

NIR SPECTROSCOPY: TECHNOLOGY READY FOR FOOD INDUSTRIES APPLICATIONS

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ABSTRACT

Recently, NIR spectroscopy has gained wide acceptance in different fields like drug and pharmaceuticals, petroleum and food industries. NIR technology is going to be the substitute technology over conventional chemical test methods due to advancements in instrumentation technology, availability of open source tools, and developments in photonics components. The ability of the spectrophotometers to record spectra in almost no time makes this technology most suitable for online analysis. Genetically Modified Organism (GMO) being the most serious concern these days can be addressed and dealt with using NIR spectroscopy. This article focuses on the potential capabilities of this technology in food industries

KEYWORDS: NIR Spectroscopy, Food Analysis

INTRODUCTION

The recent development in instrumentation has supported the extensive penetration of Near Infra Red (NIR) Spectroscopy and imaging. NIR Spectroscopy is a fast and nondestructive analytical technique that provides chemical and physical information of any complex mixture. Using multivariate analysis, both quantitative and qualitative analysis could be done in no time. NIR spectroscopy has been gaining wide acceptance in varieties of applications like Food and Drug Industries, Petroleum, Pharmaceuticals, agriculture and many more.

MOTIVATION FOR FOOD GRAIN ANALYSIS

Food producing industries are always interested in quality assurance across the supply chain. Globalization and rising competitiveness forces producers to take strategic action for quality control and product optimization. Quality control and quality assurance are typically performed using classical chemical methods. Such methods usually take considerable time to produce results and involve utilization of harmful chemicals for sample preparations. Consequently, the results are not obtained quickly enough to meet the needs of operations. Modern analysis techniques and software can provide an even better inside view into your raw materials and products within no time almost as if online.

Food composition databases provide detailed information on the concentrations of nutrients and nutritionally important components in foods. Calculating the nutrients facts from each its ingredient and its percentage is a need of food industry so crop yield would earn well for farmers as well as end users. The development of new and increasingly sophisticated techniques for the authentication of food products continues apace with increasing consumer awareness of food safety and authenticity issues. Food authentication is also of concern to food processors that do not wish to be

subjected to unfair competition from unscrupulous processors who would gain an economic advantage from the misrepresentation of the food they are selling.

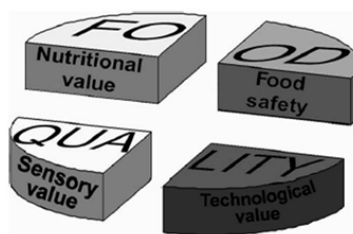


Figure 1: Factors Influencing Food Quality

It is most important to also know the biological values of the food in terms of its sensory values like Genetically Modified Organism (**GMO**). The need of knowing the GMO values are felt and being researched in most agriculture universities. Considering all such aspect, various techniques for the analysis of food are gaining significance in recent past.

CONVENTIONAL TECHNIQUES OF FOOD ANALYSIS

The conventional techniques adopted for food content analysis like fat, protein, carbohydrate etc. are standard chemical techniques developed and approved globally. Table-1 reflects standard tests adopted for some of the food constituents.

The important test for Protein which is approved is Kjeldah test which is comprised of three stages like Digestion, Distillation and Titration. The N-H bonds in protein by use of Sulphuric acid are broken and converted into ammonia (NH_3) and which in turn by means of a distillation process is separated from liquid phase. Titration is used to find the concentration of Protein in the sample. As the test itself is comprised of unit operation and process, it consumes time to deliver the results and a tedious process too. Hence in cases where online analysis is must, NIR is the only solution has been popularly accepted for food analysis.

Table 1: Recommended Chemical Tests For Food

Constituent	Method	Reference Standard
Protein	Kjeldah	Association of American Cereals and Chemists (AACC)
Oil	Single stage air oven	AACC
Moisture	Goldfisch Extraction	AACC
Crude fiber	Extraction	AACC

HISTORICAL BACKGROUND

Near Infrared radiation was discovered by Friedrich Wilhelm Herschel in 1800 (Davies, 2000) and covers by definition wavelength range from 780 to 2500 nm. When radiation hits 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.

Although *Herschel* discovered light in the Near Infrared (NIR) region as early as 1800, even spectroscopy of the first half of the last century ignored it in the belief that it lacked analytical interest. The Earliest application of NIR was reported in the 1950s, but it was not until the 1970s that the group headed by *Noriss* used it to analyze agricultural food samples. The development of equipment featuring improved electronic and optical components and the advent of computers capable of effectively processing the information contained in NIR spectra facilitated the expansion of this technique in an increasing number of fields [15].

The literature reflects the changes in the potential and the appreciation of NIR spectroscopy. Thus Wetzel in 1983 deemed it a " Sleeper among Spectroscopic Techniques " on account of high potential with scan use [14]. McLure in 1994 published a paper entitled "The giant is running strong" because of its increasing number of applications in different field. Davies in 1998, putting forward future possible advances and applications, aptly described its potential as taking it "from sleeping technique to the morning star of Spectroscopy"^[7].

The interest in NIR spectroscopy lies in its advantages over alternative instrumental techniques. Thus it can record spectra for solids and liquid samples without any pretreatment, implement continuous methodologies, provide spectra quickly and predict physical and chemical parameters from a single spectrum. These attributes make it especially attractive for straightforward, speedy characterization of samples.

BASIC PRINCIPLE OF NIR SPECTROSCOPY

The American society of Testing and Materials **ASTM** defines the NIR region of Electromagnetic spectrum as the wavelength range of 780 to 2500 nm corresponding to the wave number range $12820\text{--}4000\text{ cm}^{-1}$. The most prominent absorption bands occurring in the NIR region are related to *overtone*s and combinations of fundamental vibrations of -CH , -NH, -OH (and -SH) functional groups. Figure 2 shows the NIR spectrum and chemical composition response at effective wavelengths.

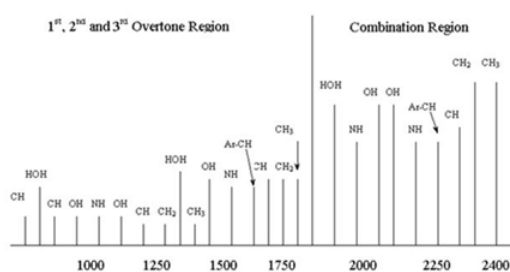


Figure 2: Constituent Response at Wavelength

The light in NIR band when irradiated on a sample under inspection, following three NIR absorbance phenomenons take place.

Transmission

As has been clear from the Figure 3, the light in the NIR range falls from the source onto the sample and while passing through the sample, the sample depending on its constituents will absorb wavelength specific light which upon measurement from other side of the source will give result analysis. It is related to 1100 - 1800 nm wavelengths and used for high water content samples.

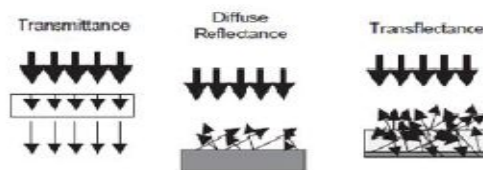


Figure 3: Transmittance, Reflectance and Transmittance

Reflectance

In case of these phenomena, the light returned back due to the reflective properties of the sample would be measured for prediction of sample analysis. This phenomena covers 1800 - 2500 nm wavelength and used for earth samples and solids analysis.

Transflectance

A Transflectance phenomenon is the combination of Transmission and Reflectance as clear from the figure. Hence reflected as well as transmitted light from the sample will be used for sample analysis predictions. Transflectance occurs in 800 - 1100 nm wavelength and used for slurries, grains and thick sample analysis.

RESEARCH TRENDS IN FOOD ANALYSIS

A comprehensive aspect on literature survey has been presented here to throw light on trends in research in the food industries.

The research article “analytical methods for detection and determination of genetically modified organism in agriculture crops and plant-derived food products” by Elke Anklam et. al. bring out significance of NIR spectroscopy for identification of GMO^[7]. The potential economic impact of GMO commingling in the supply and marketing chain has been explored and scope of NIR reflectance spectroscopic analysis in determination of GMO has been established.

Another research article titled “Remote sensing technique for predicting harvest time of tomatoes” by Haiqing Yang has touched various growing stages and harvest time of fruits and vegetables for horticulture automation^[18]. He has proposed Growing Stage index for predicting harvest time of tomatoes of three cultivars. NIR Spectral response was measured to calculate the GS index of vegetables. It has been concluded from the experiment that VIS-NIR spectroscopy combined with optimized PLSR models for GS prediction can be successfully adopted as a remote sensing technique for predicting harvest time of tomatoes, which allows for implementing autonomous fruit picking robots.

“Freshness measurement of Eggs using NIR spectroscopy and multivariate data analysis” by Hao Lin et al. At school of Food and biological engineering, Jiangsu University, China has also brought out significance of NIR techniques for food analysis at various levels^[33].

Linda M.Reid, Colm P.O'Donnell et. al. in their research article, “Recent technological advances for the determination of food authenticity” have focussed their study on European system for developing and protecting food stuff^[35]. The article has discussed in brief the relative potential of various technologies for the confirmation of food authenticity and quality. Near Infrared spectroscopy and its scope in the determination of food authenticity has been explored. They have concluded the continuing development of applications for more established techniques shall focus on potential of NIR techniques with chemometrics analysis.

Similar research study by Bart M. Nicolai et.al. at the Flanders centre of Postharvest Technology, Belgium has thrown light on usefulness of NIR technique in their research article “Non destructive measurement of fruit and vegetable quality by means of NIR spectroscopy”^[36]. The objective of the study was to identify the success of online systems for grading fruit and vegetables which in turn adds the commercial value to the fruits. The premium customers who are ready to pay premium prices for e.g. extra sweet fruit, will help them pick up the best product and in turn will allow auction house to have grading lines of their produce.

A very interesting research article “How to measure and predict the molar absorption coefficient of Protein” by C. Nick Pace et. al. At Department of Medical Biochemistry and Genetics, Texas A&M University, Texas has laid a foundation stone on content determination for multivariate content analysis in food engineering^[38].

An overview on the current research trend on food industries using NIR reflectance spectroscopy reflects various aspects of fruits, vegetables and crops. To cater to the requirement of quality based ranking of fruits and vegetables, NIR can be a handy solution. The advancements in NIR reflectance spectroscopy makes it most preferred tool for quality ranking of fruits and vegetables. From the food safety aspect, significant research in agriculture science has started and picking up the acceleration. In coming years, NIR spectroscopy is going to be the most accepted and preferred substitute over conventional chemical methods. The acceptance of NIR spectroscopy for various food application has been growing mainly because of its non-invasive and quick analysis compared to conventional chemical methods.

ADVANTAGES OF NIR SPECTROSCOPY

- It is a non-invasive, non-destructive technique.
- It requires minimal or no sample preparation. Solid samples can be directly measured with the little pretreatment, or no pretreatment, if an appropriate device is used.
- Measurement and result delivery are quite fast almost online. Dramatic developments in NIR equipment and chemo metrics used in conjunction with computers have enabled the real time extraction of analytical information from sample.
- There is no need for reagents or materials to prepare samples and the automation of technique results in increased throughput, which in turn, reduces analytical costs and decreases amortization time.
- A single spectrum allows several analytes to be determined simultaneously.
- The technique allows determination of Non-chemical (physical) parameters. In fact, the influence of some parameters on NIR spectrum allows the ready determination of properties such as density, viscosity and particle size.
- Because of great strength of optical materials and robustness of NIR equipment, which in most cases has no moving parts, NIR instrumentation is most suitable for use in process control at production level.
- Fiber optics provides robust, strong sensors for at-line, on-line and in-line analysis to control processes.
- NIR spectroscopic results are comparable in accuracy to those of other analytical techniques, also, their precision is usually higher, because there is no need for sample treatment.

- Disadvantages of NIR Spectroscopy
- NIR measurements are scarcely selective so chemometrics techniques have to be used to model data from which to extract relevant information.
- There are no accurate models to take account of the interaction between NIR light and matter. As a result, calibration is purely empirical in many cases.
- Accurate, robust calibration models are difficult to obtain as their construction entails using a large enough number of samples to encompass all variations in physical and/or chemical properties.
- The need to incorporate the physical and chemical variability of samples in calibration entails using as many as different calibration models as there are sample types, and hence more than one model per analyte.
- The technique is not very sensitive, so usually it can be applied only to major components.
- The NIR spectroscopy is a relative methodology, to construct models using it requires prior knowledge of the value for the target parameter, which must be previously, determined using a reference component.
- The construction of NIR models requires substantial investment, which can, however, be amortized by transferring calibrations from the master equipment to several slaves^[15].

CONCLUSIONS

A comprehensive study of trending research in the field of Near Infrared Spectroscopy has bring to the fore front the enormous potential of NIR chemo metrics especially in the filed of food and agriculture industries. The emphasis shall be laid down on validation and harmonization of quantitative methods to address the compliance GMO objects. Similarly, NIR chemometrics can be preferred for the analysis of multivariate content samples over conventional chemical methods.

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