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Near Infrared Spectroscopy in industries: A Review on research contribution

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Abstract — *NIR spectroscopy has gained wide acceptance in different industries like food processing, dairy products, agriculture, process analytical industry, pharmaceutics, petrochemical, environmental and soil analysis and in the diamond industry etc. The reason for wide acceptance of NIR spectroscopy as a tool over conventional analytical techniques is easy sample preparation without any pre-treatment. The determination of multivariate content from any sample can be accomplished from only single spectrum in NIR region. The technique is generally chosen for its speed, its low cost and its non-destructive characteristic towards the analyzed sample. Recently, with the development of computer science and fibre optics, the available applications of the NIRS technique become more popular and attract more attention. This paper is focused on highlighting the various applications of NIRs and brings out the importance of it in analytical instrumentation domain.*

Keywords- *Near Infrared Spectroscopy, Chemometrics*

I. INTRODUCTION

Optical radiation covers the wavelength range of 100 nm-1000 mm of the electromagnetic spectrum. It is subdivided into the ultraviolet (UV) region from 100 to 380 nm, the visible (VIS) light ranging from 380 to 780 nm, and the infrared (IR) radiation of wavelengths above 780 nm. Within the IR region, the near infrared (NIR) region covers wavelengths from 780 nm up to 2.5 mm, mid infrared (MIR) covers the region from 2.5 to 25 mm, and far infrared (FIR) the adjoining region up to 1000 mm.

When radiation strikes a sample, the incident radiation may be reflected, absorbed or transmitted, and the relative contribution of each phenomenon depends on the chemical constitution and physical parameters of the sample. Most NIR absorption is due to combinational band or first and second overtone vibrations of atoms. Any transfer of an atom from n to n+2 is first overtone and n+3 is second overtone combination. The relationship of band gap energy difference to the wavelength in case of food having multi-variant content falls under NIR region. It provides much more complex structural information related to the vibration behavior of combinations of bonds. The record of the NIR region of the electromagnetic spectrum involves the response of the molecular bonds O-H, C-H, C-O, S-H and N-H functional groups. These multilevel energy transitions are the origin of NIR overtone bands that occur at multiples of the fundamental vibrational frequency. Combination bands appearing between 1900 nm and 2500 nm are the result of vibration interactions, i.e. their frequencies are the sums of multiples of each interacting frequency.

1.1. Instrumentation setup for NIR spectroscopy

A NIR spectrometer instrument mainly consists of light source, wavelength selection device(monochromator), sample holder, optical detector, and tools for data processing as shown in Figure 1.

The ideal source of radiant energy would be one which transmits the uniform radiant intensity throughout the entire range of its application. The source used in NIR range is Pb-Ld monochromatic source which covers full NIR spectral range. For some applications in which a specific wavelength is required, tunable laser diodes are available in the market with precise wavelength which can transmit specific range of very narrow wavelength. Hydrogen or Deuterium, Mercury and Xenon arc discharge lamp are used as a light source for specific wavelength.

Sometimes for qualitative analysis of specific content light filter is placed between light source and monochromator. Beam splitter system/monochromator is used to translate multi-colored light into single-color light, such as light filter, interferometer, and gratings. The basic NIR set up can be used for the measurement of either single or combination of any of the physical absorbance band phenomena of transmittance, reflectance and transflectance which is combination of transmittance and reflectance.The selected wavelength light passes in turn through the sample where the absorbance of light based on compound and concentration

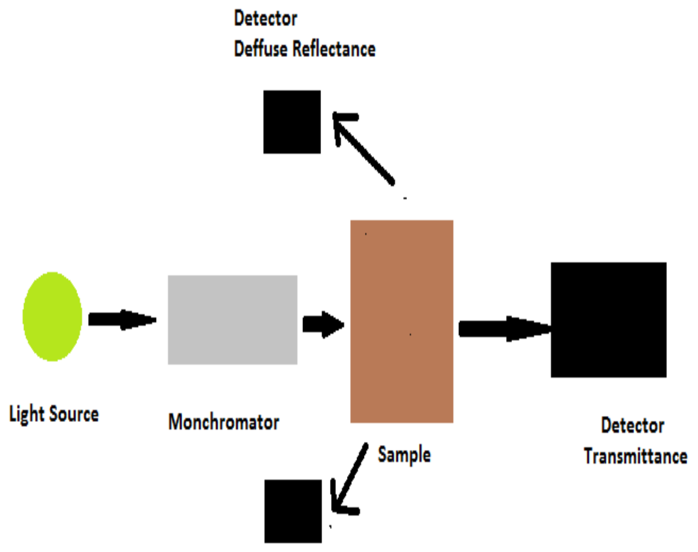

Figure 1. Instrumentation setup for NIR spectroscopy

takes place through any of the physical phenomena. Radiation detectors are the devices which convert the light from sample chamber into equivalent electrical changes. In NIR set up, they are placed after a wavelength separator to detect the selected wavelength of light. Based on an effective electromagnetic range, various types of optical detectors are available in the market. Some of these are charged coupled devices, photodiodes and photovoltaic cells in semiconductor technology, photodiode array and photomultiplier tube etc .This absorption pattern can serve to characterize uniquely the compound involved. To measure good NIR spectra, the proper sample preparation is of importance, especially when measuring solid samples, since scatter effects and stray light induced by variations in packing density of powders or sample positioning may cause large sources of error in the spectra. Therefore, several types of sample cells, such as quartz cuvettes with defined optical path length for liquids, specifically designed sample cells with quartz windows for semi-solids and powders, and adjusted sample holders have been developed. Temperature control and sample movement are other factors that have been considered.

II. REVIEW ON APPLICATIONS OF NIRS IN INDUSTRIES

This section brings out various innovative applications of near infrared reflectance spectroscopy in various industrial domain to highlight the fore future of this technique over other conventional method.

2.1 Agro and Food Industries

The economic value of any crop is based on its nutritional facts make the farmer richer and encourage them to grow yields with high nutritional values. In turn, society will benefit as a whole. Fruits and vegetables offer essential vitamins and other nutritional component those are important for human health. Various researchers applied NIR technique to detect the SSC (Soluble Solid content), Acidity, freshness, ripe time, sweetness of fruits and vegetables.

Francisco J. Rodríguez-Pulidoa, Douglas F. Barbinb, et. al. (2012) in their research article, decide appropirate harvest period of grape seed by NIR hyperspectral imaging. For this purpose, 56 samples of seeds from two red grape varieties (Tempranillo and Syrah) and one white variety (Zalema) in two kinds of soil were selected to assess their features based on the reflectance in the near-infrared (NIR) spectra by using prediction models (partial least squares regression) and multivariate analysis methods. This work has shown the capability of hyperspectral imaging to predict the variety and stage of maturation of the samples analyzed without additional analytical analysis.

Research study by Bart M. Nicolai et.al. (2007) at the Flanders centre of Postharvest Technology, Belgium has focused the usefulness of NIR spectroscopy for measuring quality attributes of horticulture produce. The objective of the study was to identify the success of online systems for grading fruit and vegetables. They recorded spectra for various fruits to reflect presence of various contents through peak values at specific wavelengths shown in Figure 2. They proved the usefulness of NIR spectroscopy to measure SSC of fruits and vegetables.

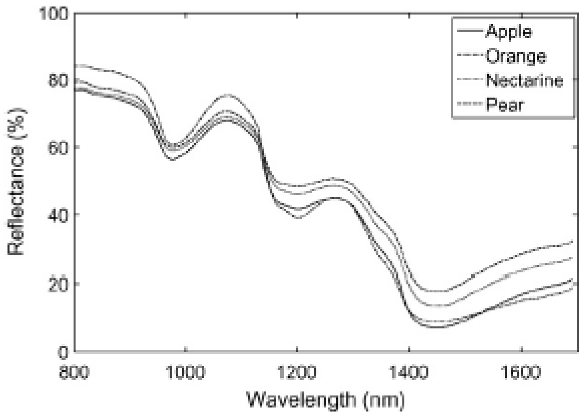

Figure 2 Typical NIR reflectance spectra of some fruit

Another research work by Ana Morales S., Victor M. et al. (2011) at University of Sevilla, Spain has determined application of NIR spectroscopy on intact olive. Spectral data over a range of 400 to 2498 nm were recorded for 448 fruit samples collected in 2008 and 2009 from seedlings of different crosses in a table olive breeding program. They developed a calibration model for some parameters like fruit weight, equatorial diameter and volume etc.The spectral response of olive fruits collected from 2008 to 2009 is shown in *Figure 3. They conclude that* NIR technology is an efficient online method for the content analysis in table olives without sample destruction but the calibration models developed were not transferable from this year to next year.

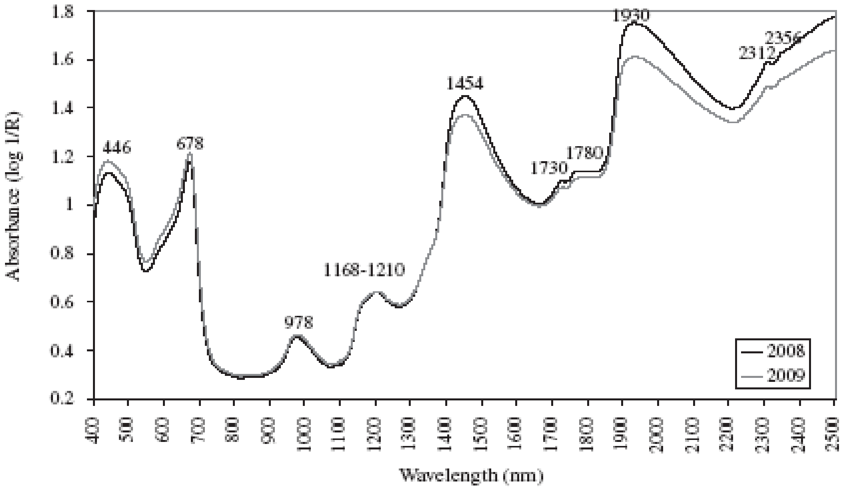

Figure 3. Spectra for olive fruits

Watermelons were assessed for their soluble solids contents by NIR/Vis transmittance spectroscopy in a spectral range of 687-920 nm by Jie, Xie, Rao, & Ying,(2014). For this purpose, on-line measurements were conducted by means of a prototype system with sample presentation in trays moving on a conveyor belt (0.3 m/s). They adopted different approaches for feature wavelength extraction and to establish calibration models for on-line prediction They developed a calibration model based on combined optimization techniques like chemometrics, Partial least squares regression (PLSR), stepwise multiple linear regressions (SMLR), Monte-Carlo uninformative variable elimination (MC-UVE) and genetic algorithms (GA) .

Yang, Hu et.al. (2015) has been using NIR spectroscopic method to detect the starch, protein, fat and water content in rice. For that 255 rice samples have been collected by diffused SupNIR-2700 NIR spectrometer. The samples were scanned 6 times and these spectra were averaged. The Kennard stone method (Zhang et al.,2012) has been used for calibration. Partial lease squares (PLS) and BP neural network were employed to build the model and MATLAB 2010 is used as a data processing tool. They conclude that for fat content analysis model built by PLS is more accurate than BP neural network and the PLS models of protein, starch and water are the same as BP neural network

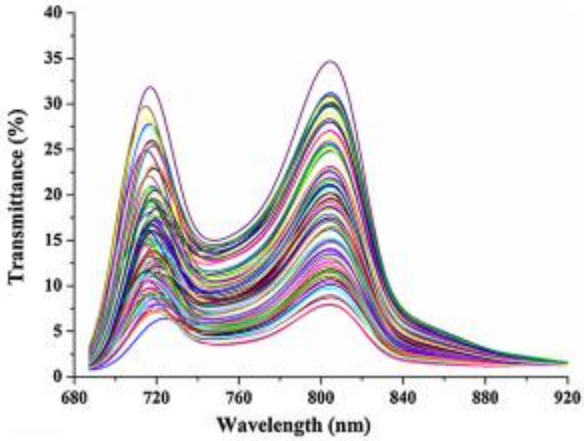

Figure 4. Spectra of Watermelons

Near infrared spectroscopy as a tool has been popularly used for fruit probable usable time calculation based on watery content accumulation. Various researchers have experimented on varieties of fruits like fruit juices of apple, banana, cherry (Singh P. et al., 1996) for processing time requirements, apple juice applied pre-heat treatment effects (Woodcock T. et al., 2005), Mango (Valente et al., 2009) for firmness determination, determination of bumpy surface of fruits (Huang L. et al., 2011). Goesaert H. and Brijs et al. (2005) provided the basics on the process ability and quality determining wheat flour constituents and presented common concepts on their fate during the bread making process. Bao, J. and Shen Y. et al. (2007) in their study, developed calibration models for measurement of thermal and retro-gradation properties built from the spectra of grain and flour samples.[1]

2.2 Dairy products

The usefulness of NIR spectroscopic in dairy products, liquid milk, cheese, butter, and fermented milk products, for the analysis of the major components and also for the detection of adulterations.
Aernouts, B. and Polshin E. et al. (2011) investigated an interesting research on characterizing based on milk NIR spectral information about the metabolism of cow. The composition of produced milk determines the economic value of the milk and provides valuable information about the metabolism of the corresponding cow. Therefore, online measurement of milk components during milking two or more times per day would provide knowledge about the current health and nutritional status of each cow individually. This information provides a solid basis for optimizing cow management. The potential of visible and near-infrared (Vis/NIR) spectroscopy for predicting the fat, crude protein, lactose, and urea content of raw milk online during milking was, therefore, investigated in this study.
Christian Huck (2016) has approached to optimize Near Infrared spectra with design of experiments and determination of milk compounds as influence factors for changing milk over time. This was illustrated with the aid of near-infrared spectroscopy and the previously optimized parameters by detection of altered fatty acids in milk, especially by the fatty acid content (number of carboxylic functions) and the fatty acid length.

Elisângela Serenato Madalozzo & Elenise Sauer & Noemi Nagata (2015) has carried out research to determine fat, protein and moisture content in ricotta cheese by NIR spectroscopy with PLSR technique. Spectra of 19 conventional and low-fat ricotta samples from different manufacturers were collected in duplicate, with 33 of the 38 spectra used as a calibration set and the remaining 5 spectra used as an external validation set. They developed robust multivariate model to predict the content of cheese with errors of less than 6.4 %. [1]

2.3 Egg and Meat

Hao Lin & Zhao J., at el. (2011) at school of Food and biological engineering, Jiangsu University, China has also brought out significance of NIR techniques for food analysis at various levels. Freshness, which is the major contributor to the quality of egg and egg product, The work presents a non-destructive method for the measurement of egg freshness, and builds a robust calibration model to improve the prediction ability. Also diffused reflectance spectroscopy as a tool to analyze freshness of shell eggs was established. The overall result indicated that the freshness of eggs can be determined by NIR spectroscopy coupled with calibration models prepared using Genetic Algorithm – Artificial Neural Network (GA-ANN).

The measurement of several beef quality traits (e.g. shear force, cooking loss, and intramuscular fat content) requires the destruction of a meat sample. The removal and destruction of meat are expensive and time-consuming and the analysis itself is often expensive. To overcome limitations of conventional technique Massimo De Marchi (2013) has investigated VIS-NIR spectroscopy to predict beef quality traits by directly applying a fiber-optic probe on carcass surface and correlating the results with meat quality traits of M. longissimus thorac. For this purpose, data were collected of 230 young bulls and beef heifers across two different trials and slaughtered in two commercial abattoirs. He concluded that vis-NIR spectrosopy promising ability to predict color, cooking loss, and pH of beef, but the weak point of this technique is standardization depends on the scheduling and position of spectra collection, and this is very difficult due to differences among animals and slaughtering conditions Finally, the accuracy of prediction of meat quality characteristics depends on a representative calibration set, which holds all the biological variability of the studied traits. So, larger data set is required for calibration model.

An experimental study by Mohammed Kamruzzaman, GamalElMasry et al. (2012) focused on the prediction and visualization of chemical composition in lamb meat using NIR multivariate regression imagines technique is an important development in the food industry. The main goal of the study was to investigate the potential of hyper spectral imagine in the near infrared range of 900-1700nm for the nondestructive prediction of chemical composition of the lamb meat. Hyper spectral images were acquired for lamb samples originated from different breeds and different muscles. The mean spectra of the samples were extracted from hyper spectra images and multivariate calibration models were built by using PLS regression for predicting water, fat and protein contents. The models developed possess good prediction abilities for this chemical constituents with determination co-efficient of 0.88, 0.88 and 0.63 with standard error of prediction (SEP) of 0.51%, 0.40% and 0.34% respectively. The resulting prediction maps provided detailed information on composition gradient in the tested muscles. The result obtained from this study clearly reveals that NIR hyper spectral images in tandem with PLSR modeling could be used for non-destructive prediction of chemical composition in lamb meat.

Various instances, researches have been focused to determine the endpoint temperature in previously heated ground beef by Thyholt K. and Enersen G. (1998), Stefano and Zardetto (2005) to identify changes in egg pasta, Lin H. and Zhao J. et al. (2011) for characterization of egg freshness.[1]

2.4 Medical diagnosis

The wavelength of NIR is such that it easily penetrates into the skin and another feature is NIR spectra is not much absorbed by water and hemoglobin .This advantage allows the use of NIR spectral imagine over other imagine techniques like X-ray etc.

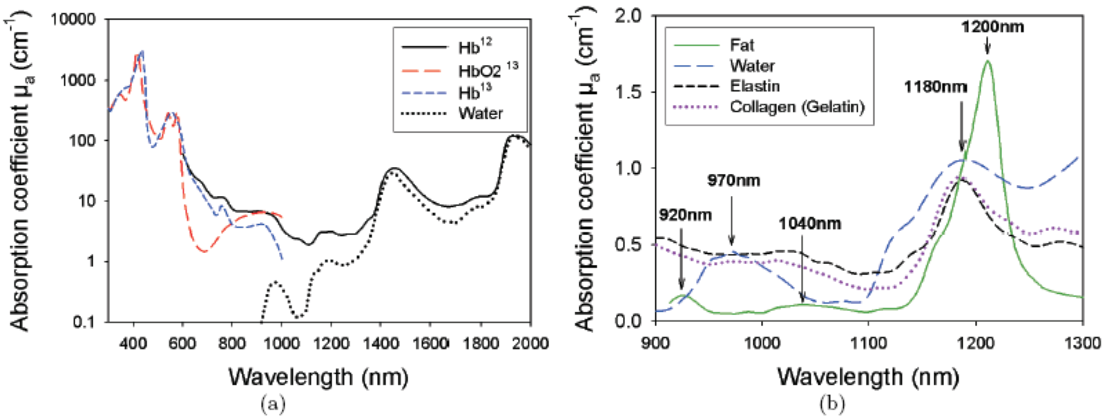

Figure 5. Photo acoustic spectra measurement of (a) normal tissue (b) fatty tissue

Thomas A. and Paul B. (2009) at Department of Medical Physics and Bioengineering, University College London, UK is important milestone in NIR spectroscopy's penetration in medical field. Photo acoustic spectroscopy has been shown to be able to discriminate between normal and athermanous areas of arterial tissue in the visible range (410 nm – 680 nm). However, at these wavelengths hemoglobin absorption is also very high. This makes it challenging to apply photo acoustic techniques using an intravascular probe, as a significant amount of excitation light will be absorbed by the blood present in the artery. Special attention was given to NIR wavelength range (900 nm – 1300 nm) as in this region blood absorption is relatively weak and there are expected to be a significant difference in absorption spectrum of each tissue type. The NIR spectral response shows spectral differences between normal and fatty tissue (*Fgiure 5.)*

NIR imaging has vast applications like monitoring time-related changes that occur during red wine fermentation by Buratti S. and Ballabio D. et al. (2011), application and interpretation of measurement devices, and procedures for determination of temperatures of the human body by Warner J. (2014), investigation of oxidative stress in irradiated skin by Albrecht S. and Ahlberg S. et al. (2016).[1]

2.5 Pharmaceutics

NIR spectroscopy in pharmaceutical environment: ranging from identification of raw materials to the online quality control of finished and packed end product. Without physical contact with compounds to be measured, NIR technique analyzes synthesis of compounds so suitable for toxic drug screening.
Work related to use of NIR spectroscopy was carried out GEA-NUS pharmaceutical Processing Research Laboratory, Department of Pharmacy, National University of Singapore, Singapore. It concentrated at in-line quantification of micronized drug and excipients in tablets by near infrared spectroscopy for real time monitoring of tableting process. The research work was carried out by Karande A., Heng.P. et al. (2010) with the objective to access utility of near infrared spectroscopy. Direct compression tablet formulations comprising micronized chlorpheniramine maleate, lactose, microcrystalline cellulose, and magnesium stearate were used. This study demonstrated that the unique feature of NIR for high speed sampling and rapid spectral acquisition enabled the quantification of individual tablet's components with a high degree of accuracy during tableting. Due to inclusion of maximum possible variability encountered during tableting at the calibration stage, more sensitive prediction models were obtained which ultimately gave predictions of high accuracy.
Many research work involving molecular chemistry in pharmaceutics and multidisciplinary domain have been recorded by various researchers and deserve mention like Rantanen J. (2000), Otsuka M. and Mouri Y. (2002), Roggo Y. and Chalus et al. (2007), Mantanus J. and Ziamons E. et al. (2009), Isao and Noda (2010), Corredor C.C. (2011), Manara D. and Bruycker F.D. et al. (2012), Begui S. and Begei G. et al. (2015) etc.

2.6 Petrochemical industry

Department of Chemistry and Applied Biosciences, Zurich, Switzerland under Balabin R., Safieva R. et al. (2010) conducted an experiment for Gasoline classification using near infrared spectroscopy data. It compares various standard chemical techniques against NIR spectrometry for multivariate content analysis in petroleum industry. The use of NIR spectroscopy for multi component chemical systems, including product of petroleum refining and petrochemicals, food products (tea, fruits, milk, wine etc.) and combustion products were studied. In all cases NIR spectroscopy is found to be the most effective. It is most effective for gasoline classification purpose, when compared with Nuclear Magnetic Resonance (NMR) spectroscopy or gas chromatography. Artificial Neural Network approach based on Principal Component Analysis was found to be most efficient tool box in the study.

At Department of Chemical Biology and Applied Chemistry, Nihon University, Japan (2011) an experiment for Quantitative analysis of alcohol-water binary solution using Raman spectroscopy has given promising results. Band ratios between Raman bands of the target molecule and that of aceto-nitrite as an external standard were calculated and found to be proportional to the mass fraction of alcohols. Raman spectra of methanol-water, ethanol-water and methanol-ethanol binary solutions were measured to determine the mass fraction of alcohol.

2.7 Environment and soil analysis

The most remarkable work to create a global spectral library to characterize the world's soil by R.A.Viscarra, Rossel T. Behrens et al. (2016) at CSIRO, Canberra, Australia in a joint venture with other 32 countries has recently been recorded. Soil provides echo system services, supports human health and habitation, stores carbon and regulates emission of greenhouse gases. Unprecedented pressures on soil from degradation and urbanization are threatening agro-ecological balances and food securities. To this end, an attempt is made to develop and analyze global soil vis-NIR spectral library. It is currently the largest and most diverse database of this kind. It shows that the information encoded in the spectra can describe soil composition and be associated to land cover and its global geographic distribution, which acts as a surrogate for the global climate variability. It shows the usefulness of the global spectra for predictive soil attribute such as soil organic and inorganic carbon, clay, silt, sand and iron contents, cation exchange capacity and pH. Using wavelets to treat the spectra, which were recorded in the different laboratories using different spectrometers and methods, help to improve the spectroscopic modeling. It finds that modeling of diverse set of spectra with a machine learning algorithm in finding the local relationship in the data to produce accurate prediction of soil property. It concludes that the spectroscopic approach should help to deal with a shortage of data on soil to better understand it and to meet the growing demand of information to access and monitor soil at scales ranging from regional to global. The outcomes of the research can be extended to soil, earth and environmental sciences towards application that have not yet been dreamed of.

Soil Organic Carbon (SOC) is an important parameter in the climate change mitigation strategies and it is crucial for the function of ecosystems and agriculture. A focused research on ecosystems by Maria Knadel, Anton Thomsen et al. (2015) at Dept. of Agroecology, Arhus University, Denmark is a noticeable work, which has generated the database of the ecosystems presentlation. Particle size fractions affect strongly the physical and chemical properties of soil and thus the SOC. Conventional analysis of SOC and particle size are costly limiting the detailed characterization of soil spatial variability and fine resolution mapping. Mobile sensors provide an alternative approach to soil analysis. They offer densely spaced geo-referenced data in a cost effective manner. In this study, two agriculture fields (Voulund1 and Volunud2) in Denmark were mapped with the Veris Mobile Sensor Platform (MSP). MSP collected simultaneously visible near infrared spectra, electrical conductivity and temperature measurements. Fuzzy k-means clustering was applied to obtain spectra to partition the field and to select representative of samples for calibration purposes. Calibration samples were analyzed for SOC and particle sizes (Clay, SILT and sand) using conventional wet chemistry analysis.

2.8 Diamond and Optics

Germological Institute of America, Carlsbad, United States under researchers Sally Eaton Magana, Troy Ardon et al. (2016) conducted a novel experiment to check the effect of temperature on luminescence in natural type of llb diamond. Blue diamond is amongst the most rarest and most valuable of naturally occurring gemstones. In this study, 12 rough, naturally sourced type llb diamonds were subjected to HPHT annealing, three different irradiation energies, and then all were stepwise annealed from 200 ˚C to 1100˚C and the optical defects were documented by changes in phosphorescence and photoluminescence spectroscopy. Several optical features that are removed from natural type llb diamonds by HPHT processing such as 3H, 648.2 nm peak, 776.4 nm peak and 660 nm band phosphorescence, can be reintroduced into these diamonds with subsequent electron irradiation and annealing at low to moderate temperature as shown in *Figure 6*.

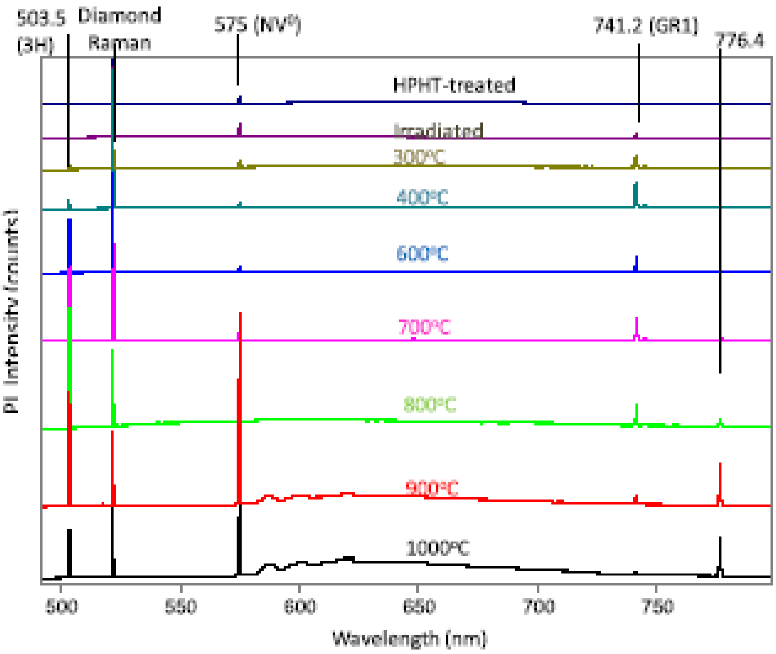

Figure 6. PL spectra at liquid nitrogen temperature after each pre-treatment and annealing steps on diamond.

The penetration of NIR spectroscopy is first recorded in optic industry in the experimental set up at the Laboratory of Material Science, Department of Chemical Engineering, University of Applied Science, Germany by Stephanie Moller and Arturas Katelnikovas et al. (2015). A new luminescent material for the conversion of blue light to near infrared radiation is presented. The new phosphor $Sr_2Si_5N_8$:Eu, Nd displays a large absorption cross section in the blue spectral region and allows stable NIR operation when combined with blue emitting (In, Ga)N LEDs, despite the high excitation densities and elevator temperature near the LED chip. The luminescence properties of the material were measured as a function of the energy, time, and temperature and excitation density. In contrast to the state of the art (Al, Ga)As NIR LEDs with suffers from severe thermal quenching and shift of the peak emission already at around 100˚ C, the presented phosphor system displays high emission intensity with constant spectral power distribution up to 180° C as shown in *Figure 7.*

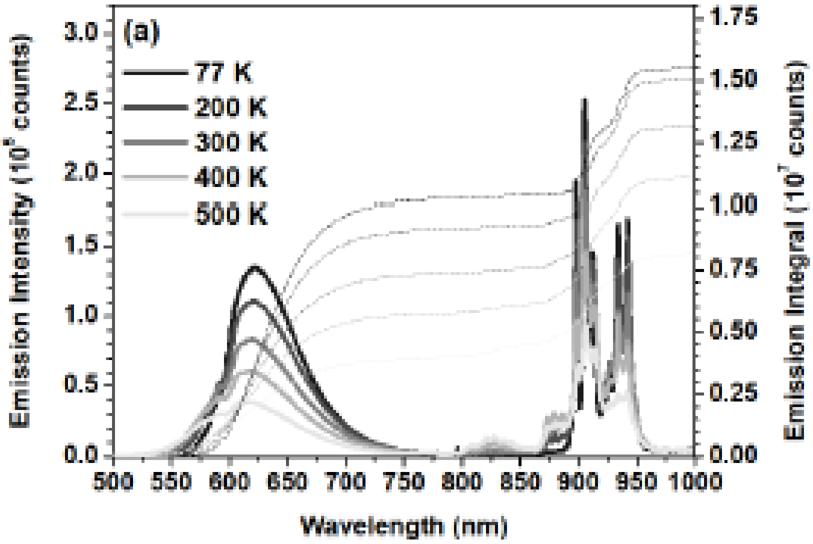

Figure 7. Emission spectra recorded at different temperatures

III. CONCLUSION

NIR spectroscopy is a valuable tool for detecting adulterant, as well as performing characterization and classification of industrial product. Due to advancement in instrumentation and computer software this technique is more popular due to faster response. Rapid analyses without the need of sample preparation present one of the main advantages of this technique. However, the accuracy of this method is directly affected by fluctuation of temperature and sample movement. NIRS combined with appropriate chemometric methods should be required for assessing quality in industrial conditions. For better accuracy for online monitoring , more robust calibrations are needed with regard to better sampling procedures and improvement of reference method and large data set is required.

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