The 18th International Diffuse Reflectance Conference

Exploring the Spectrum of Diffuse Reflectance

Wilson College
Chambersburg, Pennsylvania USA
July 30 - August 5, 2016

Index	Title	Authors and affiliation	Senior Researcher or Student	Abstract
1	Non-destructive evaluation of wood density and MFA in high-spatial resolution using NIR hyperspectral imaging	Te Ma*, Tetsuya Inagaki, Satoru Tsuchikawa Graduate School of Bioagricultural Sciences, Nagoya University, Japan	Student	Wood density and microfibril angle (MFA) are strongly correlated with its stiffness and shrinkage anisotropy. The knowledge of the spatial distraction of their values is critical for solid timber applications. In this study, softwood samples (Cryptomeria Japonica) were collected from both normal and compression parts. Near Infrared Hyperspectral imaging associated with partial least squares (PLS) regression analysis was introduced for the evaluation of wood density and MFA values at high spatial resolution. The validation coefficient of determination (R2) of density was 0.83 with a root mean squared error (RMSECV) of 105.18 kg/m3. R2 and RMSECV values of MFA were 0.77 and 5.36°, respectively.
2	Non-destructive inspection of foreign matters in food products using near infrared imaging	Te Ma*, Hikaru Kobori, Norihisa Katayama, and Satoru Tsuchikawa Graduate School of Bioagricultural Sciences, Nagoya University, Japan; Shizuoka University, Japan; and Nagoya City University, Japan	Student	Foreign matters in food products strongly affect the confidence of food production companies. To prevent such unexpected situations, fast and nondestructive online inspection methods are urgently required. In this study, we designed simple NIR transmittance imaging device to detect insects in foods equipped six LP filters. PCA was applied to reduce the spectral dimensions of the multispectral image. Insect and chocolate background parts were separated by thresholding and LoG edge detection. The classification was based on the number of white pixels in the binary images and the edge detection results. The overall classification accuracy was 93%.
3	Rapid determination of protein in whole seed common beans (Phaseolus vulgaris L.) by near infrared spectroscopy	Brad Speiss, Bert Siemens, Véronique Barthet and Ning Wang Grain Research Laboratory, Canadian Grain Commission, Winnipeg, Manitoba, Canada	Senior	A prediction model was developed using NIR to rapidly analyze the protein content of intact common bean varieties grown in Canada. The calibration set included 313 samples from 13 types of common beans differing in size, shape, and color. A modified PLS regression was used. A number of statistical treatments were applied in an attempt to optimize calibration values for SEP, R2, and bias. An independent validation set to assess the protein calibration included 209 samples from seven common bean types. The optimal calibration produced an overall SEP = 0.657, R2 = 0.888, and bias = 0.168.
4	Ridge Patterns of Blood-Transferred Simulated Fingerprints Observed on Fabrics via Mid-Infrared Laser Reflectance Imaging	Raymond G. Belliveau III, Stephanie A. DeJong, Brianna M. Cassidy, Zhenyu Lu, Stephen L. Morgan, Michael L. Myrick Department of Chemistry and Biochemistry University of South Carolina	Student	Mid-Infrared Laser Reflectance Imaging is used to detect ridge patterns of blood-transferred artificial fingerprints on acrylic, polyester, nylon and cotton fabrics. An in-house assembly using a CO2 laser as a light source, a scattering surface to spatially disperse the laser, and a microbolometer-based infrared camera as a detector is used to image large areas of fabrics for the detection of transfer prints in blood as an application in the forensic sciences.
5	Improving drying uniformity of mango slices by combined hot-air & microwave-vacuum drying, with NIR-HSI in visualizing moisture content distribution.	Yuan-Yuan Pu, Da-Wen Sun Food Refrigeration&Computerised Food Technology Center, School of Biosystems and Food Engineering, University College Dublin, Ireland	Student	Three drying approaches (hot-air drying (HAD), microwave-vacuum drying (MVD), and the combined method (HAD+MVD)) were applied to dehydrate mango slices. With the help of NIR hyperspectral imaging and multivariate data analysis, moisture content (MC) distribution on mango slices was visualized. Results showed a non-uniform drying property for mango slices dried by HAD or MVD individually, whereas drying uniformity was improved when HAD and MVD were combined. The current study may lead to the development of an effective combined HAD+MVD technique for enhancing the drying uniformity of mango slices for the industry.

6	Applying vibrational spectroscopies for quantitative studies of drug recrystallization in pharmaceutical films	Yi Li1, James K. Drennen,III1,2, Carl A. Anderson1,2; Duquesne University	Student	The aim of the study was to evaluate the ability of Raman and near-infrared (NIR) methods to estimate the amount of crystalline drug in thin films, informed by the information regarding the chemical composition and film thickness. Artificial samples with known crystallinity were prepared by coating an ibuprofen suspension onto drug-in-adhesive substrates. The developed multivariate quantitative models were applied to films with drugs undergoing non-seeded crystallization. The sensitivity of multivariate methods was compared with powder X-ray diffraction (PXRD). Vibrational spectroscopic techniques demonstrate potential benefits for accurately monitoring and controlling drug crystallization in pharmaceutical films.
7	Determination of the Limit of Detection for a Multivariate Method to Detect Polymorph Content of a Pharmaceutical Tablet	Hanzhou Feng1, Robert W. Bondi Jr.,2 James K. Drennen,1 Carl A. Anderson,1 Benoit Igne2* Duquesne University, GlaxoSmithKline	Student	The limit of detection for a multivariate method was investigated in this presented work. The goal was to detect an undesired polymorph in a pharmaceutical tablet using transmission Raman spectroscopy. Carbamazepine was studied as a model compound in this work, and polymorph form I was regarded as the analyte of interest that requires effective detection. Quantitative models were generated and optimized using multivariate regression and data preprocessing. Prediction uncertainty was estimated using the error-in-variable algorithm by accounting for all the main variables contributing to the prediction. Multivariate detection limit were calculated based on statistical hypothesis testing.
8	Selecting PLS Model Components to Develop Robust NIR Method for Quantitative Analysis	Md Anik Alam1,2, James K Drennen, III1,2, Carl A Anderson1,2 Duquesne University	Student	Robustness of the Partial Least Square (PLS) model is critical for successful implementation of a near infrared quantitative method in the pharmaceutical industry. Different sources of variations such as scale variations, physical variations and chemical variations can affect the near infrared model performance during product life cycle. A robust PLS model is required to be developed at the outset of calibration to encounter such variations and provide desired predictive performance thus avoiding the needs for recurrent model updates. Selecting the optimum number of model components (loading vectors) is critical to build a robust model with the available dataset. Cross-validation error along with the amount of model variance captured by each model component is currently used to select the optimum number of model components. A new method was developed to select the optimum number of model components based on weight co-efficient of each model component. The new method was found to be more effective in selecting the optimum number of model components and improving model robustness compared to the current selection technique. Several datasets possessing different types of critical variations to near infrared PLS model were used in this study to demonstrate the efficiency of the new method in selecting the optimum number of model components.

9	Designing a Calibration Set in Spectral Space for Efficient NIR Calibration Development of Tablets Containing Multiple APIs	Md Anik Alam1,2, Md Nayeem Hossain, Douglas Steinbach, James K Drennen, III1,2, Carl A Anderson Duquesne University	Student	Near infrared spectroscopy is a well-established analytical tool in the pharmaceutical industry for quantitative analysis of tablets. Successful development of the NIRS method depends on appropriate selection of a calibration set. There are different strategies for developing a calibration set for the NIRS method. Experimental design of synthetic samples in the concentration space is a widely used technique as these samples allow wider concentration variation compared to the commercial samples. However, this technique requires a large sample set to follow the traditional concept of incorporating as much variance as possible into the calibration set. All variance information in the calibration set may not be necessary for a successful model development. The traditional approach of using a large sample set, potentially containing redundant information, is more detrimental to the calibration process of tablets containing multiple APIs compared to a single API formulation. Designing a calibration set in spectral space is an efficient alternative to the traditional calibration development technique of tablets containing multiple APIs. Near infrared spectroscopy was used to develop a quantitative method for a model drug containing two APIs as Acetaminophen and Caffeine. The first calibration design was developed in the concentration space using a traditional concentration-based approach. A full factorial design of experiment was used to vary the APIs concentrations, each at five levels and excipient concentration ratio at 3 levels. Each tablet was compressed at three compaction pressures resulting in a total of 225 tablets in the calibration set. The second calibration set containing 33 samples was developed in the spectral space using a newly developed technique. Partial Least Square (PLS) models were developed from the respective calibration sets to predict APIs concentrations in an independent test set. Similar prediction performance was achieved from the PLS models indicating improved efficiency of the newly developed technique compared to the traditional technique in terms of sample requirements.
10	LOCAL algorithm for the prediction of optimum harvest dates in fruits of different genus	Irina Torres1*, Dolores Pérez-Marín2, Ana Garrido-Varo2, María-José de la Haba1, María-Teresa Sánchez1 Department of Food Science and Food Technology, University of Cordoba, Spain	Student	The fruit sector needs a rapid, economical and non-destructive technology for monitoring the internal quality of fruits during on-tree and on-vineripeningprocess. The aim of this work wasto evaluate the feasibility of using the LOCAL regression algorithm for the prediction of internal quality parameters (soluble solids content and titratable acidity) in different fruits using a handheld spectrophotometer based on MEMS technology. The sample set comprised1,349 samples belonging to the following genus: Citrus (oranges and mandarins), Fragaria (strawberries) and Prunus (nectarines). The results obtained demonstrate that LOCAL algorithm could be an interesting tool to estimate optimum harvest dates in intact fruits, despite the fact that the database comprised different fruit genus.
11	A performance comparison of two spatial interpolation models for evaluating processed animal proteins	Adame-Siles, J.A.1*, Pérez-Marín, D.C. 1, Guerrero-Ginel, J.E. 1, Torres-Rodriguez, I. 1, Garrido-Varo, A. 1 Department of Animal Production, University of Córdoba, Córdoba, Spain	Student	Evaluation of bulk raw materials involves a number of challenges. Typically, traditional methods have some weaknesses, mainly concerning effectiveness, operating costs or the lack of rapid tools.This work aims to exploit near-infrared spectroscopy (NIRS) analytical abilities to assess the performance of two statistical models allowing spatial distributions of analytical constituents to be inferred. For this purpose, a set of 8 lots of processed animal proteins were analysed using a fiber-optic probe interfaced with a FT-NIR instrument. The resulting distributions,based on NIR predictions, were evaluated for each sampling protocol and interpolation methodby the RMSEP statistic and density functions.

12	Development of an Efficient Robust Calibration Model of Process Analytical Systems by Incorporating Variations of Pure Components: A Pharmaceutical Tablet Assay Example	Md. Nayeem Hossain, Carl A. Anderson, James K. Drennen III Graduate School of Pharmaceutical Sciences, Duquesne University.	Student	Process analytical technology (PAT) for monitoring and controlling of pharmaceutical unit operations is an important component of a comprehensive quality management system. However, it must be ensured that PAT tools meet the performance requirements, especially the accuracy, precision, and robustness of the model. The time, expense, and effort involved in developing and validating such calibration models often detract from their implementation. Methods based on spectral characteristics of pure sample constituents, for instance Pure Component Projection (PCP), Synthetic Calibration have proved to be useful in developing efficient spectroscopic calibration techniques. However, robust model against physical and chemical variations requires samples generated from different batches to be included in the calibration model, which decreases the feasibility of PAT tools application, especially during the early stage of product development. To develop a robust and efficient calibration model, here we proposed to include variations of pure components while developing calibration model. This technique is based on augmentation of pure components with external variance (e.g. in humidity) followed by generation of in-silico spectra from the collected pure component spectra. This in-silico spectra with a small set of non-production samples is then used to create a robust model against the variance, to which pure components were exposed. This method was tested on a model drug formulation containing acetaminophen, lactose, microcrystalline cellulose, hypromellose, and magnesium stearate. During the course of study, different granule batches were manufactured using a fluid bed granulator, and subsequently tablets were prepared from these granules, in lab and pilot scale. Due to seasonal differences, large variations existed in the particle-size distribution of the granules as well as in the moisture content of the excipients and granules. The results showed that, while traditional approach required a calibration model to be updated overtime with new samples to create a robust model for prediction of API content of tablets, adding variations of pure component in the calibration model obviated the need of creating a large number of samples improving the efficiency of the calibration technique considerably. This offers advantages especially with limited resources, e.g. in the early stages of process development.
13	Quantitative Chemical Imaging of Endosperm Purity for Commercial Mill Streams	Mark D. Boatwright1,2, David L. Wetzel2; Department of Biochemistry & Molecular Biophysics, Kansas State University, Manhattan, KS	Student	Selective quantitative chemical imaging analyses of 81,920 near infrared spectra at key wavelengths are enabled by array detection with a LCTF. This is possible with partial least squares data treatment that enables determination of the analyte to be studied and the mathematical weighted summation provides the quantitative result for the field of view. This objective chemical method is applied to determine the endosperm purity profile for flour streams of a commercial flour mill and reveals distinct changes in purity as streams of descending endosperm purity are incorporated to increase the yield.
14	Development of feedforward controller for tableting incorporating NIR and Raman Spectra	Yuxiang (Henry) Zhao, James K. Drennen III, Carl A Anderson. School of Pharmacy, Duquesne University.	Student	Feedforward controller can reduce the variability of the manufactured tablet product by accommodating the raw material variability. However, material characterization, which is usually slow and destructive, can be a limitation for the implementation of feedforward controller. Rapid and non-destructive spectroscopy technologies, NIR and Raman, were incorporated in the establishment of the feedforward controller. NIR and Raman spectra successfully substituted material attributes such as particle size and apparent density, gaining similar model performance of the feedforward controller. In addition, NIR and Raman spectra captured residual moisture information which had effects on tablet quality but was not included in the previous feedforward controller.
15	Near-infrared spectroscopy using a supercontinuum laser - Application to long-wavelength transmission spectra of barley seeds	Søren Balling Engelsen, Department of Food Science, University of Copenhagen, Denmark	Senior	The supercontinuum laser is a new type of light source which combines the coherent light properties of a laser with a broad spectral region. The supercontinuum laser can therefore focus the light onto small samples without losing intensity. Barley and wheat seeds have only been studied by near-infrared transmission in the short wavelength region up to 1100 nm. In the present study, seeds from five barley genotypes were measured by near-infrared transmission from 2255-2381 nm using an experimental spectrometer based on a supercontinuum laser. The correlation between the NIR spectra and the chemical composition of the seeds was investigated.

16	2D NIR-MIR correlation spectroscopy - A useful tool to augment the interpretation of NIR spectra	T. Ringsted1, S.B. Engelsen Department of Food Science, University of Copenhagen, Denmark	Student	Two-dimensional (2D) correlation spectroscopy can be used to augment the interpretation of overlapping peaks by mapping samples that are modulated systematically (e.g. the samples vary in temperature or the chemical reaction time). Since the interpretation of mid-infrared (MIR) spectra is much simpler compared to near-infrared (NIR) – the interpretation of the NIR spectra can be significantly augmented by analyzing the 2D NIR-MIR correlation plots. This study presents generalized 2D correlation spectroscopy of NIR-MIR spectra of the bread crump staling process. A thorough assignment of the overtone spectra was performed using 2D MIR-NIR correlation spectroscopy.
17	Miniaturized versus benchtop NIR spectroscopy - a comparison	Raphael Henn1, Maria Elisabeth Grossgut1, Christian Huck1 Institute of Analytical Chemistry and Radiochemistry, Franzens University, Innsbruck, Austria	Student	NIR spectroscopy is of great interest for applications in food and pharmaceutical industry due to its quick analysis times, low consumption of chemicals and easy handling, furthermore in most cases no sample preparation is required. Recent developments of miniaturized spectrometers offer an even wider field of applications enabling off site analysis. Evaluation of the performance ability for the property of interest is therefore of great importance to decide whether using a miniaturized device is viable or not. This poster compares data of portable and benchtop NIR spectroscopy. Two datasets of different carbohydrates in syrup and melamine in milk powder were analyzed and prediction ability of different devices compared in order to analyze the influence of accessible spectral ranges and resolution.
18	Linearity of NIR Models of a Solid Dissolved in Liquid System: A Preliminary Study	Shikhar Mohan, Howard Mark, Benoit Igne, James K. Drennen, Carl A. Anderson Graduate School of Pharmaceutical Sciences, Duquesne University, Pittsburgh, PA	Student	Quantitative NIR models are used as a complimentary technique to the significantly more timely and destructive wet chemistry techniques (ex: high performance liquid chromatography (HPLC)) for pharmaceutical tablet assay assessment. The traditional practice of using gravimetric reference values for NIR model development potentially introduces non-linearity into the model because NIR absorbance is proportional to the volume fraction and not the weight percentage (gravimetric). This study investigates a solid (Urea) dissolved in a liquid (glycerol) system and compares NIR models with gravimetric versus volume based (apparent molal volume) reference values. The result suggest increase in non-linearity with gravimetric reference values.
19	Development of Non-Destructive Point-of-Sale Cannabis Grading Device	Ierud1 ranavas1 Doughty2 Meyer3 1 Portland State University Or 2 Chemical Analysis Group WA State 3 Felix Instruments WA State	Student	With the recent legalization of Cannabis in Washington State, there is now a legislative need for quality testing. To this aim a handheld swnir device was used to investigate the potential detection of active compound in extracts. Although THC and CBD do not appear to be ir active in the 700-1000nm region, there is a potential secondary correlation with chlorophyll content.
20	Blend uniformity assessment based on the simultaneous characterization of chemical composition and physical variability of a multi-grade component formulation matrix by NIR spectroscopy	Natasha L. Velez1, Shikhar Mohan1, Md. Anik Alam1, Carl Anderson 1,2, James K. Drennen III Duquesne University	Student	The distribution of drug and excipients in a pharmaceutical powder blend play a critical role in the quality and performance of the final product. Quantitative NIR models are often applied in real-time to blending operations for process monitoring/control purposes. These models use NIR chemical information to determine the amount of each constituent of interest. This work seeks to challenge the capability of NIR by using a calibration design incorporating both chemical composition and physical variability for blend homogeneity assessment. Results suggest that NIR sensitivity to particle size differences can be used to determine the degree homogeneity of different grades of the same excipient along with the other chemical components.
21	Towards a low-cost portable spectrometer for determining soil fertility	Herman Vedder*(a), Dale Fletcher(b), Frits Oukes (c), Thomas Terhoeven-Urselmans(a), Peter van Erp(a). (a) SoilCares Research, Netherlands (b) Waikato University, New Zealand (c) RedMerito, Netherlands	Senior	A low cost hand-held soil fertility sensor could have a big impact in reducing global yield gap because most farmers have no access to soil analytical services. To identify the most suitable spectrometer for this purpose a survey was carried out using about 500 Dutch agricultural soil samples. The performance for the different candidates is reported and compared to a benchmark FT-NIR spectrometer. The most promising candidate was identified and further improvements are proposed to build the low cost portable soil fertility sensor.
22	Robust Latent Variable Selection for Multiple Instrument Calibration Applications	Douglas Steinbach1, Md. Anik Alam 1, Md. Nayeem Hossain1, Carl Anderson 1,2, James K. Drennen III Duquesne University	Student	Quantitative spectroscopic model performance can be diminished when spectra from a new instrument is projected onto it. One possible cause for this is the calibration model containing variance that was specific to the subtle variations that exist between instruments. This work used a triangulated approach to partial least squares latent variable selection that helps avoid the inclusion of such distracting variance. Methods used include the area under the received operator characteristic curve calculated using the PLS scores of calibration and test spectra collected on separate instruments, calculating how each latent variable contributes to the regression vector, and the amount of variance captured by each latent variable.

23	Utilizing Parallel Factor Analysis (PARAFAC) to Qualitatively Characterize Cell Culture Growth Media using Excitation-Emission Matrix (EEM) Fluorescence Spectroscopy	Sameer Talwar1, Sayantan Bose2 1. Process Modeling Centre of Excellence, Product Development, GlaxoSmithKline 2. Advanced Manufacturing Technology, Platform technology and Science, GlaxoSmithKline	Senior	Biological culture media comprises of carbohydrates, amino acids, and vitamins which sustain mammalian/microbial cell culture growth. A capability to characterize media composition enables efficient development of culture processes for the manufacture of biological drug substances and an understanding of nutrient consumption and metabolite generation. This study used Excitation-Emission Fluorescence spectroscopy to characterize the changes in a cell culture media with time via Parallel factor analysis (PARAFAC) algorithm. The analysis revealed the consumption of specific constituents (amino acids and vitamins) and generation of metabolites. This indicated the analytical specificity and sensitivity towards the variability in physiological markers.
24	Efficiency of process spectrum methodology for preparing the calibration set in NIR pharmaceuticals quality control analysis.An innovative tool for process analytical technology	Vanessa Cárdenas*, Marcelo Blanco, Manel Alcalà. University of Puerto Rico	Senior	During pharmaceuticals manufacturing the physical characteristics of samples change due to granulation or compaction. The NIR spectrum is strongly influenced by these changes; therefore, developing appropriate NIRS calibration models requires careful selection of calibration sets containing all potential sources of variability in the production samples to be analyzed. An innovative strategy for obtaining the calibration set is proposed based on the calculation of a virtual spectral -for incorporation of physical variability- and its subsequent addition to a spectral matrix that contains the desired chemical variability. The developed methodology was evaluated in several pharmaceutical formulations in different dosage forms, and the results shows its suitability for on-line/in-line analysis.
25	Development of an NIR calibration model with temperature compensation using common temperature-difference spectra for determining the Brix value of intact fruits	P. Jannok,1,2 Y. Kamitani,2 K. Hironaka,4 M. Shibayama,2 and S. Kawano2 1 Department of Post-harvest and Processing Engineering, Rajamangala University of Technology Isan, Nakhon Ratchasima campus, Nakhon Ratchasima, 30000, Thailand 2 Faculty of agriculture, Kagoshima University, Kagoshima, 890-0065 Japan 3 Postharvest Technology Laboratory, University of the Ryukyus, Okinawa, 903-0213, Japan	Student	It is known that near infrared (NIR) spectra are affected by sample temperature. Therefore, the temperature compensation is needed for practical implementation. However, the procedure to develop a calibration model with temperature compensation is time-consuming because the calibration development method requires spectra measured at different temperatures in each sample. If it is possible to develop a calibration model with temperature compensation using spectra measured at a single temperature, it would be convenient. In this study, NIR calibration model with temperature compensation, using common temperature-difference spectra, was developed for determining the Brix values of intact fruits. The calibration model with temperature compensation using common temperature-difference spectra was called “CMTCC” hereafter. Each fruit species of peach, pear, and persimmon samples were separated into four sample sets. Calibration and validation sets were measured spectra at 25□C while prediction and difference spectra sets were measured spectra at 20□C, 25□C and 30□C. The difference spectra sets of the three fruit species were used to make the common temperature-difference spectra. The common temperature-difference spectra of averaged second derivative (2D) spectra between 20□C and 25□C, and 30□C and 25□C were calculated and used for creating artificial 2D spectra for 20□C and 30□C from the 2D spectra for 25□C. The CMTCCs in each fruit species were developed from the 2D spectra at 25□C and the artificial 2D spectra for 20□C and 30□C by partial least squares (PLS) regression. Each model developed could be used for determining the Brix values of the prediction sets of each fruit species at different temperatures with no significant difference at 95% confidence. Moreover, the common temperature-difference spectra were applied to make a CMTCC of the other species of apple from the three fruit species used for making the common temperature-difference spectra. A good result was also obtained. It was concluded that the development method of an NIR calibration model with temperature compensation using common temperature-difference spectra was useful.