techniques and by the help of two different chemometric approaches, which are Partial Least Squares (PLS) and Genetic Inverse Least Squares (GILS), multivariate calibration models were developed.

The best combination of multivariate calibration technique and spectroscopic method were aimed to be found for determination of physical parameters of light diesel samples, which are distillation temperatures, API gravity and flash point temperatures. Each developed model is then compared with each other as well as reproducibility value of standard analysis method. Study shows that multivariate calibration techniques combined with vibrational spectroscopy, FT-NIR and FTIR-ATR, results in low prediction errors for most parameters. GILS algorithm showed better predictive ability compared to PLS algorithm for low distillation temperatures. FT-NIR spectroscopy combined with PLS algorithm were selected for API gravity models while for flash point determination UV-Vis spectroscopy combined with GILS algorithm resulted in lowest prediction errors.

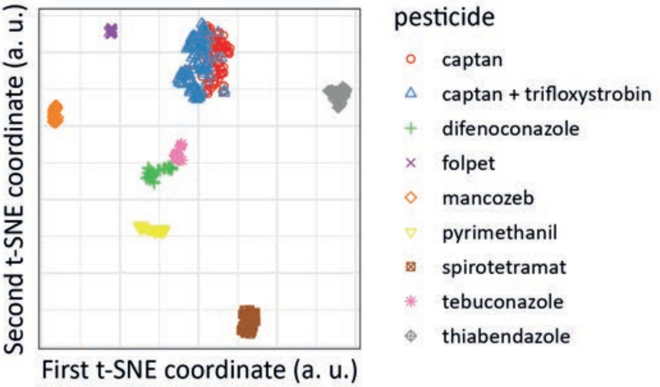
P03.17

COMBINING PCA WITH THE VISUALIZATION METHOD T-SNE FOR THE ANALYSIS OF DATA FROM NEAR-INFRARED SPECTROSCOPY

Antoine Deryck[1], François Stevens[1], Vincent Baeten[1],
Juan Antonio Fernández Pierna[1]

[1]*Walloon Agricultural Research Centre, Gembloux, Belgium*

The t-distributed stochastic neighbor embedding algorithm or t-SNE is a non-linear dimension reduction method used to visualize multivariate data. It enables a high-dimensional dataset, such as a set of infrared spectra, to be represented on a single, typically two-dimensional graph, revealing its local and global structure. t-SNE is a very popular in the machine learning community and has been applied in many fields, generally with the aim of visualizing large datasets. In NIRS, t-SNE is gaining notoriety but principal component analysis (PCA) remains by far the reference method for exploratory analysis and dimension reduction. However, t-SNE may represent a real aid in the analysis of NIRS datasets. It provides an at-a-glance global view of the dataset allowing to distinguish the main factors influencing the spectral signal and the hierarchy between these factors, and gives an indication on the possibility of performing predictive modelling. It can also provide great support in the choice of the pre-processing, by comparing rapidly different general pre-processing approaches according to their effect on the variable of interest. Here we propose to illustrate these advantages using different datasets. We also propose an approach based on a synergy between the t-SNE and PCA methods, allowing respective advantages of each to be exploited. In concrete terms, a PCA is first applied with a number of principal components large enough to ensure that all relevant variability is captured by the model. Then, the user is given the choice of excluding one or a small number of principal components suspected of masking the relevant variability. t-SNE is then applied to the scores of the other principal components. Doing this, the new t-SNE map focuses on specific sources of variability is able to more clearly display the potential influence of relevant factors.



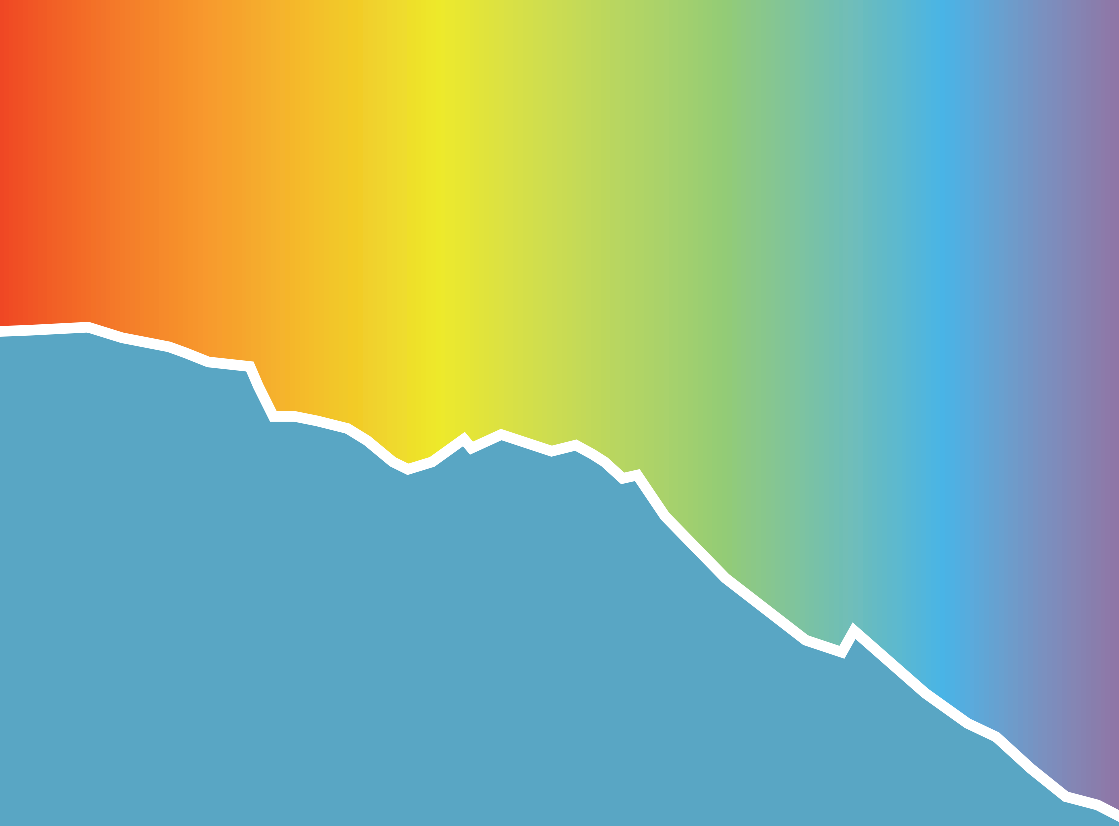
P03.18

COMPARISON OF PREDICTIVE MODEL ROBUSTNESS FOR INTACT MANGO FRUIT DRY MATTER CONTENT

Clinton Hayes[1], Kerry Walsh[1], Nicholas Anderson[1], Ryan Lerud[2]

[1]Central Queensland University, Rockhampton, Australia, [2]Portland State University, Portland, United States of America

The Derivative Quotient Method (DQM), Partial Least Squares Regression (PLSR) and Artificial Neural Networks (ANN) were compared in the estimation of dry matter content of intact mango fruit from short wave near infrared spectra, collected using an interactance geometry. Models were developed on a data set collected across three seasons (n = 10,243), with subset of 744 samples were scanned at three fruit temperatures (15, 25, 35 °C), and tested on that of a fourth season (n = 1,448), with the view for robustness across season, location and cultivar for a NIRS model for intact mango fruit dry matter content. DQM is a univariate regression method first developed by Karl Norris for the optimization of calibrations with Gap-Smooth derivatives, and implemented into MATLAB code by Dave Hopkins.. The Gap-Smooth derivative intervals are optimized for each center wavelength. A 2-term (i.e., 4 center wavelengths) model using Gap-Smooth derivatives was compared to a PLSR model utilizing an equivalent absorbance spectral region. ANN models were developed using the input of either Savitsky-Golay second derivative based on a second order polynomial with a 17 spectra window or scores of LVs of a PLSR model based on this pre-treatment of spectra. For short wave NIR based estimation of Dry Matter Content (%w/fw, DMC) of intact mangoes, the models resulted in calibrations with an R^2 values of 0.84 – 0.89 (on a population $\sigma = 2.4$ %).



THEME 04

CONSUMABLES AND LIFE-STYLE PRODUCTS

ORAL PRESENTATION: O04.01 – O04.02
POSTER PRESENTATION: P04.01 – P04.03

004.01

HIGH-PERFORMANCE MINIATURIZED NIR SPECTROMETERS FOR VARIOUS APPLICATIONS

Robert Brückner[1]

[1]Senorics Gmbh, Dresden, Germany

Senorics develops and distributes novel highly integrated spectrometers in the near infrared spectral region. The innovation is based on organic electronics as it is well known for OLED displays which conquered the consumer markets recently.
We demonstrate the latest developments for high-performance NIR sensors used in agricultural applications where extremely high precision is desired. On the other hand Senorics' sensors are implemented into hand-held devices equipped with a mobile app and various application models e.g. textile identification and quantification. The emerging field of drug testing and quality assurance in the medical Cannabis market, requires reliable analytical solution for the professional and consumer markets all over the world. We will show some recent advances in that field. Furthermore, the route towards miniaturization allows even the integration in industry 4.0 applications or low-cost consumer electronics devices such as robotic vacuum cleaners or washing machines.

004.02

QUANTITATIVE ANALYSIS OF BIC FORMATION BY NIR SPECTROSCOPY AND CHEMOMETRICS

Yusuke Morisawa[1], Nami Ueno[2], Tamio Ida[1]

[1]Kindai University, Higashi-osaka, Japan, [2]Kobe University, Kobe, Japan

The development of renewable energy and the recycling of waste has been promoted in response to the problems of global warming and rising fossil fuel prices. The use of biomass is attracting attention as one of the new energies, and one of them is biocokes. We have been developing the Kinda-Biocoke (BIC) that was developed by Ida et al. in 2005. The BIC is a solid biomass sample characterized by high density, high hardness, and hydrophobicity and is expected to be a clean fuel alternative to coal coke and charcoal. There are many studies on physical properties such as hardness and density of BIC have been reported, and applied research such as carbon addition to steel materials is also being actively conducted. However, it is necessary to judge the formation of BIC by hardness measurement, which should destroy the sample for the measurement. Thus, by convention, BIC formation has been determined by the blackness of the product. Miki et al. points out that when woody biomass is used as a raw material, the increase in black parts correlates with the degree of formation of high-density, high-hardness solids. On the other hand, Mizuno et al. found that blackness does not always correlate with density and hardness in herbaceous biomass. In this work, we developed the non-destructively analysis for the formation of BIC using near-infrared (NIR) spectroscopy. Supitchaya et al. have already investigated the formation of BIC and its analysis of compressive strength by Mid-IR spectroscopy. [1,2] In the NIR region, the vibrational transitions at X-H part in different environments are observed as an overlapping band at slightly different wavelengths. According to Lambert-Beer's law, what is reflected in the absorption spectrum is the concentration distribution for each type of chromophore. Considering that the degree of the formation reaction depends on the forming temperature in the BIC conversion of wood-based raw material biomass, it is a non-destructive measurement of the NIR spectrum that reflects the distribution of chemical components in the biomass at each temperature.

[1] Cherdkeattikul S., Morisawa Y., Tagami-Kaneda N., Ida T., Fuel, 342, 127835. [2] Cherdkeattikul S., Morisawa Y., Ida T., Anal. Sci. 2020, 36, 723-727.

POSTER PRESENTATION

P04.01

USE OF NIR SPECTROSCOPY FOR THE MONITORING AND CONTROL OF TEXTILE DYEING PROCESSES

Francisca Marques[1], Jorge Manuel Martins[2,3,4], Jorge Santos[3,4,5], Pedro Magalhães[1], Fernão D. Magalhães[3,4], Luísa Hora de Carvalho[2,3,4]

[1]TINTEX, Vila Nova de Cerveira, Portugal, [2]DEMad-Department of Wood Engineering, Instituto Politécnico de Viseu, Viseu, Portugal, [3]LEPABE—Faculty of Engineering, University of Porto, Porto, Portugal, [4]ALiCE—Associate Laboratory in Chemical Engineering, Faculty of Engineering, University of Porto, Porto, Portugal, [5]ARCP—Associação Rede de Competência em Polímeros, Porto, Portugal

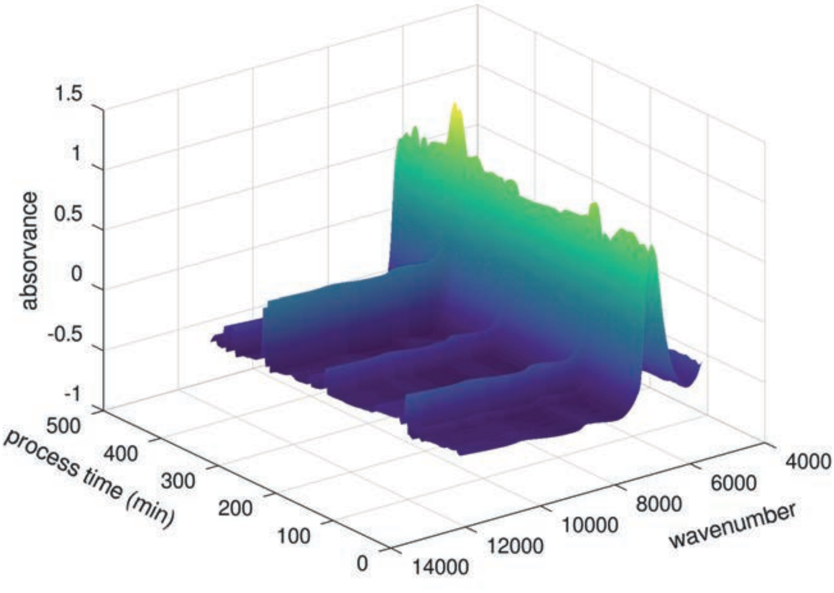
The textile industry poses a lot of environmental issues due to the number of resources it uses and the waste it produces. This inspires the constant search for technologic solutions that allow for the implementation of more sustainable and efficient industrial processes.

The application of NIR spectroscopy for the control of industrial manufacturing processes has been reported in various contexts, mostly in the agricultural and food industry, but also in fields as varied as pharmaceutical and cosmetics, petrochemical, environmental, medical, and the polymer industry. Moreover, its application has been promoted in the textile industry as well, mainly for the identification and classification of textiles fibers. Due to the flexibility of this technique, NIR spectroscopy presents itself as suitable for on-line monitoring and control of processes, by eliminating the need for sample preparation while accomplishing rapid non-destructive analysis.

With the implementation of this technology, this project intends to aid the development of high-quality textile products by automation of the textile dyeing process. The objective is to continuously monitor the dye bath and predict when the desired final color is achieved, eliminating the need for human intervention and interruption of processes. To accomplish this, NIR spectra of the dyeing operations were collected, and, due to the complexity of the NIR signal, chemometric techniques were used to obtain the values of interest and extract the most useful information out from the data. In the figure below, it is possible to observe the changes in the collected NIR spectra along a specific textile dyeing process.

With pre-processing and multivariate analysis, it was possible to develop prediction models and apply them at industrial scale to predict of a stage endpoint, with the guarantee that the desired final color is achieved. With these models, the industrial dyeing process will be more efficient. This will ultimately lead to the increase of product quality by the decrease of the average color deviation, ΔE parameter, which dictates the difference in color between the reference and the obtained product. Additionally, a reduction of operating times, percentage of reprocessed products, and operation costs is also envisioned.

The integration of NIR spectroscopy for the on-line monitoring and control of textile dyeing will unlock the production of more sustainable and higher-quality products.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wavenumber

P04.02

NEAR-INFRARED SPECTROSCOPIC EXAMINATION OF WATER INTERACTION WITH POLYMER MATRICES

Vanessa Moll[1], Krzysztof B. Bec[1], Christian W. Huck[1]

[1]Institute of Analytical Chemistry and Radiochemistry, University of Innsbruck, Innsbruck, Austria

The interactions between water and polymers represents a research field of great interest. Interplay between water and biopolymers has a fundamental function in biochemical processes, as well as an influential characteristic in materials science and industry caused by the interaction of commercially used polymers with water. The investigation of the properties of water-polymer systems has often been performed using vibrational spectroscopy techniques (infrared or Raman). In contrast, the potential of near-infrared spectroscopy (NIRS, 12,500-4000 cm^{-1}; 800-2500 nm) has received little attention in studying this problem. Although NIRS offers exclusive opportunities for the study of molecular structures and their interactions. Unique information from the overtones and combination bands is accessible with this technique. NIRS is also very well suited for the analysis of aqueous systems, as both the bands of water and the polymer can be reliably acquired in a range of concentrations, in a more straightforward manner than it is possible in MIR spectroscopy. In this study the polymer-water interactions of polymers with diverse hydrophobicity were investigated by NIRS. On the one hand, commercially used hydrophobic polymers: polytetrafluoroethylene (PTFE), polypropylene (PP), polystyrene (PS), polyvinylchloride (PVC), polyoxymethylene (POM), polyamide 6 (PA) and on the other hand biopolymers: lignin, chitin and cellulose, were analyzed. The polymer-water mixtures were examined in a mass concentration range of 1-10 % water. Analysis and interpretation of the NIR spectra included cluster analysis methods, difference spectroscopy, Multivariate Curve Resolution (MCR) and Two-Dimensional Correlation Spectroscopy (2D-COS). This research revealed clearly visible trends in NIR spectra related to the increasing hydrophilicity and the chemical nature of the polymer. It has been shown that changes in the NIR spectrum of water are also observed upon the interaction with very hydrophobic polymers (e.g. PP). Moreover, it was found that in the presence of different polymers, the revealed spectral patterns of water varied between the two main bands in the NIR spectrum of water (v1+v3 and v2+v3).

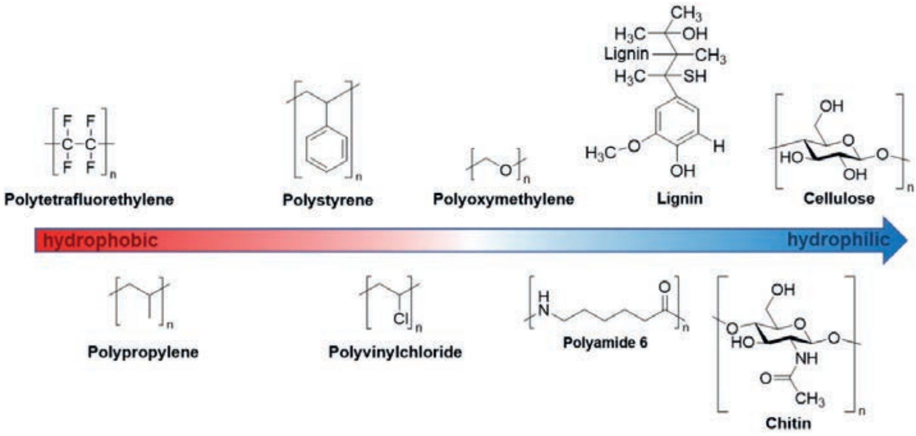


Figure 1. Approximate order of hydrophilicity of the polymers used in this study.

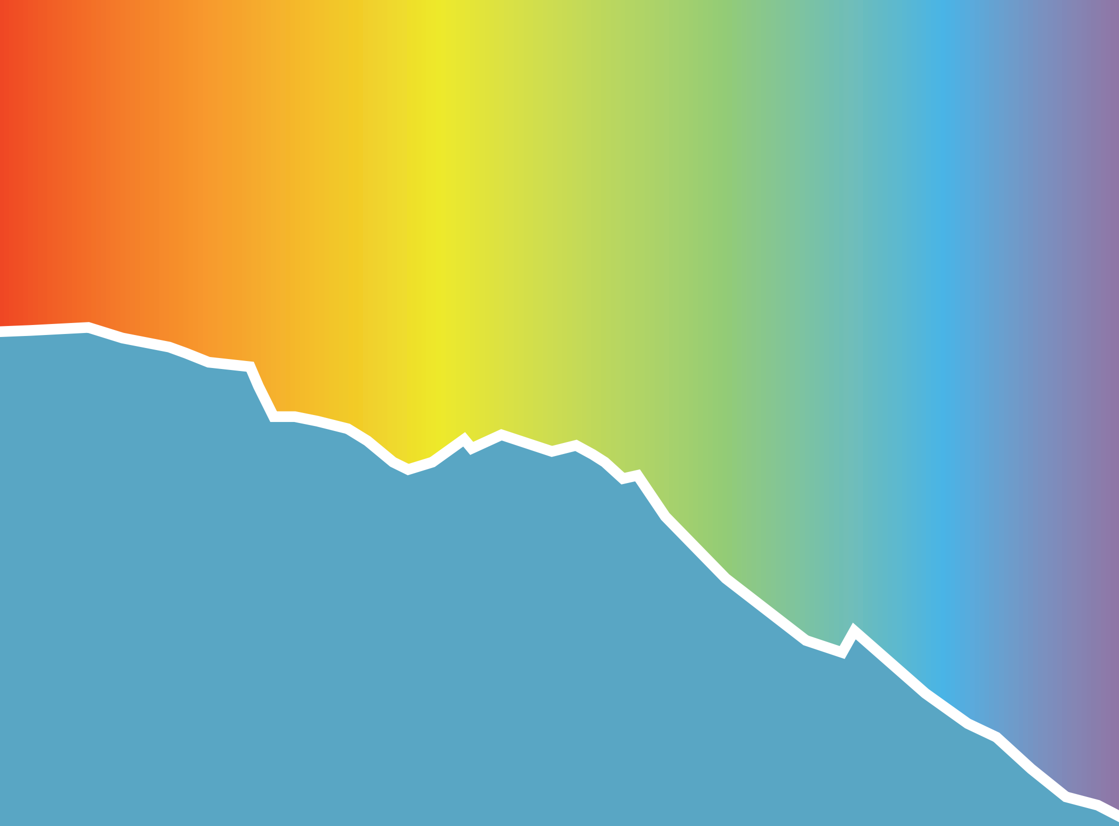
P04.03

NEAR-INFRARED SPECTROSCOPY VS. AMBIENT MASS SPECTROMETRY – METHOD COMPARISON FOR THE QUANTIFICATION OF SUCRALOSE IN COMMERCIAL E-LIQUIDS

Tobias Schlappack[1], Christoph Kappacher[1], Michela Demetz[1], Thomas Jakschitz[2], Günther K. Bonn[1,2], Christian W. Huck[1], Matthias Rainer[1]

[1]Leopold-Franzens University Innsbruck, Innsbruck, Austria, [2]Austrian Drug Screening Institute Innsbruck - ADSI, Innsbruck, Austria

E-liquids have become increasingly popular in society in recent years. A wide variety of flavors and nicotine strengths make it possible for every user to get a product according to their wishes. Many of these e-liquids are marketed with countless different flavors, which are often characterized by a strong and sweet smell. Sweeteners, such as sucralose, are therefore commonly added as sugar substitutes. However, recent studies have shown the potential formation of highly toxic chlorinated compounds. This can be explained by the high temperatures (above 120°C) within the heating coils and the used basic composition of these liquids. Nevertheless, the legal situation is composed of proposals without clear restrictions, only recommendations for tobacco products. For this reason, a high level of interest lies within the establishment of fast, reliable and cost-effective methods for the detection of sucralose in e-liquids. In this study, a number of 100 commercially available e-liquids was screened for sucralose in order to identify the suitability of ambient mass spectrometry and near-infrared spectroscopy for this application. A highly sensitive high-performance liquid chromatography coupled to a tandem mass spectrometer method was used as reference method. Furthermore, the advantages and limitations of the two mentioned methods are highlighted in order to provide a reliable quantification of sucralose. The results clearly revile the necessity for product quality due to the absence of declaration on many of the used products. Further on, it could be shown, that both methods are suiTable for the quantification of sucralose in e-liquids, with beneficial economic and ecological aspects, over classical analytical tools including high-performance liquid chromatography. Clear correlations between the reference and novel developed methods are displayed. In summary, these methods enable an important contribution to ensure consumer protection and elimination of confuse package labelling.



THEME 05

FUNDAMENTALS AND INSTRUMENTATION

ORAL PRESENTATION: O05.01 – O05.10
FLASH TALK: F05.01 – F05.03
POSTER PRESENTATION: P05.01 – P05.09

005.01

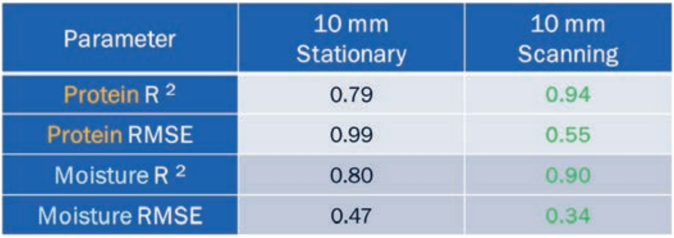
MEMS FT-NIR DIFFUSE-REFLECTANCE SPECTRAL REPEATABILITY OF SPATIALLY-SCANNED HETEROGENEOUS SAMPLES

Yasser Aboelmagd[1], Bassem Mortada[1], Samir Abozyd[1], Mostafa Medhat[2]

[1]Si-Ware Systems, Cairo, Egypt, [2]Si-Ware Systems, CA, United States

Various low-cost handheld diffuse reflection infrared spectrometers have been developed over the last decade, enabling new usage models that were not possible before. Many monitoring applications include inhomogeneous samples, such as the agriculture and food industry. The random spatial arrangement of the grains or hay samples facing the spectrometer interface, may lead to spectral variations and consequently limits the prediction accuracy. For real-time monitoring, these spatial variations should be minimized. This requires collecting a larger spot size of the diffuse-reflected light from the sample averaging the spectrospatial random variations across different locations on the sample. However the use of larger light spot size may contradict the compactness and power consumption constraints of a handheld device. Therefore, sample scanning method is used to average the spectrospatial variations, where a relative motion between the spectrometer interface and the sample is introduced during the signal acquisition to scan a wider area. A microelectromechanical systems-based spectral scanner with a 10 mm spot is built with a core spectral sensor comprising a monolithic MEMS chip, micro-optics and a photodetector in a single package; taking advantage of the high speed of the MEMS micro motor compared to the benchtop devices. The scanner covers the spectral range of 1350 to 2550 nm with a spectral resolution of 66.6 cm^{-1} and a signal to noise ratio of 4000:1 in 2 s measurement time. Spectrospatial averaging by scanning of 30 cm distance of the sample is proved where the absorbance repeatability is improved 6 times better than the stationary measurement mode. Impact of spectrospatial averaging by sample scanning on chemometrics models prediction accuracy have been evaluated using heterogeneous feed samples. The scanner is demonstrated to accurately detect protein and moisture content in spatially-scanned heterogeneous feed samples with coefficient of determination $R^2 \geqslant 0.9$ and an improvement in root mean square error RMSE of 80% for protein and 38% for moisture. Summary of Chemometric prediction models performance is introduced in Table 1.

Table 1. Feed samples prediction models showing R^2 and RMSE.



Parameter	10 mm Stationary	10 mm Scanning
Protein R^2	0.79	0.94
Protein RMSE	0.99	0.55
Moisture R^2	0.80	0.90
Moisture RMSE	0.47	0.34

005.02

BEYOND THE BLACK BOX: UNLOCKING MOLECULAR FINGERPRINTS AND ENHANCING CHEMOMETRIC MODELS WITH NIR SPECTRA SIMULATION

Krzysztof B. Bec[1]

[1]University of Innsbruck, Innsbruck, Austria

Despite the widespread use of NIR spectroscopy for qualitative and quantitative analysis of chemical compounds in fields like pharmaceuticals, food, and environmental monitoring, interpreting NIR spectra can be challenging due to complex line shapes and overlapping signals, making band assignment to specific molecular vibrations difficult. In recent years, significant advancements in the methods of computational chemistry and modelling have enabled the calculation of NIR spectra for a variety of molecules, paving the way for the integration of theoretical chemistry and NIR spectroscopy. This emerging field offers a wealth of opportunities to both basic and applied NIR spectroscopy; it elucidates a molecular fingerprint from complex spectra, deepens insights into physicochemical phenomena manifested in NIR spectra, enables chemical interpretation of chemometric models, and enhances our ability to understand instrumental factors in analysis. This presentation will overview key examples of the application of accurate simulation of NIR bands. Firstly, interpreted overtones and combination bands in NIR spectra provide unique structural information, a “fingerprint” complementary to that extracted from the fundamental bands observed in the mid-infrared (MIR) region. In the area of physical chemistry, modeling techniques unlock new potential in exploring the solvent effect or intermolecular interactions. Furthermore, essential features of chemometric models, such as loadings in multi-variate regression, can be better linked with specific molecular vibrations. Finally, this knowledge can be utilized to enhance the application of handheld and miniaturized spectrometers, which often capture only part of the molecular information from the spectra.

005.03

POTENTIAL OF THE PREDICTION OF ABSORPTION AND SCATTERING COEFFICIENTS OF A TURBID MEDIUM BY COMBINING A POLARIZED SPECKLE MEASUREMENT WITH A CHEMOMETRIC ANALYSIS

Ryad Bendoula[1], Daphné Heran[1], Maxime Ryckewaert[1]

[1]*Inrae, Montpellier, France*

Non-invasive determination of optical absorption and scattering properties of materials is a subject of considerable interest for many applications. Specific experimental techniques based on light propagation theory and radiative transfer equation resolution have been proposed to measure absorption and scattering properties separately. These techniques involve different setups based on spatially resolved, time-resolved spectroscopy as well as double integrating sphere setups.
Although powerful, these methods have their limitations and are only based on a corpuscular approach. In order to improve the determination of optical bulk properties, it is possible to explore the wave properties of light. In this study, we propose to combine the corpuscular and wave properties of light to improve the prediction of the bulk properties of materials. For this purpose, the combination of speckle pattern analysis, polarization parameters and chemometric tools was investigated.
An optical setup based on light polarization and speckle measurements was developed and turbid samples were measured at 405 nm and 660 nm. First, backscattered polarized speckle acquisition was performed on a set of 41 samples with various scattering (μs) and absorbing (μa) coefficients. Then, several parameters were computed from the polarized speckle images and prediction models were built using stepwise-Multiple Linear Regression. The first result are very promising. μs was predicted with $R^2 > 0.9$ using and $R^2 = 0.8$ for μa with only two parameters. After the validation of the proof of concept, the next step is to increase the number of wavelength.

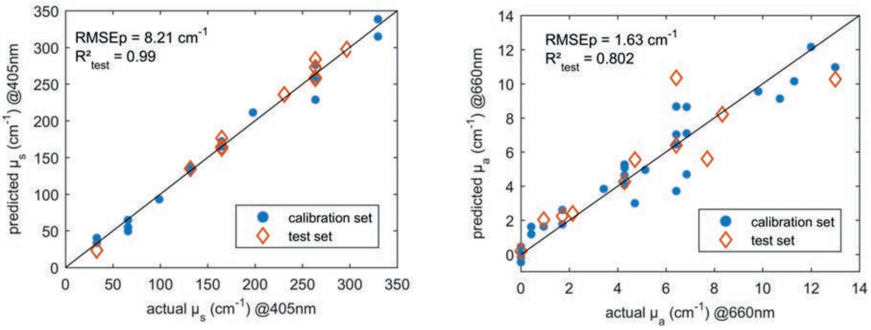


Figure 1. Stepwise MLR predicted values versus actual values of respectively us at 405nm and μa at 660 nm using two parameters, for calibration and test datasets.

005.04

COMPUTATIONAL INSIGHTS INTO PEPTIDE ANHARMONICITY: DECODING NIR SPECTRA FOR STRUCTURE AND FUNCTION

Justyna Grabska[1], Krzysztof B. Bec[1], Christian W. Huck[1]

[1]University of Innsbruck, Innsbruck, Austria

Near-infrared (NIR) spectroscopy is not only a powerful analytical technique, but it also holds immense potential in fundamental research. NIR spectra are the richest source of information on the anharmonic properties of molecules, which often play a critical role in defining their structure and function. For instance, anharmonicity in peptides can significantly impact reaction paths and energy barriers, such as enzymatic catalysis or peptide bond cleavage reactions, by influencing transition state energies and reaction rates. Anharmonicity can also dictate the dynamics of peptide reactions, including the timescales and pathways of energy flow and redistribution. Finally, it impacts the reactivity and biochemical function of peptides via couplings between vibrational modes and other molecular degrees of freedom.

This presentation showcases recent advancements in computational chemistry tools that enable the interpretation of NIR spectra for moderately complex molecules, with a focus on oligopeptides. Through simulations, we reevaluate NIR band assignments for peptides, revealing new insights and identifying important peptide bands in the NIR region. This challenges conventional NIR assignments, as the manifestation of amide bands in the NIR spectrum is less straightforward compared to the fundamental region. For example, in polyglycine, the widely speculated contribution of the Amide A mode as a broad absorption structure near 4890 cm^{-1} actually arises from the combination of amide A and amide II modes. These findings provide detailed insights into NIR band assignments for peptides, advancing our understanding of the role of anharmonicity in their spectral properties.

005.05

OPTIMIZING SPECTRAL REQUIREMENTS FOR THE HANDHELD SPECTROMETER OF THE FUTURE – A DATA-DRIVEN APPROACH

Michael Hanke[1], Robert Lovrincic[1]

[1]trinamiX GmbH, Ludwigshafen am Rhein, Germany

Applications for NIR spectrometers are manifold. They range from the quantitative analysis of animal feed to the material classification of, for example, different plastic types. Each sample type comes with its very own distinct characteristic fingerprint spectrum in the NIR region (wavelengths from 0.9 to 3 µm or 11000 to 3300 cm^{-1}) that is accessible for qualitative and/or quantitative analysis through chemometrics. Benchtop laboratory NIR spectrometers with their large wavelength coverage, high spectral resolution, and low noise properties can tackle all these applications at once. However, such solutions come at the expense of large financial investments (several 10 k€) and the need of trained lab personnel to operate them.

When striving to make this technology available to a broader market for mobile applications by reducing system complexity and form factors, one faces the non-trivial task to define the optimal spectral characteristics for a new handheld spectrometer that is capable of delivering high performances simultaneously in all of the applications mentioned above – all of which whilst being constrained by a low cost-performance ratio. Here, we present a novel optimization approach that was used to drive the requirement engineering of a next-generation handheld spectrometer. For this purpose, we have established a simulation framework that in its essence parametrizes the response function of a model spectrometer through four key parameters: wavelength range, spectral resolution, dispersion sampling rate, and noise characteristics. In connection with an end-to-end pipeline for chemometric model generation and a dataset of real-world benchtop NIR spectra to learn from, performances in the envisioned target applications can be simulated. This way, the framework enables the data-driven multivariate inference of optimal spectrometer solutions that are tailored to their target markets.

005.06

INVESTIGATION ON VARIATIONS IN PROTEIN HYDRATION AND HYDROGEN BOND NETWORK OF WATER MOLECULES INDUCED BY THE SECONDARY STRUCTURAL CHANGES OF PROTEINS USING NEAR-INFRARED SPECTROSCOPY

Mika Ishigaki[1], Yoshiki Kato[1], Eri Chatani[2], Yukihiro Ozaki[3]

[1]Shimane University, Matsue, Japan, [2]Kobe University, Kobe, Japan, [3]Kwansei Gakuin University, Sanda, Japan

This study investigated how the secondary structural changes of proteins in aqueous solutions affect their hydration and the hydrogen-bond network of water molecules using near-infrared (NIR) spectroscopy. Heating was used to denature three types of proteins, i.e., ovalbumin, β-lactoglobulin, and bovine serum albumin, from α-helix to β-sheet, and variations of NIR water bands were analyzed in relation to the secondary structural changes. Not only the changes in the hydrogen-bond network due to hydration on the protein surface but also due to the whole of water including it are disclosed. The increase in protein concentration made the hydrogen-bond network stronger due to the hydration between protein and water molecules. The variation patterns of the hydrogen-bond network depended on the type of protein and slightly on the solvent species. Such differences may be caused by the differences in hydrophobic and hydrophilic properties on the surface of protein molecules depending on the protein species and their interaction with solvents. The elucidation of the mechanism of protein hydration and the formation of the hydrogen-bond network of water molecules will afford a comprehensive understanding of the protein functioning and dysfunctioning derived from the structural changes in proteins.

005.07

CURING PROCESS OF EPOXY RESIN EXPLORED BY NIR AND MIR PCMW2D CORRELATION SPECTROSCOPY

Shigeaki Morita[1]

[1]Osaka Electro-Communication University, Osaka, Japan

A curing reaction occurring by mixing of epoxy monomer and curing agent is a complicated phenomenon due to multiple polymerization and cross-linking reactions. In the present study, a curing process of the mixture using bisphenol A diglycidyl ether as an epoxy monomer and 4,4'-diaminodicyclohexyl methane as a curing agent was investigated by means of near-infrared (NIR) and mid-infrared (MIR) spectroscopy combined with perturbation-correlation moving-window two-dimensional (PCMW2D) correlation spectroscopy. A 1-mm-thick sample sandwiched between two glass plates was used for the transmission NIR spectroscopy. Attenuated total reflection (ATR) MIR spectra were measured using a diamond crystal having an incident angle of 45°. Temperature-dependent spectra were collected during a heating process of the sample from 30 to 190 °C with a rate of 2 °C min-1. Synchronous PCMW2D correlation spectra were constructed from the temperature-dependent spectra using the 2DShige software. An exothermal chemical reaction temperature and a glass-transition-like variation of cross-linking temperature were identified at 82 °C and 100 °C, respectively, by differential scanning calorimetry (DSC). Detailed chemical reactions of the system at a functional group level under the heating process in the vicinity of the temperatures estimated by DSC will be discussed.

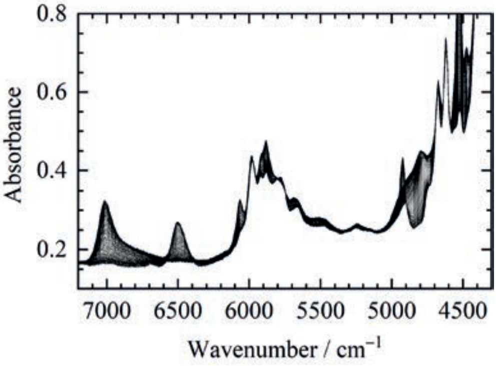


Figure 1. Temperature-dependent NIR spectra obtained during the curing process of the epoxy mixture.

005.08

NOVEL SAMPLING AND MEASUREMENT NIR SOLUTIONS IN PRACTICE PART 1

Marion O'Farrell[1], Jon Tschudi[1], Jens Petter Wold[2]

[1]SINTEF AS, Oslo, Norway, [2]Nofima AS, Oslo, Norway

Nofima and SINTEF have previously been involved in the development of the NIR instrument, QVision, which was commercialised by Tomra for measuring quality parameters of food in high-speed, conveyor belt applications. In the last 5 years, we have developed and tested a new industrial prototype, SmartSensor, which addresses more challenging applications where the outer layer weakens the light penetration so much that QVision struggled to achieve sufficiently good measurements due to lower signal to noise ratio (SNR). Both instruments use what we call optical sampling (interactance), so that they can measure stable and high-quality NIR spectra from the interior of food products. They rely on very controlled light interaction measurements, without physical contact, and this is a novelty within NIR instrumentation. The SmartSensor system allowed us to measure five different interactance geometries per measurement, which helps us understand how light interacts with different food types and decide which geometry is optimal (Figure 1). It was successfully calibrated and tested in industry by Nofima and SINTEF, in collaboration with food companies, for measuring dry matter in potatoes, fat content in whole salmon, and core temperature in sausages during heat treatment. The high performance is due to an instrument design that addresses the challenging properties of complex foods, the industrial measurement situation, and requirements for speed and signal quality. The current SmartSensor prototype is heavy and bulky and now industry would like something more compact and portable, while still maintaining good enough performance. This means that we are currently in a new design phase, where the compromise between size and performance is being further explored. In our talk, we will discuss the design process from a hardware perspective, explaining the importance of spectrometer design when developing NIR-based solutions and how it can be used to improve the quality of spectral data, and hence the calibration models. We will also discuss how the approach differs depending on the application demands.

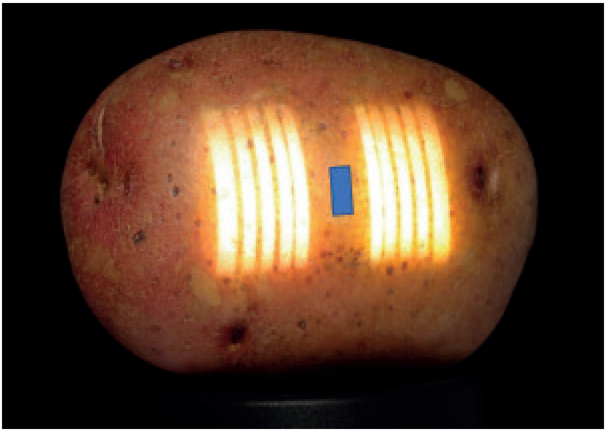


Figure 1. The light interaction geometries in SmartSensor. The Field of View (FOV) is illustrated in the photograph using the blue rectangle. The light stripe pairs, with each pair being equidistant on either side of the FOV, are turned on sequentially per measurement, to test different interactance distances.

005.09

ASSESSMENT OF TOTAL ALKALOIDS IN CINCHONA BARK USING A DEVELOPED PORTABLE NIR SPECTROMETER

Dilip Sing[1,2], Subhadip Banerjee[1], Sirsha Naskar[5], Sayanika Banerjee[2], Uden Angmu Yonzone[3], Samuel Rai[3], Amitabha Bandyopadhyay[2], Ajanto Kumar Hazarika[4], Rajib Bandyoypadhyay[2]

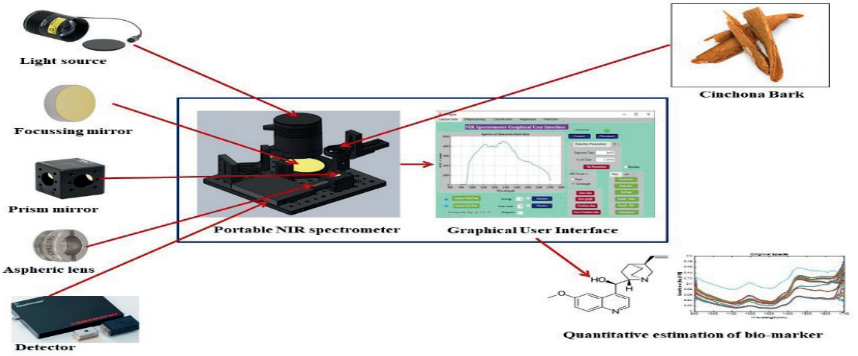
[1]MetaspeQ Division, Ayudyog Private limited, Kolkata-700040, West Bengal, India, [2]Department of Instrumentation and Electronics Engineering, Jadavpur University, Salt Lake Campus, Kolkata 700106, West Bengal, India, [3]Department of Food Processing Industries and Horticulture, Gorkhaland Territorial Administration, Mungpoo, Darjeeling-734313, West Bengal, West Bengal, India, [4]Tocklai Tea Research Institute, Tea Research Association, Jorhat-785008, Assam, India, [5]School of Natural Product Studies, Jadavpur University, Kolkata – 700032, West Bengal, India

The presence of alkaloids makes Cinchona widely produced and traded medicinal plants across countries. There are nearly 30 chemical components extracted from the bark of the trunk, the most important ones are quinine, quinidine, cinchonidine, and cinchonine. There are two types of uses for alkaloids of Cinchona. Firstly, Finished Pharmaceutical Products (FPP) for the treatment of malaria. Secondly, Active Pharmaceutical Ingredients (API) for tertain diseases, food and beverage, flavour & fragrances, and chemical industry. India has continuously led the export of Cinchona alkaloids and its derivatives. Importantly, the statistics reveal that export volume surged in 2020, owing to an increase in demand for quinine, possibly as a result of the COVID-19.

In this work, a portable NIR spectrometer was designed and developed for the estimation of cinchona alkaloids in cinchona bark. The portable NIR spectrometer was developed using a Tunsten Halogen Lamp, a concave mirror, a prism mirror and Detector in a small black box. The display unit of this portable NIR has a user-friendly Interface for the spectrum data analysis and computing system. It is used to store spectrum data, display spectrum cluster chart, make pretreatment on the spectrum data and create model with the calibration data of the spectrum samples. Eighty bark samples from cinchona tree were collected from about 26,000 acres of plantations area varying between an altitude of 1,200 ft and (approx) 6,000 ft above mean sea level of Munpoo, Labdah, Munsang, Sittong, Rungbee, Rongo, Burmeik, Dalgawn division in Darjeeling, West Bengal, India.

SNV and Savitzky–Golay preprocessing was applied before development of calibration model. A calibration model for the estimation of total alkaloids was developed using partial least square regression. Coefficient of determination (R^2)and RMSEC of 0.98 and 0.2 was obtained for the calibration set respectively. The prediction model for cinchona gave R^2, RMSEP, and RPD 0.98, 0.31 and 6.13 respectively. It fulfils the requirements according to AACC Method 39-00 (AACC in AACC Method, 39-00:15, 1999) to be used at least for screening (RPD ⩾2.5). The results establish that the developed portable NIR instrument can be used for quality assessment of bark of the Cinchona tree. The spectrometer can be used for other marker molecules in plants and their products by suitable modifications in the software.

Graphical Abstract



005.10

SENSOR FOR RAPID IN-FIELD IDENTIFICATION OF ILLEGAL CANNABIS PRODUCTS

Robert Zimmerleiter[1], Wolfgang Greibl[2], Paul Gattinger[1], Kristina Duswald[1], Ivan Alic[1], Gerold Meininger[3], Robert Holzer[1], Markus Brandstetter[1]

[1]RECENDT - Research Center For Non-destructive Testing GmbH, Linz, Austria, [2]Criminal Intelligence Service, Forensic Science, Vienna, Austria, [3]MEDS - Spath Micro Electronic Design GmbH, Graz, Austria

The cannabis plant comes in many different varieties and has long been used for manufacturing of hemp fiber, hemp seeds and oils, but also as recreational drug. Many cannabis strains have been bred, specifically to produce high levels of tetrahydrocannabinol (THC), which is the plants principal psychoactive component. To prevent the use of Cannabis as a psychoactive drug, Austrian law sets a strict upper limit for the allowed weight percentage of THC in Cannabis at 0.3%.

With the recent boom of legal cannabis products for recreational purposes, which are mostly consumed for their high content of cannabidiol (CBD), the Austrian police frequently encounters these products in the field. Since CBD-rich cannabis flowers, with THC levels below 0.3%, are visually and olfactory indistinguishable from illegal, THC-rich kinds, such samples are often confiscated and tested in police laboratories, resulting in significant bureaucratic efforts and unnecessary costs. This generates the need for a sensor device, that can be used by police officers to easily distinguish between legal and illegal cannabis flowers in the field.

In this contribution we present a hand-held sensor solution (pat. pending) for the identification of illegal cannabis products in the field, developed in close cooperation with the Criminal Intelligence Service Austria. For the sensor to be practically usable by police officers in the field, requirements for the sensor were defined concerning its form factor, ruggedness, price, and usability. These requirements are not met by any commercial sensor device and needed to be considered during the development.

The developed sensor utilizes MOEMS-based NIR spectroscopy in diffuse reflection geometry to measure untreated cannabis samples, either directly or through a transparent plastic bag, which are held in place during the measurement by a specially developed pincer mechanism. The low hardware price of the chosen spectrometer modules allowed to integrate two sensors, which helps to counteract strong inhomogeneities encountered in the untreated cannabis samples. Data evaluation is carried out using a partial least squares discriminant analysis (PLS-DA) model, trained using spectral data of different types of legal and illegal cannabis samples combined with gas chromatographic reference measurements. The PLS-DA model classifies the measured samples as either containing more or less than 0.3% THC. Measurement and data evaluation are carried out directly on the handheld sensor and the result is communicated to the user via a multi-color LED on the sensor.

This work is funded by the Austrian security research programme KIRAS of the Federal Ministry of Finance (BMF).

FLASH TALK

F05.01

BLOODSTAINS DATING BY MEANS OF NIR AND UV-VIS SPECTROSCOPY – A CRITICAL COMPARISON

Sara Gariglio[1], Alicja Menzyk[2,3], Cristina Malegori[1], Grzegorz Zadora[2,3], Marco Vincenti[4], Paolo Oliveri[1]

[1]DIFAR - University of Genova, Genova, Italy, [2]Institute of Chemistry, University of Silesia in Katowice, Katowice, Poland, [3]Institute of Forensic Research in Krakow, Krakow, Poland, [4]Department of Chemistry - University of Turin, Turin, Italy

It is widely recognized that bloodstains dating may represent a crucial evidence in resolving many cases of forensic interest. Since the non-destructive techniques are most desirable, spectroscopies appear to be particularly attractive. Even though this topic has been investigated with limited success since the 1960s, most scientific strain focused on UV-Vis spectroscopy, while few studies were conducted in the IR spectral region, with only a handful of them concerning NIR spectroscopy[1].

The final goal of the present work is to compare the performances of NIR spectroscopy for bloodstains dating with those of UV-Vis in the prospect of real-casework implementation. To reach this aim, capillary blood was collected from a healthy volunteer, deposited onto glass substrates, and stored under stable laboratory conditions. The aging of bloodstains was followed for 16 days measuring transflection NIR spectra and diffuse-transmittance UV-Vis spectra (6 bloodstains for each technique, analyzed in triplicate).

The resulting data were independently analyzed by several chemometric techniques. Preprocessing of spectral profiles was conducted first with classical row pre-processing methods, such as Savitzky-Golay smoothing and standard normal variate (SNV) transform. After conducting an exploratory analysis by principal component analysis (PCA), systematic differences between samples due to factors unrelated to aging (such as blood deposition thickness) were detected as predominant. Therefore, class centering was applied to remove this unwanted effect yielding the resulting data to be focused only on time trends. Subsequently, a partial least square (PLS) regression model was computed to evaluate the effectiveness of applied spectral methods for estimating time elapsed since blood trace deposition.

The two techniques gave comparable root means square errors in prediction (RMSEP) equal to about one day. This time uncertainty appears to be lower than any other so far reported in the literature for NIR spectroscopy. In view of the creation of a multi-instrumental platform for bloodstains dating, data fusion strategies were also tested to estimate the advantages and disadvantages of low-level, mid-level, and high-level approaches. The present results together with the regression models obtained from them looks promising, even taking into account the limit of the lack of variability of the blood donor. NIR spectroscopy is therefore seemingly worthy of increased scientific attention in the forensic field for bloodstains dating.

[1] Grzegorz Zadora, Alicja Menżyk, "In the pursuit of the holy grail of forensic science – Spectroscopic studies on the estimation of time since deposition of bloodstains", TrAC Trends in Analytical Chemistry, 2018,105, 137-165.

F05.02

NONLINEAR CONCENTRATION-DEPENDENT SCATTERING COEFFICIENT WITH A COLLOIDAL SOLUTION AND ITS ORIGIN

Yuki Inoue[1], Hiroyuki Fujii[1], **Goro Nishimura**[1]

[1]*Hokkaido University, Sapporo, Japan*

The NIR applications for thick biological, agricultural, and food samples suffer the strong scattering, and the quantifications of the sample are challenging because the scattering light is non-localized and loses spatial information. The multiple-scattering process is described by a diffusion process, allowing diffusion model based-analyses to characterize samples. The reduced scattering and the absorption coefficients determine the light propagation under the diffusion model, resulting in these coefficients representing the sample character. We focus on the scattering coefficient because the scattering coefficient reflects the structural information of the sample and are investigating the scattering property in colloidal solutions to elucidate the structural information from the scattering coefficient. Here, we discuss the interference effect of the scattering in particular and the origin of this effect.

The scattering coefficient of dilute colloidal solutions is described by a sum of the individual scattering and proportional to the concentration of the colloidal particles. However, the proportionality does not maintain anymore with concentrated solutions because the interference between the scattering fields from individual scatterings is not negligible. This effect is so-called the interference effect. We previously found that a single parameter can characterize nonlinearity. Furthermore, the experiments showed that the nonlinearity is almost corrected by the first order of the volume concentration within our experimental condition as ms(r) = m0r(1-r/r0), where ms(r) is the reduced scattering coefficient at the volume fraction r of the colloidal solution. In this presentation, we analytically derive an explicit form of the first-order correction under the local monodisperse assumption, which enables us to interpret its wavelength dependence.

Under the Rayleigh-Gans-Debye framework, the interference effect comes through the static structure factor, which consists of the two-body correlation function. In the previous work, we chose the Percus-Yevick equation to represent the correlation function, and the interference effect depends on the volume fraction. Taking the first-order term of the dependence, one can find this term is just the same as the simplest hard sphere model, which only considers the excluded volume effect. Then, we compared the reduced scattering coefficients from the experiment and the numerical calculation of the analytical expression with the Mie scattering phase function. This simple calculation can quantitatively explain the experimental results with different wavelengths. We also consider the polydisperse case, and the difference from the experiment reduces. We have concluded that the excluded volume effect in the static structure factor can explain the dominant part of the interference effect.

F05.03

TAILORING SPECTRAL SENSORS FOR SPECIFIC APPLICATIONS

Don van Elst[1], Anne van Klinken[1], Fang Ou[1,2], Chenhui Li[1],
Maurangelo Petruzzella[1,2], René van Veldhoven[1], Andrea Fiore[1]

[1]Eindhoven University Of Technology, Eindhoven, Netherlands, [2]MantiSpectra B.V., Eindhoven, Netherlands

There is a growing need for miniaturized and affordable NIR instrumentation that can perform real-time measurements in the field. However, standard spectrometers are made of discrete components, which makes them difficult to scale in size and cost. As an alternative approach, we have previously reported on an integrated, robust and low-cost near-infrared (900-1700 nm) spectral sensor that employs an array of 16 resonant-cavity-enhanced photodetectors. The sensor has a tunable response, narrow linewidth (FWHM≈60 nm), and high peak responsivity (R=0.3 A/W). The effectiveness of this approach was demonstrated on a variety of applications.

While the demonstrated sensor has wide applicability, its prediction accuracy could be improved for specific problems by using a set of optimized spectral responses. In this work, we present an approach to optimize the sensor for specific applications. We use the transfer-matrix method to simulate the spectral responses, which allows prediction of the performance for known reflection or transmission spectra from any sensing problem. By using a particle swarm optimization, we can identify a configuration that provides optimized sensing accuracy. This optimized configuration can be a non-trivial combination of resonant wavelengths, as opposed to the hand-picked designs based on the most important wavelengths.

The robustness and prediction strength depend on the chosen application, the amount of detectors used and the signal-to-noise ratio of the read-out. Here, we give a demonstration on the determination of concentration in alcoholic mixtures. This approach makes it possible to obtain a high sensing accuracy even with a minimal pixel configuration, increasing the signal per pixel and reducing the complexity and cost of the read-out. The optimized sensor can be integrated into portable devices, allowing for real-time measurements in the field.

POSTER PRESENTATION

P05.01

COMPARATIVE ANALYSIS OF NIR SPECTROMETERS FOR PHARMACEUTICAL FORMULATION PREDICTION: PERFORMANCE EVALUATION AND MODEL INTERPRETATION IN BENCHTOP AND PORTABLE FT-NIR AND LVF-BASED INSTRUMENTS

Krzysztof B. Bec[1], Justyna Grabska[1], Alexandra Warzilek[1], Christian W. Huck[1]

[1]University Of Innsbruck, Innsbruck, Austria

The analytical performance in prediction of the content of a model pharmaceutical formulation by three NIR spectrometers was dissected and interpreted. The spectrometers used in the systematic study were the Hefei SouthNest Technology nanoFTIR, equipped with a large-mirror Michelson interferometer and a miniaturized multi-channel array detector; the Viavi MicroNIR 1700ES (USB), which is LVF-based (low-volume fraction) and miniaturized with an array detector; and the Büchi NIRflex N-500, a benchtop instrument with a polarization interferometer.

Regression models based on Partial Least Squares (PLSR), Gaussian Process (GPR), and Artificial Neural Network (ANN) were constructed for all instruments and mixture components using different data pretreatments optimized for each approach and instrument. Spectral precision, reproducibility, and replicability were calculated for the instruments to evaluate their performance. Chemical interpretation of the spectra and features of the regression models of pure components and mixtures was also done with aid from quantum chemical spectra simulation to gain deeper insights into how sample properties influence the calibration models. The results showed that while all instruments delivered acceptable performance, the interplay between the measured wavelength region, spectral resolution, and the absorption features of the quantified chemical components had a clear representation in their analytical performance. This enables smart selection of a sensor tailored for the prediction of a specific compound, taking into account the unique characteristics of the sample and the instrument used.

P05.02

BENCHMARKING OF A NEW PORTABLE NIR SENSOR WITH CUSTOM WRITTEN SOFTWARE FOR APPLICATION IN POST-HARVEST TECHNOLOGY

Mercedes Bertotto[1], Yannick Weesepoel[1], Kees van Gorsel[2],
Najim El Harchioui[1], Syed Sarfaraz[2], Nithin Manuel[2]

[1]Wageningen University and Research, Wageningen, Netherlands, [2]Next Generation Sensors, Maastricht, Netherlands

The main aim of this work was to perform benchmarking of a new portable NIR sensor with custom written software for application in post-harvest technology. Therefore, we evaluated the prototype A comparing it to device B for dry matter (DM) content determination in olives samples. The device B is seen as the golden standard in portable NIRs for several post-harvest quality determinations of fresh produce.

One hundred and forty one solid olives were provided by a company in Sevilla, Spain. One hundred of them were used in this study. Moisture of solid olives was determined by gravimetry with an accuracy of 0.001 g and drying at 80 oC for 3 d in a conventional drying oven.

All samples were analysed by one prototype A, which measures in the spectral range from 920.48 - 1676 nm, allowing for 123 variables, with a resolution of 12 cm^{-1} and a spot size of 47 mm ø x 50 mm. In addition to device A, olive samples were also measured with NIR device B. Chemometrics analysis was performed with R version 4.2.2.

Pretreatments were applied in both data set, and an exploratory analysis was carried out to detect outliers. No outliers were removed. Then, both data sets were divided into calibration (n=58) and validation (n=42) sets, randomly. PLS models were cross validated in the calibration set and tested in the validation set. Finally, variance ratios between the two different devices were compared to determine if the observed differences in variances were significant or could be attributed to chance. Results can be observed in Table 1.

Table 1. Results of the comparison of regression models.

Model	Device	LVs	RMSEP	n in validation set	R^2	Variance	BIAS	Sqr (V/n)	V(B)/ V(A)	F α = 0.05
Olives, DM %	A	5	1.117	42	0.905	1.28	-0.04	0.17	1.14	1.69
	B	10	0.922	42	0.939	1.12	-0.01	0.16		

As can be seen in Table 1, prototype A, presented a slightly lower predictive performance when compared to B, but with greater complexity (more latent variables). The upper 5% point of F 41,41 is 1.69, a ratio lower than this is (1.14) was evidence that the observed difference was not big enough to be statistically significant .

To conclude, prototype A and device B allowed to accurately predict DM in olives, with no significant difference. This work was funded by OP Zuid / EFRO.

P05.03

DETECTION OF SUCCINYLATION IN CHARGE RICH PROTEINS USING A LABEL FREE APPROACH: PROCHARTS

Simangka Borsaikia[1]

[1]*Indian Institute Of Technology, Guwahati, India*

Succinylation is type of protein post-Translational modification that regulates various protein function. Dysregulation of succinylation is attributed with different diseases such as cardiovascular disease and cancer. Present techniques that detect Lysine succinylation includes mass spectrometry1which can play important roles in a variety of cellular processes. Due to an increasing number of site-specific succinylated peptides obtained from high-throughput mass spectrometry (MS and chemically labelled probes2. However, detecting succinylation using a label free intrinsic probe is limited. Recently our group discovered a new intrinsic non-aromatic chromophore in a monomeric charged rich protein. The charged residues (Lysine, Arginine, Glutamate and Aspartate) participate in photoinduced electron transfer with the peptide backbone or among themselves. This gives rise to broad UV- Vis electronic absorption ranging from 250-800 nm called as Protein Charge Transfer Spectra (ProCharTS).3monomeric proteins lacking aromatic amino acids, disulphide bonds, and active site prosthetic groups are expected to remain optically silent beyond 250 nm. Here, in a joint theoretical and experimental investigation, we report the distinctive UV-Vis absorption spectrum between 250 nm [$\varepsilon = 7338\ M{-}1\ cm{-}1$] and 800 nm [$\varepsilon = 501\ M{-}1\ cm{-}1$] in a synthetic 67 residue protein (α3C Herein we established ProCharTS as a comprehensive detection and analysis tool to study succinylated protein. We use α3W and Human Serum albumin (charge rich proteins) and titrate with succinic anhydride to obtain different degrees of succinylation. We further perform CD spectroscopy and Tryptophan fluorescence to analyse the change in the structure of protein post succinylation. Our studies show that change in the charge of amino group of Lysine after succinylation perturbs the ProCharTS profile of both the proteins by altering the pool of charge in the protein.

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P05.04

WHAT IS HIDDEN UNDERNEATH NIR LINESHAPE OF WATER?

Justyna Grabska[1], Krzysztof B. Bec[1], Christian W. Huck[1]

[1]*University of Innsbruck, Innsbruck, Austria*

Vibrational spectrum of water attracts considerable interest. While the mid-infrared (MIR) region has received considerable attention, the near-infrared (NIR) spectrum of water has not been studied to the same extent, despite its potential for providing complementary information about the structure and properties of water. In the MIR region, water strongly absorbs radiation in the region of OH stretching vibrations, resulting in distinct absorption bands in this part of the spectrum. Many efforts have been made to deconvolute the water band lineshape, with a common assumption being that all components contributing to the lineshape have a single, symmetric profile corresponding to a single water species.

NIR and MIR spectra of water provide complementary information about the structure and properties of water, and should be studied together in order to obtain a more complete picture. In this study, the NIR spectra were simulated using quantum chemical simulation of a population of water species selected through molecular dynamics. The resulting lineshape accurately reflected the well-known NIR spectral profile of water. Interestingly, the spectral lines in the NIR and MIR regions differ due to anharmonic shifts between water species. This resulted in differences in the order of appearance of the contributing bands and in the bandwidths of the spectral lines.

Furthermore, the study revealed that the contributions of overtones and combinations are particularly prominent in the NIR spectrum of water, especially in the region of OH stretching vibration. This suggested that the NIR spectrum of water could provide new insights into the structure and properties of water that are not easily accessible through MIR spectroscopy alone.

P05.05

CHARACTERIZATION OF PNIPAAM-CO-AAC DURING THE DRYING PROCESS USING NIR AND 2D CORRELATION SPECTROSCOPY

Yeonju Park[1], **Young Mee Jung**[1,2]

[1]Kangwon Radiation Convergence Research Support Center, Kangwon National University, Chuncheon, South Korea, [2]Department of Chemistry, Kangwon National University, Chuncheon, South Korea

Poly(n-isopropylacrylamide) (PNiPAAm) hydrogel is a thermoresponsive polymer that undergoes a phase transition temperature near normothermia, known as the lower critical solution temperature (LCST). Its physical and chemical properties show significant changes above LCST. Therefore, numerous studies have been conducted on its biological applications and characterizations. To expand the potential applications of PNiPAAm hydrogel, it is important to control its LCST. LCST can be adjusted pHs and temperature by introducing acrylic acid (AAc) into PNiPAAm, which is called PNiPAAm-co-AAc hydrogel. In this study, we investigated the phase transition behavior during dehydration process of PNiPAAm-co-AAc hydrogel using time-dependent NIR and Raman spectra. Figure 1 shows the NIR and Raman spectra of PNiPAAm-co-AAc hydrogel collected during the drying process. To obtain deeper insights into the dehydration mechanism of PNiPAAm-co-AAc hydrogel, we also applied principal component analysis (PCA) and two-dimensional correlation spectroscopy (2D-COS) to the time-dependent NIR and Raman spectra. The details will be discussed in this presentation.

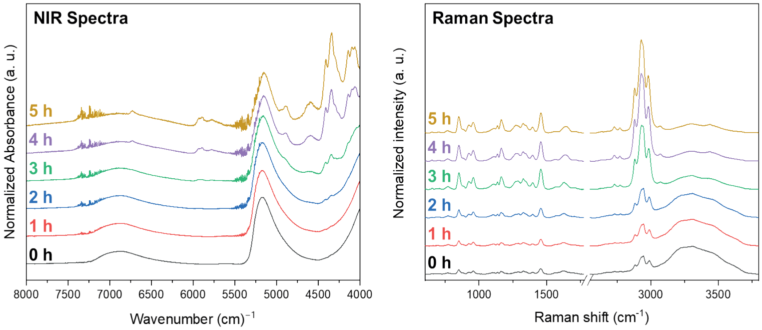

Figure 1. NIR and Raman spectra of PNiPAAm-co-AAc hydrogel during the drying process.

P05.06

A NOVEL EASY TO IMPLEMENT INSTRUMENTAL SETUP FOR NEAR-INFRARED EMISSION SPECTROSCOPY (NIRES)

Celio Pasquini[1], Jarbas José Rodrigues Rohwedder[1]

[1]State University Of Campinas, Campinas, Brazil

Despite its potential, near-infrared emission spectroscopy (NIRES) has yet to be explored for analytical purposes due to the lack of commercial instruments. The previously described instrumental setups based on tunable acoustic-optical filters employ various devices whose operation requires some knowledge of electronics and interfacing computers with analytical instrumentation. Therefore, using the technique is restricted to laboratories with instrumental skills. This work describes a new instrumental configuration of NIRES whose construction and operation can be easily implemented to extend the use of the technique to other research groups mainly dedicated to developing new applications of the technology.

The NIRES instrument is based on an interferometer (ArcoPix, Sweden - FTMIR-L1-060-4TE) operating in a free space configuration, covering the NIR/SMIR spectral range 2000 – 6000 nm (5000 – 1660 cm^{-1}). The unit is of small size (18 x 16 x 8 cm). It is connected to a microcomputer via a USB interface. It comes with a driven software capable of capturing the interferogram of a radiation source and Fourier transformation (FT) into the frequency domain with a spectral resolution of 4 cm^{-1}. The cell containing the assayed sample is placed on top of a cylindrical electric heater. Using a temperature controller and a thermocouple, the heating temperature is stabilized within ± 1° C in any set-point in the 150 – 250° C range.

The thermally excited radiation emitted by the sample passes through an iris above the sample cell to a collimating lens (focal distance = 15 cm) and impinges on a parabolic gold mirror (focal distance 15 cm and 5 cm diameter) that finally focuses it on the entrance of the interferometer.

Figure 1A shows the schematic of the NIRES setup. Results obtained for polymer isothermal degradation studies, as shown in Figure 1B, will be presented to illustrate the use of the proposed instrumental setup.

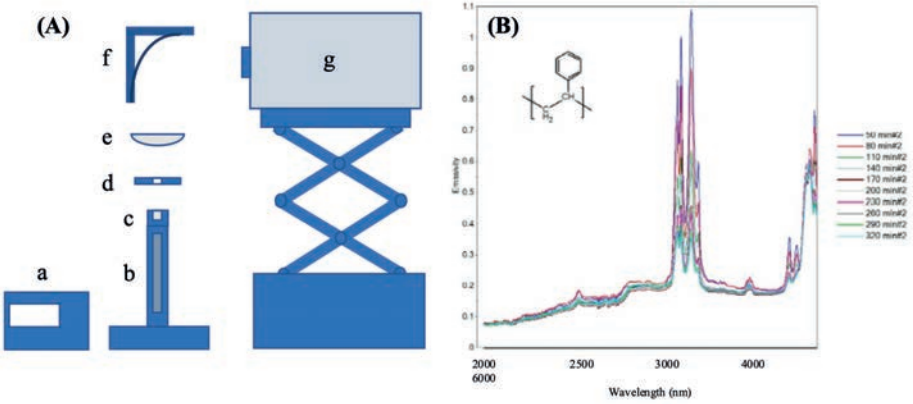

Figure 1. (A) Schematic of the NIRES setup. (a), temperature controller; (b) heater; (c), sample cell; (d), iris; (e), lens; (f), parabolic mirror; (g), FT-NIR/SMIR spectrophotometer. (B) Set of temporal spectra obtained for polystyrene degradation at 150o C using the proposed NIRES instrumental setup.

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P05.07

MODELING OF GLYCOLYTIC OSCILLATIONS IN YEAST EXTRACTS BY NIR AND METABOLITE ANALYSIS BY 1H-NMR

Miho Sesumi[1], Akifumi Ikehata[1]

[1]Institute Of Food Research, National Agriculture And Food Research Organization, Tsukuba, Japan

When quantifying target components in agricultural products and food from near-infrared spectra, we use the calibration model calculated automatically by multivariate analysis. However, it is difficult to understand this calibration model because the assignment of near-infrared spectra is not easy. Besides, it was suggested that the calibration model contains information not only on the target component but also on the related metabolites. Therefore, the correct interpretation of the calibration model requires the individual contribution of metabolites that synchronize with the target component. In this research, the glycolysis reaction in the yeast (Saccharomyces cerevisiae) extract, whose metabolic pathways and the glycolytic oscillation have been clarified, was measured over time by near-infrared spectroscopy and 1H-NMR to create the model for predicting the glycolysis reaction and to classify spectral patterns of each metabolite group. Baker's yeast was crushed and extracted to obtain the yeast extract. 1 M trehalose was added to the extract to induce glycolysis. This extract was separately measured by near-infrared spectroscopy in transmittance and 1H-NMR for 2 hours every 90 and 65 seconds, respectively. The integrated intensities of each metabolite in 1H-NMR spectra showed trends of increase/decrease or periodic oscillations. The oscillating metabolites exhibited several phase patterns, those were consistent with the result of previous studies. In addition, the partial least-squares regression model was constructed after MSC treatment for the prediction of absorbance at 340 nm, which is the absorption peak of NADH working at the glycolytic pathway. The model achieved an R^2 of 0.66 when the latent variable (LV) was 5. The same periodic oscillation with NADH were observed in some LVs.

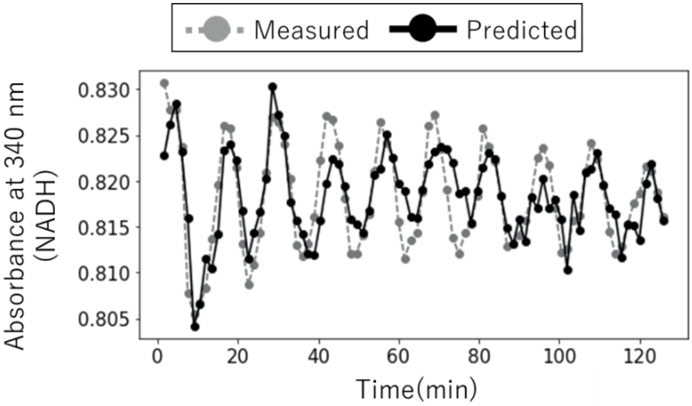


Figure 1. Predict result (Lvs=5) at 500-1050 nm by the partial least-squares regression model.

P05.08

THIN-LAYER CHROMATOGRAPHY COUPLED WITH NEAR-INFRARED SPECTROMETRY (TLC-NIR) AND ITS APPLICATION TO THE DETECTION OF SPINACH EXTRACT

Emiko Yamamoto[1], Shigeaki Morita[1]

[1]Osaka Electro-Communication University, Osaka, Japan

Thin-layer chromatography (TLC) is a technique for the separation of a mixture into its components using a thin layer of adsorbent coated on a substrate. An experimental result of the TLC is generally visualized by eye due to electronic transition of each component. In the present study, near-infrared (NIR) spectroscopic detection of the components separated by the TLC, i.e., TLC-NIR, was developed and applied to the detection of spinach extract.

A solution sample was extracted from the spinach leaves using diethyl ether. The sample was divided into two vials and the one was irradiated by UV light of ca. 365 nm for 2 hours. These samples were spotted on a TLC plate of silica gel coated on a glass substrate having a size of 25 mm × 200 mm. The sample on the TLC plate was mounted on a homemade separation chamber having two quartz optical windows. The sample separation was performed using the mobile phase of deuterated methanol-d4. Diffuse transmission spectra of the TLC plate at the position above ca. 4 cm from the sample spotting were collected as a function of retention time.

Figure 1a shows 2nd derivative of the obtained spectra using the sample before the UV irradiation. Although small signal intensity variation, the first overtone of the C-H stretching at 5785 cm^{-1} arising from the sample was clearly identified and distinguished from that of the C-D stretching at 4825 cm^{-1} from the mobile phase of methanol-d4. Figure 1b depicts the 2nd derivative intensities at 5785 cm^{-1} plotted as a function of the retention time. A relatively intense peak in the chromatogram shown in Figure 1b is observed for the sample before the UV irradiation compared to that after the UV treatment. These results will be discussed in detail.

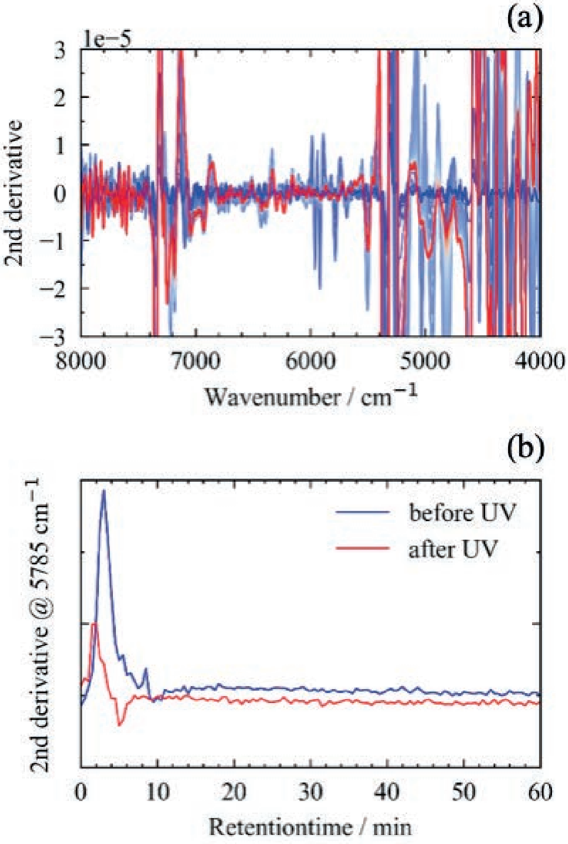


Figure 1. (a) 2nd derivative spectra calculated from the diffuse transmission spectra of the sample before the UV irradiation and (b) chromatogram of the 2nd derivative intensities at 5785 cm^{-1} plotted as a function of the retention time.

P05.09

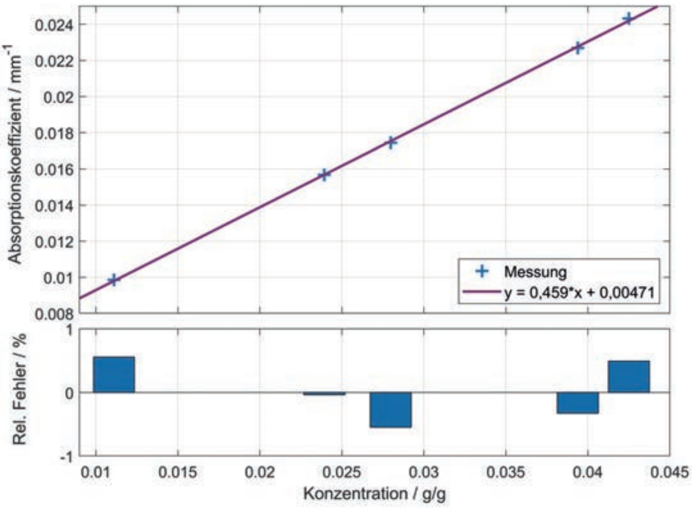
PROCESS ANALYTICAL TECHNOLOGY VIA CALIBRATION-FREE VIS-/NIR-SPECTROSCOPY

Florian Foschum[1], Ralf Zuber[2], Alwin Kienle[1]

[1]ILM, Ulm, Germany, [2]Gigahertz-Optik GmbH, Türkenfeld, Germany

A process analytical technology VIS-/NIR-spectroscopy method is presented which allows to characterize scattering and absorbing media without calibration measurements. To this end, the absolute absorption and efficient scattering spectra of the investigated sample are determined via measurement of the total diffuse reflection and transmission using exact solutions of the radiative transfer equation [1]. In general, by means of the absorption spectrum the chemical content of the sample is obtained, whereas by means of the efficient scattering coefficient the microstructure is quantified. In this contribution we apply the method for characterization of milk and milk substitute products. Spectrally resolved diffuse reflectance and transmission are determined using an integrating sphere and a spectrometer. Absolute absorption spectra and efficient scattering spectra are obtained by comparing the measurements with exact solutions of the radiative transfer equation and by considering, in principle, the whole geometry of the setup [2]. The figure shows the absorption coefficient at 530 nm of a mixture of strawberry and milk versus the strawberry powder concentration. A linear behavior is obtained with relative deviations smaller than 1 %.

In summary, a method is presented with which the concentration of food ingredients can be obtained with high precision without the need of calibration measurements by directly using the determined absorption spectra. In addition, the absorption spectrum and the spectrum of the efficient scattering can be applied to further characterize the samples, such as determination of the fat, protein or lactose concentration. We note that both spectra can also successfully be taken for chemometrics studies using calibration measurements because these spectra obtain more information than obtained with the measurements performed in conventional VIS-/NIR-spectroscopy.



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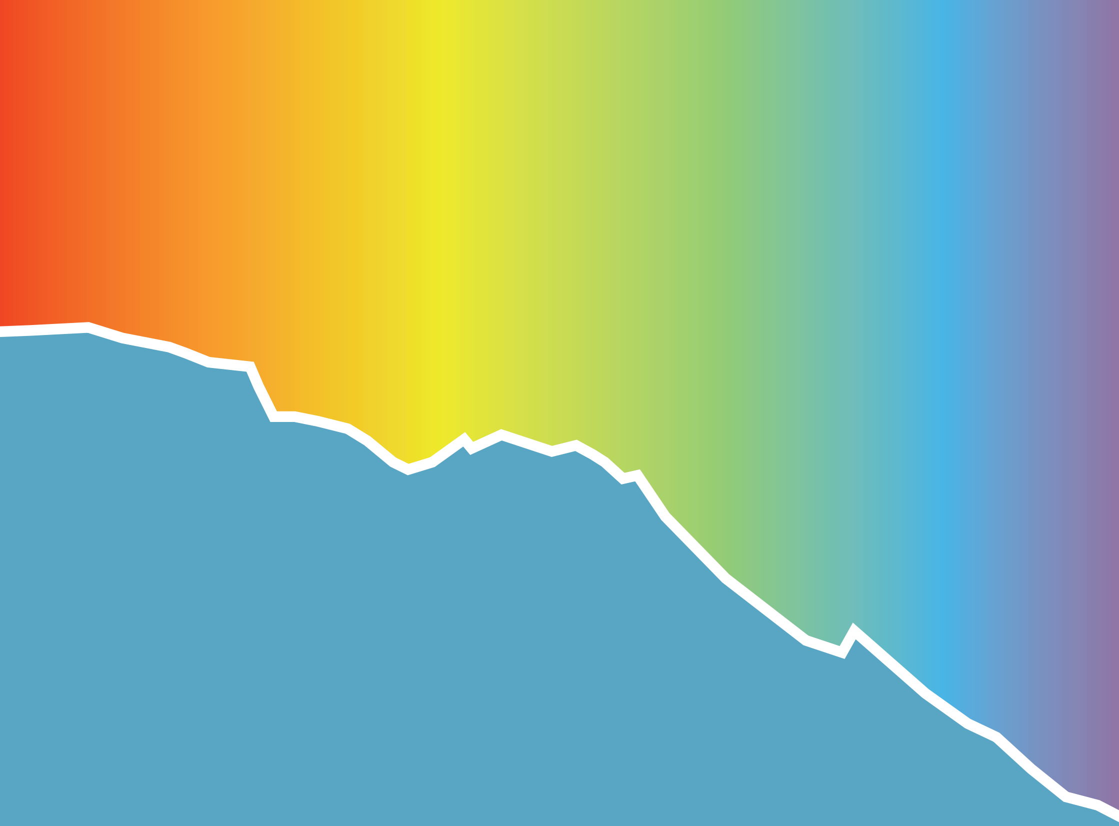
[2] F. Foschum, F. Bergmann, A. Kienle: Precise Determination of the Optical properties of Turbid Media Using an Optimized Integrating Sphere and Advanced Monte Carlo Simulations. Part 1: Theory, Applied Optics 59, 3203 - 3215 (2020).



THEME 06

FUTURE TRENDS

ORAL PRESENTATION: O06.01 – O06.05

POSTER PRESENTATION: P06.01 – P06.06

006.01

FAST AND FLEXIBLE SPECTRAL AND HYPERSPECTRAL CHEMICAL ANALYSIS THROUGH DIGITAL NIR SPECTROSCOPY

Markus Brandstetter[1], Paul Gattinger[1], Gabriel Häusler[1], Christian Rankl[1], Robert Zimmerleiter[1], Sebastian Friedl[1], Johannes Pedarnig[2]

[1]RECENDT - Research Center For Non-Destructive Testing, Linz, Austria, [2]Johannes Kepler University Linz - Institute of Applied Physics, Linz, Austria

Near-infrared (NIR) spectroscopy has progressed strongly during the last decade. This development has been driven by various technological advancements, especially through miniaturization. Nevertheless, limitations remain for fast and broadband spectroscopy and for hyperspectral imaging in particular. These limitations can be overcome by combining fully-integrated NIR optics with new measurement concepts, such as spectral coding or digital light processing.

We demonstrate a digital NIR spectroscopy approach that exploits single-pixel detection for both spectroscopy (1D) and hyperspectral imaging (2D). The centrepiece of this approach is a spatial light modulator – a digital micromirror device (DMD) – that is applied to mask a spectrum or an image with a time-varying pattern. The intensity of the masked scene is then collectively focused on a single-pixel detector for synchronized intensity measurements, which allow for the reconstruction of the spectrum or hyperspectral image. This concept brings decisive advantages in terms of speed (spectroscopy as well as hyperspectral image acquisition in the ms time regime), sensitivity (multiplex advantage – known from FTIR spectroscopy – in the spectral and spatial domain) and hardware costs (application of an inexpensive single-pixel detector with excellent detectivity instead of expensive array detectors).

The inherent flexibility of digital NIR spectroscopy in terms of measurement range and selective reconstruction is demonstrated via different practical examples – both for spectral and hyperspectral configurations. Further, we exploit the spatial light modulator for wavelength-weighted light processing, thus gaining direct optical read-out of chemical information without further data processing.

006.02

NIR IMAGING FOR TEXTILE RECYCLING

Mikko Mäkelä[1]

[1] Vtt Technical Research Centre Of Finland Ltd., Espoo, Finland

The properties of textile fibers play an important role in recycling textiles. Textile recycling is a timely topic as the EU and Switzerland alone generate 7-7.5 million tons of textile waste every year. This equates to over 15 kg per capita and could increase to almost 20 kg by 2030. Only 30-35% of generated textile waste is currently collected in the EU, roughly 10% is recycled, and less than 1% is recycled back to textile fibers. These low recycling rates have been acknowledged by the European Commission, which now requires EU member states to organize separate collection of household textile waste by 2025. Collected textile waste needs to be further sorted for recycling and most academic studies have recently focused on discriminating different synthetic and natural fibers, such polyester, cotton, and wool. Industrial sorting systems based on line-scanning near infrared (NIR) sensors are also breaking ground in the textile sector. Chemical recycling of cellulose fibers, however, requires more detailed information on fiber composition and polymer properties.

Here, we discuss how NIR imaging can be used to evaluate the properties of textile fibers specifically for chemical recycling. We show how the composition of the fiber blends can be predicted based on NIR images to reliably estimate the amount of polyester in a fabric. We then continue with classifying cotton, viscose, and lyocell, which are the main cellulose fibers for consumer clothing applications and cover over 30% of global fiber production. Finally, we conclude by showcasing our most recent findings on discriminating polymer-level differences in cotton based on the degree of polymerization of cellulose. These results could enable us to non-invasively estimate the chemical degradation of fabrics based on cellulose properties and to control the viscoelastic properties of dissolved cellulose fibers during chemical recycling. Overall, this contribution will summarize our recent work on NIR imaging for textile recycling and clarifies the important role of NIR spectroscopy in the future circular economy of textiles.

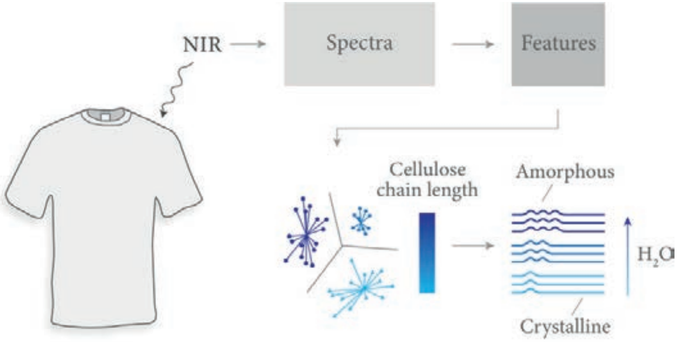


Figure 1. NIR enables estimating polymer-level fiber properties for chemical recycling of textiles.

006.03

RAPID ON-SITE DETECTION OF EXPLOSIVES AND THEIR PRECURSORS BY ULTRA-PORTABLE NIR TECHNOLOGY

Anne-Flore Prior[1], Olivier Delémont[1], Pierre Esseiva[1], Florentin Coppey[1], Bart Simoens[2]

[1]School of Criminal Justice, University of Lausanne, Lausanne, Switzerland, [2]Royal Military Academy, Brussels, Belgium

Explosives and precursors detection face significant challenges, such as the high cost and practical risks associated with traditional portable devices, making them less accessible for first responders. Consequently, there is a need for innovative approaches that enable rapid, reliable, sensitive and safe detection in the field.

Recent advancements in technology and chemometrics have led to the development of compact spectrometers with integrated communication capabilities, enabling the use of cloud computing architectures. This combination of technologies opens new opportunities of application, including the detection of explosives and their precursors.

In this context, a portable NIR (near-infrared) instrument (MicroNIR from Viavi Solutions Inc.) with cloud storage and computing, is tested. We have been interested in this approach as it offers many advantages such as a non-hazardous light source, lower costs compared to traditional devices, rapid and contactless measurements.

The reliability of this technique for explosives and precursors detection is being evaluated, particularly on the possible risks of false positives and negatives. Preliminary findings suggest that this approach is robust for the detection of peroxide-based explosives. Indeed, numerous white powders were analysed and compared to many peroxide-based explosives samples, revealing for now a very high specificity, with no false positive for those pure substances.

In addition, the scope of information that one NIR spectrum can provide is also being explored. In the case of precursors, quantitative models were developed and validated to determine if their concentrations exceed legal thresholds, indicating potential illicit usage. For explosives, specifically for triacetone triperoxide (TATP), we have seen that NIR analysis provides an insight into the humidity level for TATP samples, which could help bomb disposal experts make better-informed decisions regarding neutralisation and destruction.

In conclusion, NIR portable technology is already proving to be a valuable complementary technique for a rapid and non-hazardous analysis of explosives and their precursors, from which computing power provided by a cloud-based architecture is used for optimised results. However, it is important to better define the potential of this technology but also its limitations, such as its detection limit, its dependence on a database and internet connection, and possible data security concerns.

006.04

TOWARDS STANDARDISATION OF SOFTWARE PLATFORMS USED FOR NEAR INFRARED AND OTHER PAT INSTRUMENTATION

Brad Swarbrick, Rajani Davuluri, Joonsup Lee, Steve Hammond

[1]*Universidad de Salamanca*

Instrumentation for Near Infrared (NIR) spectroscopy and other associated Process Analytical Technologies (PAT) has experienced a technological revolution since the mid 2000's as a result of optical miniaturisation, faster computing power, wireless connectivity and a greater acceptance of technology, particularly in the regulated industries. Unfortunately, one of the drawbacks of having a wealth of instrumentation is the corresponding diversity of software applications that drive each instrument.

A general observation of the current landscape would suggest that instrument companies invest heavily into R&D to improve hardware (as should be the case), however, the software systems developed seem to play second fiddle to the excellent hardware developed. The closest analogy is that a company develops a high-performance racing car, but does not match that with a driver that is capable of extracting its true performance.

Software is as critical to instrumentation as the instrumentation itself. If users feel the software is too difficult to use, or they are burdened with having to learn 'another software' package, the overall result is less use of the instrumentation and therefore a potential loss of innovation. This too is coupled with the number of chemometrics software packages available to extract critical information out of the data.

It has become apparent that, just as communication standardisation protocols such as OPC have helped with instrument integration into processes and standardised data formats (GRAMS, JCAMP, etc.) have allowed easier data transfer between platforms, there is an overall need to have a standard, intuitive and easy to use base for spectroscopic data acquisition such that all hardware platforms can be used. Acquisition of data in a standard and secure format that can be used across a wide range of industries, while meeting the minimum data integrity requirements of each industry is highly desirable.

This presentation outlines the environment required to introduce standardisation across all instrument platforms that will not only simplify the workload of analytical and process scientists/engineers, but will also reduce lifecycle burdens on hardware manufacturers and industry practitioners alike.

006.05

DEVELOPMENT OF A COST-EFFECTIVE PROTOTYPE TO MONITOR THE MUST FERMENTATION PROCESS: FIRST TESTS IN LAB-SCALE

Alessio Tugnolo[1], Valentina Giovenzana[1], Alessia Pampuri[1], Sara Vignati[1], Daniela Fracassetti[1], Alessio Altomare[1], Andrea Casson[1], Riccardo Guidetti[1], Roberto Beghi[1]

[1]Università degli Studi di Milano, Milan, Italy

The correct management of alcoholic fermentation within the oenological process is a fundamental aspect for quality control. The current management of this operation has wide room for improvement both in terms of optimization of production factors and in terms of potential energy and economic savings.

Systems that can help the winemaker to feed the yeast during the fermentative process according to predetermined nutritional profiles are already available on the market. However, to optimize such feeding operation and to further control the fermentation process by refining the dosage of nutrients, an objective evaluation of qualitative parameters like must solids soluble content, density and developed alcohol is needed.

Therefore, this work aimed to design, build and test a monitoring system (dedicated to fermentation processes) using cost-effective sensors based on vis/NIR spectroscopy. A first prototype version for lab-scale measurements was developed. The module has been designed in-house and built using resin 3D printers (Figure 1). The structure has been equipped with a halogen lamp (20W, 12V, MR11) and a cooling system managed with a microcontroller (Arduino Nano, A000005). The spectral measurements were performed using two ultra-compact spectrometer heads taking optical signs from 340 nm to 850 nm (Hamamatsu, C12880MA) and from 640 nm to 1050 nm (Hamamatsu, C14384MA-01), respectively.

Such sensing architecture will be combined and/or used in a joint cooperation with chemical-physical sensors (temperature, time, pressure, CO2 etc.) to develop a real-time multiparameter system to be used as a PAT in yeast feeding machines to develop a quality by design approach for an enology 4.0.

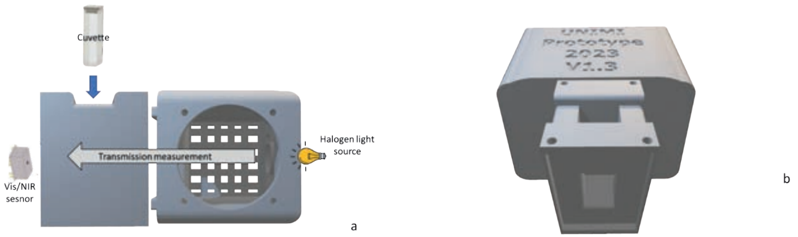


Figure 1. Prototype setup for lab-scale measurements.

P06.01

ASPARAGUS FRAUD DETECTION USING HYPERSPECTRAL IMAGING AND PORTABLE NIR SPECTROSCOPY.

Miriam Alonso[1], Javier Ramos[1], Paula Luri[1], Juan Marín[1], María José Sáiz[1], Berta Remírez[1]

[1]CNTA, San Adrián, Spain

Fraud in the agri-food industry in the European Union is estimated to create damage to around 8 to 12 billion € per year (1), including food and beverages. Usually, the inspection bodies of PGIs and Designations of Origin use systems based on traceability to control the genuineness of the products. This method is easily falsifiable and therefore most of them demand the existence of other more accurate and unequivocal methods to control these characteristics of the products subject to a Protected Geographical Indication or Designation of Origin.

In this work, machine learning models have been developed for the binary classification of origin for the Protected Geographical Indication (PGI) (2) denomination "Asparagus from Navarre", using portable NIR technology and hyperspectral imaging, on a complex plant matrix: canned asparagus.

Thanks to Navarre Institute of Agri-food Technologies and Infrastructures (NIATI), it has been possible to have a set of samples, mainly from Peru, China and Spain, but also from other countries such as Holland, Namibia and Ethiopia. These latter are non-PGI asparagus, except in the case of Spain, with samples of both types of asparagus (PGI and non-PGI).

After performing an initial pre-treatment of the data, both unsupervised and supervised machine learning classification models have been developed. Models with an accuracy greater than 0.8 have been obtained with laboratory and portable instruments. The models have been tested by an external representative set of samples, providing good results and supporting the possibility of generating fast, reliable, portable, cost-effective, and easy-to-handle anti-fraud tools.

It can be concluded from this study that spectroscopic techniques such as portable NIR spectroscopy and hyperspectral imaging could be valid technologies for classifying asparagus samples of a given origin and, specifically in this case, for identifying samples belonging to the PGI of "asparagus from Navarre", which would enable fraud in the sector to be combated. The greatest complexity of these techniques lies in computing the classification models on a representative database including all the variability inherent to each origin. Once the models have been built, the determination is quick and it is carried out on the whole asparagus without processing or destroying the sample.

1. Food Fraud | Knowledge for policy (europa.eu)
2. Agriculture and rural development | Protected Geographical Indication (europa.eu)

P06.03

IN-SITU CHARACTERIZATION OF SEIZED ECSTASY USING A COMPACT NEAR-INFRARED SPECTROPHOTOMETER

Jennifer A. Cavalcante[1], Jamille C. Souza[2], Jarbas J. R. Rohwedder[1],
Adriano O. Maldaner[3], Celio Pasquini[1], **Maria C. Hespanhol**[2]

[1]Instituto de Química - Universidade Estadual de Campinas (UNICAMP), Campinas, Brazil, [2]Departamento de Química -Universidade Federal de Vicosa (UFV, Vicosa, Brazil, [3]Instituto Nacional de Criminalística - Polícia Federal (PF), Brasília, Brazil

Near-infrared (NIR) spectroscopy based on low-cost compact spectrophotometers is an excellent option for in situ analysis. However, these instruments have not yet been evaluated to characterize seized ecstasy. The identification of the primary active substance (3,4-methylenedioxymethamphetamine — MDMA or 3,4-Methylenedioxyamphetamine — MDA, in the case of ecstasy) and estimation actives' concentration help the expert to support immediate actions.

A NeoSpectra Fourier transform infrared (FT)-NIR spectrophotometer (Si-Ware - Egypt) operating in the range of 1350 – 2550 nm was used to acquire the spectra of 394 ecstasy samples seized during the years 2021 and 2022 characterized by liquid chromatography coupled to a mass detector and stored at the headquarters of the Federal Police of Brazil in Brasília - DF. Ecstasy tablets were ground, and their reflectance spectra were acquired during 6 s integration time and expressed in absorbance using spectralon® powder as a 100 % reflectance reference.

The spectra set was transformed using Savitsky-Golay second derivative (11 points symmetrical window and 2nd-degree adjusting polynomial) and further processed by the Unscrambler 11.0 chemometric software. Principal components analysis (PCA) of the spectra set shows that the scores of MDMA and MDA define well-separated groups in the first and second PCs, as shown in Figure 1.

A linear discriminant (LDA) model based on the scores of the three first PCs was constructed using 2/3rd of samples of each class selected by the Kennard-Stone algorithm and validated using the remaining 1/3rd samples. Validation shows an overall correct classification of 98 %. Results for SIMCA classification show worse results, with 25 % of the MDA samples classified as MDMA. Externally validated partial least square (PLS) models revealed that the content in % (m/m) of MDMA and MDA can be determined with a prediction error of 5.0 % (range 6 – 98 %) and 2.5 % (range 2 – 32 %), respectively. The results show that NIR spectroscopy based on compact instruments can be used for in-situ characterization of seized ecstasy according to its active ingredient.

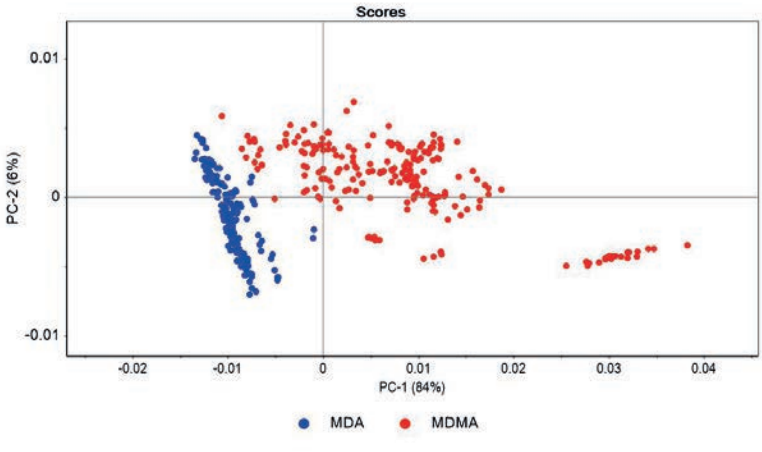


Figure 1. Scores of PCA for the first two principal components showing the separation of the groups of seized ecstasy samples containing MDA and MDMA.

Acknowledgment:
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P06.04

NEAR-INFRARED SENSOR FOR IN-LINE STATE-OF-CHARGE DETERMINATION OF ZEOLITE-BASED HEAT STORAGES

Evgeny Legotin[1,2], Gayaneh Issayan[3], Bernhard Zettl[3], Markus Brandstetter[1], Christian Rankl[1], Franz Winter[2]

[1]RECENDT – Research Center for Non-Destructive Testing GmbH, Linz, Austria, [2]TU Wien, Institute of Chemical, Environmental and Bioscience Engineering, Vienna, Austria, [3]University of Applied Sciences Upper Austria, Energy Research Group ASIC, Wels, Austria

Recent advances in thermochemical energy storage (TCES) pave the way to sustainable heat supply for domestic and industrial sectors. Among various types of TCES, high hopes are placed on sorption heat storage (SHS).

Finding a low-cost and reliable way to monitor the state-of-charge (SOC) of SHS in real-time is a relevant task to promote its application in future. The notion of SOC refers to the amount of sorbate (e.g. water) taken up by the SHS material. Up to now, a limited number of SOC sensor concepts have been proposed for SHS. Examples include sensors based on measuring the change in electrical properties of the material upon the adsorption or desorption of sorbate.

Recently, we demonstrated the applicability of near-infrared MOEMS technology to SOC estimation of diverse SHS materials in lab scale [1]. While the test set-up was simple and still providing satisfactory data, it was only suitable for water adsorption measurements but no desorption since operating under ambient conditions.

In the present study, a lab-scale set-up was built enabling both adsorption and desorption measurements under controllable conditions (temperature, air humidity and flow rate), which is considerably closer to practical applications. The tests were performed on a granulated Linde type-A zeolite material filled in a heat-resistant glass tank with NIR sensors mounted outside at the tank walls. During each experiment, the material was first "desorbed" (heated up to a certain temperature), then "adsorbed" stepwise at different temperatures and relative humidities. Spectroscopic NIR measurements were done in reflection geometry through the glass wall of the tank.

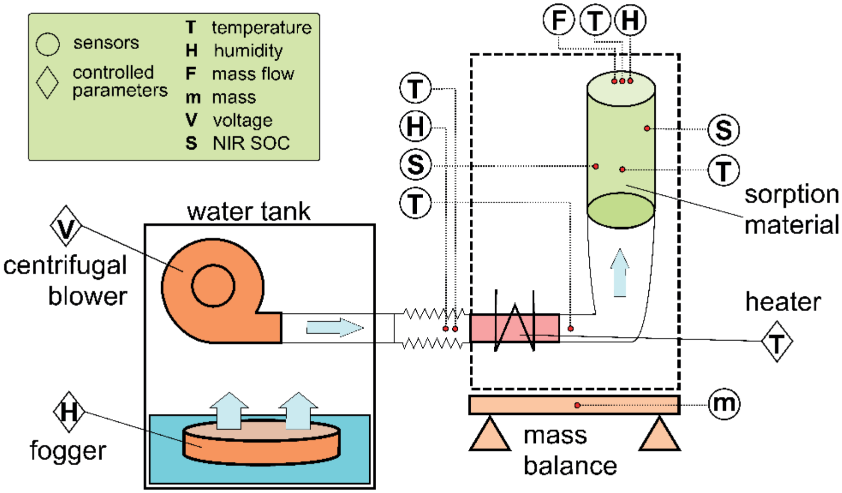

Figure 1. Schematic representation of the experimental set-up

Chemometric models were developed based on NIR spectral data with gravimetric reference to provide the means for continuous in-line SOC monitoring of SHS. The resulting models demonstrate good accuracy of SOC prediction.

This work has been supported by the government of Upper Austria and the European Regional Development Fund (ERDF) in the framework of the EU program IWB2020 with additional funds from the REACT-EU initiative (project RESINET). It is co-financed by research subsidies granted by the government of Upper Austria (project HIQUAMP).

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P06.05

INFRARED SPECTROSCOPY APPLIED TO FILO MOLLUSCA: A SYSTEMATIC REVIEW AND NEW TRENDS

Gabriela Friani[1], Vanessa Valladares[1], Mariana Guedes[1], **Clélia Mello-Silva**[1]

[1]Lapsa/ IOC/Fundação Oswaldo Cruz, Rio de Janeiro, Brazil

Infrared spectroscopy is a technique that can contribute to complex ecological studies based on changes in the spectra, which act as physicochemical fingerprints. The technique has emerged in the area of diagnostic and surveillance tool for molluscan-borne diseases and also for health and food safety. The present study was based on a systematic review of complete scientific papers published related to the application of infrared spectroscopy in organisms belonging to the Phylum Mollusca, identifying knowledge gaps and future perspectives. The descriptors used for the search were: Infrared Spectroscopy AND mollusc (and its variations), in the title, abstract and keyword in PUBMED, BVS and Web of Science databases, using PRISMA guidelines. The review of the identified studies included the spectroscopy technique used, the methodology, the class and species of mollusk and its habits, and main findings. A total of 36 studies were identified in the inclusion criteria. The class Bivalvia had the largest number of studies, especially mollusks living in saltwater. There was an increase in the application of NIR, especially in the last 5 years, with 6 articles. The topics studied were the differentiation of contaminated and uncontaminated bivalves by toxins, and also by heavy metals, including whether these animals are fresh or not. In addition, NIR spectroscopy was employed in species identification and parasitological diagnosis of Biomphalaria (intermediate host of Schistosoma mansoni). Therefore, NIR has the capacity to be a screening technique that composes surveillance strategies for schistosomiasis vectors and other molluscan-borne diseases, but studies with this focus are still scarce in the literature. Rapid and accurate diagnosis of these infections in human populations is essential to understand their transmission and initiate treatment immediately, as well as to improve and guide control and elimination strategies. NIR infrared spectroscopy is a non-invasive tool that has shown potential in species differentiation, diagnosis of parasitoses, and indication of environmental contamination by differentiating organisms with different metals and microplastics in mollusks and other aquatic organisms. Therefore, new trends and applications of NIR are related to the ability to indicate environmental changes and can be used as strategies to achieve different sustainable development goals.

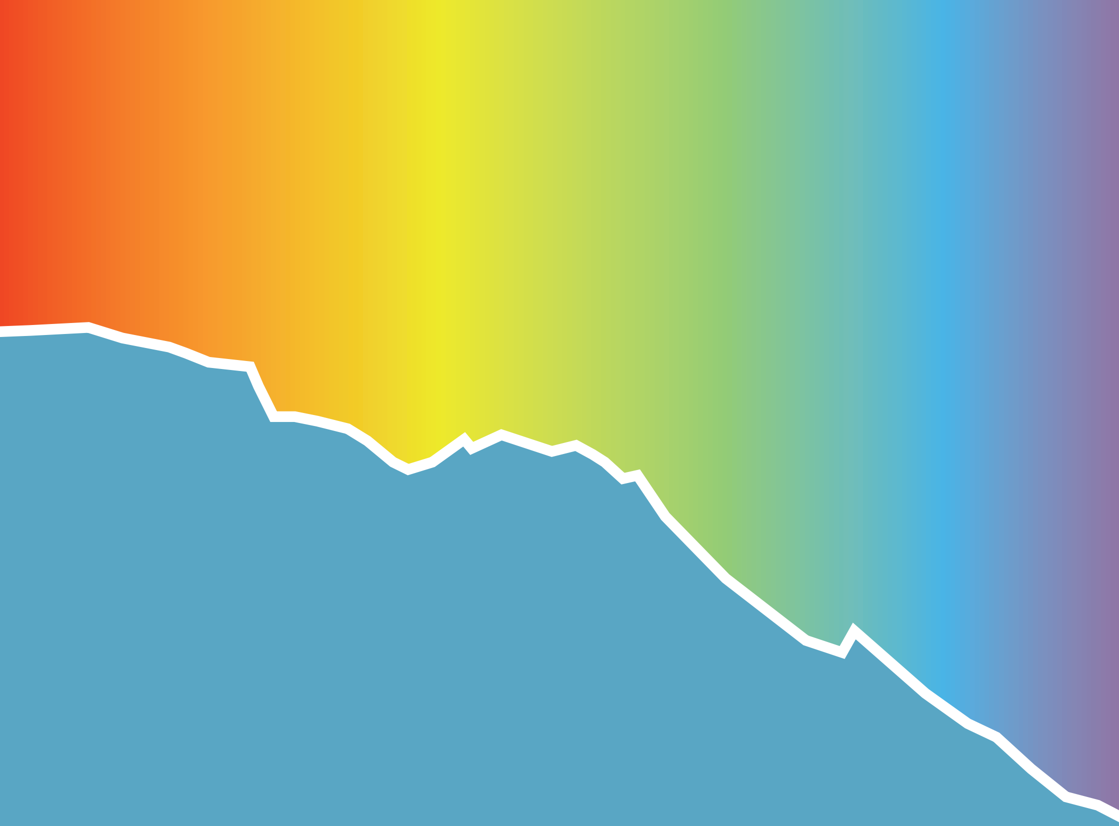
P06.06

INTRODUCING OPEN-SOURCE CHEMOMETRICS SOFTWARE AND WEB-SERVICES INTO THE TRADITIONAL NIR MANUFACTURING INDUSTRY

Leonardo Ramirez-Lopez[1], Michelle Diaz[2], Claudio Orellano[1]

[1]Buchi Labortechnik Ag, Flawil, Switzerland, [2]ETH, Zurich, Switzerland

Near-Infrared (NIR) prediction models need to be calibrated by end-users (or manufacturers) following a cumbersome process. We are using open-source software (OSS) to build Machine Learning (ML) web services that automate the calibration of NIR sensors. Based on this experience, here, we show how the NIR businesses can be transformed with the adoption of OSS and web services to minimize the complexity of chemometrics for the end-users. Although there is a vast amount of literature on OSS, information on how to put scalable OSS and NIR web services into production is scarce. Designing systems with rigorous testing and monitoring requires significant knowledge and effort. Integrating open-source developers into traditional NIR engineering teams might constitute a bottleneck. This talk describes how we have overcome the challenges that imply introducing open-source software and web services into traditional company structures.



THEME 07

HYPERSPECTRAL IMAGING

ORAL PRESENTATION: O07.02 – O07.13
FLASH TALK: F07.01 – F07.07
POSTER PRESENTATION: P07.01 – P07.23

007.02

FROM FIELD MONITORING TO POST-HARVEST SORTING: APPLICATION OF NIR SPECTRAL IMAGING SYSTEMS TO FACE HALYOMORPHA HALYS INSECT PEST

Rosalba Calvini[1], Veronica Ferrari[1], Camilla Menozzi[1], Peter Offermans[2], Lara Maistrello[1], Giorgia Foca[1], Alessandro Ulrici[1]

[1]University of Modena and Reggio Emilia, Reggio Emilia, Italy, [2]IMEC One Planet, Wageningen, Netherlands

Brown Marmorated Stink Bug (BMSB), Halyomorpha halys, is an emerging invasive pest which causes major economic losses in commercial fruit orchards. BMSB originates from East Asia but it rapidly spread in other areas, including the European continent. In Emilia-Romagna region (Italy) the spread of BSBM determined severe damages to pear orchards.

EU Project HALY.ID aims at developing innovative pest management strategies, which can be implemented both upstream to prevent BMSB activity on crops and downstream to manage fruit quality. In this context, the application of Near Infrared Hyperspectral Imaging (NIR-HSI) was evaluated at two levels: as a potential field monitoring method for BMSB detection and as a post-harvest sorting system to identify BMSB punctures on pears.

Considering the first application, NIR hyperspectral images of BMSB specimens on different vegetal backgrounds were acquired in the 980-1660 nm spectral range to mimic a field condition. Pixel-level classification models were calculated using sparse Soft Partial Least Squares Discriminant Analysis (s-Soft PLS-DA), a modified version of classical PLS-DA algorithm coupled with sparse-based variable selection which allows to identify the most relevant spectral regions to discriminate between BSBM specimens and vegetal backgrounds. Satisfactory results were obtained, with classification efficiency values higher than 95%. The spectral regions selected by s-Soft PLS-DA algorithm can be considered as a first step for the development of cheaper and faster multispectral imaging systems for on-field BSBM detection.

Moving to the second application, organic fruit samples belonging to two varieties (Williams and Abate Fètel) were collected in a pear orchard. For one week before harvesting about half of the fruits were exposed to BSBM while the remaining ones were used as control samples. NIR-HSI images (1156-1674 nm) of the fruit samples were acquired the same day of harvesting and at seven subsequent times over six weeks, obtaining a total of 1968 images.

Due to the high number of acquired images and to the blurred edges between sound and damages areas, the identification of Regions of Interest (ROIs) ascribable to punctures resulted a challenging task. To solve this issue, we proposed an innovative method to automatically identify the ROIs ascribable to punctures, which is based on combining data dimensionality reduction using the hyperspectrograms approach and feature selection algorithms.

007.03

APPLICATION OF NEAR-INFRARED HYPERSPECTRAL IMAGING AND CHEMOMETRICS IN THE EVALUATION OF DIFFERENT RICE VARIETIES

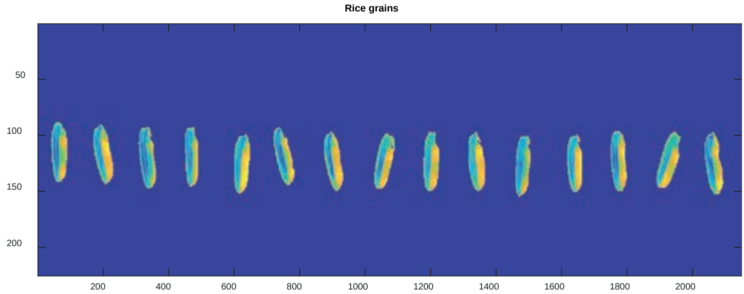
Elena Cazzaniga[1], Nicola Cavallini[1], Francesco Savorani[1], Francesco Geobaldo[1], Adrian Gomes-Sanchez[2], Anna De Juan[2]

[1]Politecnico Di Torino, Torino, Italy, [2]Universitat de Barcelona, Barcelona, Spain

Hyperspectral imaging (HSI) is a non-destructive analytical technique that provides both spectral and spatial information about the analysed sample. Each pixel of an HS image contains a spectrum, thus it is possible to obtain the chemical fingerprint of the sample across its whole surface. Moreover, morphological features such as area, circularity or roundness can be studied. HSI can be applied for analyses in many fields, such as agro-food industry, medical diagnostics, analysis of pharmaceutical products and astronomy [1].

One of the main advantages of the use of HSI is the non-invasive and non-destructive nature of the technique. As a consequence, it is possible to analyse the sample as such or with minimum preparation, allowing to save analysis time. It is possible to find HSI techniques coupled with all kind of spectroscopies and there are many agro-food applications based on the use of the visible and near-infrared (NIR) ranges of wavelengths. The data obtained by HSI can be displayed as a data cube, with two spatial dimensions and a spectral one. In order to handle the complex and plentiful amount of information, this technique is usually combined with multivariate analysis and deep-learning tools [2,3].

The present work aims at showing the power of near-infrared HSI combined with chemometrics in the agro-food field, analysing 54 different varieties of rice. For this purpose, a NIR-HSI camera (SPECIM FX17, Finland) connected with a motorized scanning bed (LabScanner Setup 40´20 cm) was used to collect 54 hyperspectral images, one for each rice variety, with 15 rice grains per image (as shown in the figure). The images were then inspected through multivariate data analysis, using unsupervised exploratory tools such as Principal Component Analysis (PCA), and classification methodologies such as Partial Least Squares-Discriminant analysis (PLS-DA). The potential of using spectral, morphological or the morphological/spectral fusion to discriminate among varieties is explored. Work at a single grain level or at a pixel level is also studied. The obtained results provided interesting insights into the chemical and physical differences occurring among the rice varieties under examination.



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007.04

NEAR INFRARED HYPERSPECTRAL IMAGING: SPECIFIC SOLUTIONS FOR SPECIFIC CHALLENGES STILL TO BE SOLVED

Juan Antonio Fernández Pierna[1], François Stevens[1], Vincent Baeten[1]

[1]*Walloon Agricultural Research Centre, Gembloux, Belgium*

More than 30 years ago, the NIR technology was coupled to the imaging technology to create what is actually the well-known Hyperspectral Imaging Spectroscopy (HIS), which today constitutes an active sector in agricultural studies. HIS allows for objective, not destructive, repetitive and rapid observation of objects and is perfectly adaptable to samples of very different nature. Thanks to HIS, the conformity of products can be constantly optimized by collecting thousands of spectra of one sample/process to obtain information about composition and spatial or temporal distribution.

The analysis of hyperspectral images for the development of analytical workflows (discrimination, quantification, quality assessment, etc.) linked to research or industrial applications needs to overcome some important challenges related to spectral noise reduction, hypercube size reduction, background removal, image exploration, image cleaning, physical effects as well as the spectral and spatial information combination.

This work shows how the HIS has managed to gain a foothold in an agro-food laboratory and even become a key part of it. It will be also shown the advantages of moving this technique to the fields and we will focus on the main challenges encountered when dealing with hyperspectral data and images. We try to propose specific solutions based on data treatment methods for the different specific challenges defined. All of this is demonstrated with real thematic from the agro-food world.

007.05

MEASUREMENT OF PAPER, COLOURED MATERIALS, AND LAMINATION FILMS USING A FOURIER-TYPE NEAR-INFRARED HYPERSPECTRAL IMAGING SYSTEM.

Shigeru Sugawara[1], Ichiro Ishimaru[2]

[1]National Research Institute of Police Science in Japan, Kashiwa, [2]Kagawa University, Takamatsu, Japan

Visible and near-infrared hyperspectral imaging (HSI) technology is used in document examination. This makes it possible to determine the authenticity of securities, detect tampering and visualize samples that have been painted over or become faded. The technology uses Si-CCD and Si-CMOS detectors in the 0.4-1.0-µm wavelength band, and relatively inexpensive devices can be built.

Light detection at longer wavelengths requires detectors such as InGaAs instead of Si, and HSI devices coupled with spectrometers have also been developed and marketed. This study uses a near-infrared HSI system capable of detecting wavelengths from 1 to 2.35 µm to measure the spectral properties of different materials, including different types of paper, printed matter, and laminated films. The ability to obtain new information which could not be determined by conventional HSI techniques with a wavelength of 0.4–1.0 µm is assessed.

There are two types of spectrometers in HSI equipment, dispersive and Fourier type, each having distinct characteristics. The Fourier type has a higher light throughput than the dispersive type and can produce a more sensitive instrument when the same detector is used. This study presents the measurement results obtained using a Fourier-type HSI instrument, showing that the amount of light required for measurements can be monitored and that measurements can be made even with samples having high absorbance, and when light is emitted from a distant light source.

007.06

GEOMETRICAL INFLUENCE CORRECTION OF APPLE'S NEAR INFRARED HYPERSPECTRAL IMAGES FOR EARLY BRUISE DETECTION

Bin Li[1], Leshang Bai[1], Te Ma[1], Seki Hayato[1], Tetsuya Inagaki[1],
Satoru Tsuchikawa[1]

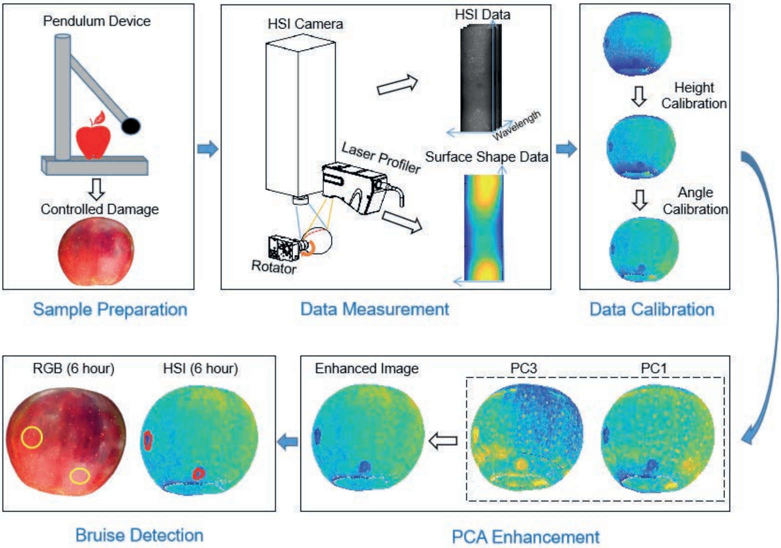
[1]Nagoya University, Nagoya, Japan

The detection of early bruising in apples presents a significant challenge, as the oxidative browning of the damaged tissue requires time to become visible. Near-infrared (NIR) hyperspectral imaging (HSI) offers a promising solution for identifying early bruising in apples due to its sensitivity to the water released by damaged tissue. However, current HSI image measurements impose considerable limitations on the detection of apple surface, including: 1) the assessment of only a restricted area on the surface from a single direction; and 2) the substantial height and curvature variations of the apple surface complicate the detection of bruises in shadowed edge regions.

To address these challenges, we propose an exploratory method for the overall detection of early apple bruising. We prepared 72 healthy apple samples and subjected them to controlled damage. By the combining use of a NIR-HSI camera and a laser profiler, We then collected HSI image data and shape data from most of the apple surface through rotational measurement. Data were gathered from the samples before damage and at one-hour intervals for six hours post-damage. With the shape data, We employed a model-based height calibration method and Lambert's cosine law to correct for surface height and angular influences on the HSI data, thereby achieving greater uniformity in the spectral data. Principal component analysis (PCA) was conducted, and by integrating images of PC1, PC2, and PC3, we reduced the secular reflectance's impact on PCA scores to stabilize the PC loading, and enhanced the differences between bruised and healthy regions. Eventually, We implemented a straightforward bruise detection method based on threshold segmentation and moving-window variation calculation, with a consistent threshold for all samples at all times.

The results highlighted a notable improvement in detection performance over time when calibration was applied. For uncalibrated samples, the overall accuracy of detecting bruised apples increased from 43% to 71% over six hours, with central bruise accuracy rising from 39% to 51% and edge bruise accuracy from 13% to 32%. In comparison, calibrated samples exhibited an increase in overall bruise detection accuracy from 65% to 90%, central bruise accuracy from 64% to 83%, and edge bruise accuracy from 19% to 61%. In conclusion, this study expands the detection area for early apple bruising and presents a potential solution for samples with diverse sizes and shapes.

Flowchart of the Early Apple Bruise Detection Program



007.07

SHINING HYPERSPECTRAL LIGHT ON AGRICULTURAL PRODUCTS: AN APPLICATION STORY FROM STRAWBERRY

Na Liu[1], Karen Wahlstrøm Sanden[2], Julio Hernandez[1], Jens Petter Wold[2], Silje Ottestad[1]

[1]Norsk Elektro Optikk AS, Oslo, Norway, [2]Nofima AS, Ås, Norway

Strawberry is one of the most popular fruits in today's market. Brix, acid, and the Brix/acid (B/A) ratio are three important traits of eating quality of strawberries. However, it is difficult to conduct massive quality inspection with these traits using traditional methods, as berries are eaten fresh and with short shelf-life, while wet chemical analysis are destructive and labor/time-cost. Innovative improvements are therefore needed to solve these challenges. Hyperspectral imaging (HSI) technique has become increasingly attractive in food and agriculture sectors because of its effectiveness and versatility for quality evaluation. Here we present a fast and non-destructive HSI solution developed and tested on fresh strawberries. The system was composed of a compact lab rack and a dual-camera setup, covering the spectral range from 400 to 2500 nm. Machine learning approaches were employed to predict the three fruit quality traits using the collected hyperspectral images.

Results showed that HSI technique with machine learning can provide reliable estimation of fruit eating quality, and furthermore, relate the chemical properties to the spatial information. Good performance of models was obtained for Brix, acid and the B/A ratio, with the prediction error rates around 4% - 12%. The spatial distribution of all three target traits within a single berry were also mapped based on the optimized regression models, without interference of calyx and background. The mapping results showed that the B/A ratio is generally higher at the apex of strawberry, in correspondence to the fact that the fruit taste is highly related to its sweet-sour balance. HSI technique could thus allow to inspect maturity and eating quality on single fruit basis and give prediction on both object- and pixel- level.

This customized HSI system, along with powerful machine learning approaches, makes automated characterization of heterogenous biological samples much faster and more efficient. In addition to revealing chemical composition and spatial distribution, this solution can also help enhance the understanding of physiological status, damage, pathogen infection, and responses to abiotic/biotic stress of agricultural products. More examples on berries, cocoa bean, tobacco, and meat were included as well, demonstrating the huge potential of HSI techniques to develop non-destructive and automatic system for real-time grading, inspection, quality control and processing in food industry.

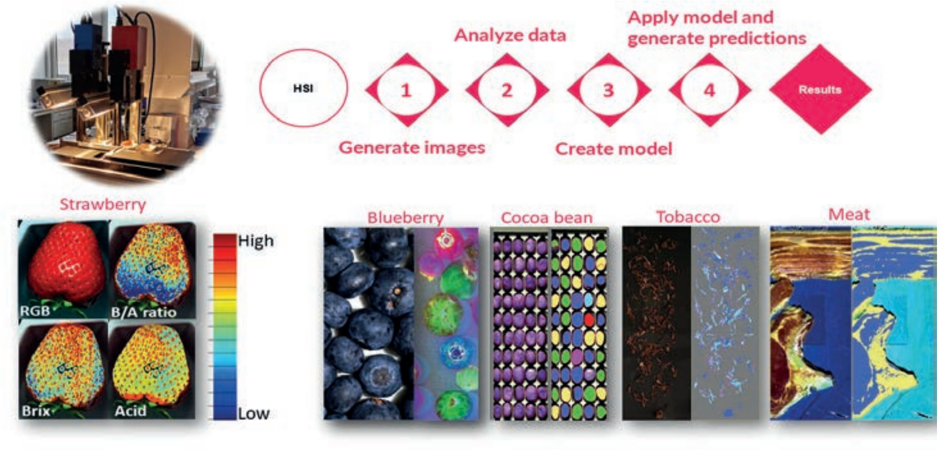


Figure 1. Applications of hyperspectral imaging system with machine learning approaches for quality control of strawberry and other agricultural products.

007.08

EXPERIMENTAL STUDY AND MODELING OF MOISTURE TRANSPORT IN WOOD BY MEANS OF NEAR-INFRARED HYPERSPECTRAL IMAGING COUPLED WITH A MASS TRANSFER SIMULATION METHOD

Te Ma[1], Tetsuya Inagaki[1], Satoru Tsuchikawa[1]

[1]Nagoya University, Nagoya, Japan

Considerable efforts have been made to visualize and simulate water movement properties in wood. However, three-dimensional (3D) thermo-hydric simulations remain challenging due to the wood's heterogeneous microstructure, which leads to intricate computational models, and the absence of suitable experimental methods for supporting and validating model construction. In this research, we initially visualized moisture distribution in wood during water adsorption and desorption processes using a high-resolution, sensitive, and stable long-wave near-infrared hyperspectral imaging (NIR-HSI) technique. Subsequently, the parameters of a mass transfer simulation code were adjusted based on the moisture visualization results. The visualization and simulation outcomes aligned well with various features, such as slower drying rates in denser wood sections. Furthermore, a significant gradient was observed on the wood sample surface layers during drying, while this characteristic was not prominent during the water adsorption process. As a result, this study suggests that the long-wave NIR-HSI method can be integrated with 3D thermo-hydric simulations and model construction, a combination warranting further exploration.

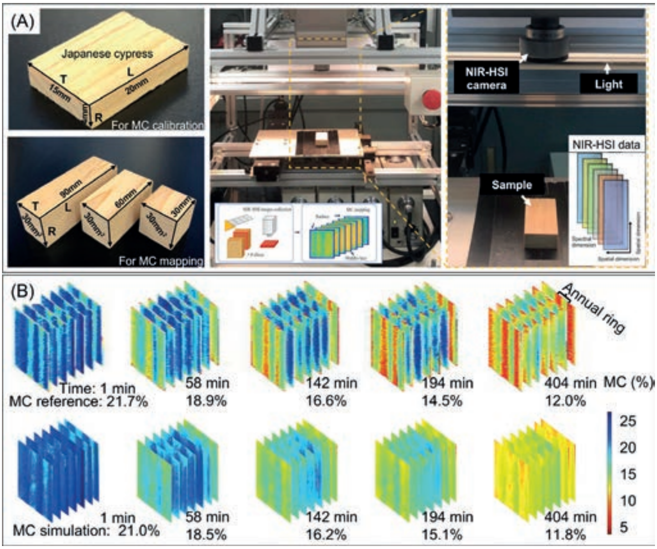


Figure 1. (A) Flowchart of the NIR–HSI experiment; (B) MC mapping and simulation results.

007.09

THE POWER OF NIR HYPERSPECTRAL IMAGING FOR VISUALIZING WATER PATTERNS DURING DEHYDRATION OF NONVASCULAR EPIPHYTIC COMMUNITIES

Cristina Malegori[1], Giulia Canali[2], Rodrigo Rocha de Oliveira[1,3], Sara Gariglio[1,4], Paola Malaspina[2], Riccardo Voccio[1,4], Monica Casale[1], Paolo Oliveri[1], Paolo Giordani[1]

[1]DIFAR – Department of Pharmacy, University of Genova, Genova, Italy, [2]DISTAV – Department of Earth, Environment and Life Sciences, University of Genova, Genova, Italy, [3]Department of Chemical Engineering and Analytical Chemistry, Universitat de Barcelona, Barcelona, Spain, [4]DCCI – Department of Chemistry and Industrial Chemistry, University of Genova, Genova, Italy

The present work is aimed at developing water maps for understanding how a micro-ecosystem composed by nonvascular epiphytic communities (mainly, lichens and bryophytes), manages water and, consequently, how the thalli can prolong water availability depending on the morphology of the species and the composition of the community itself. To achieve this aim, the water status of bark, lichens (crustose and foliose) and bryophytes was monitored by means of near-infrared (NIR) hyperspectral imaging (HSI), working in the spectral range from 1000 to 2500 nm (SWIR3 – Specim, Finland).

Regarding the sampling protocol, it was possible to analyse pure communities (composed mainly by a single organism), binary and ternary mixtures, for a total of 32 samples. All of the micro-ecosystem were naturally supported by chestnut bark. Samples of non-colonised (or as less colonised as possible) bark were also analysed.

The samples were first submitted to a full hydration protocol and, then, let dry at room temperature (19°C) and humidity (41%) for about 12 h. During the dehydration period, NIR-HSI acquisition was carried out every 45 minutes for 15 sampling points, obtaining 480 images. Before image acquisition at each sampling point, the samples were weighed to quantify their dehydration curve, relative to their weight, prior to the hydration phase. On the images, an exploratory data analysis was performed, by means of principal component analysis (PCA), with the aim of comparing the drying trajectory in the orthogonal space defined by the lowest-order components with the dehydration curve. The study of these correspondences on pure communities opens the possibility to understand the water management of each organism and then to use such information in the study of more complex microclimates (binary and ternary mixtures). The projection of the microclimate images in the space defined by the pure samples allows to map changes in the water status of each complex sample.

After this exploratory step, a supervised strategy is proposed, with the aim of extracting not only the water pattern inside the microclimate but also a numerical parameter expressing the water content in the different regions of interest inside a single map.

The study of these communities and their relative behaviour in water stress condition plays a crucial role in understanding how these organisms are facing the climate change and the subsequent water scarcity or overabundance.

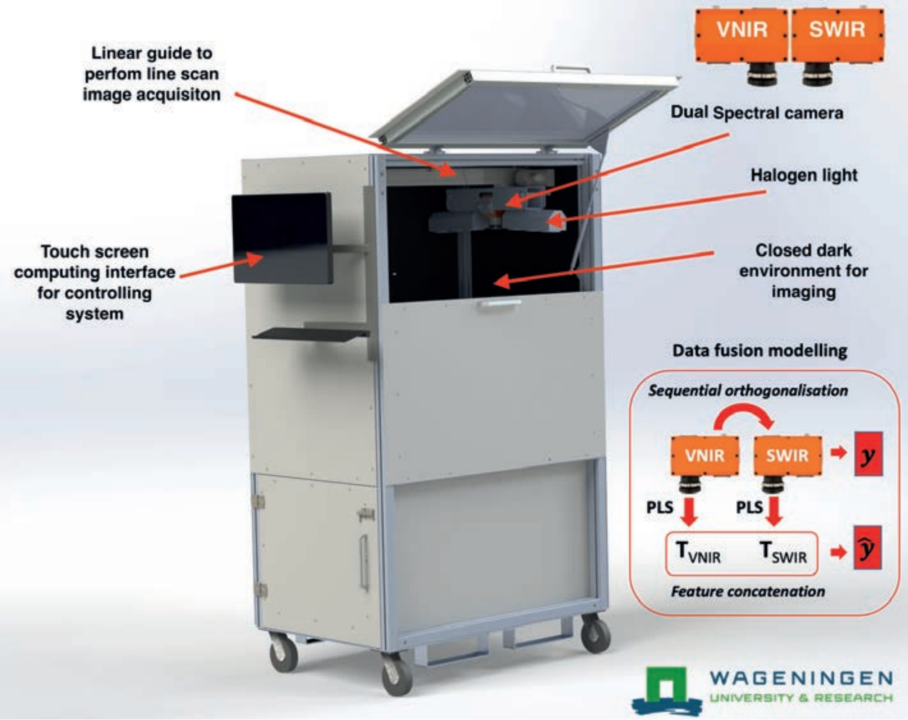
007.10

A NOVEL NEW APPROACH TO STANDARDISED PORTABLE MULTIMODAL HYPERSPECTRAL IMAGING : ALL-IN-ONE HYPERSPECTRAL IMAGING

Puneet Mishra[1], Gerrit Polder[1], Jan Verschoor[1], Mariska Nijenhuis-de Vries[1], Erik Pekkeriet[1], Aneesh Chauhan[1], Menno Sytsma[1], Martin P. Boer[1]

[1]*Wageninen University And Research, Wageningen, Netherlands*

In analytical chemistry, spectral imaging (SI) to evaluate the spatially distributed physicochemical characteristics of samples. Despite advancements in instrument and chemometrics modeling, the use of SI in daily usage by non-experts presents a significant challenge as the available sensors require extensive system integration and calibration modeling before they can be used for regular analysis. Moreover, models developed during one experiment are seldom useful when the system is reintegrated for a new experiment. This study presents an intelligent All-In-One SI (ASI) laboratory system [1], which offers standardized automated data acquisition and real-time spectral model deployment, to overcome the need for system reintegration and reuse calibrated models. The ASI system includes a standardized illumination environment, an in-built computing system, embedded software for automated image acquisition, AI for object detection, and model deployment for predicting the spatial distribution of sample properties in real-time. The paper showcases the ASI framework's ability through examples of predicting fruit properties in different fruits [2]. Additionally, the performance of ASI is benchmarked against commercially available portable and high-end laboratory spectrometers.



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007.11

DEVELOPMENT OF A PORTABLE HYPERSPECTRAL IMAGING SYSTEM FOR CITRUS DISEASE DETECTION

Jianwei Qin[1], Pappu Kumar Yadav[2], Thomas Burks[2], Insuck Baek[1], Moon Kim[1]

[1]USDA/ARS Environmental Microbial and Food Safety Laboratory, Beltsville, MD, United States, [2]Department of Agricultural and Biological Engineering, University of Florida, Gainesville, FL, United States

Early identification of citrus diseases can help growers take mitigation efforts to prevent disease spreading in orchards and thus minimize financial loss. This study aims to develop a portable hyperspectral system (Fig. 1) for citrus disease detection using both reflectance and fluorescence imaging in visible and near-infrared (VNIR) wavelength range (400–1000 nm). The system mainly consists of two LED line lights providing VNIR broadband and UV-A (365 nm) light for reflectance and fluorescence measurement, respectively, a miniature line-scan hyperspectral camera, and a linear motorized stage with a travel range of 30 cm. A sample holder was custom designed and created to hold citrus leaf and peel samples in four (2×2) identical partitions. A customized reflectance standard panel is side mounted to the sample holder for flat-field correction to the reflectance images. The linear stage translates the samples under the lights and the camera to acquire a pair of reflectance and fluorescence images in sequence during one cycle of line-scan imaging. System software was developed using LabVIEW to realize hardware parameterization and data transfer functions. The entire system was built on a 45×60 cm2 optical breadboard, which is easily transportable and suitable for on-site and field experiments. The system was used at Citrus Research and Education Center of University of Florida to collect images from citrus leaves with healthy and four diseased conditions (i.e., canker, greasy spot, melanose, and scab). Hyperspectral image processing procedures and machine learning and deep learning classification models will be developed to differentiate the citrus diseases using both reflectance and fluorescence spectral and image data. Complete experimental details and data analysis results will be presented in the NIR 2023 conference.

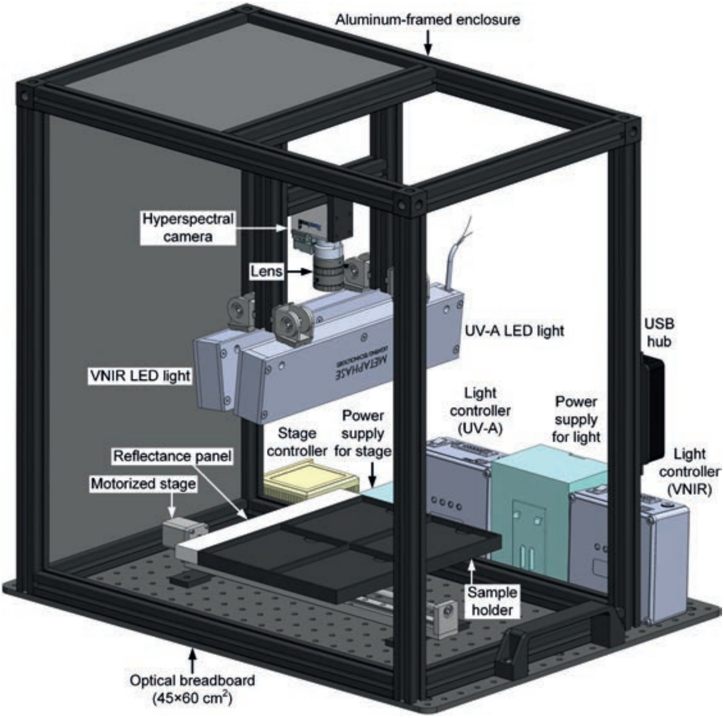


Figure 1. A portable hyperspectral reflectance and fluorescence imaging system for citrus disease detection.

007.12

HYPERSPECTRAL IMAGING AS A HIGH-THROUGHPUT METHOD FOR MONITORING FUNGAL COLONIZATION ON BUILDING MATERIALS

Karen Butina-Ogorelec[1,2], Ana Gubenšek[1,2,3], Jakub Sandak[1,2], Faksawat Poohphajai[1,2,4], **Anna Sandak**[1,2]

[1]InnoRenew CoE, Izola, Slovenia, [2]University of Primorska, Koper, Slovenia, [3]University of Ljubljana, Ljubljana, Slovenia, [4]Aalto University, Aalto, Finland

Materials exposed outdoors are prone to various deterioration processes. Architectural coatings are designed to protect surfaces against environmental and biotic degradation and/or to provide a decorative layer. Common surface treatments often include mineral oil binders and other ingredients that are known to have negative impacts on the environment. The use of toxic substances is increasingly restricted by legal regulations due to awareness of their negative environmental impacts. An alternative bioinspired concept for materials protection based on fungal biofilm is recently investigated in the ARCHI-SKIN project. In the first phase the morphological, functional, and social interactions in fungal communities naturally appearing on building materials are deeply investigated. The assessment of a variety of fungi that can thrive on exposed surfaces and understanding their interactions with various building materials is crucial to identify fungal species with a protective potential.

A set of 34 wood-based cladding materials were exposed and monitored in outdoor conditions. Hyperspectral imaging (HI) as a high-throughput method was used to understand the effect of fungal growth on material properties as well as to objectively quantify the infested area. Hyperspectral images were acquired with the FX17 camera (Specim, Finland) and LumoScanner program (Middleton Spectral Vision, Wisconsin). Data were analysed using Evince (Prediktera, Sweden). Spectral data were pre-processed using SNV before PCA. The loadings of the first principal components were plotted using Prism 9 (GraphPad Software, Massachusetts).

Simultaneously to hyperspectral imaging microbial growth was evaluated by wet swab technique and molecular identification of prevalent species. The surfaces were sampled using the wet swab and plated on DG-18 agar, which prevents the growth of bacteria and limits the growth of fast-growing fungi. Pure cultures were then isolated and identified through PCR amplification and Sanger sequencing of specific DNA regions/genes. Multi-sensor and multi-scale measurements were performed to assess the influence of surface properties (wettability and roughness) and material structure on fungal growth, species variability, and dominance. Proposed techniques enabled the identification of features that promote/inhibit fungal colonization and revealed the preference of certain fungi for specific materials. Both the material type and the climate condition at the exposure site influence fungal colonization and the variability of microorganisms. The samples in close spatial proximity exhibited different fungal microbiota, demonstrating that fungal colonization across samples is not random. This study is a starting point for more exhaustive assays that aim to develop novel solutions for materials protection based on controlled and optimized fungal biofilm formation.

007.13

ENHANCING POULTRY MEAT SAFETY: REAL-TIME FOREIGN MATERIAL DETECTION THROUGH HIGH-PERFORMANCE DEEP LEARNING-ENABLED NIR HYPERSPECTRAL IMAGING

Seung-Chul Yoon[1], Bosoon Park[1], Brian Bowker[1]

[1]USDA - Agricultural Research Service, Athens, United States

This study focuses on the development of inline sensing technology using hyperspectral imaging (HSI) and high-performance deep learning (DL) for the real-time detection of foreign materials (FMs) on chicken breast meat. HSI and DL are useful tools for assessing agricultural and food products' safety and quality features. However, most HSI-based DL models for food quality and safety assessment lack real-time sensing capabilities critical for industrial deployment, due to the high computational demands of both HSI and DL. Therefore, we propose near-infrared hyperspectral imaging (NIR-HSI) coupled with a DL model based on the generative adversarial network, suitable for high-speed food production applications such as inline sensing for detecting FMs during poultry processing. To achieve real-time HSI-based DL inferencing, the hardware and software should be capable of acquiring and analyzing high-volume data in a timely manner (approximately 0.25s per fillet). A line-scan NIR-HSI camera (1000-1700 nm) with a frame rate of 670 Hz is used for data acquisition. For real-time imaging and high-performance DL inferencing, the software system implemented in C++ employs a double-buffer memory architecture for parallelization of data acquisition, utilizing a multithreaded procedure and two buffers that flip-flop between the receiving and processing of hyperspectral data. The acquired and pre-processed hyperspectral data are sent to a GPU for FM detection, utilizing CPU threads to mitigate the transfer latency by asynchronously creating multiple streams and overlapping multiple transfers with kernel execution. Inside the GPU, tensor cores are utilized to perform inference on a 1D U-net classifier, trained as an anomaly detector. The high-performance DL algorithm utilizes the weights of the previously trained model to detect abnormal spectral responses of any FMs, combining global and local classification for a final prediction. A detection map is obtained by computing the loss value of the network's prediction. The FM detection framework can perform pixel-wise detection as well as object-level detection. The proposed model is evaluated for its speed and accuracy with a hyperspectral image dataset comprising 12 boneless skinless breast fillets and 30 different types of FMs. When fully developed into a real-time HSI system and tested, it is expected to achieve high-performance DL inferencing for online hyperspectral image classification of FMs in real-time.

F07.01

EARLY DETECTION OF PENICILLIUM DIGITATUM USING HYPERSPECTRAL IMAGING AND DEEP LEARNING

Salvador Castillo Girones[1], Ricardo Felipe Lima de Souza[1], Sandra Munera[3], Alejandro Rodríguez[3], Sergio Cubero García[1], Juan Gómez-Sanchís[2], Nerea Martinez Onandi[1], Marina Lopez Chulia[1], Jose Blasco[1]

[1]Instituto Valenciano De Investigaciones Agrarias, Moncada, Spain, [2]Universidad de Valencia, Burjassot, Spain, [3]Universitat Politècnica de València, Valencia, Spain

Fungal infections are a main concern in fruit packing houses since one infected orange can cause a whole storage room to be infected in case the infection is not detected. For that reason, early detection before the infection is visible by the human eye is crucial. In many packing houses, UV light is used to manually sort oranges, but it can be dangerous for the eyes. Besides, at the very beginning of the infection, it can be difficult to detect it even using UV light, and since the human eye gets exhausted when working for hours, that makes human early detection not suitable for the task. However, hyperspectral imaging can solve that problem since it avoids the use of UV light and humans due to its ability to detect not only external but also internal information of the fruit.

For that reason, a preliminary study was carried on using 100 oranges of Navel-late variety. 50 of them were inoculated with distilled water as a control, and 50 of them with Penicillium Digitatum with a concentration of 106 spores ml-1. After that, they were stored at 20ºC and 90% relative humidity for 4 days. Images were taken at the day of inoculation and every day for 4 days after inoculation, taking a total of 500 images.

Hyperspectral images of these fruits were acquired with a VIS/NIR pushbroom hyperspectral imaging system (900-1700 nm) and were corrected with the white and dark reference and the background was removed. Images were then divided into train (60% of the images), validation (20% of images) and test (20% of images) datasets of healthy and inoculated images.

Unlike most authors who use 2D CNNs, we have used a 3D customized CNN which is designed for 3-dimension data, and which allows obtaining more spectral information than 2D CNNs. Images were resized to 100 x 100 pixels due to computational limitations and data augmentation was applied before training to increase the number of samples.

Results showed that after one day, the model was able to classify correctly 100% of the control oranges and 80% of the inoculated ones, proving that this technology might be able to predict infection after it is visible by the human eye and even when using UV light. However, this should and will be studied in a future using more samples and better computational resources as to improve the model and the image resolution.

F07.02

NON-DESTRUCTIVE MONITORING AND CLASSIFICATION OF POTATO QUALITY DURING STORAGE USING HYPERSPECTRAL IMAGING

Ye-Na Kim[1], Haeun Kim[1], Hangi Kim[1], Byoung-Kwan Cho[1]

[1]Department of Biosystems Machinery Engineering, Chungnam National University, 99 Daehak-ro, Yuseong-gu, Daejeon, South Korea

Potatoes are one of the most important food crops worldwide. As they are consumed throughout the year in the form of chips and French fries, long-term storage is required after harvest. The biggest challenge in the utilization of potatoes is the difficulty in controlling sprouting during storage. Although properties such as moisture content and sprouting capacity of potatoes are important factors during storage, sorting based on these properties is not performed because it is impossible to confirm by visual inspection. In this study, the quality of potatoes was monitored through spectroscopic imaging during the storage period, and the models were developed to classify potatoes. While potatoes of the same variety and harvest time were stored under the same conditions, weight change, moisture content, sprouting capacity, and spectroscopic images through Vis/NIR and SWIR hyperspectral imaging systems were confirmed during the storage period. Partial Least Squares models that quantitatively predict the moisture content and sprouting of potatoes using the spectra extracted from spectral images and classify potatoes according to quality were constructed. The sprouting capacity calculated as the ratio of the sprout weight to the initial tuber weight showed differences for each potato even though the potatoes were stored under the same conditions. These findings will contribute to the development of non-destructive methods for pre-screening potatoes unsuitable for long-term storage and monitoring their quality during storage period.

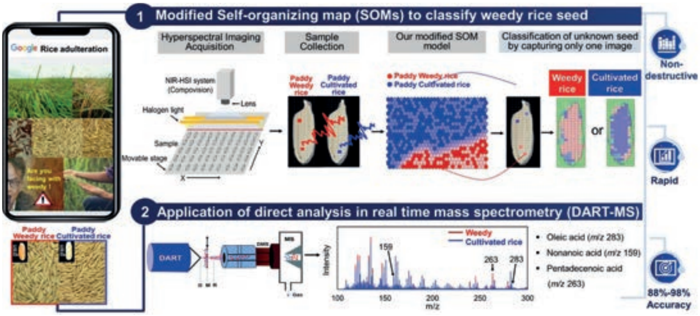
F07.03

MAPPING HYPERSPECTRAL NIR IMAGES USING SUPERVISED SELF-ORGANIZING MAPS: DISCRIMINATION OF WEEDY RICE SEEDS

Sureerat Makmuang[1], Sanong Ekgasit[1], Kanet Wongravee[1]

[1]Chulalongkorn University, Bangkok, Thailand

Weedy rice (Oryza sativa f. spontanea) is one of the aftermaths of the mutagenesis. It is the most problematic weeds occurring in rice-growing regions worldwide, particularly in Southeast Asia. Unlike other types of weeds, it is extremely difficult to distinguish weedy rice from cultivated rice directly from paddy seeds as they exhibit similar morphological features. As such conventional weed management can be a difficult, time-consuming, and inaccurate process that is often carried out manually. This study offers a novel classification approach based on an artificial neural network (ANN), utilizing self-organizing maps (SOMs), to directly discriminate weedy rice from cultivated rice on paddy seed in situ using the near-infrared (NIR) hyperspectral imaging (HSI) technique. The physical attributes, thermal behaviors, and chemical profiles of the weedy and cultivated rice were thoroughly investigated by a range of analytical techniques, including optical microscopy, scanning electron microscopy, thermogravimetric analysis, and direct analysis in real-time mass spectrometry (DART-MS). For direct sample analysis by HSI, the global self-organizing map was generated with the optimized parameters (scaling value and map size). The color indices (Red, Green, Blue values) of the sample image were defined to obtain a color ratio that can be used to classify unknown samples which different classes representing in different color shades. The optimal threshold for classification was carefully determined using a receiver operating characteristic (ROC) curve. Performance metrics (sensitivity, specificity, precision, accuracy, and misclassification error) were used to evaluate the performance of the model. Classification accuracies of 98% (Weedy vs PL2) and 88% (Weedy vs RD49) were obtained with the balanced sensitivity and specificity. The classification was assessed from the whole sample image, which was completely independent of the selected region of interest. As far as we know, this is the first instance where SOMs have been utilized to appraise seed quality by means of authentic HSI images. In the future, this work could be expanded to apply to other economic seed systems as well as develop an end-to-end functionality of a low-cost multispectral camera as a portable NIR device instead of expensive, standard laboratory instruments to reach a larger user community for seed quality inspection.



F07.04

DIFFERENTIATION OF *LISTERIA MONOCYTOGENES* SEROTYPES USING NEAR INFRARED HYPERSPECTRAL IMAGING

Rumbidzai Matenda[1], Paul Williams[1], Diane Rip[1]

[1]Stellenbosch University, Stellenbosch, South Africa

Among the severe foodborne illnesses, listeriosis resulting from the pathogen Listeria monocytogenes exhibits one of the highest fatality rates. In this study, near infrared hyperspectral imaging (NIR-HSI) was investigated for the detection and classification of three L. monocytogenes serotypes. The serotypes L. monocytogenes 4b (ATCC 23074), L. monocytogenes 1/2a (ATCC 19111) and L. monocytogenes 1/2c (ATCC 7466), which attribute to most listeriosis clinical cases, were grown on brain heart infusion agar for single colonies. After incubation, a HySpex short wave infrared pushbroom imaging system was used to collect images in the spectral range 950-2500 nm. Principal component analysis was used to remove unwanted pixels of the petri dish, agar, and background pixel noise. Standard normal variate and the Savitzky-Golay smoothing (1st derivative 2nd order polynomial with 11 points) were applied for scatter correction and noise reduction The score plots using five principal components (PCs) indicated that PC1 and PC3 accounted for 57% and 4% of the total variation respectively. There were three clusters that could be noticed, with almost a clear separation between L. monocytogenes 1/2a and L. monocytogenes 1/2c along PC3. An overlapping of the three serotypes along PC2 was attributed to the similar carbohydrates and proteins found in the bacterial cell wall. Partial Least Squares Discriminant Analysis was investigated for classification, and the results showed that the model achieved an overall classification accuracy of 82%. Sensitivity values of prediction for L. monocytogenes serotypes 4b, 1/2a, and 1/2c were 0.76, 0.87 and 0.98, respectively. Results were indicative of the inability of the model to correctly identify L. monocytogenes 4b. Variable importance in projection scores were used to assess the spectral region with the most relevance. Bands at 1415 nm (lipoteichoic acids), 1580 nm (carbohydrates), and 1660 nm (proteins) were identified as the most important for class differentiation. The findings of this study suggest that NIR-HSI is a promising technique for detecting and classifying L. monocytogenes serotypes on agar media. The identification of relevant spectral regions for class differentiation could contribute to more accurate, fast, and effective detection of this pathogen, potentially improving food safety and public health. Further research could focus on the application of this technology on food products.

F07.05

FEASIBILITY OF SPECTRAL IMAGING AND DATA FUSION FOR CLASSIFICATION OF TURKISH WHEAT KERNELS

Gozde Ozdogan[1], Aoife Gowen[2]

[1]University College Dublin, Dublin, Ireland, [2]University College Dublin, Dublin, Ireland

Wheat is the second most used grain type in the world. Identification and verification of wheat grain samples is of crucial importance because wheat grains can change in nutritional value and processability according to their origin. Moreover, verification is an obligation in seed purification. In this study, the potential of spectral imaging for the classification of wheat kernels and the effect of the data fusion via data concatenation were investigated. Spectral images of 38 different wheat varieties were collected in Vis-NIR and SWIR ranges with seeds in the crease-up and crease-down positions. Linear Discriminant Analysis (LDA) of mean seed spectra showed 93% and 75.8% classification accuracy for the samples in the up position in the Vis-NIR and SWIR wavelength range, respectively, while it was 89.5% and 81.4% for the down position. Horizontal concatenation was applied to combine wavelength ranges, and then, vertical concatenation was performed for the fusion of data in the all positions which achieved the highest classification accuracy of 95.4%. This study highlights the appropriateness of spectral imaging as an objective and fast tool for the identification of wheat kernels and the promising results of data fusion to improve the classification accuracy.

P07.01

COMPARISON OF PRETREATMENT METHODS IN USE OF VIS/NIR SPECTRAL IMAGING TO DETECT AND QUANTIFY DIFFERENT MODES OF MICROBIAL BIOFILMS ON ALUMINIUM AND HDPE SURFACES

Nazan Altun[1,2], Ana Herrero-Langreo[1], Aoife Gowen[1]

[1]University College Dublin (UCD), School of Biosystems and Food Engineering, Dublin, Ireland, [2]University of Oviedo, Department of Molecular and Cellular Biology, Oviedo, Spain

Spectral imaging, providing a spectrum at each pixel of a sample image, is a promising choice for heterogeneous organic samples. This technique has the potential to detect and identify complex biological samples and the components of interest [1-2]. An important application, with use of visible and NIR ranges, is the detection of microbial biofilms on food contact surfaces, which are highly difficult to detect and localise with conventional methods in industry. A bacterial biofilm is an organized community of cells embedded in a self-produced protective and complex matrix called extracellular polymeric substances (EPS) [3].

Biofilms of Escherichia coli were prepared in 2 modes: at air-liquid interface on aluminum substrate (as a strong form) and at areal and highly irregular mode on high density polyethylene (HDPE) (as a weak form). They were imaged in visible and NIR spectral regions. The possibility to determine the presence of biofilms and the extensions of contaminated surface was explored with PLS-DA classification. Performance of different pretreatment methods as SNV and Savitzky Golay 1st and 2nd derivatives before classification were compared. Biofilm presence was detected with an accuracy over 0.85 when applied to external test sets. Discussions on the roles of pretreatment methods and the effects of other factors will be presented.

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P07.02

HYPERSPECTRAL IMAGING AS A TOOL FOR QUALITY CONTROL OF BRICKS

Emilie Giltvedt Langeland[1], Arnoud Jochemsen[1], Arnkell Jonas Petersen[1], Thomas Thiis[1], Jorunn Tyssø[2], **Ingunn Burud**[1]

[1]Norwegian University of Life Sciences, Aas, Norway, [2]Høine, Oslo, Norway

Bricks are widely used as building materials and the production of new bricks requires high energy in order to burn the clay at high temperatures. Reuse of old bricks is increasing, however, a proper quality control of the old bricks needs to be carried out in order to guarantee their required physical characteristics. The goal of the present study was to investigate if hyperspectral imaging can be used as a non-destructive characterization technique for bricks. A set of 17 old bricks were characterized with standard measurement techniques and they were scanned with a hyperspectral camera in the SWIR wavelength region. The bricks were scanned both in raw condition, and then rescanned after drying and polishing.

After preprocessing, the spectra were explored with a PCA and compared with a PCA of the physical measurements carried out. Figure 1. displays the PCA score plot of component 1 and 2 colored by the measured pore filling capacity of the dried bricks. This capacity says something about the bricks ability to resist frost and it is therefore useful to be able to predict a threshold value of this ability.

A PLS regression analysis was carried out to investigate whether the measured physical characteristics of the bricks could be predicted from the hyperspectral images. A correlation of R2=0.78 was found for prediction of pore filling capacity on the dried bricks. In addition to wavelengths related to water, the wavelength around 2200 nm was found to be significant for the model.

Given that the moisture content in the bricks influenced the prediction model, it was investigated whether the moisture could be modelled and corrected for when analyzing raw bricks. An EMSC algorithm with a pure water spectrum as one of the input reference spectra was applied with the aim of correcting for the water content in the bricks. The number of samples in the study was too small and to conclude on this issue.

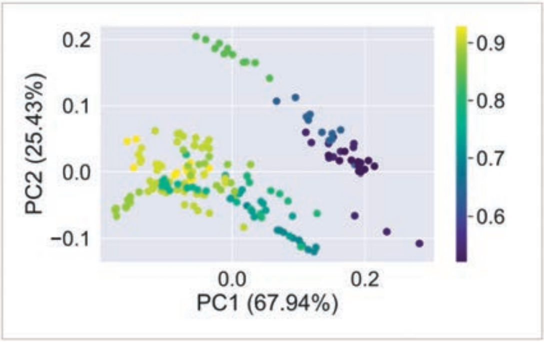


Figure 1. PCA score plot of spectra from dried bricks colored by the bricks' pore filling capacity.

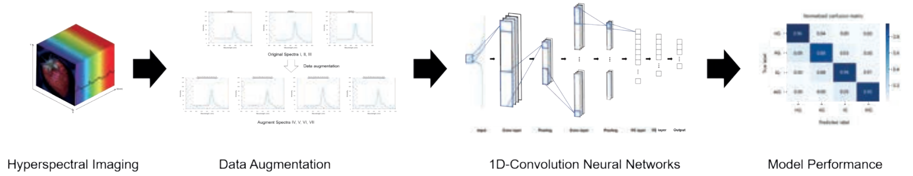
P07.03

DATA AUGMENTATION EFFECT OF DEEP LEARNING MODEL BASED ON HYPERSPECTRAL FLUORESCENCE IMAGING DATA FOR EARLY DETECTION OF BOTRYTIS CINEREA IN STRAWBERRY

Seungwoo Chun[1], Doo-Jin Song[1], Kwang-Ho Lee[1], Min-Jee Kim[3], Kyoung-Su Kim[1,2], Cheon-Soon Jeong[1,4], Changyeun Mo[1,5]

[1]Department of Interdisciplinary Program in Smart Agriculture, Kangwon National University, Chuncheon, South Korea, [2]Division of Bioresource Sciences Department of Applied Biology, Kangwon National University, Chuncheon, South Korea, [3]Agriculture and Life Sciences Research Institute, Kangwon National University, Chuncheon, South Korea, [4]Department of Horticulture, Kangwon National University, Chuncheon, Chuncheon, South Korea, [5]Department of Biosystems Engineering, Kangwon National University, Chuncheon, South Korea

Botrytis cinerea is a plant pathogenic fungus that causes infections to more than 1,000 plant species worldwide and is a disease that mainly occurs in strawberries. In particular, strawberries for export have a longer storage and transportation period than strawberries for domestic consumption, so export strawberries cause greater losses when Botrytis cinerea occurs. Therefore, it is required to develop a technology to detect asymptomatic strawberries in which Botrytis cinerea has occurred but no symptoms have appeared. In this study, a deep learning model was developed using hyperspectral fluorescence image data for the early detection of Botrytis cinerea, and the effect of data augmentation methods was investigated in developing the deep learning model. The Keumsil variety of strawberries, which is mainly exported from South Korea, was used in the experiments. The stage of Botrytis cinerea infection, was composed of Healthy group (HG), Asymptomatic group (AG), Infected group (IG), and After Infected group (AIG) through the results of infection for a week. Deep learning algorithms based on VGG-19 and Resnet-50 were developed. Six spectrum preprocessing methods (Smoothing, Normalization, SNV, and MSC) were investigated to improve detection performance. Three data augmentation methods were applied to each infection stage. As a result of comparing the classification performance of the developed deep learning algorithm for early detection of infection according to the ratio of original data and augmented data, the detection performance was improved by making the ratio of data for each infection stage constant. The results of this study demonstrated the possibility of early detection of Botrytis cinerea by converging hyperspectral fluorescence imaging technology and deep learning algorithm applied with data augmentation.
Graphical Abstract



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P07.05

PREDICTION OF IN VITRO DISSOLUTION PROFILE OF SUSTAINED RELEASE TABLETS BASED ON NIR AND RAMAN IMAGING AND ARTIFICIAL INTELLIGENCE

Dorián Galata[1], Szilveszter Gergely[2], Rebeka Nagy[2], Lilla Mészáros[1], Ferenc Ronkay[3], Zsombor Nagy[1], Attila Farkas[1]

[1]Department of Organic Chemistry and Technology, Faculty of Chemical Technology and Biotechnology, Budapest University of Technology and Economics, Budapest, Hungary, [2]Department of Applied Biotechnology and Food Science, Faculty of Chemical Technology and Biotechnology, Budapest University of Technology and Economics, Budapest, Hungary, [3]Department of Innovative Vehicles and Materials, GAMF Faculty of Engineering and Computer Science, John von Neumann University, Kecskamét, Hungary

Thanks to recent advances in vibrational spectroscopic techniques, the comprehensive real-time quality analysis of pharmaceutical tablets is now in the realm of possibility. Some critical quality attributes of a tablet such as its dissolution profile is influenced by the particle size of the components. Chemical imaging is a great way to characterize the spatial distribution of the components on the surface of a tablet, consequently it also yields information about particle size. The development of instrumentation has greatly increased the measurement speed of vibrational imaging techniques, recently even real-time line scan imaging setups have been demonstrated.

This introduces the possibility that a chemical map can be obtained from each tablet leaving a tablet press. By obtaining such an enormous amount of information, the quality attributes of the tablets can be predicted more reliably. For example, in sustained release tablets utilizing a gel matrix, the main factor that determines dissolution rate is the concentration of the polymer. This can be predicted with a single spectrum representing a large portion of the tablet. However, the particle size of this polymer can vary between tablets because of various reasons, including segregation of the powders during manufacturing processes. The particle size of the polymer can also influence the dissolution rate, larger sizes result in faster dissolution, therefore it is beneficial to know the particle size of the polymer in each tablet. This can be accomplished with chemical imaging techniques.

Our goal was to use NIR and Raman imaging to predict the in vitro dissolution profile of sustained release tablets using hydroxypropyl methylcellulose (HPMC) as matrix polymer. A chemical map was obtained from each tablet covering an 1.2 × 1.2 mm2 area with steps of 40 μm in the case of Raman and 25 μm in the case of NIR imaging. 112 training and 28 validation tablets were used, the chemical maps were processed using convolutional neural networks, and the dissolution profile of the tablets was predicted with simple artificial neural networks.

The prediction of the in vitro dissolution profile was accurate with both NIR and Raman imaging. While Raman imaging yielded slightly better results, its instrumentation is less capable of real-time quality analysis. Consequently, NIR imaging has the best potential in the future of pharmaceutical quality assurance.

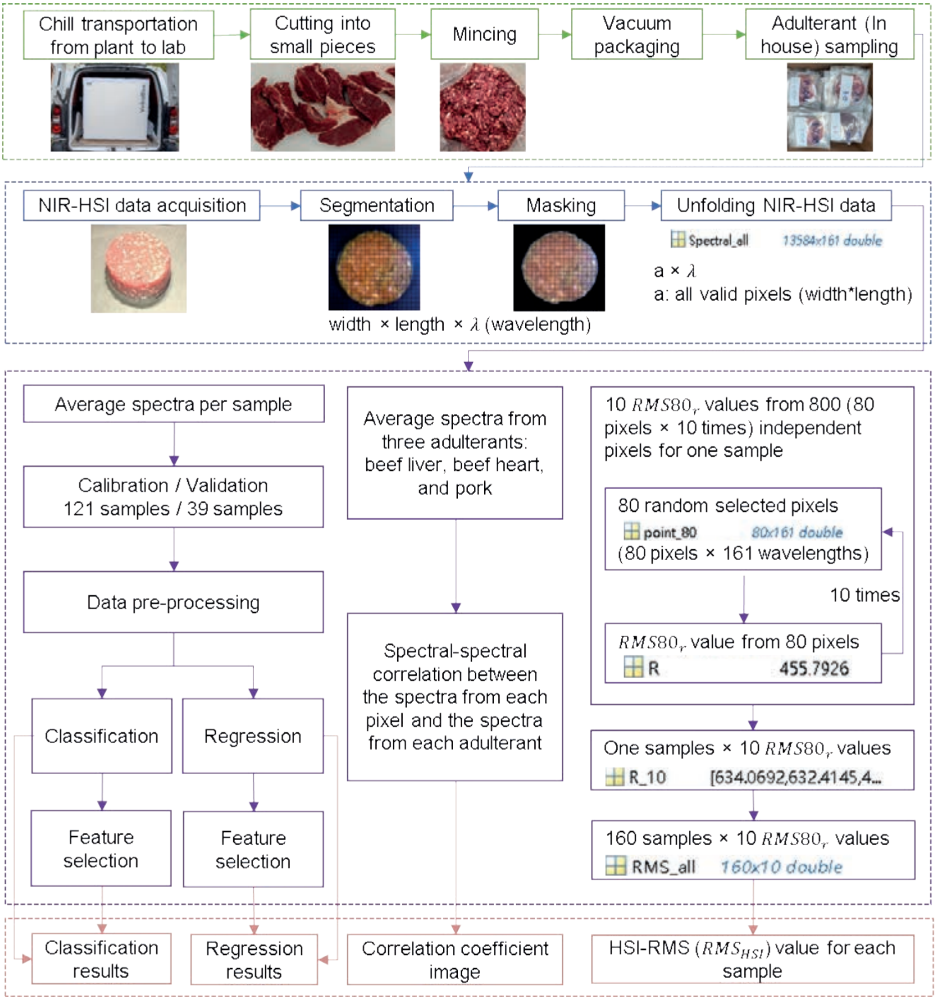
P07.06

HYPERSPECTRAL IMAGING (HSI) COMBINED WITH CHEMOMETRICS AND STATISTICAL METHODS FOR IDENTIFYING LOW-LEVEL ADULTERATION IN GROUND BEEF: CLASSIFICATION, REGRESSION, AND VISUALIZATION STUDIES

Wenyang Jia[1], Alessandro Ferragina[2], Anastasios Koidis[1]

[1]Institute for Global Food Security, School of Biological Sciences, Queen's University Belfast, 19 Chlorine Gardens, Belfast BT9 5DL, Northern Ireland, United Kingdom, [2]Teagasc Food Research Centre, Food Quality and Sensory Science Department, Dublin, Ireland

Owing to the inherent characteristics of ground beef, adulteration presents a substantial risk for subsequent users, including downstream suppliers and consumers. This study developed a robust and novel method for simultaneously identifying replacement fraud in ground beef with beef liver, beef heart, and pork using NIR-HSI (900 - 1700 nm) coupled with chemometric and univariable statistical methods (Figure 1). The classification study demonstrated that NIR-HSI provided an efficient and accurate means of identifying each type of adulteration using the classification model (Raw spectra - Genetic Algorithm - Backpropagation Artificial Neural Network), with sensitivity and specificity exhibiting a value of 1.00 for the calibration and the independent validation sets. The regression model based on the Raw spectra - Interval Partial Least Squares Regression - Artificial Neural Network) pipeline implemented in PLS-Toolbox (Eigenvector Research Inc, Wenatchee, USA), provided a systematic approach to quantify the adulteration levels in ground beef with high accuracy (R^2 for both calibration and validation is over 0.95, and the RMSE value is 1.37% and 2.08%, respectively). As an alternative to multivariate analysis, the correlation coefficient and HSI-RMS (Hyperspectral Imaging - Root Mean Square) value were calculated in order to discriminate the type of adulteration with less computational resources. Correlation analysis was conducted between the external spectra of pure adulterants and the spectra of each pixel. The HSI-RMS approach proposes a method to clarify the complexity of HSI data, based on the RMScut-off value which was used to understand the similarity between different scans of one sample. Different types of adulteration show noticeable differences under HSI-RMS value, which drives the concept of HSI-RMS to be a valuable metric for assessing the HSI data and facilitating the identification of adulterants. This adulteration study highlights the effectiveness of HSI in identifying adulteration in beef products, which could develop novel strategies to combat meat fraud and ensure the quality of meat products when using spectral-imaging techniques.


Chill transportation from plant to lab
Cutting into small pieces
Mincing
Vacuum packaging
Adulterant (In house) sampling
NIR-HSI data acquisition
Segmentation
Masking
Unfolding NIR-HSI data
Spectral_all 13584x161 double
a × λ
a: all valid pixels (width*length)
width × length × λ (wavelength)
Average spectra per sample
Calibration / Validation
121 samples / 39 samples
Data pre-processing
Classification
Regression
Feature selection
Feature selection
Average spectra from three adulterants: beef liver, beef heart, and pork
Spectral-spectral correlation between the spectra from each pixel and the spectra from each adulterant
10 $RMS80_r$ values from 800 (80 pixels × 10 times) independent pixels for one sample
80 random selected pixels
point_80 80x161 double
(80 pixels × 161 wavelengths)
10 times
$RMS80_r$ value from 80 pixels
R 455.7926
One samples × 10 $RMS80_r$ values
R_10 [634.0692,632.4145,4...
160 samples × 10 $RMS80_r$ values
RMS_all 160x10 double
Classification results
Regression results
Correlation coefficient image
HSI-RMS (RMS_{HSI}) value for each sample

P07.07

LAYERED DATA FUSION APPROACHES USING SPECTRAL IMAGING TO ASSESS THE CHANGES IN MYOGLOBIN CONTENT OF PACKAGED VEAL PRODUCTS DURING STORAGE

Wenyang Jia[1], Sara Erasmus[2], Joseph Peller[3], Saskia van Ruth[1,2,4], Anastasios Koidis[1]

[1]Institute for Global Food Security, School of Biological Sciences, Queen's University Belfast, 19 Chlorine Gardens, Belfast BT9 5DL, Northern Ireland, United Kingdom, [2]Food Quality and Design Group, Wageningen University and Research, P.O. Box 17, 6700 AA, Wageningen, the Netherlands, [3]Greenhouse Horticulture, Wageningen University & Research, Wageningen, the Netherlands, [4]UCD School of Agriculture and Food Science, University College Dublin, 4, Dublin, Ireland

Myoglobin is an essential indicator of the quality and freshness of red meat. The content and status of myoglobin change during storage due to various factors, which could result in spoilage and a decrease in meat quality. This study aims to predict the myoglobin profile (total myoglobin, deoxymyoglobin (DeoMb), oxymyoglobin (MbO2), and metmyoglobin (MetMb)) of packaged veal products during storage using two sensors which include the visible range (400 - 1000 nm) and shortwave infrared range (900 - 1700 nm) (Figure 1). Here, three levels of data fusion (data-level, feature-level, and decision-level) were employed to evaluate packaged veal products. This is a vertical experiment where the derived meat is coming from Zwartbont calves of controlled sex and age from a single farm near Wageningen (the Netherlands). After slaughtering, the veal samples (Psoas major and Longissimus thoracis) were vacuum packaged at day 0. The fused spectral data were then subjected to a chemometric analysis using the data-level fusion approach. Results showed that MbO2 content (%) is predicted more successfully among the rest of the myoglobin profile elements (R2CV=0.63, RMSECV=1.46%). Regarding the feature-level fusion, different features were extracted from each sensor: selected wavelengths from average spectral, standard deviation (STD) spectral from valid pixels, spatial information (Gray-Level Co-Occurrence Matrix (GLCM)) from PCA analysis, and Hyperspectral Imaging-Root Mean Square (HSI-RMS) value. Among them, 110 wavelengths were used to obtain the best performing model (R2CV=0.68 and RMSECV=1.37%) for MbO2 content (%). Finally, for decision-level fusion, Bayesian Decision was used to determine the weight of results from data-level and feature-level fusion. Similarly, MbO2 content achieves the highest performance: R2CV=0.71, RMSECV=1.11%. The model performance with total myoglobin, DeoMb, and MetMb remained unsatisfactory under the three data fusion approaches (R2CV=0.56, 0.53, 0.68, respectively). Despite its limitations, this study highlights the efficacy of Spectral Imaging in rapidly evaluating myoglobin content, especially MbO2 in packaged veal products during storage. By employing data fusion approaches, the accuracy of the analysis is enhanced, offering valuable insights into the changes that occur during storage. These findings can be further developed into a more general model that, if validated, may one day help meat processors determine meat quality rapidly and on-site.

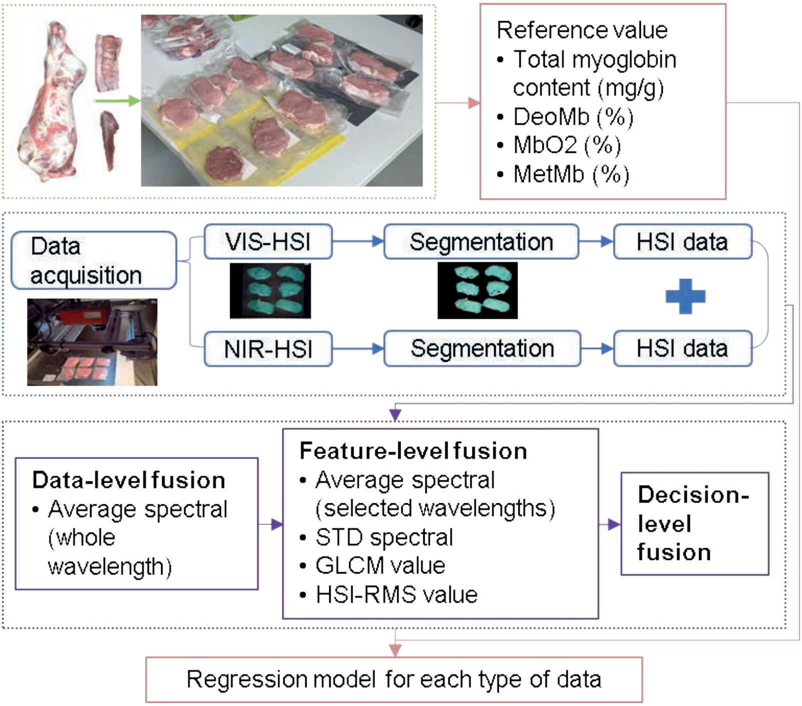


Figure 1. Visualised experimental design

P07.08

REAL-TIME DETECTION OF NITROGEN OVER-SUPPLY ORIENTAL MELON PLANTS USING HYPERSPECTRAL VISIBLE AND NEAR INFRARED IMAGE

Hangi Kim[1], Minseok Park[1], Byoung-Kwan Cho[1]

[1]Department of Biosystems Machinery Engineering, Chungnam National University, 99 Daehak-ro, Yuseong-gu, Daejeon, South Korea

Excessive nitrogen supply to oriental melons can lead to reduced fruit yield due to delayed flowering and increased susceptibility to pests and diseases. Therefore, precise management of nitrogen supply is essential for optimal oriental melon cultivation. This study proposes a Convolutional Neural Network (CNN)-based waveband selection method to detect nitrogen over-treated oriental melon plants using visible near-infrared (VIS-NIR) hyperspectral image and develops deep learning algorithms for real-time object detection. Three levels of nitrogen treatment, 1.0, 1.5, and 2.0 times of the fertilization recommendations were applied to the soil and hyperspectral canopy images for the plants were acquired. An one-dimensional (1D-CNN) classification model was developed and evaluated its performance for the classification of the melon plants defends on the nitrogen treatment. Based on the learning weight of the model, priority wavebands of high importance for the classification were selected. Based on the selected wavebands of the hyperspectral image, a CNN-based backbone network was optimized and used for the real-time detection of the presence of nitrogen over-supply in oriental melon plants. The proposed approach demonstrated potential of hyperspectral VIS-NIR imaging combined with deep learning model for measuring nitrogen level in oriental melon plants.

P07.09

IMPROVED SORTABILITY OF PLASTIC PACKAGING FILMS THROUGH ADAPTED NEAR-INFRARED SPECTROSCOPY AND CUSTOM MACHINE LEARNING MODELS

Gerald Koinig[1], Nikolai Kuhn[1], Thomas Fink[1], Alexia Tischberger-Aldrian[1]

[1]Montanuniversität Leoben, Leoben, Austria

Around 150,000 tons of packaging films are disposed of through separate collection in Austria alone, representing about 50% of the annual usage of lightweight packaging in the country. Most of these film packaging wastes are currently thermally treated and thus do not contribute to the recycling quotas required by the European Union.

One reason for this low recycling rate is the difficulty in separating recyclable single-layer plastic films from multi-material multi-layer films. Multi-layer films are particularly useful for food packaging as each layer serves a protective role for the product. Subsequently, reducing food waste and the CO2 footprint of Austria. As the use of film packaging is expected to increase in the future, there is a need to develop methods to improve their sortability.

A core problem in sorting single-layer and multi-layer films is the low spectral quality in near-infrared spectroscopy used in sensor-based sorting. It is caused by the low film thickness which leads to insufficient interaction between the near-infrared radiation and the material itself. This study presents an adaptation of near-infrared spectroscopy by adapting existing aggregates from traditional reflection measurement to transflection measurement. Transflection allows the near-infrared radiation to penetrate the material twice, thus capturing more information and hence improves the material sortability.

Another problem in sorting plastic packaging waste is the constantly changing composition of multi-layer films, as commonly used evaluation models need to be adapted to the specific composition. To address this, custom machine learning models, including a support vector machine, a shallow neural net, and a deep neural net, were developed to recognize and classify multi-layer films regardless of their material composition. These models were compared in terms of prediction accuracy and latency and evaluated for their applicability for inline classification in commercial material recovery facilities. Results show accuracies of up to 90% on unknown test sets and prediction speeds of up to 120,000 observations per second.

The combination of adapted near-infrared spectroscopy and custom machine learning models is a promising method to improve the sortability of packaging films. It contributes to closing the loop of plastic waste and increasing the recycling rate.

P07.10

DIMENSIONAL REDUCTION OF SPECTRAL DATA THROUGH FEATURE SELECTION FOR THE REAL-TIME CLASSIFICATION OF PLASTIC WASTE

Nikolai Kuhn[1], Gerald Koinig[1], Thomas Fink[1], Alexia Tischberger-Aldrian[1]

[1]Montanuniversitaet Leoben, Leoben, Austria

Sensor-based sorting machines equipped with near-infrared (NIR) sensors are commonly used for sorting plastic packaging waste in Europe. New laws and economic trends require improved particle characterisation, which can be achieved with more powerful sensor systems. For this, hyperspectral NIR sensors with high spectral and spatial resolutions are used. Hence, large amounts of data are generated, which must be evaluated within milliseconds for mechanical separation in real time. This development necessitates both potent hardware and efficient evaluation algorithms.

We investigate how large spectral data sets can be algorithmically pre-processed in a way that only the data relevant for classification are passed to the classification model. The principle behind this idea is demonstrated on an exemplary separation of multi-material multilayer from monolayer plastic packaging films from the separate collection in Austria. For this purpose, we collected raw data on a commercial sensor-based sorting machine equipped with a hyperspectral camera with a resolution of 320 pixels and 220 channels. The multilayer nature of the films was determined with an FTIR spectrometer. For the present modelling, 17 single-layer films and 22 multilayer films made from standard plastics such as PP and PE were used. A principal component analysis and pareto distribution of the resulting principal components was used to prove that the data set can be divided into both classes. Afterwards, we identified relevant wavelength ranges by feature selection using minimum-redundancy maximum-relevance feature selection. The spectral bands identified as relevant were used to train an artificial neural network (ANN). For inference, channels were selected to minimise the inference time without negatively influencing prediction accuracy. Finally, the accuracy and inference time of the trained ANN were determined. Of the 220 spectral channels, 57 channels are sufficient to achieve a prediction accuracy of the ANN of 97%. By reducing the number of predictors, a short inference time of only 0.02 s per data batch (about 3000 spectra) could be achieved while maintaining a high prediction accuracy.

We could show that using a level of abstraction above the actual material composition allows classification that does not rely on teaching every material combination and their respective spectra. Hence, overlying spectral information that is characteristic for the desired overarching class can be considered.

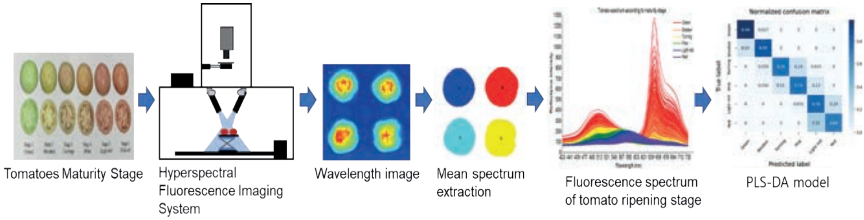
P07.11

TOMATO MATURITY CLASSIFICATION BY COMBINING PLS-DA MODEL AND HYPERSPECTRAL FLUORESCENCE IMAGING TECHNOLOGY

Bo-Young Lee[1], Min-Woo Baek[1], Doo-Jin Song[1], Min-Jee Kim[2], Cheon-Soon Jeong[1], Chang-yeun Mo[1,3]

[1]Department of Interdisciplinary Program in Smart Agriculture, Kangwon National University, Chuncheon, South Korea, [2]Agriculture and Life Sciences Research Institute, Kangwon National University, Chuncheon, South Korea, [3]Department of Biosystems Engineering, Kangwon National University, Chuncheon, Korea

Tomatoes are characterized by a change in maturity even after harvesting, so the storage period varies depending on the maturity. In the distribution process, the tomato maturity stage should be considered according to the distribution period. Tomato is sorted based on weight or size, and the external quality such as scratches, which is visually determined by the operator. Visual sorting of tomatoes requires a lot of labor, and sorting errors occur depending on the skill level of the worker. Due to these errors, the screening accuracy is lowered, and the reliability of consumers is reduced as products of uneven quality are distributed. In order to improve the accuracy of quality measurement and uniformity of quality, a technology capable of quickly and non-destructively measuring quality is required. Therefore, this study aims to develop a model for determining the maturity of tomatoes using hyperspectral fluorescence imaging technology. A total of 672 TY Amazon cultivar tomatoes and TY Yeolgang cultivar tomatos harvested in July and August of 2022 were classified into six maturation stages and used for the experiment. The maturation stages were classified according to standard of the US Department of Agriculture (USDA): green, breaker, turning, pink, light-red, and red. The hyperspectral imaging systems consists of hyperspectral image camera (micro HSI™ 410 Hyper-spectral Sensor, Corning®, USA) and UV-A LED of the 365nm excitation. The exposure time and step interval were measured as 950 ms and 1 mm, and 700 ms and 1 mm, respectively, for the tomatoes of the Amazon and TY Yeolgang cultivars. Partial least squares - discriminant analysis (PLS-DA) models for determining tomato maturity were developed. Evaluation metrics of classification models were used for the performance evaluation. The accuracy, recall, and precision of the PLS-DA model with mean normalization were the highest at 92.1%, 79.75%, and 80.24%, respectively. The results of this study show that hyperspectral fluorescence imaging technology can be used to determine maturity of tomatoes. In addition, this technology is expected to be utilized in the development of internal and external quality discrimination devices for tomatoes in the future.



This research was supported by the Korea Institute of Planning and Evaluation for Technology in Food, Agriculture, Forestry and Fisheries (Project no. 3220525-5)

P07.12

VARIABLE SELECTION METHODS TO DETECT ESCA DISEASE IN GRAPEVINE LEAVES USING NEAR-INFRARED HYPERSPECTRAL IMAGING

Sara León-Ecay[1], Claudia Pérez-Roncal[2], Carmen Jarén[2],
Ainara López-Maestresalas[2], Silvia Arazuri[2]

[1]Universidad Pública De Navarra, IS-FOOD (Institute of Innovation and Sustainable Development in Food Chain), Department of Agricultural Engineering, Biotechnology and Food, Pamplona, Spain, [2]Universidad Pública De Navarra, IS-FOOD (Institute of Innovation and Sustainable Development in Food Chain), Department of Engineering, Pamplona, Spain

Esca is one of the grapevine trunk diseases with more impact in Europe and in the Mediterranean countries. It is a fungal disease considered as a complex of diseases that affects the crop productivity and can produce the early decay of plants. One of the main characteristics of this disease is that symptoms may not appear even if the plant is infected. There is not chemical treatment for curative or control purposes, so it is important to detect the disease as early as possible to prevent its spread throughout the vineyard. Therefore, the aim of this work is to evaluate different variable selection methods to make early Esca prediction methods in grapevine leaves more applicable. To carry out the evaluation, the hyperspectral images of 360 leaves of cv. Tempranillo were recorded: 180 corresponding to asymptomatic leaves from Esca non-affected vines, named Control (Control), and 180 to asymptomatic leaves from Esca-affected vines (Esca). Hyperspectral images were recorded using an NIR-HSI system consisting of an InGaAs camera with a resolution of 320 x 256 pixels (Xeva 1.7-320, Xenics, Belgium) coupled to a spectrograph (ImSpector N17E, Specim, Spectral Imaging Ltd., Finland), both sensitive in the 900–1700 nm range. This line-scanning imager was mounted 400 mm above a linear translation stage (LEFS25, SMC Corporation, Tokyo, Japan). Images were then processed and Partial Least Squares Discriminant Analysis (PLS-DA) were performed. Different variable selection methods were carried out: automatic, interval PLS (iPLS) and genetic algorithm (GA). The multivariate analysis was performed using the PLS_Toolbox v. 8.8 (Eigenvector Research Inc., USA) under MATLAB® computational environment. The parameters used to evaluate the performance of the different models obtained were the percentage of explained variance, and the percentage of correctly classified samples (%CC). As it can be seen in table 1, the best results were obtained by applying the iPLS method, with a significant reduction in the number of wavelengths needed to achieve values above 95 %CC. Furthermore, although the %CC values obtained by GA were lower (95% vs. 91%), the number of wavelengths selected was also lower and the obtained %CC could be sufficient if it translates into higher applicability of the equipment. In conclusion, variable selection methods could allow equipment to be adapted to specific spectral ranges to improve the portability of spectrophotometers.

Table 1. Classification results obtained with the different variable selection methods compared to the model without variable selection (raw model).

VS	λ	LV	Variance (%)	Control (%CC)	Esca (%CC)	Global (%CC)
Raw Model	224	4	95.8	95.0	96.1	95.5
Auto	116	2	93.8	91.5	98.8	95.2
iPLS	50	3	97.1	96.4	97.6	97.0
GA	32	4	96.7	91.1	92.2	91.7

P07.13

FRAUD DETECTION IN THE FISHING SECTOR USING HYPERSPECTRAL IMAGING

Paula Luri Esplandiu[1], Juan Marín Méndez[1], Miriam Alonso Santamaría[1], Berta Remírez Moreno[1], María José Saiz Abajo[1]

[1]Centro Nacional De Tecnología Y Seguridad Alimentaria (cnta), San Adrián, Spain

Nowadays, more and more consumers are interested in the quality, safety, and authenticity of food products. The fishing sector is the second food category with the highest risk of fraud and solving authentication problems in fish. There are many traditional analytical techniques available to tackle authentication challenges and ensure product quality (chromatography, mass spectroscopy, protein and DNA-based techniques, elemental profiling and isotopic analysis). However, most of the abovementioned techniques are associated with several drawbacks, such as being destructive, time-consuming, unable to handle many samples, and sometimes requiring lengthy sample preparation. There are non-destructive, fast, accurate, robust and high-throughput techniques for real-time authentication, highlighting hyperspectral images (HSI). In this context, the main aim of this work is to explore the viability of HSI in the VNIR (400-1000 nm) and NIR (900-1700 nm) ranges for the detection of fraud by origin and by non-declaration of the previous freezing process, in anchovies.

Fifteen batches of fresh anchovies from two origins (Mediterranean Sea and Cantabrian Sea) were purchased in two supermarkets in La Rioja, Spain. After the image was acquired, the region of interest (ROI) was extracted and then, one spectrum was obtained from each pixel that was part of the ROI and finally, the mean reflectance spectrum was acquired by each anchovy. It was created a spectral matrix of around 1400 samples × 224 bands. Four spectral pretreatment methods of Savitzky–Golay smoothing (SG), Standard Normal Variate (SNV), 1st and 2nd derivatives and two classifiers of SIMCA (Soft Independent Modeling of Class Analogy) and PLS-DA (Partial Least Squares Discriminant Analysis) were used to build the classification models.

After analysis, it was found that the modelling results using the VIS-NIR range were always better than those using the NIR range, and the best performance model was by PLS-DA with a recall of 0,97 for fresh and 0,98 for frozen-thawed in anchovies, and 0,98 for Cantabrian anchovies and 0,96 for Mediterranean anchovies. An advantage of the obtained model is the ability to classify the anchovies measuring on the skin side of fish without the need to pretreat or grind the sample. Overall, the results demonstrated that HSI combined with PLS-DA is a promising solution for rapid and non-destructive detection of adulteration in freshness and origin in anchovies.

P07.15

MATURITY DISCRIMINATION MODEL OF CITRUS USING HYPERSPECTRAL FLUORESCENCE IMAGING AND CONVOLUTIONAL NEURAL NETWORK

Changyeun Mo[1,2], Min-Jee Kim[3], Woo-Hyeong Yu[1], Seung-Woo Chun[2], Doo-jin Song[2], Soo-Hwan Park[2], Jae-EUN Lee[2], Soon-Hwa Kwon[4]

[1]Department of Biosystems Engineering, Kangwon National University, Chuncheon, Republic of Korea, [2]Department of Interdisciplinary Program in Smart Agriculture, Kangwon National University, Chuncheon, Republic of Korea, [3]Agriculture and Life Sciences Research Institute, Chuncheon, Republic of Korea, [4]Citrus Research Institute, National Institute of Horticultural and Herbal Science, Jeju, Republic of Korea

Citrus fruit, one of the representative fruits in the Republic of Korea, are climacteric fruits and that has quality changes during the postharvest storage. Maturity is one of the factors that affects quality changes such as soluble solids content and total acidity. Currently, citrus maturity is assessed by human observation of the color and texture of citrus. Therefore, objective criteria for evaluating maturity of citrus fruits are demanded. Hyperspectral fluorescence imaging technique serve as a rapid and non-destructive technique to estimate various quality of fruits. The object of this study was to evaluate the applicability of hyperspectral fluorescence imaging to determine the maturity of citrus fruit. Convolutional neural network (CNN) model to determine the maturity of citrus fruit was developed using hyperspectral fluorescence imaging. 313 citrus fruits (Citrus unshiu Marcow) were produced in Jeju, Republic of Korea were classified into 6 stages of harvest period and used in the experiment. The hyperspectral imaging systems consist of hyperspectral camera (micro HSI™ 410 Hyper-spectral Sensor, Corning®, USA) with wavelength range of 460 ~ 800 nm and violet LED of the 405nm excitation. Hyperspectral fluorescence images were acquired from each citrus fruit in two orientations (i.e., blossom end and calyx side). The CNN model for citrus maturity discrimination was developed. The performance of each model was evaluated through the coefficient of determination (R^2)and root mean square error (RMSE). The accuracy of these algorithm was above 95%. The results of this study showed that the hyperspectral fluorescence imaging technique has the potential for maturity discrimination of citrus fruit.

Keyword: Hyperspectral fluorescence imaging, maturity discrimination, citrus fruit, convolutional neural network (CNN)

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P07.16

DEALING WITH THE CHALLENGE OF INSUFFICIENT LABELLED DATA IN THE TASK OF CLASSIFYING POTASSIUM STATUS OF RUBBER LEAVES USING NIR HYPER-SPECTROSCOPY

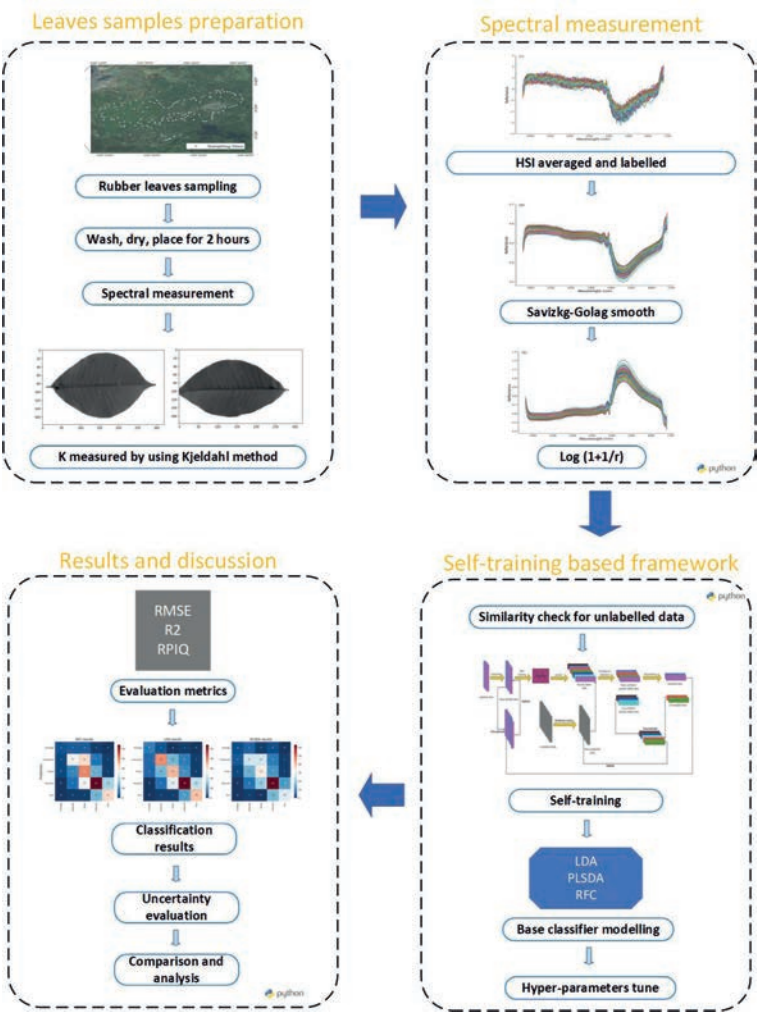
Tang Rongnian[1], Tang Weihao[1], Hu Wenfeng[1,2], Li Chuang[1]

[1]Hainan University, Hainan, Haikou, China, [2]Tianjin University, Tianjin, China

Potassium (K) is crucial macronutrient for crop growth and development. Using near-infrared spectroscopy to monitor the status of STN implementing scientific fertilisation management can effectively increase the yield of natural rubber. In establishing a spectral detection model, the number of modelling samples is key to the model accuracy. Because rubber trees are perennial crops, the cost of chemical analysis and the time consumption are too expensive for collecting thousands of leaves samples. In this sense, using traditional modelling methods leads to a poor performance of the model. To address it, solutions from a new perspective of mining more valuable information from hyperspectral images (HSIs) data were proposed in this study. Therefore, semi-supervised learning (SSL) method was implemented to extract spectral information from pseudo-labelling (PL) HSI pixels data with high confidence, so as to complement the limited labelled mean spectrum data. A five-classification model of the K statuses of rubber leaves was established by using the framework of self-training and machine learning (ML) base classifiers. The weighted average precisions (WAPs) of the self-training-LDA model on the test set were 64.6%, and the macro-averaged precisions (MAPs) were 59.3% in terms of the K statuses, respectively, which were 7.6% and 9.3% higher than those of the model established using traditional classifier and mean spectrum data. Therefore, the proposed method shows great potential in rapid and accurate classification of K status in leaves of perennial crops with small samples sizes.

Highlights

- The mean spectrum data from region of interests in HSI was used as labelled data for the modelling.
- Unlabelled pixel data from the HSIs are considered potentially in alleviating the costs of annotations by using chemical methods.
- Self-training algorithm were used to mine more valuable information from HSIs data as a supplement to limited labelled data.


Leaves samples preparation
Rubber leaves sampling
Wash, dry, place for 2 hours
Spectral measurement
K measured by using Kjeldahl method
Spectral measurement
HSI averaged and labelled
Savizkg-Golag smooth
Log (1+1/r)
python
Self-training based framework
python
Similarity check for unlabelled data
Self-training
LDA
PLSDA
RFC
Base classifier modelling
Hyper-parameters tune
Results and discussion
RMSE
R2
RPIQ
Evaluation metrics
Classification results
Uncertainty evaluation
Comparison and analysis
python

P07.17

DETECTION OF BLACK HEART INTERNAL DEFECT OF POMEGRANATE FRUIT (PUNICA GRANATUM L.) BY USING VIS-NIR HYPERSPECTRAL IMAGING

Lucia Russo[1], Danial Fatchurrahman[1], Maria Luisa Amodio[1], Giancarlo Colelli[1]

[1]Dipartimento di Scienze Agrarie, Alimenti, Risorse Naturali e Ingegneria (DAFNE), Università di Foggia, Foggia, Italy

Internal Black heart is a disease occurring in pomegranate fruit, due to the infection caused by Alternaria Spp. and/or Aspergillus Niger, which is not noticeable externally; some trained people can detect slight colour changes and differences in weight, if compared to sound fruit. In this, work we assess the potential of hyperspectral imaging method in the range of Visible-Near Infrared (VIS-NIR) (400–1000 nm) to predict non-destructively the defect. To this aim, 70 fruits were scanned by using a hyperspectral line-scan scanner (version 1.4, DV s.r.l., Padova, Italy) equipped with a spectrograph in the Vis-NIR range (400-1000nm, spectral resolution of 5 nm and spatial resolution 315dpi). As soon after image acquisition, fruit were then cut to verify the presence of internal defects, and to label the fruit. In total 27 fruits were found to be defective. Partial Least Square Discriminant Analysis (PLS-DA) was conducted with 2 classes (sound and damaged) by using mean cantering as spectra pre-processing technique. From the total amount of the fruits in the data set, 70 % of samples were used for calibration model and 30 % were used for the external prediction, by using Kennard stone algorithm. The model was able to classify defective fruit from sound fruit, reaching R^2 of calibration and prediction at 98.5 % and 100 %, respectively. The outcome of this study indicates the very high potential of visible-NIR to provide non-invasive, rapid, and reliable early detection of internal defect of black heart in pomegranate fruit, even if it’s desirable to enlarge the dataset with more samples, belonging from different harvest conditions.

P07.18

USE OF NIR HYPERSPECTRAL IMAGING FOR THE CHARACTERIZATION AND EVALUATION OF TRIPTYCH FROM THE VENETIAN GOTHIC PERIOD

Jakub Sandak[1,2], Saša Dolinšek[3], Ana Oblak[3], Lea Legan[4,5], Maša Kavčič[4], Polona Ropret[4,5], Anna Sandak[1,2]

[1]InnoRenew CoE, Izola, Slovenia, [2]University of Primorska, Koper, Slovenia, [3]Zavod za varstvo kulturne dediščine Slovenije, Ljubljana, Slovenia, [4]Institute for the Protection of Cultural Heritage of Slovenia, Ljubljana, Slovenia, [5]University of Ljubljana, Ljubljana, Slovenia

Several conventional characterization techniques implemented to study art objects' authenticity, composition, and conservation state are rather time consuming, expensive, or (to certain extent) destructive. An alternative method having entirely non-destructive character and high reliability is within great interest of restorers and researchers. This work demonstrates the implementation of hyperspectral imaging (HI) for evaluation of cultural heritage object. Four hyperspectral cameras produced by SPECIM (Oulu, Finland) that operate in different spectral ranges were used for scanning triptych from the Saint Dominic church in Izola. All cameras were operating in push broom mode, allowing line by line spectral measurement. White (spectralon) and black (detector noise) reference backgrounds were measured before each scanning of the panel. Halogen lamps were used as a light source for VNIR, NIR, and SWIR cameras, while heat radiation was used as a source for the MWIR system. Evince software by Prediktera (Umea, Sweden) was used for analysing and exploring the hyperspectral images. The main objective was the examination of hidings below the triptych's surface for identification and reconstruction of past intervention processes.

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P07.19

MODULAR SNAPSHOT HYPERSPECTRAL IMAGING IN THE NIR

Jan Stegemann[1,2], Franziska Gröniger[1], Sebastian Kruss[1,2]

[1]Fraunhofer Institute for Microelectronic Circuits and Systems IMS, Duisburg, Germany,
[2]Ruhr University Bochum, Bochum, Germany

Hyperspectral imaging in the NIR provides a huge level of additional information compared to monochrome cameras. By extending the pixels with wavelength data the imaging quality in many different sectors like medical diagnostics, waste management or agriculture is improved. Commercially, for hyperspectral imaging the image is either scanned spatially or spectrally, where the procedure can take several seconds to several minutes. Hyperspectral snapshot cameras have been created as a solution to this issue because they only need one snapshot to produce spectral data. Yet, these cameras also represent expensive and complex optical systems with in the end lack of spectral or spatial resolution. We present a novel optical approach to generate hyperspectral snapshot images. The design makes use of a regular InGaAs camera as the sensor element, which is transformed into a hyperspectral camera by the addition of our optical module. As a result, all pixels of the camera can be preserved for imaging. The spectral range is modular, and it can be adjusted for unique measurements. The approach was characterized with fluorescent carbon nanotubes, which show emission from 800 to 1200 nm depending on their chirality. Additionally, NIR hyperspectral images of materials like plants, food and plastics were taken, revealing chemical fingerprints, which are the result of specific absorption, reflection and transmission spectra.

P07.20

CLASSIFICATION OF POST-CONSUMER TEXTILES USING NIR SPECTROMETERS

Hana Stipanovic[1], Tanja Baeck[1], Alexia Tischberger-Aldrian[1]

[1]Montanuniversitaet Leoben, Leoben, Austria

Textile waste is one of the most rapidly growing waste streams. European households consume a large amount of textile products. According to European Commission every person buys 15 kg of textile per year. Yearly 5.8 million tons of textiles are thrown away, about 11.3 kg per person. As it is expected to have continuous growth of textile waste, European Commission has identified textiles as a "priority product for the circular economy" and that by 1st January 2025, Member States shall set up a separate collection for textiles. Most textile waste is incinerated, landfilled or exported to developing countries. In order to reach circular economy goals, it is required to higher the textile recycling rates. One of the most critical issues in textile recycling is the identification of fibre typologies constituting the waste. NIR technology is imposed as the solution to the recognition problem of textile waste materials. Using both benchtop and portable spectrometers have been analysed 466 (88 kg) samples of post-consumer textile waste. All the samples obtained the original labels, which were used as a reference for analyses. As referred to on the labels, the used textiles comprise different material compositions, a maximum of five fibres in one material. For further analysis, samples of pure fibers (e.g. 100% polyester) and composits of two different fiber materials (e.g. cotton/polyester) were used. The spectra were acquired in the wavelength of 991-1677 nm when analysed with a NIR benchtop spectrometer and in the wavelength of 1600-2400 nm when analysed with a handheld NIR spectrometer. Acquired spectra had to go through signal pre-treatment, where signal correction methods are used to remove effects on spectroscopic data, which could affect the performance of the chemometric analysis. The resulting spectra of both benchtop NIR and handheld NIR were derived and normalised. The spectra were further processed using chemometric tools like PCA and classification tools using MATLAB (R2022b) with chemometric toolboxes. The obtained results showed promising results when separating most of the samples. When analysing pure materials, differences and possible separation can be examined between all the materials apart from cotton and linen. In the case of material composites, it is observed that the correct material characterisation depends on the fibre content of specific material in the composition.

P07.22

NEAR INFRARED SPECTROSCOPY AND HYPERSPECTRAL IMAGING AS A TOOL FOR ANALYZING CARBON AND NITROGEN IN SOIL CORES

Philippe Vermeulen[1], Donatienne Arlotti[2], Bruno Huyghebaert[1],
Vincent Baeten[1]

[1]Cra-w, Gembloux, Belgium, [2]IFEL-W, Gembloux, Belgium

Near infrared spectroscopy (NIRS) is routinely used on conditioned soil samples (dried and crushed) to assess several parameters such as the organic carbon total (C) and the nitrogen (N) contents [1]. The study aimed to assess the potential of NIR handheld spectrometers and hyperspectral imaging combined with chemometrics to analyse the in-situ C and N contents of dried soil cores and to determine the ploughing sole, without the need to condition the samples (e.g. drying, grinding, etc.).
To achieve this goal, 36 dried soil cores of 1 m long including 12 horizons from a fertilization trial were analysed. Spectra were acquired on a total of 432 samples using an ASD Fieldspec 4 spectrometer equipped with a contact probe (350-2500 nm) and a Viavi MicroNIR 1700 device (900-1700 nm). NIR images were also acquired on the 36 cores using a Specim NIR hyperspectral imaging camera (1100-2500 nm) used with a conveyor belt (BurgerMetrics SIA, Riga, Latvia). From the 432 samples, 80 were selected for N and C reference analysis using Dumas method.
All data treatment was conducted using the Matlab R2007b (The Mathworks Inc., Natick, MA, USA) and the PLS toolbox 7.0.2. (Eigenvector Research Inc., Wenatchee, WA, USA). For NIR imaging dataset, mean spectra was calculated for each horizon. PCA analysis was performed on the three spectral datasets to identify the soil horizons. PLS models were also developed to predict N and C content on a calibration set of 80 samples for the three devices.
In summary, for measurements on conditioned soil samples (dried and crushed) ,using the FOSS XDS in the laboratory, the C and N calibration models gave a RMSECV of 0.12 for N content and of 0.16 for C content. These performances allow to identifying 5 soil type groups. For direct measurements on dried cores using the ASD FieldSpec 4 portable spectrometer or the NIR hyperspectral imaging system, RMSECV of 0.3 for N content or 0.3 for C content is obtained allowing to identifying 2 soil type groups. The measurements using Viavi handheld NIR spectrometer gave models with lower performance. Additionally, PCA analysis performed on the NIR images allowed to identify the ploughing sole directly on the dried cores.

Reference

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ACKNOWLEDGEMENTS
The authors wish also to thank the CRA-W teams for providing samples and for performing NIR analyses.

P07.23

DEVELOPMENT OF A COST-EFFECTIVE HYPERSPECTRAL CAMERA FOR FOOD QUALITY MONITORING

Sara Vignati[1], Alessio Tugnolo[1], Alessia Pampuri[1], Marco Menegon[1], Andrea Casson[1], Roberto Beghi[1], Riccardo Guidetti[1], Valentina Giovenzana[1]

[1]*University of Milan, Milan, Italy*

Hyperspectral imaging (HSI) is widely used for the quality assessment of various agri-food products thanks to the capability to retrieve useful information (both spatial and spectral) which can be qualitatively and/or quantitatively modelled for the prediction of several physical and chemical attributes.

Even though HSI technique can collect a large amount of data, the application of only one device (in some cases) is not enough to cover all the critical points that the company has to handle. Both the production process in the firm and the field monitoring could require distributed systems to collect data and provide information. Under these circumstances, considering the application of several hyperspectral devices, the costs become prohibitive for most of the companies and, therefore, the research is moving toward the development of hyperspectral solutions taking into account a considerable cost reduction for distributed stand-alone applications.

Therefore, a prototype of a HSI camera has been designed and built starting from a previous work proposed by Salazar and Mendez (2020). The final system showed the capability to pick the spectral information from 400-1000 nm within a spatial surface of 41x41 pixels.

The whole system has been fine-tuned using low-cost optical and electronic components (i.e., lenses, transmissive holographic diffraction gratings, Raspberry Pi 3b+ and a Raspberry Pi NoIr camera) and parts 3D printed using a photopolymer resin (Figure 1).

A first trial has been performed in lab scale conditions in order to verify the sensibility and sensitivity of the HSI device to acquire hyperspectral images which can be handled and analysed in MATLAB environment.

Figure 1. The hyperspectral camera prototype

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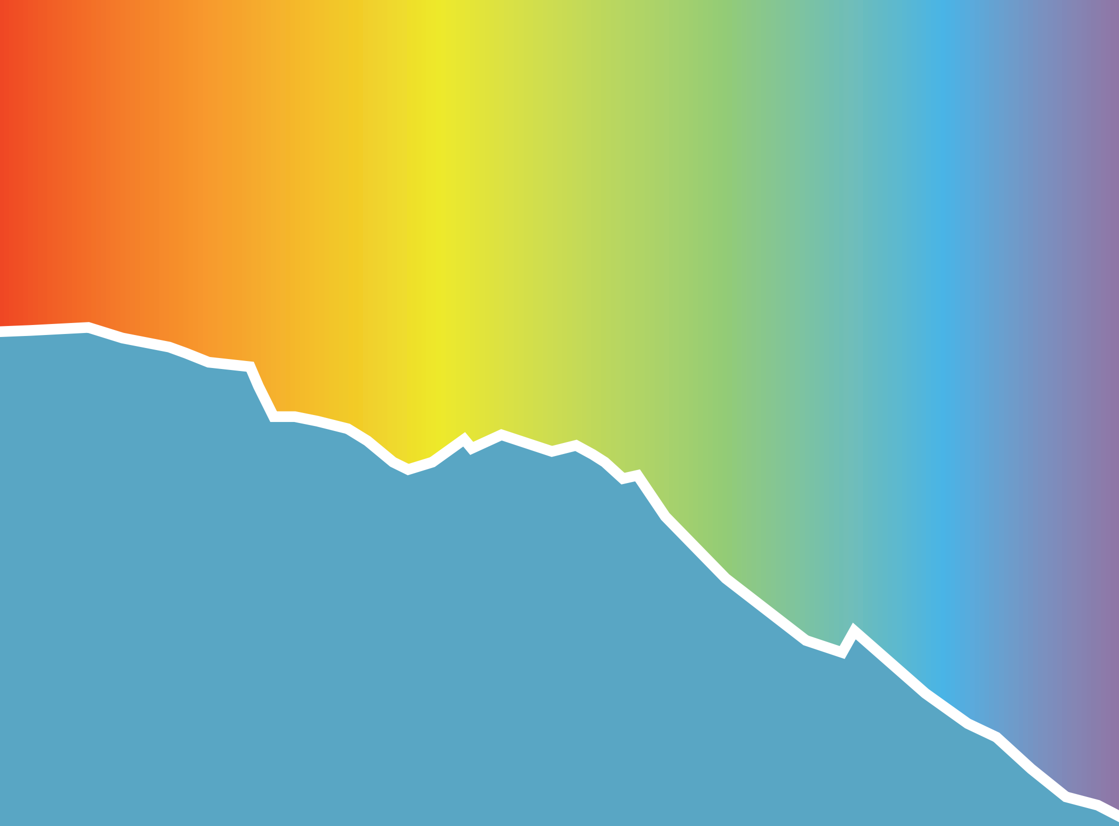
Salazar-Vazquez, J., & Mendez-Vazquez, A. (2020). A plug-and-play Hyperspectral Imaging Sensor using low-cost equipment. HardwareX, 7, e00087



THEME 08

IMPACTS IN PHARMA, DIAGNOSTICS AND BIOTECHNOLOGY

ORAL PRESENTATION: O08.01 – O08.05
FLASH TALK: F08.01
POSTER PRESENTATION: P08.01 – P08.11

008.01

NEAR-INFRARED ANALYSIS OF BILE JUICE FOR IDENTIFICATION OF GALL BLADDER CANCER

Hoeil Chung[1], Yunjung Kim[1]

[1] *Hanyang University, Seongdong-Gu, South Korea*

Infrared (IR) spectroscopy was previously adopted for the identification of gall bladder (GB) cancer using bile as a specimen. An organic phase extracted from raw bile juice using a methanol:chloroform solution was dried on a Si wafer and then transmission IR spectrum of the dried sample was collected for the discriminant analysis. Meanwhile, a simpler method such as without the sample-drying procedure is desirable for practical clinical application. To meet the goal, bile samples extracted in deuterated-chloroform phase (CDCl3) were directly measured using transmission near-infrared (NIR) spectroscopy. The bile samples obtained from the normal, GB polyp, hepatocellular cancer, and GB cancer subjects were involved in this study. The acquired NIR spectra were comparatively analyzed and the discriminability of GB cancer was assessed. Next, for sample pretreatment-free analysis, a non-NIR absorbing polytetrafluoroethylene (PTFE) providing a hydrophobic surface was chosen as a substrate to form raw bile juice droplets of a consistent shape. To ensure representative spectroscopic sampling, NIR radiation illuminated the whole area of the dried sample for spectral acquisition. In addition, two-trace two-dimensional (2T2D) correlation analysis was employed to recognize dissimilar spectral features among diverse bile samples for potential improvement of the discrimination. The differentiation of GB cancer samples was mediocre in the case of analyzing the raw spectra; while, with using the 2T2D slice spectra, the discriminability was improved. The sensitive recognition of dissimilar spectral features in GB cancer by 2T2D correlation analysis was responsible for the enhanced discrimination.

008.02

ENSURING GOOD ANALYTICAL SAMPLING OF PHARMACEUTICAL BLENDS BY NIR FIBER OPTIC PROBES INSERTED INTO MOVING POWDER BEDS

Stephen Hammond[1]

[1]Expo Pharma Engineering Services, Cork, Ireland

The foundation of any analytical method is good sampling. If sampling is not representative of the material to be analyzed the whole measurement system is compromised. This basic rule holds true for PAT measurements, but is sometimes neglected, especially when considering moving powder beds.

Engineering solutions have been developed that allow the insertion of diffuse reflection probes into powder streams. Each probe interface is designed to be compatible with the characteristics of the sample material. The capability to rapidly sample the powder bed of different processing steps, measure performance and understand the how the process is operating.

In the same way, the use of a NIR probe inserted into the feedframe of a tablet press, as a means to support the fast release of the product has emerged as a viable option for a continuous process, that can easily be applied to common batch processes. By use of an off-line device that simulates the feedframe of a tablet press, the sampling characteristics and capability of diffuse reflectance probes inserted into a tablet feedframe can be assessed using a minimum amount of material outside of a GMP manufacturing environment.

Key drivers considered for development of the measurement systems were speed of sampling, relevant (unit dose) contributing mass of material, and the ability to detect and react to disturbances in the system. In this area information on sampling characteristics have been captured and used to integrate a probe into at multiple points is SOD manufacturing.

An important facet of the practical implementation of these engineering devices is the ability to detect and eliminate real-life disturbances in the sensor response unrelated to the product quality but are related to processing equipment, stops and starts for example. A software platform has been developed that can in realtime filter out such disturbances allowing only “clean” spectra to be used for prediction of powder characteristics.

This presentation will describe the spectroscopy, engineering development and supporting software that has contributed to a highly capable measurement and control system deployed in SOD manufacturing.

008.03

DEVELOPMENT OF SKIN PHANTOMS FOR IMPROVEMENT OF A NONINVASIVE BLOOD GLUCOSE ASSAY USING DIFFUSE REFLECTION NIR-SPECTROSCOPY

Herbert Michael Heise[1], Elina Rose[1], Sven Delbeck[1]

[1]South-Westphalia University of Applied Sciences, Iserlohn, Germany

Noninvasive blood glucose measurement technology is an important field of medical research as it would enable the increasing number of diabetic patients worldwide to monitor their blood glucose levels without painful punctures and blood sampling. Diffuse reflection NIR spectroscopy of skin is a promising approach to realize such a noninvasive assay.[1] To determine and improve the performance of a custom-built noninvasive NIR spectrometric measurement system for blood glucose analysis, using a special accessory with a rotational ellipsoidal mirror to focus backscattered photons, skin phantoms were produced, which simulated optical properties of various skin tissues. Gel-building gelatin was chosen as matrix scaffold for the tissue phantoms produced with easily adjusted water and glucose content. Scatter was introduced either with particles of TiO2 or glass fiber paper, which were characterized by laser scanning and electron microscopy. Absorption and reflection characteristics of phantoms with different gelatin, water, and pigment concentrations, as well as from the effects of the gel-soaked glass fiber materials were studied in the NIR spectral range of 10000 to 4000 cm^{-1}. To estimate challenges with glucose quantification, also phantoms with D2O as main constituent were prepared. Especially for the simulation of scattering by the stratum corneum, an NIR-transparent porous Teflon film could be implemented as superficial layer. Further spectroscopic investigations were carried out on the effect of optical clearing by application of an NIR-transparent perfluorinated polyether liquid, which reduces scatter from the horny skin layer and allows refractive index matching to the immersion lens of the reflection accessory used for reproducible skin presentation. In particular, spectra of the phantoms with fiberglass showed high agreement with lip spectra measured in diffuse reflectance. Overall, the phantom preparation recipes developed bear considerable potential for optimizing the noninvasive NIR spectrometric skin measurement technique and quantitative multivariate determination of glucose, whereby additional water-soluble blood components such as lactate and urea could be added to approximate the relevant in-vivo conditions for skin measurements.

1. Heise HM, Delbeck S, Marbach R, Noninvasive monitoring of glucose using near-infrared reflection spectroscopy of skin - constraints and effective novel strategy in multivariate calibration. Biosensors 11, 64 (2021)

008.04

NIR-SIGNAL ENHANCED BIOSENSING WITH SENSITIVE SILICON DETECTORS

Jan Stegemann[1,2], Julia Ackermann[1,3], Nour el Houda Fizzi[1], Sebastian Kruss[1,2]

[1]Fraunhofer Institute for Microelectronic Circuits and Systems IMS, Duisburg, Germany, [2]Ruhr University Bochum, Bochum, Germany, [3]University of Duisburg-Essen, Duisburg, Germany

In the context of biosensing, optical signals in the near infrared (NIR) region are superior than those in the visible range. Because of reduced absorption and scattering, as well as minimized autofluorescence, signal-to-noise ratios are high and interfering background is low. Single walled carbon nanotubes (SWCNT) serving as optical biosensors exhibit fluorescence in the NIR range (870 – 2000 nm). SWCNTs' high photostability and the ability to functionalize their surface make them ideal for sensing applications. This allows for the creation of optical biosensors that are extremely sensitive and specific. Biochemical signals are sensed by the functionalized SWCNT, which subsequently converts the biochemical signal into an optical signal (change in fluorescence intensity or wavelength shift of the emission peak). The emission peaks of SWCNTs with (6,4)- and (6,5)-chirality fall exactly on the edge, where the sensitivity of silicon (Si) detectors ends and the sensitivity of InGaAs-detectors begins. Due to the several advantages of silicon detectors, like low noise and low-cost, it is of interest to evaluate them for SWCNTs emitting between 850 to 1000 nm. Simulations for NIR signals, background and noise with different detectors were performed. Results show that for sensing applications Si-detectors are more sensitive than InGaAs-detectors, due to their lower background and noise. This is especially interesting for functional NIR-imaging, when using (6,4)-SWCNTs (880 nm) with common Si-based cameras. Due to the significant drop of the quantum efficiency towards 1000 nm, (6,5)-SWCNTs (990 nm) on the other hand are better imaged with an InGaAs-camera. In other contexts, like screening wellplates or arrays of different biosensors, spatial information is not necessary when scanning the elements. By utilizing pin-photodiodes, silicon as detector material outperform InGaAs for (6,5)-SWCNTs. This was evaluated in experiments with different SWCNT concentrations and further by testing the biosensing with analytes, which reveal similar responses between InGaAs and low-cost Si-photodiodes. To conclude, we show the technology to detect beneficial NIR-signals (<1000 nm) with low-cost commercial cameras and detectors, which opens many opportunities for NIR-biosensing.

008.05

FAST QUALITATIVE AND QUANTITATIVE ANALYSIS OF PEUCEDANUM PRAERUPTORUM DUNN BY THE HANDHELD NIR SPECTROMETERS

Hui Yan[1], Fang Wang[2]

[1]Jangsu University of Science and Technology, Zhenjiang, China, [2]Wanxi University, Lu'an, China

1. Introduction

Peucedanum praeruptorum Dunn (PPD) is a traditional Chinese medical herb of high medical and economic value. It is widely used to treat a cough with thick sputum and dyspnea, and upper respiratory infection. Presently, there are two urgent issues that need to be addressed for PPD.

Firstly, many provinces within China commonly use Maoqianhu and Hongqianhu, which are high-yield, low-cost, and inefficient alternative medicines. Additionally, there are many phenomena of adulteration on the market. For example, PPD is often adulterated with Peucedanum japonicum Thunb, Peucedanum wawrae (Wolff) Su, Peucedanum medicum Dunn, and so on. Because they are similar to PPD in appearance, it is difficult to distinguish with the naked eye.

Secondly, with climate change and excessive fertilization for the pursuit of yield during the artificial planting process, the content of effective components of PPD procyanidin A decreases, many of which do not meet the requirements of the pharmacopeia of the People's Republic of China. In this work, handheld NIR spectrometers were used for the qualitative and quantitative analysis of PPD.

2. Qualitative analysis

The samples, including PPD, Maoqianhu, and adulterated products, were collected, and 82 and 41 samples were randomly selected as the calibration set and the test set, respectively. A handheld NIR spectrometer, NeoSpectra module SWS62221, was used to record the NIR spectra. After the spectral pretreatment, PLSDA model showed the highest performance for the identification of PPD, the prediction accuracies were 95.1%, 94.2%, and 93.4% for the calibration set, cross-validation set, and prediction set, respectively.

3. Quantitative analysis

70 genuine PPD were collected as the calibration set for modeling, and 35 PPD were collected as the test set for the performance validation of the built model. The content of procyanidin A in each sample was measured by HPLC.

A handheld NIR spectrometer, MicroNIR 1700, was used to record NIR spectra. After the combination of the first derivative and SNV was applied to pretreat the spectra, PLS was used for the calibration. The results showed that the RC2, RCV2, and RP2 were 0.9170, 0.8687, and 0.8254, respectively, and the corresponding RMSEC, RMSECV, and RMSEP were 0.098%, 0.122%, and 0.138%, respectively. The RPDcv was 2.79, which indicated that the calibration performance was not high, at a normal level (2< RPDcv<3), it was because the content of PPD procyanidin A was low, from 0.057% to 1.431%.

4. Conclusions

Based on the experiment results, we draw the conclusion that it is feasible that the handheld NIR spectrometer can identify the quality of PPD and quantitatively determine the content of PPD procyanidin A.

FLASH TALK

F08.01

NEAR-INFRARED SPECTROSCOPY (NIRS) AS A SCREENING TOOL FOR LETHAL CHYTRID FUNGUS (BATRACHOCHYTRIUM SALAMANDRIVORANS) IN EASTERN NEWTS (NOTOPTHALAMUS VIRIDESCENS)

Li-Dunn Chen[1], Merrie Urban[2], Carmen Merolle[2], Debra Miller[2], Matthew Gray[2], Devin Chen[3], Andrew Kouba[3], Carrie Kouba[2]

[1]Department of Biochemistry, Molecular Biology, Entomology, and Plant Pathology, Mississippi State University, Mississippi State, United States, [2]Center for Wildlife Health, University of Tennessee Institute of Agriculture, Knoxville, United States, [3]Department of Wildlife, Fisheries & Aquaculture, Mississippi State University, Mississippi State, United States

The emergence of a skin-eating fungal disease, chytridiomycosis, nearly three decades ago has driven hundreds of amphibian species to the brink of extinction. Despite the wide-spread loss of amphibian diversity globally due to chytrid fungus, techniques for effectively detecting the pathogenic fungus B. salamandrivorans (Bsal) in caudates remain limited. As Bsal has yet to invade free-ranging amphibian populations outside of Europe and Asia, research into technologies capable of rapidly assessing prevalence of this fatal fungal pathogen is urgently needed to mitigate its transmission. Traditional techniques (e.g. histology, qPCR) are crucial for diagnosing Bsal but are time consuming and limited to laboratory settings, restricting their use in conservation programs where quick, on-the-ground decisions are needed. Near-infrared spectroscopy (NIRS) provides a solution as a rapid, non-invasive biophotonic technique for screening various diseases in wildlife taxa. Once spectra are collected and a reliable prediction model has been established, the disease status of unknown individuals can be assessed in real-time. The objective of this study was to develop NIRS as a screening tool to rapidly and reliably detect the presence of Bsal in wild caught eastern newts, Notopthalamus viridescens (N=50). Healthy newts were inoculated with a Bsal concentration of 5x103 zoospores/mL. Bsal(-) controls consisted of 10 individuals who received a sham treatment. Live individuals were scanned in triplicate in four regions (chin, cloaca, tail, and foot) using an ASD FieldSpec®3 and small-diameter reflectance probe, then swabbed for qPCR analysis. Following the application of mathematical pretreatments (SNV, detrending, Savitsky-Golay derivative and smoothing), distinct biochemical patterns were observed between the spectra associated with Bsal(+) and Bsal(-) newts. A multi-model benchmarking approach utilizing seven machine learning algorithms with 5-fold cross validation was then applied to calibrate prediction models and subsequently evaluate their predictive efficacies. Bsal genomic copies/ µL detected via qPCR varied by individual and time point of sample collection with average loads of 80170 ± 187506. The seven algorithms tested varied significantly ($p < 0.05$) with regards to predictive performance, such that random forest (79.2 ± 0.4%) yielded the highest and partial least squares the lowest

(66.7 ± 1.5%) mean classification accuracies. Our results reveal that Bsal(-) and strongly Bsal(+) individuals were classified with a promisingly high level of accuracy overall, with sensitivity >50% and a specificity >90% achieved, the former associated with classification rate of Bsal(-) individuals and the latter with Bsal(+) individuals. This work highlights NIRS as a practical diagnostic tool for assessing deadly fungal pathogen occurrence in susceptible amphibian species.

P08.01

NIRS GLOBAL MOLECULAR FINGERPRINT OF BIOBANK SERA OF HEPATITIS C PATIENTS: A PROMISING TOOL FOR RAPID DIAGNOSIS AND PROGNOSIS OF VIRAL HUMAN DISEASES

José Gómez[1], Jennifer Gonzalo[1], Sergio Salguero[3], Óscar Barquero-Pérez[2], María L. Casas[3], María L. Gutiérrez[3], Elena Jaime[3], Enrique Pérez[1], Rafael García-Carretero[4], Javier Ramos[2], Conrado Fernández[3], **Myriam Catala**[1]

[1]Department of Biology and Geology, Physics and Inorganic Chemistry, ESCET, Universidad Rey Juan Carlos, Móstoles, Spain, [2]Department of Signal Theory and Communications, ETSIT, Universidad Rey Juan Carlos, Fuenlabrada, Spain, [3]Hospital Universitario Fundación de Alcorcón, Alcorcón, Spain, [4]Hospital Universitario Móstoles, Móstoles, Spain

The COVID pandemics has highlighted the need for rapid and non-destructive methods for diagnosis and prognosis of viral diseases. Hepatitis C virus (HCV) infection is an often asymptomatic disease in its early stages, where viremia persists, and the infection becomes chronic. HCV can cause varying degrees of fibrosis, which even can lead to cirrhosis and hepatocellular carcinoma. In addition, advanced stages of HCV infection can alter some serum parameters such as circulating proteins and glucemia. However, there is no rapid and inexpensive technique available for the accurate detection of these types of viremias. NIRS is an accurate technique, easy to use, with a low-cost analysis, it performs a fast measurement, does not destroy the sample, and does not generate polluting residues. Therefore, the development of methods for rapid diagnosis of viremias by NIRS would be a major breakthrough in clinical medicine. Our general goal is to develop a diagnostic and prognostic tool of HCV-infected patients based on the NIR spectrum of human skin. For this purpose, we obtained biobank serum samples from HCV patients (N=151), collected between 2000 and 2010. Then, we obtained serum NIRS global molecular fingerprint (GMF) and related each peak to a functional group using NIRS absorption band tables of different functional groups. The HCV serum GMF representative peaks correspond to water (several molecular species), glucose, proteins and saturated/unsaturated lipids. The Principal Components Analysis shows clear differences between sera with detectable HCV levels and those negative. This study establishes the basis for future lines of research that will allow the identification of biomarkers for the diagnosis, prognosis and follow-up of HCV-infected patients based on serum NIRS spectra.

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P08.02

A SIMPLE NEAR INFRARED SPECTROSCOPY SYSTEM FOR QUANTIFICATION OF GLUCOSE IN SOLUTION AND INTRALIPID SAMPLES.

Nicholas Davison[1], Christopher Gaffney[1], Jemma Kerns[1], Qiandong Zhuang[1]

[1]Lancaster University, Lancaster, United Kingdom

Near Infrared (NIR) spectroscopy offers several potential applications in the field of biosensing due to the depth of penetration of NIR radiation in tissue compared to other spectral regions. Glucose sensing is a regular part of the management of diabetes, but current monitoring is performed using invasive needle-prick methods or subcutaneous sensors. A non-invasive, continuous glucose sensing device could benefit patients in the self-management of the condition. However, sensing accuracy depends heavily on the design and choice of components for the system. We aimed to develop a NIR absorption spectroscopy system capable of determining liquid glucose concentrations in the physiological range, using simple NIR detectors and light sources. Two different light sources (a halogen lamp and a globar) and three NIR detector assemblies (a photodiode array, a Czerny-Turner monochromator with an InGaAs photodiode and a miniature Fourier Transform infrared spectrometer) were systematically tested by collecting spectra from the same set of aqueous glucose concentrations, from 10-100mmol/L in 10mmol/L increments. Partial Least Squares (PLS) regression was used to determine the Standard Error of Prediction (SEP) and coefficient of determination (R2), which were compared for each component. The optimal system was determined to be the halogen lamp with the InGaAs photodiode and further concentrations with decreasing concentration step were tested to identify the limit of detection, which was determined to be 2mmol/L. Finally, normally reflected light was collected from glucose samples in 10% intralipid suspension from 2-10mmol/L. The relationship between intensity and concentration indicated that below 1300nm the spectra were dominated by scattering, which varied with glucose concentration as predicted by Mie theory, whilst above 1300nm the spectra were dominated by absorption. In intralipid, the data showed an SEP of 3.15mmol/L, and this suggests that the system offers potential to collect reflectance spectra from more advanced intralipid-based tissue phantoms and form the basis for a prototype NIR glucose-sensing device.

P08.04

A NEW PERSPECTIVE IN UNDERSTANDING THE PROCESSING MECHANISMS OF TRADITIONAL CHINESE MEDICINE BY NEAR-INFRARED SPECTROSCOPY WITH AQUAPHOTOMICS

Lele Gao[1], Liang Zhong[1], Lian Li[1], Hengchang Zang[1]

[1]Shandong University, Jinan, China

1 Introduction. The processing of traditional Chinese medicine (TCM) is a critical pharmaceutical technology that enhances the efficacy and safety of TCM. However, the underlying scientific principles of processing have remained largely unknown, hindering the development of TCM. Current studies on the mechanisms of processing primarily focus on analyzing specific chemical components and biochemical indicators, ignoring the holistic theory of TCM, which is inconsistent with the TCM characteristics of network targets and multi-component therapy. Aquaphotomics provides an opportunity to study the mechanisms of processing, particularly blood circulation related, by exploring the changes in the hydrogen-bonded network structure.

2 Materials and methods. This study examined Crataegi Fructus, which has four processed products (raw, fried, charred, and carbonized), as an example to investigate the processing mechanisms that cause differences in their clinical use. The differences between the four processed products were analyzed in terms of physical properties and chemical compositions using a metallographic microscope, scanning electron microscope, and high-performance liquid chromatography. The study also proposed a new method for investigating processing mechanisms using near-infrared spectroscopy with Aquaphotomics, including analysis of raw and continuous wavelet transform spectra, difference spectra, principal component analysis, and Aquaphotomics.

3 Results and discussion. The results showed that there were significant differences after processing, with a loose and porous structure formed, and the 5-hydroxymethylfurfural content increased. Additionally, raw Crataegi Fructus was found to promote an active hydrogen-bonded network, which may account for the circulation-promoting effect, while carbonized Crataegi Fructus formed a stable hydrogen-bonded network structure, thus exerting its hemostatic effect.

4 Conclusion. The scientific principle of “Carbonized-processing promoting hemostatic effect” in the processing of TCM was first revealed by Aquaphotomics in this study, providing a valuable reference for future studies on the processing mechanisms of TCM.

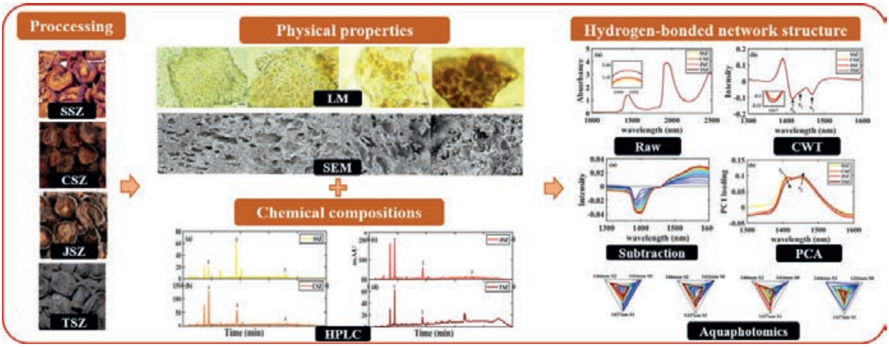

Figure 1. Graphical abstract of this study

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P08.06

ASSESSMENT OF X-IRRADIATION DOSE EXPOSURE IN MICE USING NEAR INFRARED (NIR) SPECTROSCOPY AND AQUAPHOTOMICS

Jelena Muncan[1], Masaru Yamaguchi[2], Nikolai Iordanov[1], Ikuo Kashiwakura[2], Roumiana Tsenkova[1]

[1]Aquaphotomics Research Department, Graduate School of Agricultural Science, Kobe University, Kobe, Japan, [2]Department of Radiological Life Sciences, Graduate School of Health Sciences, Hirosaki University, Hirosaki, Japan

Accurate and rapid assessment of radiation exposure doses is crucial not only for predicting health effects in victims of radiation accidents and disasters but also in other scenarios such as occupational exposure in nuclear power plants, medical diagnostic and therapeutic procedures involving ionizing radiation, and accidental or intentional exposure to radiation from sources such as radioactive materials. The development of a method for large-scale biological dosimetry that can provide accurate results quickly would be highly beneficial in improving the management of radiation exposure in various scenarios. This research investigates the potential of using near infrared (NIR) spectroscopy and aquaphotomics [1] for such purposes.

The study was performed using seven-week-old female mice (CLEA, Tokyo, Japan). Mice were randomly divided into 6 groups with 8 mice per group and subjected to total body irradiation using doses of 0, 0.5, 2, 4, 6 and 6.5 Gy (MX-160Labo, MediXtec, Tokyo). NIR measurements were performed using microNIR portable spectrometer (VIAVI solutions, USA) at the following time points: one day before irradiation, immediately after the irradiation, 6, 12, 24, 48 and 72 hours, and 2, 5, 10, 15, 20, 25 and 30 days after irradiation. The assessment of X-irradiation dose was performed using Partial Least Squares Regression [2] analysis by creating models separately for each of the different time points. Several different strategies were tested in the terms of different pre-processing techniques and different spectral ranges. The validation was performed using active-class validation, where active class was “animal”, meaning that in each iteration all the spectra of one mouse were excluded from calibration and used as a test set. These strategies resulted in creation of 16 models for each of the measurement time points and the optimal strategy was chosen based on the lowest root mean square errors of validation RMSE and highest coefficients of determination R^2.

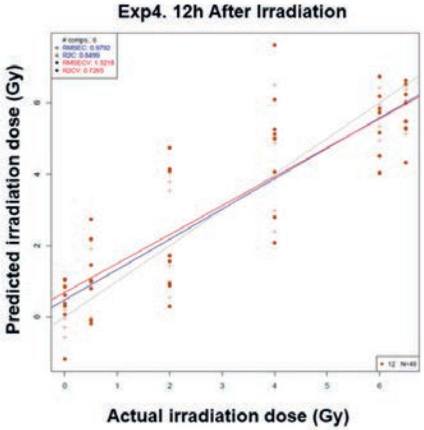
The best results were achieved for the following conditions: range 900 – 1100 nm, smoothing using Savitzky-Golay filter, detrend and standard normal variate transformation. The best prediction of irradiation dose exposure was achieved using spectra acquired 12 hours after the irradiation, with $R^2=0.73$ and RMSE=1.32 Gy (Fig.1). The prediction of irradiation dose was successful in the time window from immediately after up to 12 h after the irradiation, and then 5 to 30 days after the irradiation. The PLSR statistics for the 24 to 72-hour period after irradiation were unsatisfactory, possibly due to the presence of distinct phases in the biological response to ionizing irradiation, as suggested by studies on cells [3].

This research shows that it is possible to predict the dose of irradiation to which the mice have been exposed to with reasonable accuracy immediately after the irradiation up to 12 h after the irradiation. This shows that applied method has a great potential for rapid early biodosimetry and efficient triage in the cases of irradiation accidents.

PLSR statistics

Time after irradiation	No. of Factors	R^2 Calibration	R^2 Validation	RMSE (Gy) Calibration	RMSE (Gy) Validation
Exp.2 – 0 h	6	0.81	0.70	1.09	1.39
Exp.3 – 6 h	6	0.82	0.70	1.06	1.37
Exp.4 – 12 h	**6**	**0.85**	**0.73**	**0.98**	**1.32**
Exp.5 – 24 h	5	0.63	0.47	1.53	1.84
Exp.6 – 48 h	4	0.59	0.46	1.63	1.85
Exp.7 – 72 h	4	0.53	0.41	1.73	1.94
Exp.8 – 5 days	5	0.78	0.68	1.20	1.42
Exp.9 – 10 days	6	0.81	0.66	1.10	1.46
Exp.10 – 15 days	5	0.74	0.58	0.74	1.65
*Exp.11 – 20 days	1	0.10	0.07	2.27	2.31
Exp.12 – 25days	6	0.69	0.69	0.96	1.30
Exp.13 – 30 days	6	0.82	0.62	0.96	1.40

* *Model could not be developed due to many faulty measurements*



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P08.07

COMPENSATING-VOTING METHOD FOR VIS-NIR SPECTRAL PATTERN RECOGNITION

Yongqi Huang[1], Lu Yuan[1], Jiemei Chen[1], **Tao Pan**[1]

[1] *Jinan University, Guangzhou, China*

Spectral discriminant analysis is based on spectral similarities among samples within the same group and spectral differences between samples of different groups, using pattern recognition method to classify samples, which is often easier and more effective than quantitative analysis for the identification of samples with similar components. Models fusion, using the complementarity of multi-model, is expected to achieve better integrated discrimination results than its sub-models. Drawing on the ideas of game theory and integrated learning, in the present study, the strategy based on compensating and voting was proposed, and two examples of blood discriminant analysis of Vis-NIR spectra were used to examine the proposed compensating-voting method.

Three-model voting strategy based on compensation: (i) determined the model $\Phi_{A,B}$ according to the maximum total recognition-accuracy rate (RAR_{Total}) and taking into account balance of the two types of discrimination; (ii) determined the compensation models Φ_A and Φ_B according to the respective maximum recognition-accuracy rate (RAR_A, RAR_B); (iii) used $\Phi_{A,B}$, Φ_A and Φ_B to judge the samples three times and determined the final category of samples based on the principle of "two wins in three games", which is denoted as the joint model Φ_{Joint}. Classifier: partial least squares-discriminant analysis (PLS-DA). Wavelength selection: equidistant wavelength combination with initial wavelength, number of wavelengths and number of wavelength gaps as screening parameters (Appl Spectrosc, 68(3), 2014). Wavelength range: 400-2498 nm.

Blood discriminant analysis of α-thalassemia (A) and ß-thalassemia (B): divided into calibration (A100, B50), prediction (A70, B35) and validation (A70, B35) sets. For modeling, the RAR_{Total} of $\Phi_{A,B}$, Φ_A, Φ_B, and Φ_{Joint} were 91.2%, 88.7%, 90.4%, and 94.3%, respectively. Serum discriminant analysis of breast cancer (A) and normal control (B): divided into calibration (A25, B25), prediction (A20, B20) and validation (A20, B20) sets. The RARTotal of $\Phi_{A,B}$, Φ_A, Φ_B, and Φ_{Joint} were 96.7%, 92.2%, 96.7%, and 100%, respectively. The modeling effect of ΦJ_{oint} of both examples were significantly superior to its three sub-models. For independent validation, the RAR_{Total} of their Φ_{Joint} were 90.4% and 95%, respectively.

The results indicated that, the proposed compensating-voting method achieved the model fusion effect of complementary advantages. This idea can be extended to n-category discriminant analysis.

P08.08

NIR SPECTROSCOPY IN PHARMACEUTICAL BLEND CHARACTERIZATION: SAMPLE PREPARATION & DATA ANALYTICS

Amrit Paudel[1], Prakash Muthudoss[2]

[1]Research Center Pharmaceutical Engineering (rcpe) Gmbh, Graz, Austria, [2]A2Z4.0 Research and Analytics Private Limited, Chennai, India

We here present our two recent articles published at AAPS PharmSciTech that make significant contributions to understanding the factors that impact near infrared (NIR) analysis in pharmaceutical development and propose methods for improving the reliability and robustness of NIR spectroscopy data using machine learning and AI techniques. The first article, by Muthudoss et al. (2022) conducted a comprehensive study on the factors affecting the performance of NIR spectroscopy in quantitative analysis of pharmaceuticals. The study proposes a data analytical quality by design (AQbD) framework to optimize NIR performance by selecting appropriate pre-processing methods, chemometric models, bias-variance decomposition, and machine learning techniques. This work provides insights into the complex factors impacting NIR spectroscopy's accuracy and precision and offers a roadmap for its performance optimization in pharmaceutical analysis. The follow up article by Hussain et al. (2023) describes a systematic approach for selecting representative subsamples from open-source data and using quality measures and visualization techniques to improve the reliability and robustness of NIR spectroscopy data for pharmaceutical analysis. The focus here is on an open-source tablet dataset and demonstrates how selected subsamples can be used to investigate model transferability across different scales and improve the performance of ANN-MLP models through hyperparameter tuning. This work makes a significant contribution to enhancing the accuracy and reliability of NIR spectroscopy in pharmaceutical analysis.

The work published in these two articles provides significant contributions to improving the reliability and robustness of NIR spectroscopy in non-destructive testing across different industries. They offer insights into the factors that impact its performance and propose methods for optimizing machine learning models and selecting representative subsamples.

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P08.09

FORENSIC INTELLIGENCE THROUGH INSTANT NON-DESTRUCTIVE ANALYSIS OF FALSIFIED MEDICINES VIA ULTRA-PORTABLE NIR TECHNOLOGY

Hervé Rais[1], Pierre Esseiva[1], Olivier Delémont[1], **Anne Flore Prior**[1]

[1]School of Criminal Justice, University Of Lausanne, Lausanne, Switzerland

Falsified medicines represent a significant illicit market that is, at least in part, a matter of organized crime. In addition to potentially endangering the lives of those who use them, they can lead to significant economic losses and a drop of confidence in the health system. Although the scale of the problem varies greatly from region to region, it shows no sign of slowing down. It is therefore critical to maintain, strengthen and diversify strategies to understand and disrupt this phenomenon.

In this context, ultra-portable NIR technology can make a major contribution by providing a real time, portable, non-destructive and highly informative approach to tackle various problems associated with medicines. This may include, for example, control of authenticity, establishment of links between counterfeits, screening of imported medicines and quality check.

This presentation showcases the results of several NIR analysis campaigns conducted on falsified and suspect drugs. The instrument used is an OnSite-W MicroNIR from Viavi Solutions (Scottsdale, USA) coupled with a mobile application (via Bluetooth) that send the NIR spectra to a server where the statistical data treatment takes place.

Two case situations are studied. One on medicines used for erectile dysfunction (Viagra and others), the other on medicines used for HIV preventive treatment (HIV PrEP). In both cases, we explore the wide range of information that can be obtained. We also look at some fairly simple but already effective implementation models. Finally, we discuss future analyses and perspectives that should confirm and develop the practical uses of this technology in the field of falsified medicines.

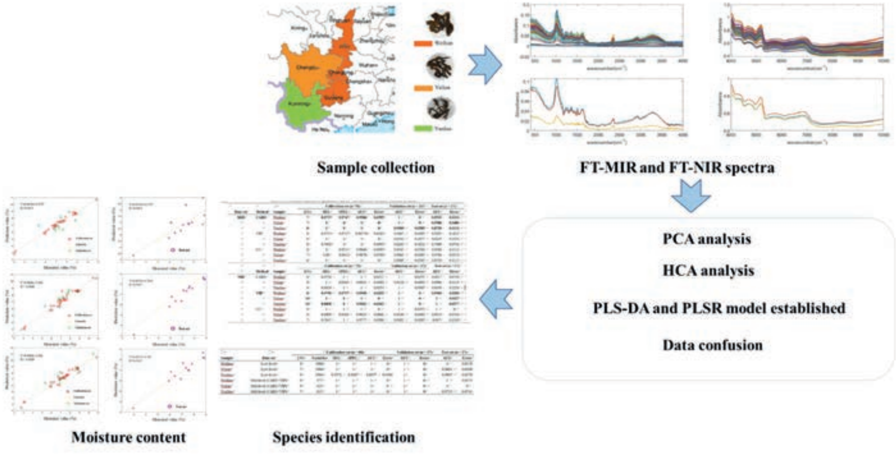
P08.10

APPLICATION OF RAPID IDENTIFICATION AND DETERMINATION OF MOISTURE CONTENT OF DIFFERENT SPECIES OF RHIZOMA COPTIDIS BASED ON COMBINED NEAR-INFRARED AND MID-INFRARED SPECTROSCOPY

Mengyin Tian[1]

[1]Shandong University, Ji Nan, China

Background
Traditional Chinese medicine (TCM) has been clinically proven for thousands of years. The difference in utility between different species has been highly valued by doctors of all dynasties. species identification relies mainly on manual experience, with methods that are difficult to manipulate and highly specialized.
Objective
The purpose of this study is to replace the complex methods used for empirical identification of by compensating the information loss of a single sensor through data fusion.
Methods
With Rhizoma coptidis (RC) as the research object, NIR, and MIR spectra were collected based on spectral optimization and data fusion technology. PLS-DA (n = 134) and PLSR (n = 63) models were established to identify the medicinal materials and determine the moisture content in the medicinal materials.
Results
For the identification of three species of RC, the mid-level fusion model is better than the single-spectrum model. The coefficients (sensitivity and specificity of prediction set) of near infrared (NIR), mid-infrared (MIR), and data fusion qualitative models were all higher than 0.95, and the AUC value was 1 in detail. The model of NIR data is superior to the MIR data. The result of low-level fusion is similar to that of NIR optimization model. The RPD of the test set of NIR and low-level fusion model is 3.98 and 3.02, respectively, indicating that the model has good prediction ability.
Conclusion
NIR and MIR data fusion technology can be applied to the demand of RC species identification, the analysis speed is fast, without complex pretreatment methods. It could provide technical support for the rapid determination of the moisture content of RC.


Sample collection
FT-MIR and FT-NIR spectra
PCA analysis
HCA analysis
PLS-DA and PLSR model established
Data confusion
Moisture content
Species identification

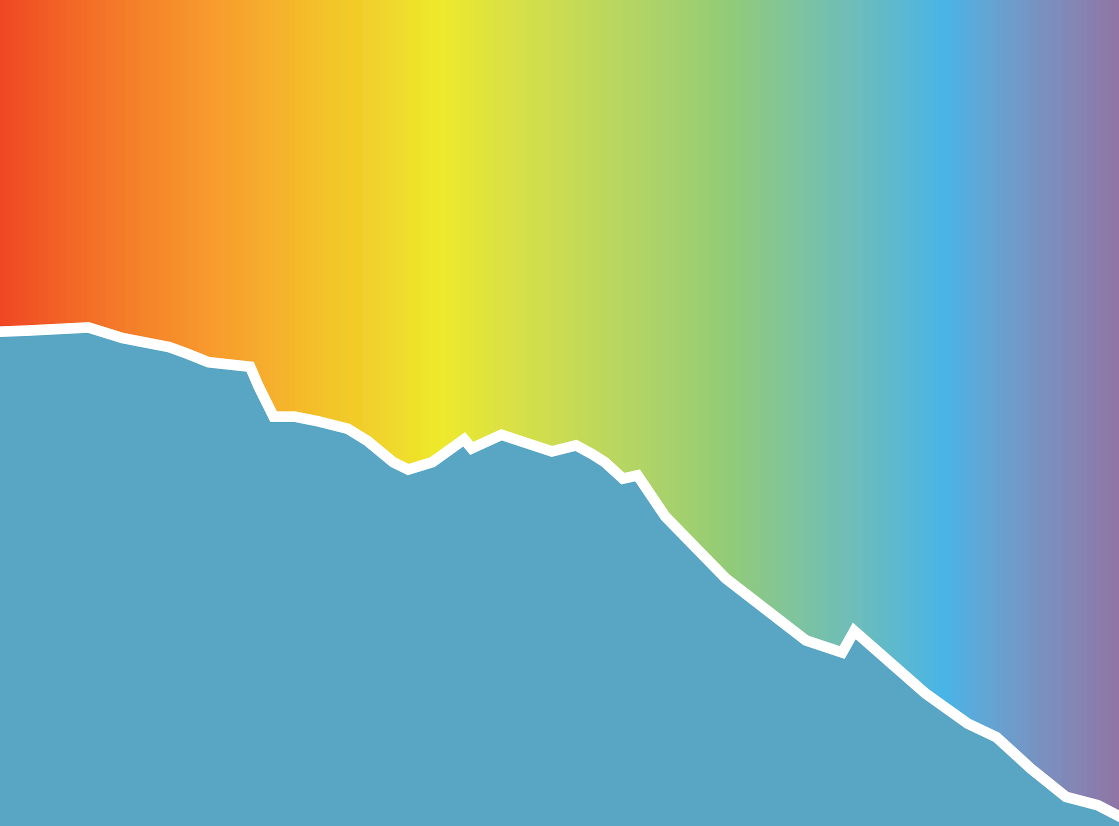
P08.11

INNOVATIVE POTENTIAL OF PORTABLE NIR: FIELD USE APPLIED TO THE MOLLUSK BIOMPHALARIA

Vanessa Valladares[1], Gabriela Friani[1], Clélia Christina Mello-Silva[1]

[1]*Fundação Oswaldo Cruz, Rio De Janeiro, Brazil*

NIR spectroscopy can contribute to complex ecological studies based on changes in spectra, which act as physical-chemical fingerprints. However, traditional benchtop NIR spectrometers considerably limit usage scenarios. Accordingly, the miniaturization of spectrometers with high level performance has become a research trend. A portable version of the NIR enables spectrophotometric analyses to be performed directly in the field and makes it possible to know quickly and effectively the different adaptations undergone by the host in relation to the environment. This work proposes to establish and standardize the procedure for collecting spectra using a portable NIR, building a spectrophotometric model of Biomphalaria sp. directly in the field at different seasons of the year. One hundred and eleven mollusks of the genus Biomphalaria were collected in the city of Sumidouro/RJ/Brazil, transmission area of schistosomiasis, in the months of January, February and March 2023, totaling 3 collections, during the summer season. The spectra of live mollusks were obtained individually in the field using a portable NIR equipment connected to a notebook. To standardize the spectra obtained in the field, a wooden box covered with black material was built to maintain the equipment during the analysis, in order to stabilize the equipment and minimize the influence of ambient light. The animals were dried on absorbent paper and placed individually on the window of the equipment, and only when the wooden box was closed, the obtaining of the spectrum was activated by the notebook. Principal component analysis of these first results showed changes in the snail group between the different collections throughout the summer season. It is possible to verify the gradual change in the expression of the chemical phenotype of these animals throughout the season, where the spectra obtained in the first collection showed differences from the other groups, with the scores of the second collection being the most mixed with the others, showing the transition period. These preliminary results demonstrate not only the possibility of applying portable NIR as a fast, simple and innovative tool directly in the field on species of the genus Biomphalaria, but also emphasize the influence of the environment on the chemical phenotype of these individuals, and their ability to adapt to small environmental changes they are subjected to in a short period of time, such as seasons.



THEME 09

PAT/INDUSTRIAL APPLICATIONS

ORAL PRESENTATION: 009.01 – 009.05
FLASH TALK: F09.01
POSTER PRESENTATION: P09.01 – P09.13

ORAL PRESENTATION

009.01

THE SAMPLING INTERFACE: A CRITICAL SUCCESS FACTOR IN PROCESS SAMPLING AND PAT

Kim Harry Esbensen, Nawaf Abu-Khalaf, **David Honigs**

[1]KHE Consulting, København, Denmark, [2]Department of Process, Energy and Environmental Technology, University of South-Eastern Norway, Norway, Porsgrunn, Norway

Methods and equipment of process sampling are front and center in the realm of the Theory of Sampling (TOS). TOS supplies a comprehensive, well-proven framework that derives all principles and implementation demands needed for **how to** extract representative samples from moving lots, i.e. from a conveyor belt or from ducted material streams. The sampling interface comes to the fore. PAT aspires to take this situation over to the situation in which the task is **how to** extract representative sensor signals.

For 'sensor sampling' i.e. Process Analytical Technologies (PAT), there is no similar foundational framework. Instead, a pronounced practical approach is evident in this realm, in which the question of **how to** achieve representative sensor signals is not so much related the design and implementation of an appropriate sampling interface between the sensor and the streaming flux of matter. Rather, a survey of the gamut of sensor interfaces presented in industry and in the literature reveals a credo that appears to be: "Get good quality multivariate spectral data – and chemometrics will do the rest", exclusively relying on multivariate calibration of process sensor signals (multi-channel analytical instruments). There is a tacit misunderstanding that the admittedly powerful chemometric data modelling is able to take on and correct for any kind of sensor signal uncertainty - including 'sampling errors'. However, this leaves analytical representativity the victim of imperfect understanding of the nature of data analytical errors (ε) vs. sampling errors (TOS errors).

The current PAT focus, in which NIR is the preeminent analytical modality, representativity is wholly related to spectral and, reference sample measurement uncertainty (MU), and to possible data modeling errors, unfortunately ignores the geometric specifics of sensor signal acquisition in relation to the full cross-section of the streaming/ducted flux of matter even though this is the very domain where sampling errors occur in the exact same fashion as when extracting physical samples. The process sampling interface again comes to the fore. In fact, the principles behind proper sampling from stationary lots and process sampling are identical, which means that they must <u>both</u> comply with TOS' stipulations to be able to guarantee representativity. Sampling error effects for sensor and probe signal acquisition are largely terra incognito in the PAT realm.

This situation invites all parties to take a closer view of the interconnections between TOS and the way sensor sampling is performed in PAT, using NIR or any other suitable spectroscopic modality. It turns out that there is a complete duality between TOS and PAT, which must be complied with if representative sensor signals is the goal. TOS' usefulness is its stipulation of Governing Principles (GP), Sampling Unit Operations (SUO) and sampling error management covers all demands for sampling both stationary as well as dynamic, moving lots – whether extracting physical samples of spectral signals. Aside from NIR and chemometrics, TOS constitutes a missing link in PAT.

009.02

DEVELOPMENT OF A COST-EFFECTIVE NEAR-INFRARED SENSOR FOR INLINE MOISTURE MONITORING FOR ENHANCED DRYING (EDDY) PROCESSES

Sebastian Friedl[1], Robert Zimmerleiter[1], Michael Lauermann[2], Jovana Kovacevic[2], Daniel Lager[2], Abdulrahman Dahash[2], Michal Rezucha[2], Reinhard Jentsch[2], Martin Fischer[3], Raphael Priesner[4], Robert Holzer[1], Markus Brandstetter[1]

[1]Research Center For Non Destructive Testing Gmbh, Linz, Austria, [2]Austrian Institute of Technology GmbH, Vienna, Austria, [3]Fischer Brot GmbH, Linz, Austria, [4]AGRANA Stärke GmbH, Gmünd, Austria

About 12-25 % of the industrial energy consumption in developed countries is used for drying and dehydration processes. They are among the most wide-spread processes in industry and are currently predominantly relying on fossil fuels. Most of these processes are carried out without knowledge about the actual moisture content of the product. Further, differences in air humidity, temperature, and moisture of raw material, caused by seasonal fluctuations, are typically not considered when it comes to drying parameters.

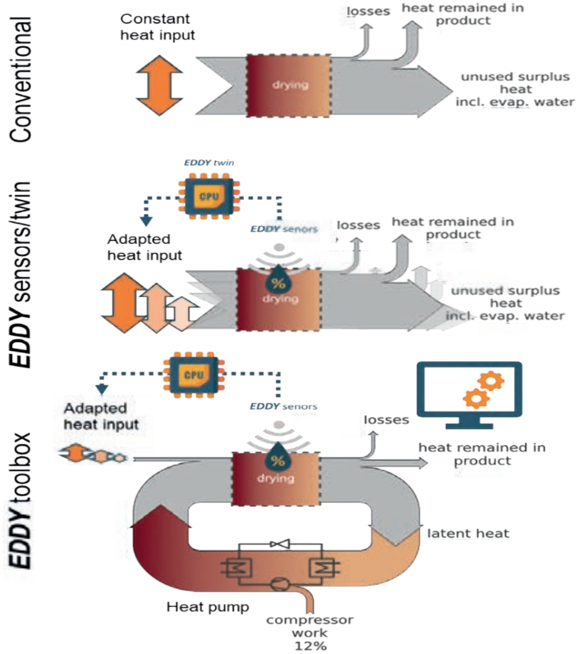


Figure 1. Variable heat input with the EDDY concept

We present the development of a cost-effective sensor for contact-less, real-time moisture monitoring employing a near-infrared (NIR) spectrometer that is based on Micro-Opto-Electromechanical System (MOEMS) technology. This kind of fully-integrated spectrometers not only allow significant reduction in hardware costs, but also show increased robustness due to the lack of moving parts when compared to conventional process spectrometers. The moisture sensor is deployed at production sites for bakery products and saccharification products. In the former a conveyor belt dryer is used for product drying. The challenges for this product where the high temperature and limited space in the oven. A reflection measurement head has been designed to deal with this challenging environment. In the latter a spray dryer is used to dry the product. In this application the moisture sensor is equipped with an automatic sampler which drops the product into a sample chamber where it is analysed. A multi-mode optical fiber is used for illumination and collection of the reflected light.

The data gained by the real-time inline measurements are combined with advanced numerical models to achieve a digital twin ("EDDY twin", Fig. 1) of the whole drying system. With this twin in place, a feed-back control will be established on the real dryers to account for fluctuations in product characteristics, weather conditions, etc. Therefore, the operating conditions of the drying processes can be optimized, which leads to higher productivity and more constant product quality.

The work was performed in the project "EDDY – Enhanced Drying" (FFG project number 880778) within the NEFI innovation network. This project is supported with the funds from the Climate and Energy Fund and implemented in the framework of the RTI-initiative "Flagship region Energy".

009.03

NEAR-INFRARED SPECTROSCOPY FOR THE MONITORING OF CONTINUOUS FLOW SYNTHESIS OF GRIGNARD REAGENTS

Alexis Rousseaux[1]

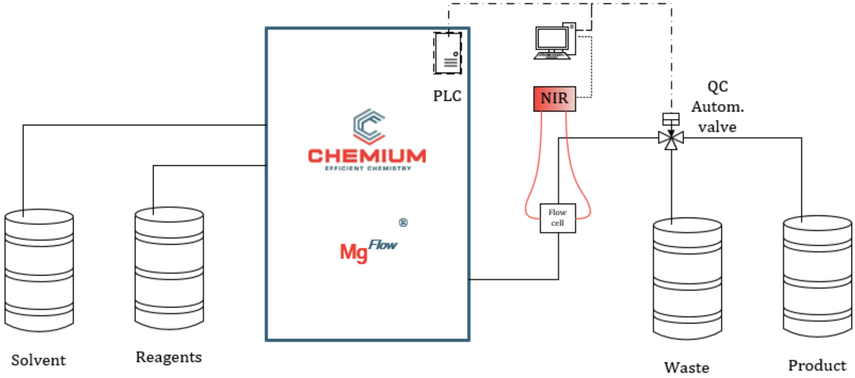
[1]Chemium, Louvain-la-neuve, Belgium

Flow chemistry is increasingly gaining interest in industrial applications due to its many advantages. It provides an accurate control over reaction parameters, enhances process safety, helps in process automation, facilitates the scale-up operations, etc (1).

Flow chemistry is particularly suitable for dealing with exothermic reactions such as those observed in the synthesis of Grignard reagents. These compounds are "building blocks" molecules used in a wide range of industries like pharmaceutical, cosmetics and agro-chemicals (2,3). They are traditionally produced in batch mode in which it is difficult to handle the high reaction exothermicity.

In order to solve common batch-related issues, Chemium, a Belgian ChemTech specialized in fine chemistry, has developed the MgFlow® Technology, a continuous flow reactor monitored by in-line near-infrared (NIR) spectroscopy for Grignard reagents synthesis. Continuous flow synthesis and in-line NIR monitoring combination offers better reaction control and ensures a high product quality at any given time.

NIR monitoring has previously been applied in the follow up of a specific Grignard reagent batch synthesis using Partial Least Squares (PLS) regression, the most common multivariate technique, to quantify reaction compounds (4,5). In the current study, the first application of continuous flow Grignard reagents synthesis monitored by NIR spectroscopy is presented. To achieve this goal, reference methods have been developed to quantify halide reagents and Grignard reagents. In addition, multiple spectra pretreatments and different multivariate techniques like Ridge, Lasso, ElasticNet, PLS, interval-PLS, etc. have been investigated. The resulting models have been selected according to cross-validation results and have been tested on an external validation dataset. Thanks to this work, Chemium is able to define specific models for each Grignard reagent developed on its MgFlow Technology. Moreover, a model transfer methodology has been implemented to allow the use of developed models on all MgFlow reactor scales and locations.



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009.04

QUALITY ASSURANCE IN GLUE-LAMINATED TIMBER PRODUCTION ASSISTED WITH NEAR INFRARED SPECTROSCOPY

Jakub Sandak[1,2], Andreas Hänsel[3], Juana Mai[3], Anna Sandak[1,2],
Destin Bamokina Moanda[4], Martin Lehmann[4], Thomas Volkmer[4],
Florian Schinle, Gordian Stapf[5], Benoit Canoine[5], Sebastien Josset[5],
Cuauhtli Campos Mijangos[1,2], Ender Hazir[1], Peter Niemz[4]

[1]InnoRenew CoE, Izola, Slovenia, [2]University of Primorska, Koper, Slovenia, [3]Saxon University of Cooperative Education, Dresden, Germany, [4]Berner Fachhochschule, Biel, Switzerland, [5]Henkel & Cie. AG, Sempach Station, Switzerland

Engineered timber products are a great alternative to traditional construction materials, such as concrete or steel. However, due to its nature, wood is not an easy material to handle, especially considering its durability and susceptibility to deterioration due to abiotic factors. Glue-laminated timber is a composite with at least four timber lamellae glued together that were manufactured, controlled and marked according to certain rules. One of the greatest advantages of glulam is that it can be manufactured in a wide variety of shapes, sizes and configurations to provide elements for wide-span but light construction. Unfortunately, it is extremely difficult to assure the proper performance of engineered timber product along its service life. The glue connection between lamellae is particularly important and only minor (less than 5%) delamination is accepted in timber structures. The quality assurance of the gluing process is, therefore, critical for the optimal product's performance. Near infrared spectroscopy was identified here as a candidate solution for sorting of timber before gluing aiming to eliminate extensive future delamination.

The results from the pilot tests confirm high suitability of NIR spectroscopy for supporting quality assurance before gluing operation of engineered timber products. The evaluated MicroNIR sensor is a portable, resistant and easy-to-operate instrument. It allowed measuring>2500 spectra used for calibration of various chemometric models. A direct assessment of wood properties is straightforward after integrating prediction models with the scanner software. A single measurement cycle takes less than 3 seconds, enabling this method to become an alternative routine quality assurance solution for off-line implementation at the production facility. The best performance was noticed for assessing wood moisture content, with a lower capacity to estimate wood density and mechanical properties. NIR spectroscopy was modestly capable of predicting surface roughness. However, the traceability of the raw resources and the automatic classification of diverse wood defects were successfully demonstrated. The developed chemometric model could predict the total delamination and detailed delamination length. The manual NIR spectroscopy assessment of wood quality before gluing is a highly recommended solution for implementation in glue-laminated timber production plants. However, further improvement of the chemometric models and measurement routines is indispensable before the broad practical implementation.

009.05

IMPLEMENTATION OF DYNAMIC AND STATIC MOISTURE CONTROL IN FLUIDIZED BED GRANULATION

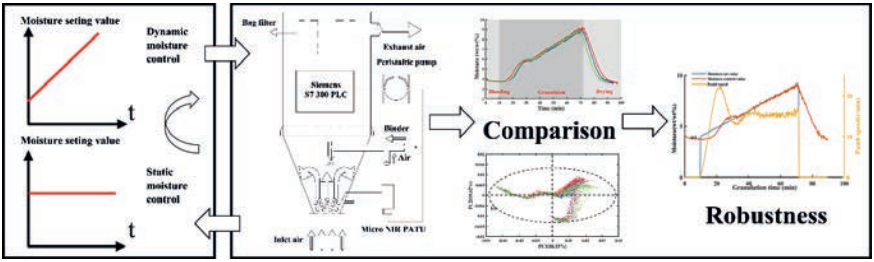
Liang Zhong[1], Lele Gao[1], Hengchang Zang[1]

[1]*Shandong University, Jinan, China*

Abstract: The application of process analysis and control is essential to enhance process understanding and ensure output material quality. The present study focuses on the stability of the feedback control system for a fluidized bed granulation process. Two strategies of dynamic moisture control (DMC) and static moisture control (SMC) were established based on the in-line moisture value obtained from the near-infrared sensor and control algorithm. The performance of these strategies on quality consistency control was examined using process moisture similarity analysis and principal component analysis. The stable moisture control performance and low batch-to-batch variability indicated that the DMC method was significantly better than other granulation methods. In addition, the investigation of robustness further showed that the implemented DMC method was able to produce predetermined target moisture values by varying process parameters. This study provides an advanced and simple control method for fluidized bed granulation quality assurance.

Keywords: Fluidized bed granulation; Near-infrared spectroscopy; Quality consistency; Dynamic moisture control; Static moisture control.

Graphical abstract:



F09.01

RAPID END-POINT DETERMINATION OF THE EXTRACTION PROCESSES BY NEAR-INFRARED SPECTROSCOPY

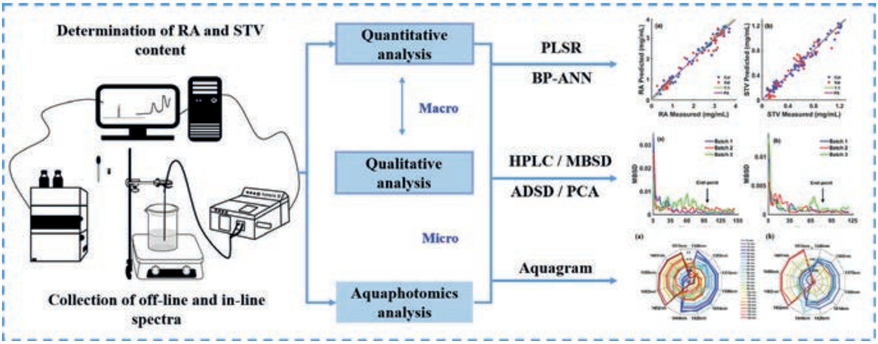
Lele Gao[1], Liang Zhong[1], Lian Li[1], Hengchang Zang[1]

[1]Shandong University, Jinan, China

Extraction process is not only a critical manufacturing unit but also the initial process of various extracts and preparations. Taking the Stevia rebaudiana Bertoni leaves as an example, Rebaudioside A (RA) and Stevioside (STV) are abundant steviol glycosides (SGs) contained in Stevia rebaudiana Bertoni leaves, which exhibit good stability and various pharmacological activities that have been widely developed in the food and pharmaceutical industries. However, the stevia industry still suffers from high consumption, low efficiency, and long-term dependence on the operational experience of workers. The extraction process of Stevia rebaudiana Bertoni leaves one of the fundamental units for the production of SGs, which is crucial for the homogeneous stability of the final product. The applications of near-infrared (NIR) spectroscopy combined with chemometrics in determining the end-point of the extraction process were proposed in this study. Firstly, the quantitative models were established by partial least squares regression (PLSR) and back-propagation artificial neural network (BP-ANN) to rapidly detect the changes of content RA and STV, which have good prediction results. Secondly, the qualitative analysis methods were established by moving block of standard deviation (MBSD), absolute distance of standard deviation (ADSD) and principal component analysis (PCA) to rapidly determine the extraction end-point, which MBSD method was consistent with the high-performance liquid chromatography (HPLC) method. Finally, the variation of the extraction process was revealed by Aquaphotomic to provide a microscopic perspective of water molecules for end-point determination. The HPLC method requires 30 min to determine the content, whereas whereas NIR spectroscopy requires only 18 s to obtain a spectrum. These results indicate that the extraction end-point of Stevia rebaudiana Bertoni leaves can be determined rapidly and accurately using NIR spectroscopy, which provides a significant reference for other food, medicinal plants, and agricultural products production processes.

Keywords: Extraction process; Stevia rebaudiana Bertoni leaves; Near-infrared spectroscopy; End-point determination; Aquaphotomics.

Graphical abstract:



References: Chemometrics and Intelligent Laboratory Systems 230 (2022) 104668.

POSTER PRESENTATION

P09.01

DEVELOPMENT OF A PAT PLATFORM FOR THE PREDICTION OF GRANULE TABLETING PROPERTIES

Tibor Casian[1], Cristiana Lazurca[1], Victor Marcu[1], Brigitta Nagy[2], Zsombor K Nagy[2], Radu Oprean[1], Ioan Tomuta[1], Eva Katalin Kelemen[3], Erzsebet Orsolya Tokes[3], Katalin Zoldi[3], Bela Kovacs[3,4]

[1]Iuliu Hatieganu University Of Medicine And Pharmacy, Cluj Napoca, Romania, [2]Budapest University of Technology and Economics, Budapest, Hungary, [3]Gedeon Richter, Tirgu Mures, Romania, [4]George Emil Palade University of Medicine, Pharmacy, Science, and Technology of Târgu Mureș, Tirgu Mures, Romania

To compensate for the multivariate nature of drug manufacturing, the applied quality management strategies have evolved over time. The highest level of quality management that is considered to revolutionize industrial manufacturing of medicine, relies on the use of soft sensors, that can enable the future of smart manufacturing in a Quality by Control (QbC) environment.

This work proposed the development of a multi-instrument PAT platform for the prediction of granule downstream processability in terms of tabletability, detachment (DS) and ejection stress (ES). Predicting these attributes, by fusing data derived from NIR spectra and particle size distribution (PSD) is expected to provide a better control of the tablet manufacturing process.

The complementarity between NIR spectra and particle size distribution (PSD) data was demonstrated through the identification of joint and unique sources of variation. Results revealed that 69.7% of variability in the NIR data was correlated with 50.5% of changes in PSD. Further, the decomposed sources of variability present in the NIR data were regressed against the factors varied during sample preparation. As the binder percentage and granulation temperature had an impact on granule growth, the latent variables correlated with PSD data were significant. Also, multiple unique sources of variation (orthogonal components) from the NIR dataset were correlated with the binder content, demonstrating the complementary nature of the data sets.

The use of Mid Level Data Fusion (MLDF) enabled the development of models with an improved predictive performance compared to individual methods (Fig.1). The optimal feature extraction strategy was dependent on the investigated response.

The good predictive ability of the models was demonstrated on external prediction sets. The calculated RMSEP values were further analysed to evaluate the model performance. The prediction errors for ES and DS were influenced positively by the applied compression load, suggesting that the prediction of these parameters is less accurate towards higher forces.

The results of this work demonstrate the advantages of fusing complementary sources of data for the control of complex quality attributes relevant for the tableting process.

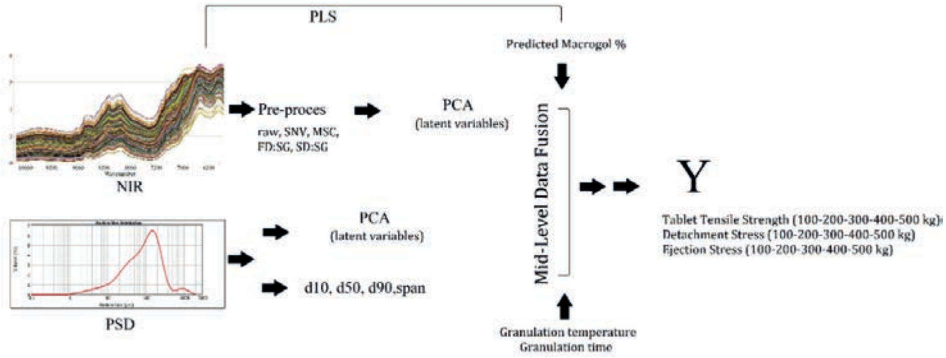


Figure 1. MLDF of NIR and PSD data for predicting granule tableting properties

Acknowledgements: This work was supported by a grant of the Ministry of Research, Innovation and Digitization, CNCS—UEFISCDI, project number PN-III-P1-1.1-PD-2021-0420, within PNCDI III.

P09.02

INTERPRETABLE AND INTERACTIVE TRANSFER LEARNING IN PROCESS ANALYTICAL TECHNOLOGY

Valeria Fonseca Diaz[1], Bianca Mikulasek[2], Christoph Herwig[2],
Ramin Nikzad-Langerodi[1]

[1]Software Competence Center Hagenberg, Hagenberg, Austria, [2]TU Vienna, Vienna, Austria

The project "Interpretable and Interactive Transfer Learning in Process Analytical Technology" (INTRAL), funded by the Austrian research promotion agency FFG, aims at exploring the potential of transfer learning (TL) and domain adaptation (DA) as technologies to increase the efficiency of calibration/process model development and maintenance for bio-pharmaceutical applications. The consortium consists of the technical university of Vienna (Bioprocess technology), the Software Competence Center Hagenberg (SCCH) GmbH (Chemometrics and Machine Learning), as well as Sandoz Austria and Takeda Austria.

This contribution is devoted to providing a high-level overview of the R&D activities that have been undertaken in the scope of INTRAL, including the milestones achieved so far, and the lessons learned from employing TL to solve real-world PAT challenges involving spectroscopic data. A particular emphasis will be placed on how to determine if a (calibration) model can be maintained to a new condition (e.g., for application under different media composition) and efficiently integrate all relevant data sources, including labelled and unlabeled data, into the model building process. In addition, we systematically explored for which types of dataset shift (unsupervised) DA is applicable, and the conditions under which model maintenance can succeed.

Our preliminary conclusion is that while TL still needs to represent the silver bullet to solve the model maintenance problem in PAT, it provides important new insights into the problem in general and a solution under certain conditions which we have started to understand.

The research reported in this contribution has been funded by BMK, BMDW, and the State of Upper Austria in the frame of the SCCH competence center INTEGRATE (FFG grant no. 892418) part of the FFG COMET Competence Centers for Excellent Technologies Program and the FFG project Interpretable and Interactive Transfer Learning in Process Analytical Technology (Grant No. 883856).

P09.03

PHARMACEUTICAL EVALUATION BY NEAR-INFRARED SPECTROSCOPY USING A HIGH-INTENSITY LIGHT SOURCE

Yasuto Fujimaki[1], Tomoaki Sakamoto[2,3]

[1]Tokyo Metropolitan Industrial Technology Research Institute, Tokyo, Japan, [2]National Institute of Health Science, Tokyo, Japan, [3]Research Institute of Electronics, Shizuoka University, Shizuoka, Japan

Near-Infrared spectroscopy is a spectroscopic method that observes the absorbance of near-Infrared light (NIR: 800 - 2,500 nm). It is known that overtones and combination tones of organic compounds are observed in this region. It has excellent permeability through objects and enables non-destructive analysis, because near-infrared light has low energy. Therefore, even a thick sample such as a pharmaceutical tablet can be measured inside without destruction. However, it is difficult to apply the detailed spectra analysis and quantitative analysis because the many signals appear overlapping in this region. In general, complex data analysis methods such as multivariate analysis are often used.
On the other hand, the short wavelength regions less than 1,200 nm are regions without overlap of the band, although it has very weak absorption. The peak separation and the interpretation of the spectra are relatively easily, and it is possible to extract a characteristic band. Therefore, it is considered that it will become possible that the simple quantitative analysis if signals in this region can be observed.
In this study, the applicability was examined to the quantitative analysis for the model tablet by the imitation manufacturing process as application to the practical quantitative analysis of the pharmaceutical ingredient.

The model tablets including levofloxacin (LVFX) (optical active material) and ofloxacin (OFLX) (racemic form) were prepared.
Concentrations of the active pharmaceutical ingredients (API) are three levels, 80%, 100% and 120%.
LVFX and OFXN are quinolone antimicrobials which are universally used.
NIR spectra were measured by NIR spectrometer that adopted high-density InGaAs photodiode array detector.

The CH second overtone of LVFX was observed to around 1,240nm, and that of OFLX was observed at the position that shifted to the about 20nm long wavelength side.
The quantitative capability of API of model tablets was investigated using the absorption of the CH second overtone.
As a result, it is considered that the quantitative analysis of API is possible using NIR region of the short wavelength if using high sensitive spectrometer and bright light-source.

This study was supported in part by a research grant from the Japan Agency for Medical Research and Development (AMED) under Grant Number 22mk0101202h302.

P09.04

PREDICTION OF DISSOLUTION BY NIR SPECTROSCOPY AND TRADITIONAL PROCESS CONTROL - A DATA FUSION APPROACH

Béla Kovács[1,4], Erzsébet-Orsolya Tőkés[4], Éva Katalin Kelemen[4], Katalin Zöldi[4], Brigitta Nagy[3], Zsombor Nagy[3], Tibor Casian[2]

[1] "George Emil Palade" University Of Medicine, Pharmacy, Science and Technology of Târgu Mureș, Târgu Mureș, Romania, [2] "Iuliu Hatieganu" University of Medicine and Pharmacy, Cluj-Napoca, Romania, [3] Budapest University of Technology and Economics, Budapest, Hungary, [4] Gedeon Richter Romania, Târgu Mureș, Romania

Introduction: The rise of green chemistry and in-silico process modelling in pharma R&D arises new, fast, and reliable methods for product QC, deemed for the continuous achievement of QTTPs. The present work aimed the modelling of dissolution of Clopidogrel from tablets based on traditional and chemometric analysis and fusion of data (DF) derived from the aforementioned QC methods.

Materials and methods: The manufacturing process implied the fluidized hot-melt granulation of the API, with Macrogol as plasticizer, according to an experimental design (DoE), varying three factors: quantity of plasticizer, time and temperature of granulation. Granules were lubricated for 2 and 15 minutes, and compressed at three different compression forces. Granules and tablets were characterized besides conventional IPC, also by recording the NIR spectra in reflectance (NIR-r) and transmittance (NIR-t) mode. API dissolution was recorded at 5-10-15-20-30-45 minutes. Prediction of API dissolution was carried out based on six different mathematical models using SIMCA17 software. PCA-X models were built for NIR-r, NIR-t and IPC results. A LLDF model was created for all NIR spectra. MLDF strategy was applied for (1) data extracted from NIR-r and NIR-t spectra, (2) data extracted from NIR-r and NIR-t spectra complemented with raw IPC results, (3) data extracted from NIR-r, NIR-t spectra and IPC results.

Results and discussion: The results of DoE revealed that the Macrogol content shows a negative correlation with the dissolution of the API in all time-points, whereas the granulation temperature shows an inverse proportionality up to 20 minutes, afterward positively correlating with dissolution data. Granulation time is only relevant at the latest time-points, reducing API dissolution. The best predictability of dissolution in terms of F2 assessment was observed in the case of NIR-r and MLDF1/MLDF3 models, with RMSEP values below 10% in all cases for all time-points.

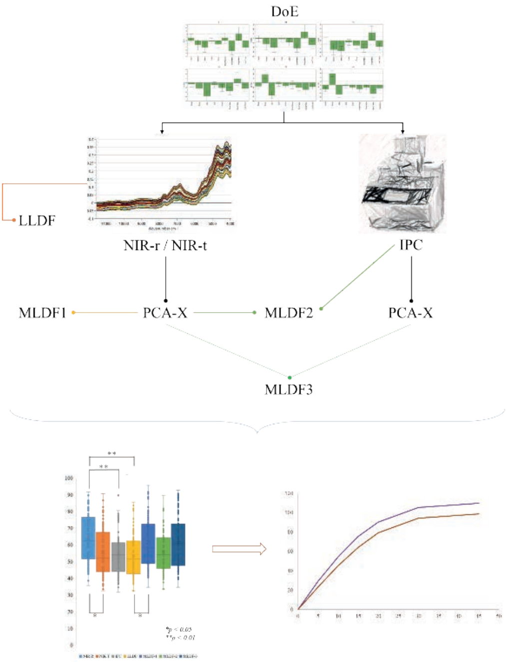


Figure 1. DF strategies used for dissolution prediction

Conclusion: This study evaluated the applicability of chemometric analysis and DF strategies in the manufacturing of tablets. The results indicate that NIR analysis is more advantageous compared to traditional analysis, and DF might complement these results for a more appropriate RTRT of APIs.

Funding: This work was supported by the University of Medicine, Pharmacy, Science and Technology „George Emil Palade“ of Târgu Mureș Research Grant number 510/8/17.01.2022

P09.05

IN-LINE MONITORING OF A BIOPHARMACEUTICAL FERMENTATION PROCESS

Félix-Antoine Lavoie[1], Lucas Vann[2], John Sheppard[2], Frederic Despagne[1]

[1]ABB, Québec, Canada, [2]North Carolina State University, Raleigh, United States of America

Quality and yield of virtually any manufactured product can be optimized through understanding and measuring the critical aspects of the process. This precious information turns into feedback for process control and triggers actions to maintain or adjust the process to the desired result. The chemical industry has been using sensors and measurement techniques such as near-infrared spectroscopy (NIR) for decades to operate and optimize process aiming at maximum quality, efficiency and cost reduction.
In recent years, biotechnology evolved to the point where bio-organisms are used to synthesize proteins, vaccines, and monoclonal antibodies on large-scale production. While the goals and quality interest will be similar, the biopharmaceutical reality is quite different from the chemical reality when it comes down to measuring sample properties. Biopharmaceutical processes deal with complex mixtures of water, nutrients, sugar, dissolved gases and suspended solids which represent a significant challenge for real-time analysis. Careful sampling techniques, spectral pre-processing and modelling are required to overcome these difficulties and result in reliable real-time measurements.
This presentation will illustrate the advantages of the combined use of in-line NIR spectroscopy and soft sensors to perform advanced process control on a 300L bioreactor for production of green fluorescent protein. The control platform was able to execute strategies that automated protein induction and decreased batch-to-batch variability so that "golden-batch" performance could be consistently achieved.

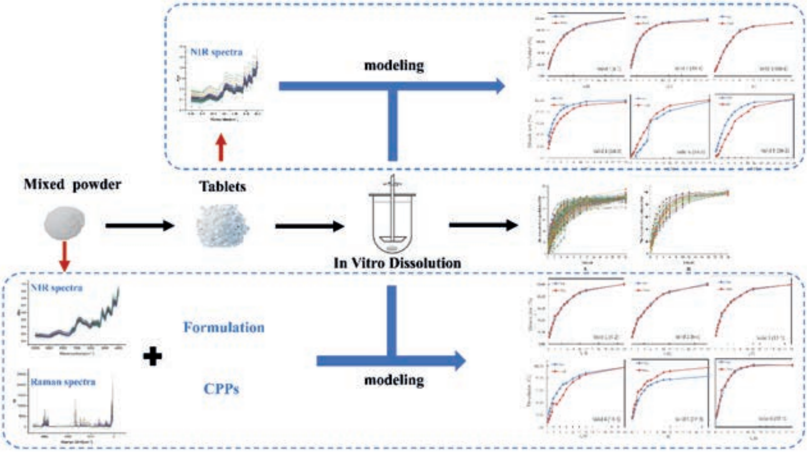
P09.06

STUDIES ON THE DISSOLUTION PROFILES PREDICTION OF SINOMENINE HYDROCHLORIDE SUSTAINED-RELEASE TABLETS USING NEAR-INFRARED SPECTROSCOPY

Long Wang[1], **Wenlong Li**[1]

[1] *Tianjin University of Traditional Chinese Medicine, Tianjin, China*

The objective of this study was to predict dissolution profiles of sinomenine hydrochloride sustained-release tablets using near-infrared (NIR) spectroscopy and multi-source data fusion technology. In this study, N=36 batches of representative sinomenine hydrochloride sustained-release tablets were prepared, using a D-optimal design, to introduce adequate dissolution variability, and the NIR spectral data of each tablets were acquired. The percentages of dissolution of each sample were correlated with the NIR spectra of five tablets of each batch, through the PLS regression algorithm and BP-ANN algorithm. The results showed that PLS regression algorithm could accurately predict dissolution profiles. To improve prediction performance, 180 samples of mixed powder before tablets pressing were scanned using a NIR spectrometer and a Raman spectrometer. The PLS models were trained using 3 predictor sets including (1) NIR spectra, formulation and CPPs, (2) Raman spectra, formulation and CPPs (3) a combination of both. It was found that the comparison among the predictive models revealed that the incorporation of Raman spectral information in calibration reduced prediction error. In summary, NIR spectroscopy was successfully applied to predict dissolution behavior of sustained-release tablets. By fusing the NIR with Raman spectra data, formulation and CPPs, the accuracy of dissolution model was improved.



P09.07

REAL-TIME MONITORING OF MULTISTEP SYNTHESIS PROCESS OF A KEY INTERMEDIATE OF LIFITEGRAST BY A COMBINED SPECTROMETER SYSTEM WITH BOTH NIR AND RAMAN

Quan Liu[1], Chuanjun Wu[2], Chuanmeng Zhao[2], Huiting Liu[3]

[1]Shanghai Jiaotong University, Shanghai, China, [2]Shanghai Institute of Pharmaceutical Industry, Shanghai, China, [3]Shanghai PROXS Chemical Technology Co. Ltd, Shanghai, China

Lifitegrast is an innovative product targeting the mechanism of ocular inflammation. As a novel small-molecule integrin antagonist, it can inhibit the interaction between lymphocyte function-associated antigen-1 (LFA-1) and intercellular adhesion molecule-1 (ICAM-1). Compared with other drugs for the treatment of dry eye, it has the advantages of faster effect, higher safety and less side effect.

3-(Methylsulfonyl)-L-phenylalanine phenylmethyl ester 4 is one of the key intermediates of Lifitegrast. Its synthetic process involves three reactions. It begins from Boc-protective Phe 1 with a coupling reaction catalyzed by CuI/L-Proline, where high temperature and insulation of air are required. The traditional method to monitor the process is to by offline sampling and HPLC analysis. Taking samples from reaction vessel under high temperature is dangerous and can damage gas tightness of the reactor. If the reaction time is too long, there will be impurities generated. Real-time analysis will help decide reaction end point and control the reaction time. In the reaction, Br was substituted with methyl sulfonyl group. Sulfur-oxygen double bond of methyl sulfonyl group has excellent Raman signal at 600-750 cm^{-1}. The calibration model by Raman spectra shows results of 0.972 of R^2 and 0.4% of RMSECV for product 2.

The following 2 steps are EDCI-catalyzed condensation reaction and deprotection reaction. These 2 reactions can be done in one reactor sequentially. Because one of the components methylsulfonyl-substituted Boc-Phe ester 3 has strong fluorescent effect, NIR showed better spectral quality than Raman for online calibration. In addition, NIR distinguishes well between ester group and hydroxyl group. The used NIR probe meets the process parameter of both reaction and don't need to be reset during the switch of the reactions. Calibration models were developed separately for the 2 reactions. The results of the calibration model are 0.999 of R^2 and 0.57% of RMSECV for product 3, 0.999 of R^2 and 1.12% of RMSECV for final product 4. The calibration models of the 2 reactions can be switched on the software by a defined model code, which provides easy operation for the production monitoring.

In multistep synthesis, real time analysis of the complex reaction mixtures is a significant challenge, but provides an opportunity to enhance reaction understanding and control. In this study, a combined multi-channel spectrometer system with both NIR and Raman was built and calibration models were developed to quantify the desired products, intermediates and impurities in real time at multiple points along the synthetic pathway. The capabilities of the system have been demonstrated by operating dynamic experiments and represents a significant step forward in data-driven multistep pharmaceutical ingredient synthesis.

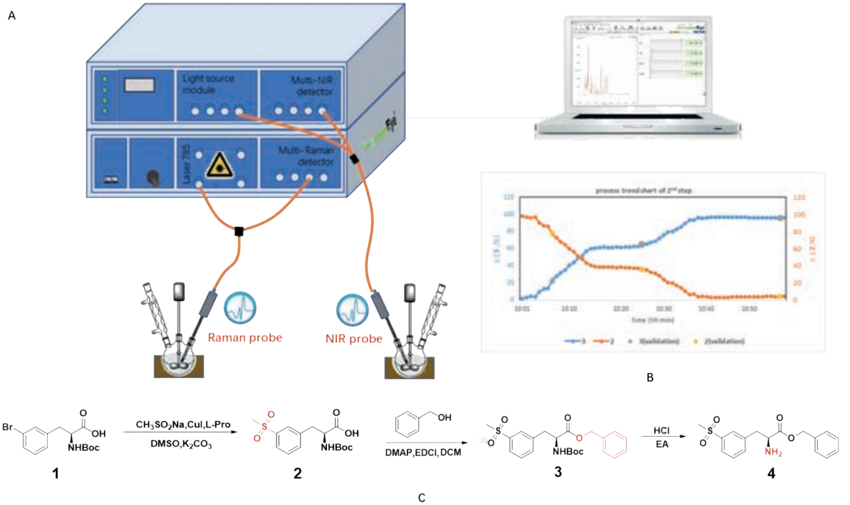


Figure 1. A, The combined spectrometer system setup for the multistep synthesis; B, Real-time monitoring data from second step reaction process; C, Synthesis route of 3-(Methylsulfonyl)-L-phenylalanine phenylmethyl ester.

P09.08

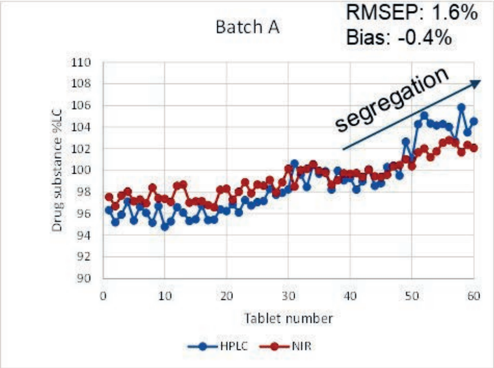
PAT CASE STUDIES FOR PHARMACEUTICAL PROCESS SCALE-UP AND OPTIMIZATION OF SOLID DOSAGE FORMS

Lizbeth Martinez[1], Matthieu Clavaud[1]

[1]Novartis, Basel, Switzerland

The implementation of Process Analytical Technology (PAT) tools in pharmaceutical manufacturing has shown great advantages and numerous applications from development to routine production; covering process understanding, scale-up support, troubleshooting, minimizing process variability, enhancing the control strategy, regulatory enabler in the context of ICH Q12 and integral part for real-time release of pharmaceutical products. These goals can only be achieved by clearly understanding the manufacturing process and by implementing the suitable technology for manufacturing and for process control. Each unit operation brings challenges that need to be assessed to prevent compromising the quality of the final product. NIR spectroscopy, as one of the major PAT tools, has attracted a lot of attention from the pharmaceutical industry since it can analyze bulk solids without previous treatment, therefore reducing or eliminating wet chemistry analysis. The motivation of this work is to present two cases, based on real PAT applications during pharmaceutical process development. These cases show the advantages of PAT implementation and their contribution to a robust process development and desired product quality.

One case considers the blending monitoring by means of a hybrid PLS model generated with data collected at different manufacture scales. Second case tackles the challenges of process development in an international environment, with different locations for manufacture and traditional analytics. Showing how PAT enables a lean evaluation of the product quality, better understanding and supports process transfer by in-line monitoring of the blending process, at-line content uniformity determination on a high number of tablets and by helping the troubleshooting of segregation. In conclusion, NIR proved to be a versatile and valuable tool for the pharmaceutical development and manufacturing of different unit operations.



P09.09

HONIGS REGRESSION METHOD TO TRANSFER SPECTRA FROM AN AT- LINE TO AN ON-LINE DIODE ARRAY INSTRUMENTS FOR PROCESS CONTROL AT AN OLIVE OIL PLANT

Cecilia Riccioli[1], Dolores Pérez-Marín[2], José A. Entrenas[2], Ana Garrido-Varo[2]

[1]Perkinelmer, Madrid, Spain, [2]Universidad de Córdoba, Córdoba, Spain

The ripe stage of the olive fruits and their quality are usually performed by manual sampling and off-line laboratory analysis, which is labour intensive, time consuming, and may suffer from sampling bias. As olive oil mills are becoming bigger, the chances for automation are increasing; for instance, current large mills have several differentiate processing lines fully automatedCompliance checks at the reception stage of olives is an essential step for obtaining high quality virgin olive oils, to meet quality standards and to pay producers accordingly. Near Infrared Spectroscopy (NIRS) could be an ideal technology to enhance efficiency and decision-making processes1.
This study explores the possibility of calibration transfer from a lab-based instrument to an online NIR sensor using the Honig Regression method2 to enable the rapid quantification of moisture and oil content in intact olives. NIR spectra of approximately 2000 samples of intact olives, with different geographical origins and collected during several harvest seasons, were recorded using different PerkinElmer Instruments DA7250 diode array devices placed at different sites (olive mills, laboratories) and models were built using the Honigs regression method (HR). Models were then transferred to an on-line system (DA 7440, PerkinElmer). Afterwards, it was evaluated whether the performance or accuracy of the process NIR instrument could be compared to the benchtop NIR instrument. This process was carried out through a fitting procedure, which compared the calibration parameters of the benchtop and process instrument, and the sample.
An amount of 20 samples were analyzed in one of the DA7250 instruments and in one DA 7440 and models were applied to the corresponding spectra. The fitting procedure revealed a high correlation (R2>0.70 for oil content and 0.90 for moisture) between the two instruments thus demonstrating that the HR method hold promise for the transfer of calibration models from at-line to on-line diode array instruments and for the quality control and for automation at the olive oil mill reception point for grading olives according to quality for further processing into separate lines.

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P09.10

REAL-TIME MEASUREMENT OF BIOMASS AND WASTE-BASED FUEL PROPERTIES AND PREDICTION OF FLUE GAS CONCENTRATIONS TO IMPROVE THE DYNAMIC OPERATION OF BOILER AND CCS UNIT

Jan Skvaril[1], Ali Ahmad Shahnawazi[1], Beibei Dong[1]

[1]Future Energy Center, Schools of Business, Society and Engineering, Mälardalen University, Västerås, Sweden

As society strives to expand the use of renewable energy sources to counter dwindling fossil fuels and reduce carbon dioxide emissions, biomass and waste provide a sustainable carbon-based alternative. Its physical and chemical diversity poses a challenge for energy use, especially since biomass conversion processes are sensitive to the properties of the feedstock, such as moisture content, heating value, and elemental composition. Moreover, the use of biomass and waste, especially in thermochemical energy conversion processes such as combustion combined with carbon capture and storage (CCS) unit, has significant potential for reducing or even reversing greenhouse gas emissions. The variable feedstock properties have a negative impact on the operation and control of these plants, creating inconsistencies in volume flow and gas concentration. Therefore, there is a high demand for a fast and effective method to measure the fuel properties and predict flue gas concentration and volume flow as combustion products.

The presented work aims to develop a soft sensor enabling real-time measurements of biomass and waste-based fuel properties, including moisture content, ash content, elemental composition, and prediction of flue gas concentrations. The work includes the acquisition of NIR spectral data and the reference property determination by standardized laboratory methods as well as flue gas concentration calculations. Different chemometric methods were used for spectral data pre-treatment and regression model training and validation to achieve the best results in prediction. The real-time soft sensor signal can be used to enable the introduction of appropriate operational measures to improve the combustion process's stability and to predict flue gas concentration and flow, which is essential for performance optimization and control of the CCS unit.

Acknowledgment: This work is supported by VEMM Group, including Mälarenergi AB, Eskilstuna Energi och Miljö AB, and VafabMiljö AB under project Renaissance - Real-time characterization of material flows for optimal operation of combined heat and power-plants, wastewater treatment plants and waste management facilities and sustainable use of resources within the circular economy.

P09.11

ONLINE MEASUREMENT OF THE REACTION DEGREE DURING POLYMERIZATION AND CURING PROCESS OF THERMOSETTING RESINS BY NIR SPECTRA AND MCR-ALS

Chunfeng Song[1], Long Hai Guo[1], Hong Fu Yuan[1], Xiaoyu Li[1]

[1]*Beijing University Of Chemical Technology, Beijing, China*

Thermosetting resins have been extensively applied in many fields owing to their excellent properties. During the polymerization and curing process of these resins, the molecular weight increases with the polymerization reaction, and then a three dimensional polymer is produced due to cross-linking. The measurement and control of the reaction degree are important to form appropriate molecular structure, qualified products, avoid gelation, and study reaction mechanism. Here, a method of near infrared spectroscopy combined multivariate curve resolution—alternating least square (MCR-ALS) are opposed to online measure the reaction degree of thermosetting polymer resins.

Phenolic resins and epoxy resins are the frequently used thermosetting resins. The Free formaldehyde is the key component for the reaction degree of synthesis process of phenolic resins using phenol and formaldehyde as reactants, and the primary and secondary amines are for the curing process of using amines as curing agents.

The NIR absorption of free formaldehyde in the phenolic resin polymerization system seriously overlapped with those of phenol, products and water. There is also a serious overlap between the characteristic bands of primary and secondary amines during curing process of epoxy resins. The noise and the light scattering drift due to the change of polymer structure during the polymerization were removed by the continuous wavelet transform (CWT) and an angle vector spectral transformation method. The main components of the system were included by exploratory factor analysis (EFA). The spectra of free formaldehyde, primary and secondary amines, and their concentrations were obtained by applying MCR-ALS to eliminate the spectral overlap. Then, the the reaction rates were calculated. The conversion results of free formaldehyde calculated by MCR-ALS are consistent with those obtained by the traditional titration method. This result shows that NIR spectroscopy combined with MCR-ALS is an effective method for on-line detection of reaction degree during the synthesis process of phenolic resin. The reaction rates of primary and secondary amines were used to study their reaction activity and reveal the kinetic of epoxy curing reaction.

P09.12

DEVELOPMENT OF A NIR BASED PAT TOOL FOR QUANTIFICATION OF IBUPROFEN ACID-SALT TRANSFORMATION DURING A WET GRANULATION PROCESS

Elizabeta Atanaskova[1,3], Veronika Angelovska[1], Natasha Anevska Stojanovska[1], Jelena Lazova[1], Gjorgji Petrushevski[2], Nikola Geskovski[3]

[1]Research and development, Alkaloid AD, Skopje, Republic of North Macedonia, [2]Quality control, Alkaloid AD, Skopje, Republic of North Macedonia, [3]Ss. Cyril and Methodius University in Skopje, Faculty of Pharmacy, Institute of Pharmaceutical Technology, Skopje, Republic of North Macedonia

Ibuprofen is one of the most widely used non-steroidal anti-inflammatory drug for treatment of mild to moderate pain. As a BCS class 2 drug is likely to be dissolution rate limited. A large number of studies indicate an improved bioavailability when it is applied in the form of a salt such as Ibuprofen sodium, due to the improved solubility of the salt form. The acid-salt transformation may occur during the wet granulation of Ibuprofen with a sodium-containing base. Thus, monitoring and the possibility of quantification of the transformation of Ibuprofen into Ibuprofen sodium is of particular importance.

The purpose of this research was to develop a PAT tool for monitoring the transformation process of the active ingredient ibuprofen into fast-dissolving salt ibuprofen sodium during the wet granulation process. A set of 35 standard mixtures containing active ingredients ibuprofen and ibuprofen sodium in a concentration (w/w) range from 0% to 100% were prepared and a calibration model using PLS regression was developed and compared for both benchtop (model 1) and portable (model 2) NIR spectrometers. Both models yielded high correlation coefficients and adequate predictive ability (R2X=0.894, R2Y=0.992, Q2=0.99, and R2X=0.874 R2Y=0.989, Q2=0.989 for models 1 and 2, respectively). However, the model based on the portable NIR spectroscopic data was chosen for a further application allowing PAT monitoring of different steps during the granulation process. Model 2 was further upgraded with introduction of the spectral data obtained during the analysis of the standard mixtures after the addition of different amounts of water (R2X=0.993, R2Y=0.978, Q2=0.973).

The assessment of the model's predictive ability involved analyzing laboratory trial batches produced using different amounts of disodium carbonate, which resulted in variations in the concentration of ibuprofen sodium at the end of the process. Finally, tablets were produced from each laboratory trial batch, and dissolution analysis was performed. The results from the dissolution analysis were in a good agreement with the NIR predicted concentrations of ibuprofen sodium (the trials with larger concentration of ibuprofen sodium presented higher ibuprofen dissolution rates). Based on the results, the proposed model provides an excellent tool to monitor the Ibuprofen acid-salt transformation process, determine the end-point of the reaction, and efficiently control the wet granulation process.

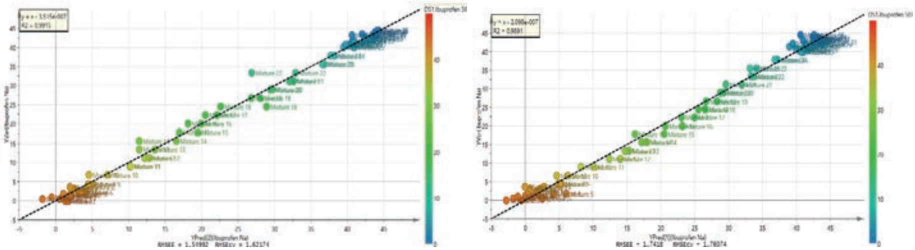


Figure 1. Observed vs. Predicted plot

a) Model 1 – Benchtop NIR

b) Model 2 - Portable NIR model

P09.13

NIR SPECTROSCOPY AS A TOOL FOR ESTIMATION OF MOISTURE CONTENT / WATER CONTENT / WATER ACTIVITY IN CANNABIS FLOWERS

Irena Slaveska Spirevska[1], Maksim Osman Nikolov[1], **Olga Gigopulu**[2], Gjoshe Stefkov[2], Nikola Geskovski[2], Petre Makreski[3], Jelena Acevska[2]

[1]REPLEK FARM Ltd, Quality Control Department, Skopje, Macedonia, [2]Ss. Cyril and Methodius University in Skopje, Faculty of Pharmacy, Skopje, Macedonia, [3]Ss. Cyril and Methodius University in Skopje, Faculty of Natural Sciences and Mathematics, Institute of Chemistry, Skopje, Macedonia

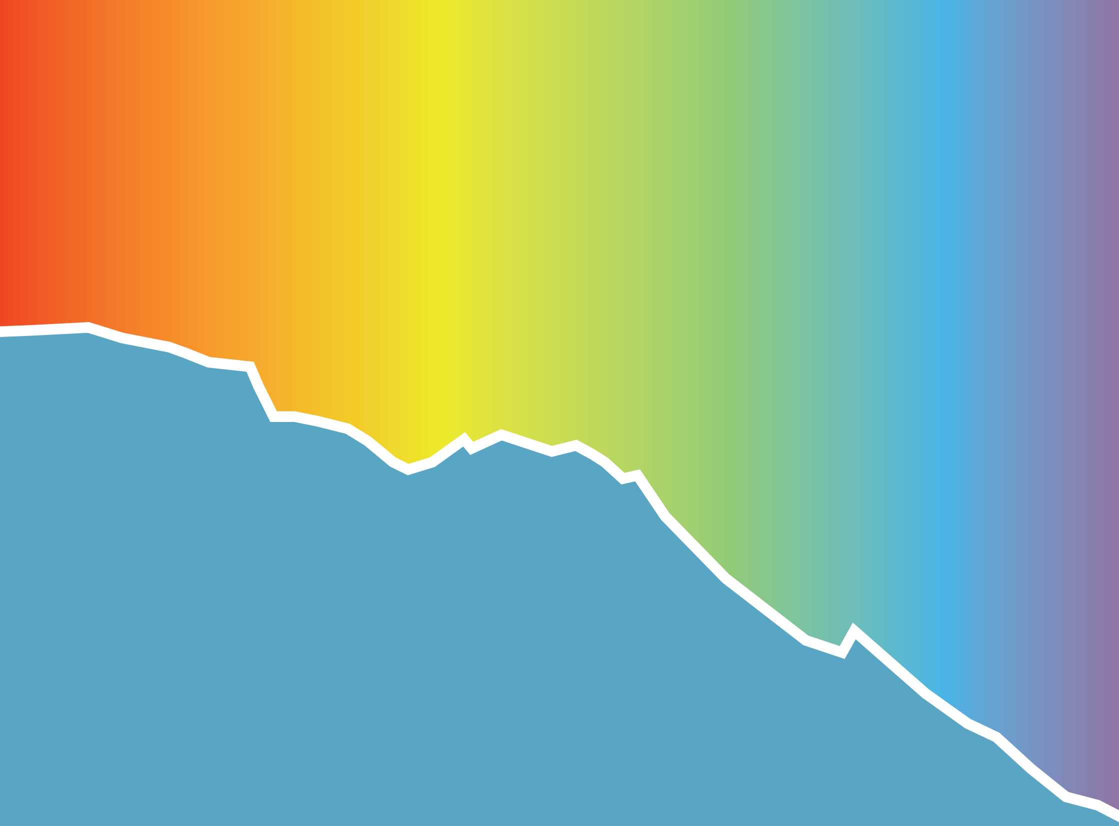
Total water is an important quality attribute and consists of both unbound ("free") and unavailable ("bound") water. The quality and stability of the herbal substances and preparations are affected by the available water that enables microbial growth, moisture migration, chemical and physical reactions. Therefore, it is crucial to understand which specification parameter(s) related to water content / moisture content / water activity is critical to the quality, stability and safety of Cannabis flower, since it affects the actual potency of cannabinoids, susceptibility to contamination and product consistency.

There are several methods for determination of water/moisture content in Cannabis flowers: loss on vacuum drying (DAB method), determination of water content by distillation of the sample dispersed in toluene (Ph.Eur. 2.2.13, the alternative pharmacopeial method recommended for dried herbal drugs with a high content of volatile compounds), Karl Fisher titration (Ph.Eur. 2.5.12, proposed in the specifications as a selective method that excludes the possibility of misinterpreting other volatile compounds, such as terpenes and some cannabinoids, as moisture). Despite the disadvantages, in terms of durability and large sample amounts, the main problem with these methods is that the information on the water content or moisture content may not be directly related to the quality and stability of the herbal preparations. The water activity (Aw), as a measure of the energy status of water in the system (calculated as the ratio of the vapor pressure of the sample and the vapor pressure of pure water at the same temperature, USP<912>), indicates the risk of physical-chemical and microbiological deterioration.

The aim of this research was to employ the NIR spectroscopy as an environmentally friendly, non-destructive and fast tool to estimate water activity, simultaneously with water content in Cannabis flowers.

Based on NIR spectra of over 40 samples of powdered Cannabis flowers of different cultivars and a comparative analysis of the results of conventional methods for determination of water content in the same samples of cannabis flowers, a multivariate partial least square (PLS) model was developed for predicting water activity.

The model exhibited a reasonable correlation coefficient and root mean square error of estimation, proving that the proposed methodology can be applied for in-process control of cannabis herbal substance.



THEME 10

WATER, SOIL AND ENVIRONMENT

ORAL PRESENTATION: O10.01 – O10.05
POSTER PRESENTATION: P10.01 – P10.10

ORAL PRESENTATION

010.01

METABOLOMIC ANALYSIS OF THE GLOBAL MOLECULAR FINGERPRINT AND AQUAPHOTOM-IC ANALYSIS OF THE DEHYDRATION-REHYDRATION CYCLE OF THE SYMBIOTIC AEROTERRES-TRIAL MICROALGA ASTEROCHLORIS ERICI

Rosa María de las Heras[1], Enrique Pérez[1], Eva Barreno[2], **Myriam Catala**[1]

[1]Rey Juan Carlos University, Móstoles (Madrid), Spain, [2]Valencia University / ICBIBE, Burjassot (Valencia), Spain

Aeroterrestrial microalgae can survive in a state of dehydration (anhydrobiosis), with very low levels of metabolic activity. Very little progress has been made in understanding their molecular mechanisms of tolerance to dehydration. Near-infrared spectroscopy allows the analysis of the metabolomic profile of cells, providing a unique molecular fingerprint that allows the association of metabolomic changes to stress situations. Recently, together with aquaphotomics, it has allowed in-depth analysis of molecular and water structure differences between common and “resurrection” plants. However, the molecular structure of water during dehydration of aeroterrestrial algae is unknown. To assess these changes, spectra of cultured Asterochloris erici, a green microalga isolated from lichens, were obtained throughout the dehydration process and after rehydration. When analyzing the water loss, a relative water content of 16% is reached after 180 min under silica gel atmosphere, remaining stable up to 48 h. As dehydration progresses, free water molecules decrease, and hydrogen bonded molecules increase. The sharp decrease of free water at 100 min and the increase of molecules with one and four hydrogen bonds seems to indicate the preparation for anhydrobiosis in agreement with a previous “resurrection” plant study. The spectra of microalgae dehydrated for 24 h show increased absorbance of polyols, saturated lipids and proteins, which are important for maintaining the stability of cell structures. On the other hand, the spectra of microalgae rehydrated for 24 h show few differences with respect to fresh algae, while at 48 h the spectra appear indistinguishable. Modulation of polyols, proteins, lipids and the molecular structure of water seem critical in anhydrobiosis.

Keywords: Anhydrobiosis, NIRS, phycobiont, metabolome, poikilohydry.

Acknowledgements: Thanks to Ms. Patricia Alonso and Nutrilab (RedLabu URJC), for technical support and use of the Perkin Elmer FT-NIR spectrophotometer of the 100N series. This research was funded by the Generalitat Valenciana (PROMETEO/2021/005), Spain.

010.02

RECENT DEVELOPMENTS IN AQUAPHOTOMICS: INSIGHTS INTO WATER STRUCTURE AND FUNCTIONALITY

Jelena Muncan[1], Roumiana Tsenkova[1]

[1]Kobe University, Graduate School of Agriculture, Aquaphotomics Research Department, Kobe, Japan

Aquaphotomics, a new "omics" science, was established in 2005 (Tsenkova 2009). After seeing that water absorbs light at various frequencies, the aim of aquaphotomics was to define these frequencies and find a relation between the spectral pattern called aquagram, based on these frequencies and the respective water characteristics including its functionality. While unravelling the water - light interaction in living and aqueous systems at various frequencies the ultimate goal of complete understanding of water as a life matrix was foreseen (Tsenkova 2009). The first systematizations of aquaphotomics knowledge resulted in the publication of 12 identified regions named WAMACS - Water Matrix Coordinates. These absorbance bands correspond to particular water molecular species found repeatedly and consistently in diverse bio-aqueous systems and related to certain functionalities.

Recent developments in aquaphotomics have been focused on more in-depth investigations of how certain water structures are connected to the properties and functionality of the examined systems. One of the first publications where this is presented in a systematic manner was about water activity and rice germ storage monitoring (Malegori et al. 2022). This study was the first to uncover the water absorbance bands of water vapour (1364 nm, 1375 nm and 1382 nm) in connection with high water activity. Further, high-water activity was also found to be associated with the strongly bound water (1518 nm), reflecting the state of the food matrix in terms of porosity and space available for highly mobile water vapor species to move. This study, also for the first time published how certain water species are connected with textural, mechanical, and biological properties. The studies that followed had different research topics but the same line of investigation was followed, aimed at uncovering how the water molecular structure of the samples is related to their function. Despite very different research topics such as cement mixing and drying (Muncan et al. 2022b), storage of strawberries under influence of electric field (Muncan et al. 2022a)SCF, waters perturbed by the sound of different frequencies (Stoilov et al. 2022)water is influenced by all internal and external influences, changing its molecular structure accordingly. The water molecular structure and its changes can be observed as a whole by measuring its electromagnetic (EMG and others, common water absorbance bands were recognized and related to the common functionalities in those systems. These studies contributed to a more complete picture of water as a poly-phasic system encompassing vapour, liquid, amorphous, liquid-crystalline, semi-crystalline and crystalline phases. It is also now better understood that there are at least 19 WAMACs in the first overtone of water, and within the existing WAMACs there is a difference between the water molecular species (Figure 1).

The ongoing development of aquaphotomics offers great potential for advancing our understanding of life and water, and could have implications for various fields such as food science, materials science, and medicine.

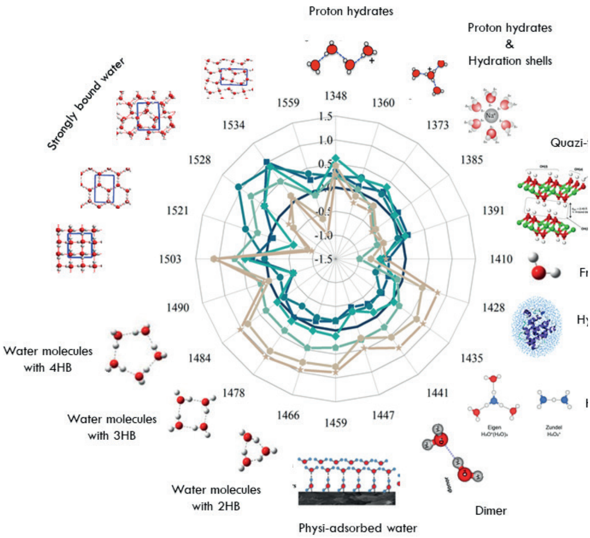


Figure 1. The example of aquagram showing water spectral pattern defined by the specific water absorbance bands (WAMACs). In the 1st overtone of water, it is currently recognized that there are actually 19 WAMACS, each corresponding to specific water molecular structure having different functionality.

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010.03

FUSION OF ACTIVE HYPERSPECTRAL IMAGING AND RAMAN SPECTROSCOPY FOR CLASSIFICATION OF PLASTICS WITH BROMINATED FLAME RETARDANTS

Tuomas Sormunen[1], Ilkka Rytöluoto[2], Anna Tenhunen-Lunkka[3],
Francisco Senna Vieira[3]

[1]VTT Technical Research Centre Of Finland, Oulu, Finland, [2]VTT Technical Research Centre Of Finland, Tampere, Finland, [3]VTT Technical Research Centre Of Finland, Espoo, Finland

Whilst legislation in the EU emphasizes greater recycling rates for all waste categories, the plastic in waste electrical and electronic equipment (WEEE) is problematic due to the presence of hazardous substances such as brominated flame retardants (BFRs). To recycle this fraction, BFR-free and BFR-rich plastics must be segregated. X-ray fluorescence spectroscopy (XRF) is often used to quantify bromine content in plastics. However, it uses ionizing radiation and is limited to elemental analysis. In previous studies, near infrared (NIR) and Raman spectroscopy have been applied to this task, but lacked a robust classification scheme and a large sample set.

In this study, we go beyond the state-of-the-art by utilizing a rigorous machine learning scheme for 235 samples, including 210 plastic pieces from actual WEEE stream. Fig. 1 shows the overview of our sensor fusion approach. The aim was to classify these samples to high- or low-bromine (Br) content fractions according to a Br concentration segmentation threshold. We collected spectral data with a commercial Raman spectrometer (TimeGate PicoRaman) and an active hyperspectral sensor (AHS) prototype developed at VTT operating in the NIR range (1950-2500 nm). We developed classification models using both Raman and AHS datasets individually, and by data fusion. The pre-processing steps for each dataset are depicted in Fig. 1. We conducted two data fusion approaches, both of which used datasets with reduced dimensionality generated using principal component analysis (PCA). In one approach, the PCA was applied to the spectral data of both instruments separately before concatenating the datasets (SPCA), and in the second approach after concatenating the datasets (JPCA). We used a robust nested 5-fold cross-validation scheme for finding the optimal number of PCs, segmentation threshold, and the best performing classifier out of 33. Reference Br concentrations were determined by XRF (Niton XL3t 900S GOLDD).

The best performing classifier over all datasets was Extremely Randomized Trees. The mean balanced accuracy of AHS, Raman, JPCA and SPCA fusion approaches were 81.6%, 82.0%, 82.9% and 85.2%, respectively. Our results show that either of the data fusion approaches yield superior results compared to each of the individual spectroscopies. The segmentation threshold in these cases was 7 wt-%.

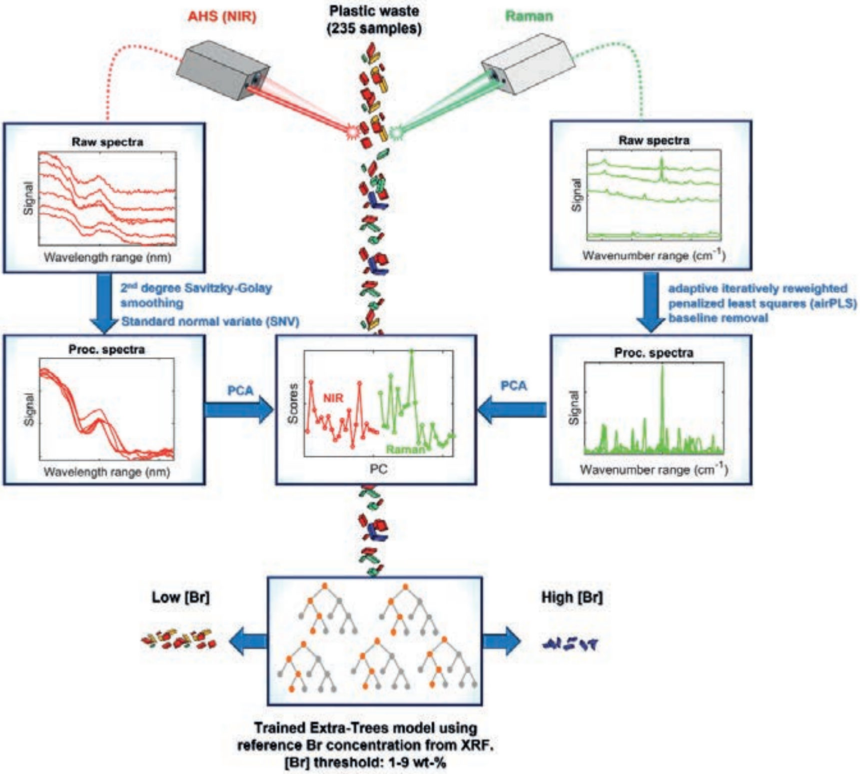

Figure 1. Overall flowchart of the sensor fusion approach for sorting plastic waste according to the Br content.

010.04

WATER AND ITS MOLECULAR CONFORMATIONS AS QUALIFIERS OF AQUEOUS SYSTEMS

Aleksandar Stoilov[1]

[1]Yunosato Aquaphotomics Lab, Hashimoto, Japan

Aquaphotomics is a novel discipline that was introduced by Prof. Roumiana Tsenkova at the Laboratory of Bio Measurement Technology at Kobe University, Japan, with a focus on understanding the role of water in biological and aqueous systems by monitoring the water spectrum at various perturbations.

Near-infrared spectroscopic (NIRS) studies were conducted on various soil types at different moisture content levels at Yunosato Aquaphotomics Lab (Wakayama, Japan), and their spectral data investigated by aquaphotomics methods. Soil, as an active biological porous medium, is rich in water molecules, which reflect the presence of all organic and mineral components while constantly rearranging corresponding to the environment. Acquiring particle density parameters gave the opportunity to build accurate models on particle size and organic matter content. Clay, silty and sandy soils were distinguishable through their water spectral patterns in the first overtone of the water molecule at 1300-1600nm.

Unique water spectral patterns were shown for agricultural, garden and forest soil types, defined by water molecular species with high light absorbance variations. The more uniform human-processed soils were characterized with the light absorption at water bands related to water solvation shells, trapped and free water molecules. Forest soil, having a higher amount of organic matter, was seen with more hydrogen bonding between water molecules due to hydrophobic interactions. After rewetting all soil samples, a strong correlation was discovered between soil and water, where linear regression models on moisture content were characterized with water bands related to trapped and free water molecules, hydronium ions and strongly-bounded water molecules. Moisture content levels were discovered to be characteristic for each respective type of soil and its properties.

In order to better understand if and how soil and plant communicate between each other through water, NIRS monitoring was conducted over the span of several months. Specific water molecular conformations and water ions were seen to play a huge role in the growth of carrots, strawberries and onions, while trapped and free water molecules were seen to interchange complementary between the two aqueous systems. It was demonstrated that aquaphotomics can be applied as a holistic approach for investigating soil, its crops and their properties.

010.05

DEVELOPING NIR-AQUAPHOTOMICS FOR RAPID SCREENING OF RESPIRATORY DISEASE IN LIVESTOCK

Carrie Vance[1], Marianna Santos-Rivera[1], Li-Dunn Chen[1], Amelia Woolums[1], Merrilee Thoresen[1], Florencia Meyer[1]

[1]Mississippi State University, Mississippi State, United States

Bovine Respiratory Disease complex (BRDC) is a multi-factorial, multi-pathogen condition that causes billion-dollar losses to the US cattle industry. We aim to develop and validate NIR spectrometry in combination with Aquaphotomics as a diagnostic tool for BRDC by analyzing NIR spectra of biofluids from cattle challenged with acute viral (BRSV; bovine respiratory syncytial virus) or bacterial (MH; Mannheimia haemolytica) BRD infection. Dairy calves (minimum 5/trt) were challenged with MH or BRSV, and nasal secretions (NS), breath condensate (BC), saliva (SA) and blood (serum (SE) and plasma (PL)) were collected daily for a minimum of 2-weeks and stored at -80oC (IACUC 19-037). Using an ADS Fieldspec®3 + Indico®Pro (Malvern Panalytical) and 1.0mm matched quartz cuvettes, transmittance NIR spectra were obtained from each thawed biofluid held at 22oC. Following the application of mathematical pretreatments (SNV, detrending, Savitsky-Golay derivative and smoothing), distinct biochemical patterns were observed between the spectra associated with biofluids collected from calves infected with BRSV or MH compared to non-infected animals. Aquaphotomics assesses the molecular organization of the aqueous phase (1300-1600nm) and is a potential biomarker for BRSV or MH infection. For the WAMACS barcode, Water Absorbance Bands and aquagrams, absorbance was normalized by subtracting the mean transformed spectrum of millipore purified water and divided by the SD. Subsequently, conventional machine learning algorithms (PLS, PCA-LDA, GLM, Support Vector Machine, Random Forest, KNN, eXtreme Gradient Boosting) with 5-fold cross validation were applied to calibrate prediction models and evaluate the predictive efficacies between control, MH and BRSV infections. Balanced data sets were generated by stratified random sampling and analyzed in a leave-one-animal out approach for external validations. WAMACS barcodes reveal strong shifts in the C3 and C11 WABS in blood SE and PL from BRSV and MH treated animals, while the largest shifts in the BC, NS and SA spectra were observed in the WAMACS C1, C4 and C5 compared to non-infected control. Unique shifts in the C8 WABS were observed for samples collected from MH infected animals, but not BRSV infected animals. Prediction models for each biofluid using PCA-LDA to compare accuracy, sensitivity and specificity for BC (78.5%, 70%, 85%) and NS (71%, 70%, 72%) collected from animals infected with MH, and BC (78.5%, 70%, 85%) and NS(78.5%, 70%, 85%) collected from animals infected with BRSV when directly compared to non-infected control. Analysis with the multimodel approach yielded a direct comparison of all three treatments as high as 75%, 92% and 74% accuracy for control, BRSV and MH treated calves, respectively. This work highlights NIRS as a practical diagnostic tool for assessing BRDC infection in cattle..

P10.01

DEEP LEARNING FOR SOIL CARBONATES CONTENT PREDICTION FROM NEAR-INFRARED SPECTRAL DATA

Lykourgos Chiniadis[1], Petros Tamvakis[1], Nikolaos Kazakis[1], Nestor Tsirliganis[1], Chairi Kiourt[1]

[1]Athena - Research and Innovation Centre In Information, Communication And Knowledge Technologies, Xanthi, Greece

We propose a rapid and efficient way to predict carbonates content in soil by means of Fourier Transform Near-Infrared (FT-NIR) reflectance spectroscopy [1,2] and by deep learning methods. We train: 1) a Multilayered Perceptron (MLP) and 2) a Convolutional Neural Network (CNN) on the combined dataset of two near-Infrared (NIR) spectral libraries: Kellogg Soil Survey (USDA), a dataset of soil samples reflectance spectra collected nationwide, and LUCAS TopSoil (European Soil Library) which contains soil sample absorbance spectra from all over the European Union, and use them to predict carbonates content on never-before-seen soil samples.
MLP regression method achieves excellent carbonates content prediction accuracy (R^2 = 0.84, Residual Prediction Deviation (RPD) = 2.14) when tested on NIR absorbance spectra of 19 soil samples. Respectively, CNN model trained not on spectral absorbances related to each wavelength but on their respective spectrograms, scores R^2 = 0.68 and RPD = 1.47.
Our work contributes to rapid carbonates content prediction in soil samples in cases where: 1) no volumetric method is available to the researcher and 2) only vis-NIR spectral absorbance data are available.

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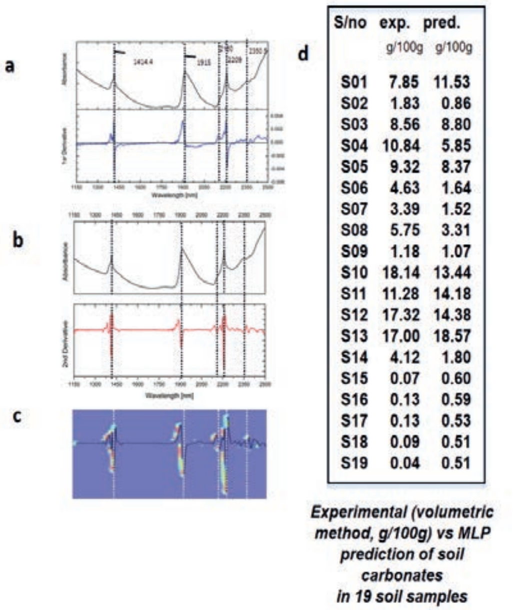


S/no	exp. g/100g	pred. g/100g
S01	7.85	11.53
S02	1.83	0.86
S03	8.56	8.80
S04	10.84	5.85
S05	9.32	8.37
S06	4.63	1.64
S07	3.39	1.52
S08	5.75	3.31
S09	1.18	1.07
S10	18.14	13.44
S11	11.28	14.18
S12	17.32	14.38
S13	17.00	18.57
S14	4.12	1.80
S15	0.07	0.60
S16	0.13	0.59
S17	0.13	0.53
S18	0.09	0.51
S19	0.04	0.51

Experimental (volumetric method, g/100g) vs MLP prediction of soil carbonates in 19 soil samples

Figure 1. (a,b) NIR absorbance spectra aligned to the first derivative of NIR spectrum and to the second derivative of NIR spectrum of S01. (c) Saliency map derived from CNN. (d) Experimental vs prediction values of soil carbonates content in 19 soil samples.

P10.02

NEAR-INFRARED SPECTROSCOPY FOR ESTIMATION OF MICROPLASTIC IN SOILS - SPIKING EXPERIMENT

Maria Knadel[1]

[1]Aarhus University, Tjele, Denmark

Soil microplastic (MP) pollution is a global concern. It has been shown to affect physical, chemical, and biological processes in soils posing risks to food safety, human health, and the environment. However, there is a lack of standardized analytical methods for isolation, detection, and quantification.

Here, a fast way of estimating the amount and type of MP present in soil samples is suggested based on near infrared spectroscopy (NIRS) in conjunction with multivariate data analysis, and without the common step of MP extraction.

Eight Danish soils differing in soil texture and amount of soil organic carbon (SOC), were spiked with four common polymers (polypropylene; PP, polyamide; PA; polyethylene; PE, polyethylene terephthalate; PET) at eleven levels. The MPs with no additives were prepared in the laboratory by cryogen grinding in the presence of liquid nitrogen, followed by sieving to 1mm. The MPs were mixed into the air-dried and 2 mm sieved soil samples. NIRS measurements using a bench top sensor covering 400-2500 nm were conducted. The measurements were done in triplicates directly on the soil samples spiked with MPs. A total of 1080 spectra was generated. In order to estimate the MP levels partial least squares regression (PLSR) was performed for each of the polymer type individually. Models' performance was compared and estimated using root mean square error of cross validation (RMSECV) and R^2 values.

The obtained RMSECV and R^2 values for PET, PE, and PP were very comparable (~2.5g/kg and 0.84, respectively). Whereas the estimation of PA was less accurate (RMSE=3.2g/kg, $R^2 = 0.75$). Regression coefficients indicated that the important wavelengths explaining variability in the MPs were associated mainly with the first and second overtone region and stretching of carbo-hydrates molecular bonds, as well as the combination band region originating from stretching and bending vibrations of CH bonds.

Despite, generally good correlation between the spectral and MP reference data, the obtained errors indicate possible challenges for soils with relatively low MPs concentration. As the investigated soils were highly variable in terms of soil texture (clay content: 4- 31%) and the range of soil organic matter (SOC content: 1-10%) further studies with MP models dedicated to a certain type of soil should be tested in search for accuracy improvements.

P10.03

UNDERSTANDING THE PROCESS OF COLD ATMOSPHERIC PLASMA TO WATER WITH AQUAPHOTOMICS

Junsha Luo[1], Wenshuo Ding[1], Xiaoying Wei[1], Xiaolong Wang[2], Hengchang Zang[1], **Lian Li**[1]

[1]School of Pharmaceutical Sciences, Cheeloo College of Medicine, Shandong University, Jinan, China, [2]School of Electrical Engineering, Shandong University, Jinan, China

Introduction

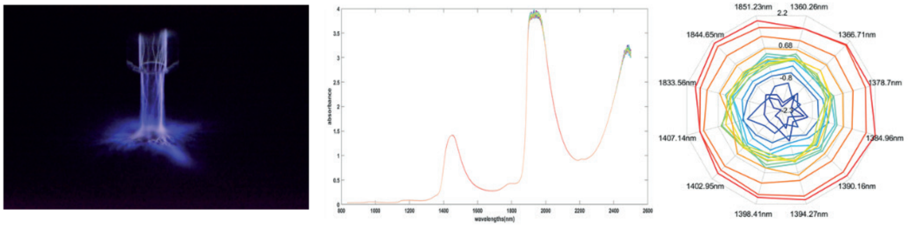
Cold atmospheric plasma (CAP) is a kind of ionized gas which could induce the formation of reactive species in solution such as reactive oxygen and reactive nitrogen species. With the help of CAP, plasma-activated-water (PAW) shows good potential in the clinical application. Therefore, it is of great important to understand the process of CAP to water. Aquaphotomics could provide a powerful tool to investigate minor changes in the water system to elucidate mechanism caused by CAP. Therefore, in this study near infrared spectroscopy combined with aquaphotomics was introduced to visualized the PAW process.

Materials and methods

A self-made jet devise (Fig. 1) consisting of a dielectric layer covered high-voltage electrode and copper coil ground electrode was used to produce PAW at different time intervals (5, 10 and 15 min). Near infrared spectra were collected with 1 mm cuvette at 25 oC. And the sample treated with 15 min was selected to collect the NIR spectra in 8 h with 30 min intervals. Different data treatment methods such as derivatives, PCA and PLS were used to find specific water matrix coordinates (WAMACS).

Results and discussion

The results showed that the longer the treatment time, the greater the spectral change, which is directly related to the increase of reactive species with the increase of treatment time. SNV, Savitzky-Golay second derivative (setting the smoothing window length to 13 and using quadratic polynomial) were used to improve the significance of spectral change. The specific WAMACS are found by using PLS and PCA and most of these wavelengths fall within the wavelength range of weak hydrogen bond vibrations (1300nm-1400nm), as shown in Fig. 1. It can be seen that the general trend of spectral change is increasing, but there are fluctuations in the intermediate time period (2.5h-6h). According to previous studies, it is speculated that this is because the reactive species in PAW are unstable and will react with each other. With the increase of time, reactive oxygen species will continue to react to form H_2O_2. Generally, aquaphotomics can indeed reflect the formation of reactive species in PAW and the change of reactive species with time, and it is a powerful tool to study the interaction process between CAP and water.



CAP jet devise

PAW NIR spectral

Aquagram of PAW

Figure 1. Results of CAP processed water

P10.04

SPECTRAL LIBRARY OF AUSTRIAN SOIL SAMPLES AND ITS APPLICATION IN THE PROBEFIELD PROJECT

Maximilian Lippl[1], Taru Sandén[1], Elisabeth Reiter[1]

[1]AGES - Austrian Agency for Health and Food Safety, Vienna, Austria

Faster and high-throughput soil characterisation methods are needed to meet soil policy requirements, such as assessing changes in soil condition, organic carbon and erosion rates under agricultural management, or assessing soil nutrient status in the context of farm-to-fork objectives.

Soil spectroscopy offers promising opportunities to speed up soil surveying and reduce costs.

The spectral library of Austrian soil samples consists currently of around 750 agricultural soil samples. The soil spectral library has been built up from representative agricultural air-dried soil samples from farmers and agricultural long-term experiments that have first been analysed with wet and dry chemistry for soil organic matter characteristics including total organic carbon (TOC), total nitrogen and among other soil fertility characteristics. The soil spectral library is continuously being extended by more representative agricultural soil samples from farmers and long-term experiments. This is to ensure generic, robust and well performing models that could be used in a simple and fast manner on local, regional and national scales in Austria, as well as to be connected to larger geographical and soil type coverage on a European and global scales through ProbeField and GLOSOLAN networks.

In addition to the development of the spectral database with a lab instrument the project ProbeField focuses on the use of proximal sensing for soil monitoring in the field and aims to validate proximal sensing techniques for soil property estimation.

P10.05

ANAEROBIC FUNGI IDENTIFICATION BY NIR SPECTROSCOPY

Markus Neurauter[1], Christoph Kappacher[1], Tobias Schlappack[1], Jovan Badzoka[1], Julia Vinzelj[2], Sophia Strobl[2], Matthias Rainer[1], Sabine Podmirseg[2], Christian W. Huck[1]

[1]Institut für Analytische Chemie und Radiochemie, Universität Innsbruck, Innsbruck, Austria, [2]Institut für Mikrobiologie, Universität Innsbruck, Innsbruck, Austria

Neocallimastigomycota are a phylum of anaerobic fungi (AF) that inhabit the gastro-intestinal tracts of herbivores. Their ability to break down lignocellulosic biomass by enzymatic and mechanical means in anaerobic conditions, makes them of interest for the optimization of biofuel and biogas production from organic waste. AF possess high inter- and intra-species length variability in 18S rRNA and ITS1 rRNA standard marker gene regions, which poses challenges for commonly used molecular fungal identification tools such as sequencing. Therefore, identification methods for AF strains, which do not rely on molecular approaches are of high interest. Hence, we have used near-infrared spectroscopy (NIRS) to characterize dried fungal cultures to differentiate between four different AF strains and draw conclusions about the molecular composition of fungal cells. Linear discriminant analysis of fungal NIR-spectra revealed a prediction accuracy of up to 100 % for AF strain identification. Further, differences in the molecular composition of fungal cells between strains, such as amino acid and carbohydrate content, could be uncovered by interpretation of NIR absorption intensities. Our results demonstrate that NIRS of AF biomass not only allows for identification of AF strains, but also allows for further downstream analysis of samples, as they are non-destructive. With these results in mind, further investigation of AF cell composition, especially differences over time related to the complex life cycles of AF and differences caused by varying growth media, could lead to a better understanding of this yet poorly understood group of fungi. Our findings further highlight the efficiency of NIRS as a non-destructive analytical tool to answer microbiological research questions.

P10.06

AQUAPHOTOMICS INVESTIGATION OF THE DIFFERENCES IN DEEP SEAWATER AT VARIOUS DEPTHS

Yoko Osafune[1,2], Roumiana Tsenkova[2], Jelena Muncan[2]

[1]Dr.Recella Co., Ltd, Osaka, Japan, [2]Aquaphotomics Research Department, Graduate School of Agricultural Science, Kobe University, Kobe, Japan

The purpose of this study was to investigate Deep Sea Water (DSW) collected at different depths (600 m, 800 m, 1000 m, 1400 m, and 1500 m) using near-infrared (NIR) spectroscopy and aquaphotomics [1].

Measurements were done using the XDS-RLA spectrophotometer (Foss NIR-Systems, Inc. Laurel, MD, USA), at a controlled temperature of ~23.8°C. Two replicate measurements were conducted for each sample, with all measurements performed in a random order. Each measurement involved acquiring 30 spectra by consecutively irradiating the sample with NIR light while acquiring spectra, without removing the sample from the holder throughout the process. The temperature of the sample was recorded at each measured spectrum using temperature sensor (UX100-014 M, HOBO) placed on the external surface of the cuvette. The acquired spectra were analyzed using aquaphotomics [1], [2].

The Soft Independent Modeling of Class Analogies (SIMCA) analysis classified samples based on depth with an accuracy of 91.67%. The temperature of each sample increased linearly during the first five irradiations, and then stabilized to 23.8°C. Therefore, only the spectra collected at the stabilized temperature (from the 10th to the 30th irradiation) were chosen for further analysis using aquagrams [2], as presented in Figure 1 (average aquagram of the sample DSW).

The DSW collected at different depth shows different water molecular structure depending on the depth. Up to the level of 800 m, the water is characterized by presence of proton hydrates and water solvation shells (1342, 1363, 1375 and 1385 nm) [1]. At 1000 m depth, DSW is characterized by the presence of physi-adsorbed water (1453 nm) [3], water molecules with 2 - 4 hydrogen bonds (1466, 1472, 1484 and 1490 nm) and strongly bound water (1503 nm) [1]. The DSW collected at 1400 m has similar structure, but with more proton hydrates and hydration shells. DSW at 1500m was abundant in proton hydrates and water solvation shells and small water clusters with 1-2 hydrogen bonds (1435, 1441, 1466 nm). [1].

In conclusion, the results revealed distinct differences in water molecular structures among different DSW depths, highlighting the unique characteristics and respective functionality at each level. The study results will inform novel applications of DSW based on variations in depth.

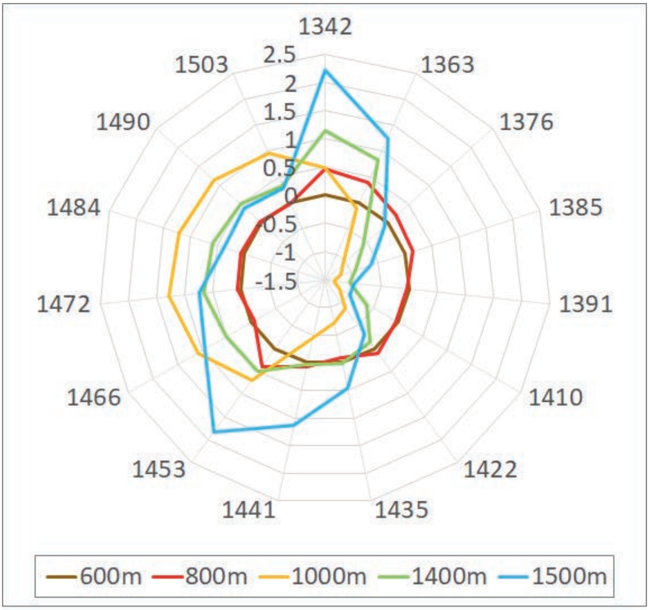


Figure 1. Aquagrams of DSW at different depths: 600 (zero line), 800, 1000, 1400 and 1500 m.

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P10.07

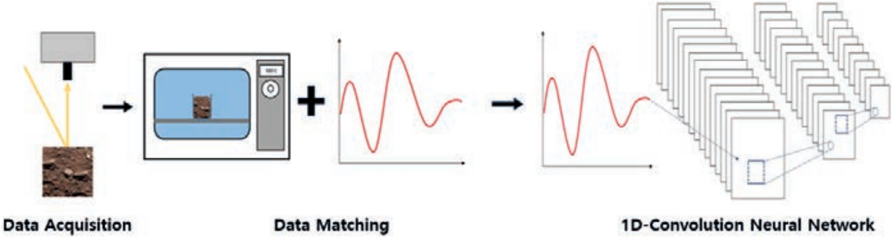
1D-CNN MODEL FOR TOPSOIL MOISTURE PREDICTION BASED ON GROUND REMOTE SENSING

Soohwan Park[1], Jae-Eun Lee[1], Hye-In Lee[1], Min-Jee Kim[2], Kyoung Jae Lim[3], Changyeun Mo[1,4]

[1]Department of Interdisciplinary Program in Smart Agriculture, Kangwon National University, Chuncheon, South Korea, [2]Agriculture and Life Sciences Research Institute, Kangwon National University, Chuncheon, South Korea, [3]Department of Regional Infrastructure Engineering, Kangwon National University, Chuncheon, South Korea, [4]Department of Biosystems Engineering, Kangwon National University, Chuncheon, South Korea

The moisture content, one of the crucial environmental factors of topsoil, is closely related to topsoil erosion and loss, agricultural productivity, and water resource storage function. The topsoil moisture content needs to be continuously monitored to preserve the topsoil from erosion and loss and to manage sustainable water resources. In this study, a deep learning model to predict soil moisture content prediction model was developed using the near-infrared spectrum of soil remotely measured in the open field. The reflection spectrum of topsoil was remotely measured in the field with ASD Field-Spec-4, and the moisture content of topsoil was analyzed using a dry induction method. Based on these data, the models Partial Least Squares Regression (PLSR) model and a 1 Demention-Convolutional Neural Network (1D-CNN) model for predicting the moisture content were developed and their performance was compared. Various preprocessing was applied to the spectral data to remove noise. R^2 (Coefficient of Determination) and RMSE (Root Mean Squared Deviation) were used as performance validation indicators of the model. As a result of model validation, the performance of the deep learning-based topsoil moisture content prediction model was excellent. The results of this study showed that remote sensing spectroscopy can predict soil moisture content in open fields. In addition, this technology can be used as a non-destructive soil moisture content monitoring technology combined with IoT technology, and is expected to be used to build deep learning-based soil moisture information management technology.

Graphical Abstract



Keywords: Topsoil, Moisture Contents, NIRS, Deep Learning, Ground Remote Spectroscopy

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P10.08

EPO OR TRANSFER LEARNING? A COMPARISON OF SOIL CHARACTERIZATION TECHNIQUES FOR SOIL AT FIELD CONDITION

Matteo Poggio[1], Samuel Sarjant[1], Stefan Franciscus Catharina Vaessen[1]

[1]Agrocares, Arnhem, Netherlands

For soil characterization, it is more practical to apply prediction models directly to field soil spectra than to lab-dried soil spectra. Instead of sending soil samples to a lab, which can be time-consuming, costly and subject to lab error process, the spectrometer is brought directly to the field. However, this convenience comes with considerable cost: dry sample calibration models are typically more accurate, few spectrometers can be deployed directly in the field and building a field spectra database requires more cost and effort in collecting calibration samples. Across the soil spectroscopy community, it is common practice to build calibration models on dried and sieved samples. Thus, spectra at field conditions need to be corrected for the effect of the moisture if they are to be predicted with a dry soil calibration model. One of the most common correction algorithms, external parameter orthogonalization (EPO), is commonly used for this procedure. However, uncertainty and error arise from the application of such algorithms.

Deep learning models commonly take advantage of a technique called transfer learning. Transfer learning allows a model to be trained from an advantageous starting point, rather than from tabula rasa. We hypothesize that training a model on lab-dried soil spectra, then use transfer learning to retrain the model on field spectra will produce better results than applying EPO.

We performed an experiment to compare the results for five important soil parameters (Organic Carbon [SOC], Cation Exchange Capacity [CEC], Clay Fraction, Total Nitrogen [TN] and pH after water extraction) between deep learning models on dry, field-transferred and EPO-corrected spectra. We used 855 spectra to create the orthogonal projection for the EPO algorithm and 6986 other spectra to build the models. The dataset was split into calibration (80%), validation (10%) and test (10%) set.

The results show that the transfer models built on spectra from samples at field conditions produce better results on average compared to the EPO-corrected spectra, but less accurate compared to the models on dry samples. The results suggest that the transfer models automatically compensate for moisture levels and that no additional correction is needed. Therefore, calibration models built on field soil spectral libraries should be preferred compared to spectral correction if the final aim and standard practice is collecting spectra in the field.

		Performance statistics				
	Model	Bias	RMSE	R^2	RPIQ	CCC
CEC	Laboratory	9.13	57.13	0.81	3.23	0.89
[mmol/kg]	Field	7.05	58.47	0.80	3.16	0.89
	EPO	3.84	61.97	0.78	2.98	0.88
Clay	Laboratory	1.18	9.34	0.70	2.70	0.83
[%]	Field	1.47	9.22	0.71	2.73	0.84
	EPO	1.47	9.92	0.66	2.54	0.81
SOC	Laboratory	0.15	8.14	0.60	1.73	0.79
[g/kg]	Field	0.21	7.80	0.63	1.81	0.79
	EPO	0.40	8.14	0.60	1.73	0.78
TN	Laboratory	0.07	0.66	0.69	1.79	0.82
[g/kg]	Field	0.05	0.76	0.59	1.57	0.77
	EPO	0.05	0.75	0.60	1.59	0.78

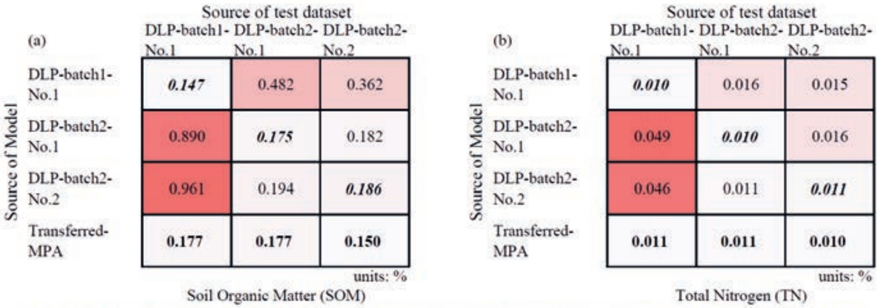
P10.09

ENHANCING THE DEPLOYMENT EFFICIENCY OF MINIATURIZED NIR SPECTROMETERS FOR SOIL ORGANIC MATTER AND TOTAL NITROGEN ESTIMATION USING A LABORATORY MASTER SPECTROMETER

Zhuolin Shi[1], Zhaoxia Ren[1], Linglong Wang[1], Yuanping Huang[1], Chenjun Ge[1], Zengling Yang[1], Lujia Han[1]

[1]*China Agricultural University, Beijing, China*

Miniaturized near-infrared (NIR) spectrometers allow instantaneous estimation of soil traits at low cost. However, the differences across devices are also magnified due to the simplification of components, such as dispersive elements, calibration units, and cooling systems. Therefore, even with the identical type of miniaturized NIR spectrometers, chemometrics models cannot always run well across devices, and it's required to build models individually for each device, with scanning all samples in the library. The primary goal of this research is to alleviate this issue by using a laboratory master spectrometer for enhancing the deployment efficiency of miniaturized NIR spectrometers for soil organic matter (SOM) and total nitrogen (TN) estimation. Firstly, several DLP-based miniaturized NIR spectrometers from same or different batches and a Bruker MPA II laboratory spectrometer were used to acquire the spectra of representative soil samples to establish partial least squares (PLS) models of SOM and TN independently. The models developed separately for DLP and MPA achieved high accuracies, with RMSEP reaching lowest 0.147 % and 0.119% for SOM, 0.010% and 0.009% for TN, indicating the feasibility of using miniaturized NIR spectrometers for soil traits estimation. Then, the models of each DLP spectrometer were applied to the datasets of other DLP spectrometers respectively. However, across-DLP model failures appeared widely, particularly crossing batches, with RMSEP reaching highest 0.961% for SOM and 0.049% for TN. Therefore, the MPA models were transferred to DLP spectrometers by the direct standardization (DS) method respectively. Comparatively, the transferred model has better performance than across-DLP models, with RMSEP reaching highest 0.177% for SOM and 0.011% for TN, which are close to the results of the independent models. Furthermore, the amount of reference samples required for DS transfer was explored. It results that the transfer effect reaches stable relatively with no more than 30 reference samples. It's demonstrated that introducing a laboratory master spectrometer with the DS method can be considered as a feasible method to lighten the workload for deploying miniaturized NIR spectrometers.



(a)

Source of Model \ Source of test dataset	DLP-batch1-No.1	DLP-batch2-No.1	DLP-batch2-No.2
DLP-batch1-No.1	*0.147*	0.482	0.362
DLP-batch2-No.1	0.890	*0.175*	0.182
DLP-batch2-No.2	0.961	0.194	*0.186*
Transferred-MPA	**0.177**	**0.177**	**0.150**

units: %

Soil Organic Matter (SOM)

(b)

Source of Model \ Source of test dataset	DLP-batch1-No.1	DLP-batch2-No.1	DLP-batch2-No.2
DLP-batch1-No.1	*0.010*	0.016	0.015
DLP-batch2-No.1	0.049	*0.010*	0.016
DLP-batch2-No.2	0.046	0.011	*0.011*
Transferred-MPA	**0.011**	**0.011**	**0.010**

units: %

Total Nitrogen (TN)

Fig. RMSEP values of the results of miniaturized NIR spectrometers applying various models. The color of grids from white to red represents the relative RMSEP from low to high.

P10.10

COMBATTING THE YELLOW CRAZY ANT: USING NEAR-INFRARED SPECTROSCOPY TO IDENTIFY AND MANAGE INVASIVE ANT SPECIES IN THE WET TROPICS

Russell Withers[1], Bronson Philippa, Lori Lach

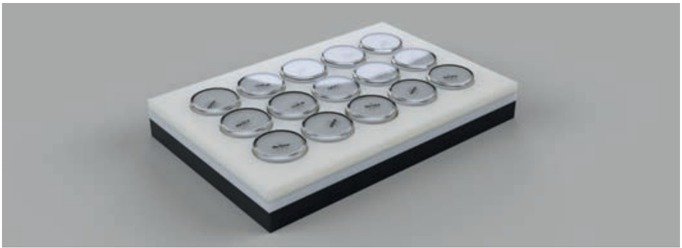
[1] *James Cook University*

Invasive insect species are a potent threat to ecological and economic systems, causing damage to natural habitats, disrupting agriculture, and displacing native species. One key challenge in the management of invasive insects is the detection of infestations over a broad area as well as the monitoring of infestations after treatment. There is a need for innovative techniques to improve the detection of invasive ant species, and ultimately work towards wide-scale deployment of passive monitoring systems.

The yellow crazy ant (YCA), Anoplolepis gracilipes, presents an insidious biosecurity threat. Native to Southeast Asia, it has since been introduced to many parts of the world, primarily in the tropics. YCA is particularly harmful due to a lack of mutual aggression between colonies, which can lead to the formation of continuous networks of high-density colonies that can devastate the host ecosystem.

Previous research has shown near-infrared spectroscopy (NIRS) to be a powerful tool in distinguishing cryptic ant species. It is specific enough to be able to discriminate between colonies of a single ant species, making NIRS an enticing potential tool to aid in the management of the invasive yellow crazy ant. This study evaluates the use of NIRS as a tool to distinguish invasive ants from native ants in north-eastern Australia by creating a NIRS calibration that classifies YCA against the green tree ant, Oecophylla smaragdina, a species ubiquitous in the region and frequently misidentified as YCA. The study also reports on the accuracy of a NIRS calibration to predict the colony of origin of YCA, providing insights into how NIRS may help us better understand YCA colony composition without requiring expensive and slow genetic analysis.

NIRS for insect taxonomy has predominantly been applied to dried or otherwise preserved specimens due to their shelf stability and because live insects generally do not cooperate with the scanning procedure. Some studies have been performed on live insects, however this is typically on the pupal or larval stage where the insect is dormant or stationary. We developed a methodology for NIRS measurement of live adult worker ants using both hyperspectral cameras and handheld spectrometers by means of a custom-built apparatus to immobilise the ant (Fig. 1). Our study demonstrates the potential for near infrared spectroscopy to be used as a rapid and non-destructive method for differentiating live ant species and colonies.



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Konrad-Lorenz-Straße 20
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6020 Innsbruck
www.carpemedia.at
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