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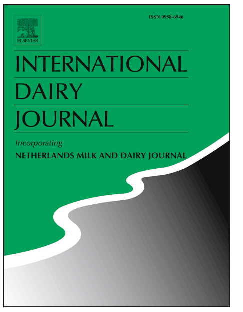

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Yuan-Yuan Pu, Colm O’Donnell, John Tobin, Norah O’Shea

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Review of near-infrared spectroscopy as a process analytical technology for real-time product monitoring in dairy processing

Yuan-Yuan Pu [a], Colm O'Donnell [b], John Tobin [a], Norah O'Shea [a]*

[a] *Food Chemistry and Technology Department, Teagasc Food Research Centre, Moorepark, Fermoy, Co. Cork, Ireland*

[b] *School of Biosystems and Food Engineering, University College Dublin, Belfield, Dublin 4, Ireland*

*Corresponding author. Tel.: + 353 1 8059717

E-mail address: norah.oshea@teagasc.ie (N. O'Shea)

ABSTRACT

Real-time process/product monitoring can be achieved using suitable process analytical technologies (PAT) to improve process efficiencies and product quality. In the dairy industry, near infrared (NIR) spectroscopy has been utilised as a laboratory analytical method (off-line) for compositional analysis of dairy products since the 1970s. Recent advances in NIR technology and instrumentation have widened its applications from a bench-top analytical instrument to a promising PAT tool for on-line and in-line implementation. This review focuses on the use of NIR technology for real-time monitoring of dairy products, by briefly outlining the measurement principle, NIR instrument configurations, in-line sampling methods, calibration models development, some practical considerations for process installation, and current state of the art in on-line and in-line NIR applications (2012 to date) for continuous process monitoring in the production of dairy products. The challenges and additional resources required to improve production efficiencies using NIR spectroscopy are also discussed.

Contents

1. Introduction

The consumption of dairy products is expected to increase by 25% from 2015 to 2024 (IDF, 2019). This increasing demand is driving dairy processors to become more competitive and streamline processes to be more efficient. To achieve this, it is essential for dairy plants to implement real-time process monitoring to allow corresponding production lines to be controlled and optimised, while also ensuring the production of a high quality and consistent final product. The acquisition of real-time process information (i.e., operating conditions, quality attributes of processed materials) can be achieved by implementation of process analytical technologies (PAT) in the manufacturing processes. The concept of PAT was first introduced to the pharmaceutical industry by the US Food and Drug Administration (FDA, 2004), with the aim of supporting innovation and efficiency in pharmaceutical development, manufacturing, and quality assurance. Since then, the adoption and promotion of PAT initiatives have been widely spread across other related areas (Chew & Sharratt, 2010), particularly in the food industry including the dairy sector (O'Donnell, Fagan, & Cullen, 2014).

In the dairy industry, common PAT tools such as pH, temperature, pressure and flow instrumentation are installed at critical control points of a processing line to provide real-time operating information of the process (Tajammal Munir, Yu, Young, & Wilson, 2015). In recent decades, the importance for real-time measurement of chemical composition of in-process materials has been recognised by dairy processors as an important component to ensure end products meet desired quality specifications (Munir et al., 2017). For example, the protein-to-fat ratio (PFR) of cheese milk governs the coagulation process, cheese yield and final composition of the cheese (Sturaro, De Marchi, Zorzi, & Cassandro, 2015). It is important to maintain batch-to-batch consistencies during cheese manufacturing with the use of standardised cheese milk. The milk standardisation process can be optimised and controlled by real-time measurement of fat and protein content using suitable PAT tools, such as an in-line NIR spectrometer or in-line milk standardisers. The majority of in-line milk standardisation currently is carried out with at-line middle infrared (MIR) instruments (i.e., the MilkoScan™ or more recently MilkoStream™ instruments from FOSS, Hillerød, Denmark), as a result, the fat and protein content can be continuously adjusted to achieve a target PFR.

Near-infrared (NIR) spectroscopy has been identified as a powerful analytical technology for compositional analysis of a variety of dairy products, since its first application in the dairy industry in the late 1970s (Cattaneo & Holroyd, 2013). This technique can overcome the disadvantages of time-consuming and tedious laboratory analysis, offering a rapid (results derived in seconds), non-destructive (no sample damage), cost-effective (simultaneously measure multiple quality parameters) and environmentally-friendly (chemical-free and no waste disposal required) solution to meet the requirements of a fast-paced processing supply chain. In 2006, the International Organisation for Standardisation (ISO) together with IDF published an international standard, providing guidelines for the application of NIR spectrometry as an off-line analytical technology for quality measurement of several milk products (ISO & IDF, 2006). This standard was substantially updated and revised in 2019 to cover a wider range of dairy samples in different forms (i.e., liquid, semi-solid and solid). In addition, a Bulletin of the IDF (No. 497/2019) entitled "Applications of NIR spectrometry for the analysis of milk and milk products" was recently released (Niemöller & Holroyd, 2019). This document summarised unpublished calibration statistics originating from global dairy companies and NIR instrument vendors, to provide comprehensive and up-to-date information on NIR performance in the dairy industry. To meet current process requirements of quality by design (QbD), the latest NIR instruments are equipped with features that are suitable for real-time (on-line or in-line) process monitoring. The application and implementation of NIR technology has also started to move from the laboratory (off-line measurement) to production lines (at-line, on-line and in-line analysis), and from scientific research to industrial applications.

Currently, NIR technology has been successfully implemented in the pharmaceutical industry for process understanding, monitoring and control. Guidelines offered to the pharmaceutical industry regarding ‘development and submission of near infrared analytical procedures’ have been provided by the European Medicine Agency (EMA, 2014) and the FDA (FDA, 2015). However, the majority of the NIR implementation in the dairy industry is still off-line or at-line measurements.

Several other spectroscopic methods such as Raman spectroscopy (Yang & Ying, 2011), fluorescence spectroscopy (Shaikh & O'Donnell, 2017) and hyperspectral imaging (Manley, 2014) have been applied in the diary sector. This review will focus on the NIR technology and its recent applications as a PAT tool for on-line and in-line measurements during dairy processing. The review will cover several aspects including the basic principle of NIR spectroscopy, NIR instrumentation, in-line sampling, calibration development and some practical considerations for on-line and in-line implementation. Information listing global companies that provide on-line and in-line NIR solutions and the instrument specifications is summarised in Supplementary material Table S1.

2. Principle of NIR spectroscopy

2.1. Spectral region

Spectroscopy studies the interaction between light and matter. Light is a form of electromagnetic radiation, which contains a certain amount of energy. The energy (E) of the light depends on its frequency; the higher the frequency f of the light (or the shorter the wavelength λ), the higher the light energy E, as shown in Equation 1.

$$E = h \cdot f = \frac{h \cdot c}{\lambda} \qquad (1)$$

where h is the Planck constant (m^2 kg s^{-1}), f is the frequency of the light (s^{-1}), c is the light speed (m s^{-1}) and λ is the wavelength of the light (m).

Fig. 1 illustrates the electromagnetic spectrum that is divided into several spectral regions. Increasing the wavelength of the light from gamma waves to radio waves will result in the corresponding light energy decreasing. Visible light that can be perceived by the human eye covers the wavelength range from 350 nm to 800 nm. The whole infrared (IR) region is from 800 nm to 100 µm, and is sub-divided into three main regions: near infrared (NIR, 800–2500 nm), middle infrared (MIR, 2500 nm – 25 µm) and far infrared (FIR, 25–100 µm) (Dufour (2009)). The NIR region is the region that is closest to the visible right, thus, it is called 'near' infrared. Generally, the wavelength (in nm) is most frequently used for the NIR region and the wavenumber (in cm^{-1}) is used for the MIR region. The conversion between wavelength and wavenumber is given in Equation 2.

$$wavenumber\ (\mathrm{cm^{-1}}) = \frac{10^7}{wavelength\ (\mathrm{nm})} \qquad (2)$$

As a sample is exposed to a beam of IR light, the sample will absorb part of the light energy that can cause molecular vibrations. Each chemical bond (i.e., C–H, N–H, O–H, S–H) of a molecule has unique vibration modes (i.e., stretching, bending and rocking) and vibration frequencies (i.e., fundamental, overtones and combination; see Supplementary material for description and explanation). When the chemical bonds receive the light frequency that matches its vibrational frequency, the light energy will be absorbed. The concentration of absorbing chemical bonds in the light path influences the absorbance signal. As a result, the higher concentration, the more pronounced absorption signals being observed in the corresponding spectrum.

2.2. Why NIR spectroscopy

Both NIR and MIR spectroscopy are considered as rapid and chemical-free technologies. Table 1 compares NIR and MIR spectroscopy. NIR spectroscopy is more attractive for on-line and in-line applications since: (i) the NIR region has higher energy than MIR (as known from Equation 1) resulting in a larger penetration depth into the sample; (ii) NIR light can pass through materials such as glass, films and plastic materials without losing much energy, giving a major advantage over the MIR in measuring samples through these materials (Lin, Rasco, Cavinato, & Al-Holy, 2009); (iii) The NIR spectrum contains a large amount of information related to the overtone and combination bands of hydrogen bonds (i.e., C–H, O–H and N–H). Most organic materials are made of these bonds, allowing a wide range of organic samples in chemistry, pharmaceutical and agri-food industries to be suitable for NIR analysis (Manley, 2014). For example, the N–H vibration bands presented in the NIR spectrum of a milk sample are mainly corresponding to the protein molecules.

Nevertheless, NIR spectroscopy has its limitations. The NIR spectra are made up of overtones and combination bands of chemical bonds, the peaks are broad and overlapped which makes the spectral interpretation more difficult. NIR data exploration and interpretation rely heavily on the use of multivariate data analysis (chemometrics). The NIR technology cannot measure constituents/contaminations (e.g., melamine) that are lower than 0.1%, as a trace amount of contamination will have no measurable effect on the scatter properties of the samples (Norris, 2009).

2.3. Measurement modes

The interactions between light and matter include absorbance, reflectance and transmittance, as shown in Fig. 2. When the light (located in the NIR region) is passing through the sample, some parts of the light can be absorbed by the sample causing molecular vibrations, and the rest of the light is either reflected back towards the sample or transmitted through the sample, or a combination of both depending on the sample properties (i.e. sample density and chemical compounds).

According to the specific light-output captured by the detector in an NIR instrument, NIR spectroscopy can be performed in transmittance, reflectance, and transflectance (a mixture of transmittance and reflectance) mode, as shown in Fig. 3. The transmittance mode measures the light transmitted through a sample, with the detector located at the opposite side of the light source. The reflectance mode measures the light reflected by the sample, with the detector located on the same side of the light source. For the transflectance mode, a gold reflector (functioning as a mirror) is placed at the bottom of the sample, the transmitted light will be reflected back once it reaches the gold reflector (Núñez-Sánchez et al., 2016). As the light travels through the sample twice, the optical pathlength is double the sample thickness.

In dairy applications, for example, NIR light can penetrate through skim milk liquid easily, thus, the transmission spectra will provide more detailed compositional information on the sample. Compared with whole milk that has a fat content of 3~5%, light scattering effect could be observed due to the presence of different fat globules (Chen, Iyo, Terada, & Kawano, 2002), as a result, NIR spectrometers operating in either reflectance or transmittance can be considered for whole milk compositional measurement (Aernouts, Polshin, Lammertyn, & Saeys, 2011). Milk powders have a strong diffuse reflectance of the light, as a result, the reflectance spectrum carries more sample information compared with the transmission spectrum. The transflectance mode is very useful in applications where there is a significant physical and chemical change in the in-process materials. For example, in the fermentation process of yoghurt, the rheology of the in-process materials changes from liquid milk to semi-solid gel after the addition of microorganism for a certain period of time (Grassi et al., 2013). It should be noted that the penetration depth of the light is also wavelength-dependent (Aernouts et al., 2011), therefore, the selection of an optimal measurement mode of a NIR analyser is mainly based on the physical and chemical properties of a target sample (i.e., sample thickness, liquid or powder, clear liquid or emulsion) and the instrument specifications (i.e., spectral range and resolution). Supplementary material Table S1 gives more details on the acquisition modes of NIR instruments and their suitable applications.

2.4. NIR instrumentation

There are five main types of NIR instruments available on the market, according to the wavelength selection methods (AB Vista, 2018; Agelet & Hurburgh, 2010); these are as follows.

(1) Filter-based device, which uses optical filters that are mounted in a rotating wheel to generate the specific wavelengths required

(2) Dispersive type of spectrometer, which employs an optical prism or a grating element to generate a set of continuous wavelengths, e.g., as a ray of white light passes through a prism, it can be split into a whole spectrum of wavelengths due to different light refractions

(3) Fourier-transform (FT) spectrometer, which is based on the theory of Michelson interference, the light source is split and reflected by two mirrors, and then re-combined to interact with the sample. The resulting signal recorded by the detector is the light intensity as a function of time (called 'interferogram'). Using Fourier-transform, the optical signal can be converted from time to frequency domain, as a result, a spectrum (light intensity as a function of frequency or wavelength) can be obtained. Many of the modern NIR instruments are FT-NIR instruments since the spectra generated are of a higher spectral resolution and quality.

(4) Diode-array type of instrument, which measures all the wavelengths at the same time using a fixed grating element with a dedicated diode detector for each wavelength (AB Vista, 2018), resulting in a high measurement speed.

(5) Micro-electro-mechanical systems (MEMS) type of spectrometer (Schuler, Milne, Dell, & Faraone, 2009), which is a small, compact and cost-effective NIR device based on semi-conductor technologies (Agelet & Hurburgh, 2010). This type of device can be used to fabricate portable / handheld

NIR instruments that are suitable for on-site measuring applications. As an example, Fig. 4 shows the working principles of the filter-based, dispersive and Fourier-transform types of NIR instruments.

3. Analysis of NIR data

3.1. Spectral profiles of dairy products

Fig. 5 illustrates the typical NIR spectra of three different dairy powders: skim milk powder (SMP: protein content, 34%; moisture content, 4.0%), milk protein concentrate (MPC: protein content, 86%; moisture content, 5.6%) and whey protein isolate (WPI: protein content, 90.1%; moisture content, 6.9%). The spectra were collected using a FOSS-NIR 6500 system operating in the reflectance mode (FOSS UK Ltd., Warrington, UK). The spectral range is from 1100 nm to 2500 nm with a spectral interval of 2 nm. As observed in Fig. 5, the absorption bands in the NIR region are broad and overlapped. The water absorption bands due to the vibration of O–H bonds are found in the region of 1440–1470 nm and 1920–1940 nm (Manley, 2014). The protein absorption band at around 2172 nm can be attributed to the combination of C–O stretching, N–H bending and C–N stretching (Manley, 2014). The absorption band at 2274 nm is possibly due to C–H and O–H vibration from lactose (Holroyd, 2013). More specifically, the NIR band assignments for different dairy products (cheese, liquid milk and milk powder) were summarised in a table in the review paper by Holroyd (2013).

3.2. Multivariate data analysis

An NIR spectrum generally consists of hundreds of wavelengths and the wavelengths are considered as variables. For example, there are 700 variables (wavelengths) in the NIR spectra of dairy powders as shown in Fig. 5. In addition, the spectra are very complicated due to the overlapping of the overtones and combination bands. Thus, multivariate data analysis (MDA) is required to extract meaningful information from the NIR spectra of the sample. Common spectroscopic data analytical approaches include spectral pre-processing, spectra exploration, regression (quantitative analysis) and classification (qualitative analysis), as summarised in Table 2. More details about multivariate data analysis and chemometrics in relation to spectroscopic data analysis can be referred to in papers by Rinnan, Berg, and Engelsen (2009), Roggo et al. (2007) and Xiaobo, Jiewen, Povey, Holmes, and Hanpin (2010).

3.3. Development of a calibration model

Before NIR spectroscopy can be employed in industry, a robust calibration model should be developed to ensure prediction results are accurate. A number of factors can influence the robustness of the model, i.e., the accuracy and reproducibility of the reference method, the sampling method used to collect representative samples, the spectra pre-processing methods, the quantitative or qualitative modelling methods. Fig. 6 illustrates a general procedure for the development of a calibration model. Samples in the calibration set are used to develop the calibration model, an external set of samples are used to evaluate and validate the model performance. For quantitative modelling, the quality composition parameter (i.e., protein, moisture, fat and total solids) is the dependent variable Y (results obtained from the reference method), the spectral signal/intensity at each waveband is the independent variable X (X_1, X_2, …, X_n). For qualitative analysis (i.e., classification analysis), the sample class would be the dependent variable Y.

According to the IDF standard (ISO & IDF, 2006), typically at least 120 calibration samples are required for a robust model with the use of MLR and PLS techniques. For validation of the calibration models, at least 25 samples (a test set) that are independent of calibration samples are needed. A global or a local model can be developed based on the data used in calibration development. For example, Melenteva, Galyanin, Savenkova, and Bogomolov (2016) developed a global model (i.e., resistant to seasonal, geographical and genetic variation on milk composition) for measurement of fat and total protein content in raw milk, based on historical spectroscopic data that were collected from different cow breeds located in different regions over a specific time period.

Once a robust calibration model is developed, it should be regularly validated and updated to ensure model prediction performance. A model that is developed on one NIR instrument might not be used directly in another similar NIR instrument due to variations in instrument components and the detecting environment. In this case, standardisation and calibration transfer techniques as mentioned in the review paper by Fearn (2001) can be applied to ensure model transferability for a wider model application.

3.4. Evaluation of a calibration model

The model prediction performance can be evaluated using statistical criteria such as coefficient of determination (R^2), root mean square errors for prediction (RMSEP), bias, standard error of prediction (SEP), and the ratio of prediction to deviation (RPD; RPD = standard deviation (SD) / SEP or RMSEP). The calculations behind these prediction indices are outlined by Porep, Kammerer, and Carle (2015). For the same calibration range, a higher R^2 value that is close to 1 (or a lower prediction error that is close to 0) indicates a greater prediction performance of a model. Table 3 provides guidelines for the model prediction performance in terms of the R^2 and RPD values. An R^2 of over 0.92 is generally accepted for most applications including quality control (Williams, 2017).

4. NIR applications

4.1. NIR application in dairy processing

NIR spectroscopy applications in dairy processing include off-line (laboratories), at-line, on-line and in-line installations (FDA, 2015); the main differences between which are as follows.

(1) Off-line: samples are manually taken-off the process line and tested in a bench-top NIR system located in the quality assurance/quality control (QA/QC) laboratory located away from the process line.

(2) At-line: samples are removed from the process line and analysed in an NIR system placed near the process line.

(3) On-line: a sample by-pass is created at a sampling point to divert the process materials from the mainstream for NIR analysis.

(4) In-line: the NIR analysis is directly integrated into the processing line by the use of different sampling strategies (in-line sampling will be discussed later).

Off-line and at-line measurements require manual sampling and consequently there is a time delay between sampling and measurement that does not facilitate real time monitoring. On-line and in-line analysis does not require manual sampling, which is a major advantage for measuring samples that are processed under high temperature and pressure conditions. In addition, the on-line and in-line measurement results can be obtained in seconds, which provides a real-time and continuous process measurement by 'bringing the instrument to the sample' (Cattaneo & Holroyd, 2013).

4.2. In-line sampling

The configuration of in-line NIR sampling contains three parts: (i) the NIR analyser; (ii) hygienic-design optical interface (i.e., probes, flow cells, or non-contact sensor heads) to be used in the process line; (iii) fibre optic cables for the connection between the NIR analyser and the sensing elements to achieve remote control and analysis (Hitzmann et al., 2015). During dairy processing, raw materials, intermediates and final products are either stored in tanks / vats or transported by pipes / conveyor belts around the plant. Thus, in-line spectroscopic sensors can be mounted in the production line with either an immersion probe, by a flow cell or through an optically transparent window in the NIR range. Fig. 7 summarises a variety of in-line sampling methods in specific applications.

4.3. Practical considerations for process installation

Currently, most of the NIR applications in the dairy industry are off-line or are at-line measurements. Moving NIR technology from a well-controlled measuring environment (samples are measured at static state and at a controlled temperature) to a more practical environment (samples are measured in flow conditions) can present many challenges, i.e., the effect of process conditions (e.g., temperature and flow) on the prediction performance and transferability of the calibration models. For example, the temperature of the samples and operating environment may vary during the production, which can influence the repeatability of the spectral signals collected, resulting in a reduction in the prediction accuracy of the calibration model. Therefore, it is essential to identify variables that might affect the spectra and these variables (i.e., temperature) must be considered when developing a calibration model.

For on-line and in-line implementation, NIR spectrometers should be able to bear harsh operating environments such as high temperature, high humidity, process vibrations (Nikos, Serafim, & George, 2004). To avoid the effect of humidity, the NIR spectrometer can be placed in a protective cabinet. The installation of the NIR probes or sensors should not affect the processing line (e.g., not introduce external contaminants, not disturb the process flow). The probes and sensors should meet an appropriate hygienic standard (i.e., 3-A Sanitary Standards (3-A SSI), European Hygienic Engineering & Design Group (EHEDG) standards (EHEDG)) to ensure a high standard of food safety is upheld. For some specific process / product applications where a frequent internal cleaning and sterilisation is required, the sensors applied should always be fully capable for cleaning-in-place (CIP) and sterilisation-in-place (SIP).

The result generated from NIR analysis can be integrated into an industrial control system (i.e., the Supervisory Control and Data Acquisition (SCADA) system) for monitoring and to achieve continuous process control and optimisation. However, the cost for integration and communication between the NIR system and the control system should also be considered for real-time industrial applications.

5. Recent applications in dairy processing

NIR spectroscopy can be applied across the whole dairy processing chain, i.e., (i) to check the quality of raw milk on a farm and at milk intake points, (ii) at-line, on-line and in-line monitoring of products along production lines, (iii) routine off-line quality measurement conducted in QA/QC laboratories, (iv) determination of final products to meet quality specifications. This section of the review focuses on the application of NIR technologies for on-line and in-line measurement in the dairy industry. Most of the industrial-level applications are rarely reported in literature, possibly as the data are confidential and commercially sensitive (Munir et al., 2017). Therefore, this review summarises the information available from literature and companies' application notes, to provide an overview of the on-line and in-line NIR applications during processing at industrial-level, close to industrial-level (i.e., pilot scales), or laboratory-scale research which indicates the potential for future on-line and in-line industrial applications.

5.1. Compositional analysis of milk

Raw milk is the starting material of all dairy products. The changes in quality and composition of incoming milk can be due to seasonality, the animal feeding system used and stage of lactation, which can have a significant impact on the subsequent processes and products. As a result, continuous monitoring of incoming milk quality is required. Protein and fat are two nutritional components in liquid milk with high economic value. Bogomolov, Dietrich, Boldrini, and Kessler (2012) investigated the use of visible-NIR spectroscopy (TIDAS E, J&M Analytik AG, Essingen, Germany, 400–1000 nm) to quantitatively analyse fat and total protein in bovine milk. Milk samples were prepared using cream (fat source), skim milk (protein source) and a 10%-solution of lactose and water. The samples had a protein content ranging from 2.6 to 3.2% and fat content ranging from 3.0% to 4.0%. PLS regression was conducted on the raw spectra, an excellent prediction performance (R^2 > 0.94 and root mean square errors < 0.05%) for the fat and protein content prediction was derived from the model, demonstrating the potential of NIR spectroscopy to be suitable for raw milk analysis either in the laboratory or in-line measurement.

An in-line NIR instrument (PSS-1720, Polytec GmbH, Waldbronn, Germany) using diffuse reflectance was applied to a milking parlour on a farm in Germany (Melfsen, Hartung, & Haeussermann, 2012). The NIR spectra (n = 785) covering the spectral range of 851–1649 nm were recorded to predict fat (%), protein (%), lactose (%), urea content (mg L^{-1}) and somatic cell count. Promising results were obtained for the prediction of fat, protein and lactose content, with R^2 = 0.99, 0.98, 0.92 and a standard error = 0.09, 0.05, 0.06, respectively, while satisfying results were obtained for the prediction of urea (R^2 = 0.82) and somatic cell count (SCC) (R^2 = 0.85). Melfsen, Hartung, and Haeussermann (2013) investigated the robustness of in-line NIR calibration models for the prediction of raw milk composition (fat, protein and lactose) in three different farms during the milking process and over a six-month period (n = 3119). The authors reported that the prediction accuracy of the NIR calibration models on each farm could be improved once a validation set was completed using spectra from an external farm. Further improvements were observed after the inclusion of data sets from additional farms in the calibration set. The improved model performance was attributed to the diverse model developed from the farms that included more sample variations.

5.2. Dairy powders

Milk powders [i.e., whole milk powder (WMP), skim milk powder (SMP) and whey protein concentrate (WPC)] are widely produced final products in the dairy industry, as the milk powders have a high-value and are easy to store and transport. Holroyd, Prescott, and McLean (2013) reported on a number of industrial trials conducted in the Fonterra Co-operative Group Ltd (a major dairy processor in New Zealand) in which different on-line and in-line NIR instruments have been implemented and tested in the milk powder plants since the 1990s. Four on-line NIR systems (5500 series) from the FOSS company (Laurrel, MD, USA) were installed to measure powder samples from the surge hopper after the sifter. Two of the instruments were calibrated for moisture prediction only, giving an SEP of 0.07–0.09%. One instrument was also calibrated for fat and protein prediction; however, the result was not stable and difficult to maintain overtime. The authors also pointed out other challenges / issues experienced with the on-line NIR systems, including the sample presentation system and instrument tolerance to high operating temperatures. A fifth NIR system from the FOSS company was an in-line system consisting of a probe, a spectrometer and fibre-optic cables. The instrument was calibrated for moisture prediction of different milk powders, giving a standard error of validation from 0.09% to 0.14%. It has been highlighted that the calibration stability of the system was related to the powder type, flow and production rate. A fixed-filter NIR system from NDC Infrared Engineering (Maldon, Essex, UK) was installed in the plant for a number of years to measure moisture of skim milk powder and butter milk powder, the prediction accuracy (SEP) was 0.13% for skim milk powder and 0.17% for butter milk powder. A recent study carried out in Fonterra was the use of a Matrix-F in-line FT-NIR system from Bruker Optics for a two-month trial of

WMP. The system consisted of a spectrometer that was located in a temperature-controlled cabinet far from the process line, two reflectance probes and 10 m fibre optic cables to connect the probes to the spectrometer. Results demonstrated that the system was stable throughout the trials and could be used to measure the moisture (SEP = 0.03%), fat (SEP = 0.11%) and protein content (SEP = 0.07%) of milk powder accurately. For optimal or best results, powder flow over the probe area must be uniform and stable to ensure consistent powder sampling.

Among a variety of dairy powders, infant formula production requires the highest standard of quality. Cama-Moncunill, et al. (2016) employed multipoint NIR spectroscopy (MultiEye, Innopharma Labs., Dublin, Ireland, 1515–2170 nm) at laboratory scale to evaluate the carbohydrate and protein content of powdered infant formula under static and motion conditions. It was expected that an improved prediction performance would be achieved at the static condition (R_p^2 = 0.89~0.92) compared with in-motion conditions (R_p^2 = 0.73~0.90), which are more realistic to industrial applications. A greater prediction accuracy was obtained for carbohydrate prediction rather than protein, results indicated the potential of this NIR instrument for future in-line or on-line measurement of infant milk formula.

5.3. Cheese manufacturing

During cheese manufacturing, the determination of the optimal cutting-time of a formed milk gel is very important for producing high-quality cheese products. Lyndgaard, Engelsen, and van den Berg (2012) applied in-line NIR spectroscopy (Antaris MX FT-NIR Process Analyzer, Thermo Scientific, Waltham, MA, USA, 1000–2500 nm) for real-time measurement of the milk coagulation process. NIR spectra were collected during the coagulation process using a reflectance probe. PCA was applied to the spectral data to extract meaningful process information. Using component scores as a function of time, two kinetic models were developed, with one describing the whole coagulation process and the other describing three milk coagulation processes (k-casein proteolysis, micelle aggregation, and network formation). The models were successfully evaluated and validated using an additional 12 cheese batches to determine the cheese cutting-time. Nicolau, Buffa, O'Callaghan, Guamis, and Castillo (2015) used an in-line NIR light backscattering fibre optic sensor (CoAguLab, at 880nm, Reflectronics Inc., Lexington, KY, USA) to predict the clotting and cutting times during sheep cheese manufacture. However, the study was carried out at laboratory scale using 300 mL of sheep milk to simulate the coagulation process, further studies on validating the models at a larger scale were required. An improved in-line sensor (FluorLite^{TM} Milk Coagulation, Reflectronics Inc) integrating NIR (880 nm) and fluorescence (350 nm) was applied by Panikuttira, O'Shea, O'Donnell, and Tobin (2017) to optimise the milk coagulation process during cheese-making. Validation of the sensor at industrial level is currently under investigation. Another application of NIR spectroscopy in terms of cheese manufacturing was quantification of casein fractions and their genetic variants in reconstituted casein samples (Marinoni, Monti, Barzaghi, & de la Roza-Delgado, 2013). Results illustrated that the NIR techniques could potentially be used to select milk products for cheese-making, as milk rennet properties and cheese yield are affected by milk casein fractions and its content. Additional PAT tools based on different operating principles used on cheese manufacturing can be referred to in the review by Panikuttira, O'Shea, Tobin, Tiwari, and O'Donnell (2018).

5.4. Yoghurt and other products

In the production of yoghurt, the milk lactic acid fermentation process requires real-time monitoring for optimal control of the microbial counts, lactic acid and sugar concentration to ensure the consistency of the final yoghurt product. Grassi et al. (2013, 2014) used an FT-NIR spectrometer (MPA, Bruker Optics, Milan, Italy) with a fibre optic transflectance probe to directly monitor inoculated skimmed milk during the fermentation process. Though the study was conducted at laboratory scale, the results indicated that the NIR technology coupled with chemometrics [i.e., PCA and multivariate curve resolution-aternating least squares (MCR-ALS)] could capture critical process information that matched the off-line rheology and conventional quality parameters, demonstrating a greater potential of NIR technology for further application in the production of yoghurt. Svendsen, Cieplak, and van den Berg (2016) carried out seven milk fermentation batches at pilot scale using a 15 L in-house glass single-wall fermenter vessel. Batches 1–4 were maintained at nominal fermentation temperature of 35 °C, batch 5 was kept at 32 °C and batches 6-7 were conducted at 37.5 °C. A fibre optic reflectance probe (ABB Bomen, Quebec, Canada) was inserted in the fermentation broth for in-line and real-time acquisition of the NIR spectra

(1000–1800 nm). The spectra were pre-processed by SNV to remove the scaling effect caused by sample differences, then modelled by PCA for each fermentation batch. In addition, an in-line pH meter was also placed in the fermentation broth for pH measurement, as it is currently used in the dairy industry for yoghurt fermentation monitoring. By comparing the pH profiles and the scores plot derived from the PCA modelling of the NIR data, it can be observed that NIR spectroscopy is preferable for process monitoring as it provides more information related to the physical (i.e., textural differences of gel formed) and chemical changes (i.e., conversion of sugar to lactic acids) during fermentation, rather than using the an in-line pH meter that only gives uni-variate pH information of the process.

As reported in literature and from NIR companies' application notes, other applications of NIR spectroscopy in dairy processing include prediction of total solids, protein and the protein-total solids ratio after the final ultra-filtration process in whey protein concentrate (WPC) and milk protein concentrate (MPC) production (FOSS). In addition, an in-line NIR system (DA 7300, Perten Instruments, Stockholm, Sweden) was installed at the outlet of a butter churn for continuously measuring of moisture, butter-fat and colour during butter production. The real-time results could be used for quick adjustments of moisture content during butter production as well as for a complete documentation of the product quality (https://www.perten.com/Publications/Articles/DA-7300-In-line-NIR-for-butter-production).

6. Current challenges and future work

Currently, most of the NIR research reported for the dairy sector has been carried out in the laboratory (off-line) or at-line. For a successful implementation of in-line NIR spectroscopy at an industrial level, the following challenges have to been addressed.

(1) The NIR instruments need to be stable over a 10-year life time. They should also be able to cope with vibrations and variations in processing conditions (i.e., the temperature / humidity fluctuation of the process environment). Alternatively, the spectrometer can be placed in a protective cabinet with temperature control to reduce the effect of the process environment on the NIR signal.

(2) For those batch to batch type processes as mentioned in section 5.3 and 5.4 (i.e., milk coagulation in cheese manufacturing, milk fermentation in yoghurt production), the cost of the individual

NIR sensor should be taken into consideration, as a dairy processor may have many batches to be monitored at the same time. Alternatively, a multiplexed spectrometer which allows several process probes / sensors to be connected to the same spectrometer could be used for monitoring different batches.

(3) For calibration development, the in-line sampling points must be as close to the sensor as possible. Thus, the spectra captured by the NIR are matched to the reference samples taken from the process stream.

(4) Calibration models developed at laboratory-scale should be validated for further implementation at industrial scales to provide more reliable and accurate measurement results. Transferring calibration models between different on-line or in-line NIR instruments is also a challenge, as variations in instrument components (optics or new replacements) and detecting environment (temperature and humidity) may result in spectral differences between the same samples. Different standardisation techniques can be applied to adjust the calibration model by slope and bias correction, or to correct the spectra by spectral processing (i.e., SNV or MSC pretreatment) or standardisation (i.e., direct standardisation and piecewise direct standardization) (Fearn, 2001; Pu et al., 2017).

(5) Seasonal variations, geographical differences and changes in an animals' diet can have an effect on the chemical composition of raw milk, resulting in changes to the spectra of raw milk (Melenteva et al., 2016). As a result, the developed calibration models need to be routinely validated and updated to ensure accurate and robust prediction performance.

(6) Due to the complexity of NIR spectra, interpretation of the data requires the use of multivariate data analysis. Efforts need to be made for integration of the NIR result to the process control system for successful PAT implementation to achieve process monitoring and control.

7. Conclusions

The higher demand for global consumption of dairy products requires dairy processors to increase process efficiency, product quality and yield. NIR spectroscopy has been seen as a fast, non-destructive and chemical-free process analytical technology for real-time measurement of a variety of dairy products, from liquid, semi-solid to solid samples. Successful implementation of NIR technology in the production lines can bring many benefits to the dairy industry by improving the knowledge and understanding of the process through real-time process monitoring. Additional personnel upskilling is required for development, evaluation and maintenance of robust calibration models at industrial level to ensure the performance of a NIR analytical solution. Also, future work on the connection and communication between the NIR measuring system and the control system are required to close the loop of ‘process, monitoring and control’, and thus to achieve the goal of quality-by-design by real-time and continuous process automation.

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Figure legends

Fig. 1. The electromagnetic spectrum.

Fig. 2. The interactions between light and matter. (This figure is extracted from the Application Note – TIDAS P Milk Inspector, J&M Analytik AG, Essingen, Germany; http://www.j-m.de/2/main-navigation/applications/industry/milk/milk-inspector.htm).

Fig. 3. Three spectrum acquisition modes of NIR spectroscopy.

Fig. 4. Three main NIR instruments according to the wavelength selection methods: (a) filter-based instrument, (b) dispersive instrument, (c) FT-NIR instrument.

Fig. 5. Typical NIR spectra of three different dairy powders.

Fig. 6. Development of a calibration model.

Fig. 7. In-line sampling examples: (a) diffuse reflectance sampling for in-pipe homogeneous process streams, (b) spoon probe sampling for powders, (c) transmission probe sampling for in-tank clear liquids, (d) transmission cell sampling for in-pipe liquids [Images in this figure are extracted from Q-interline brochure- InSight Pro (http://www.q-interline.com/)].

Table 1

Comparison between NIR and MIR spectroscopy.

Parameter	NIR	MIR
Spectral region	800–2500 nm (shorter wavelength)	4000–400 cm^{-1} (longer wavelength)
Light energy	Higher	Lower
Vibrational frequencies	Overtones and combination	Fundamental
Spectral peaks	Broad and overlapped, weak intensity	Sharp, strong intensity
Peak assignment	Not straightforward	Can be assigned to specific functional groups, providing 'fingerprint' information of the sample
Cost	Lower	Higher

Table 2

Analysis of NIR spectral data.

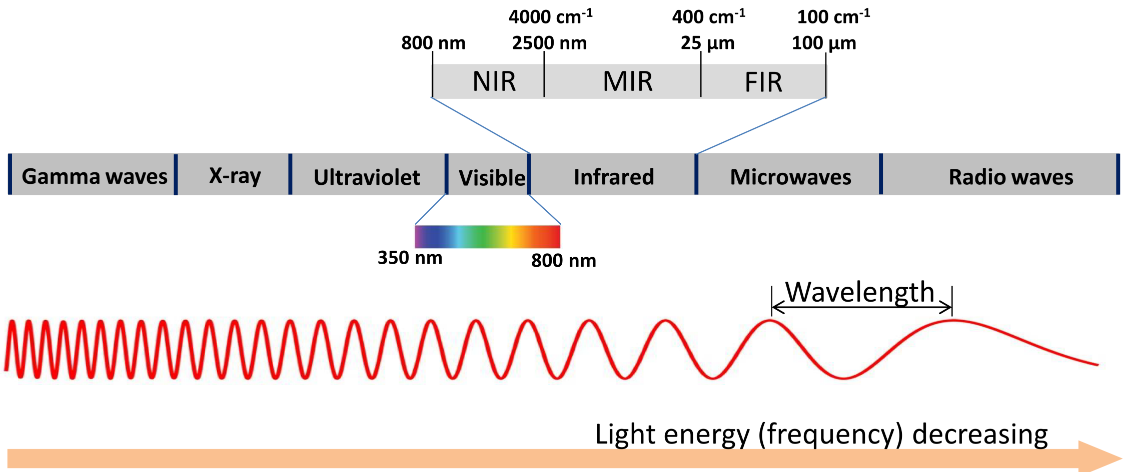
Analysis	Objectives	Most frequently used methods
Spectral pre-processing	To remove undesired information (i.e., noise), to improve the signal to noise ratio	Savitsky-Golay smoothing, Multivariate scatter correction (MSC), Standard normal variate (SNV)
Spectral exploration	To investigate the potential relations (i.e., grouping trends) between samples, to reduce data variates	Principal component analysis (PCA)
Regression (quantitative analysis)	To correlate the spectral data with the quantities of particular constituents (i.e., protein, fat, moisture) in the sample	Multiple linear regression (MLR), Partial least squares (PLS), Principal component regression (PCR), Artificial neural network (ANN) for regression
Classification (qualitative analysis)	To classify samples into different groups based on spectral differences, to identify out-of-specification samples (i.e., adulterated milk powders)	Partial least squares discriminant analysis (PLS-DA), K-Means clustering, Soft independent modelling by class analogy (SIMCA); Support vector machine (SVM)

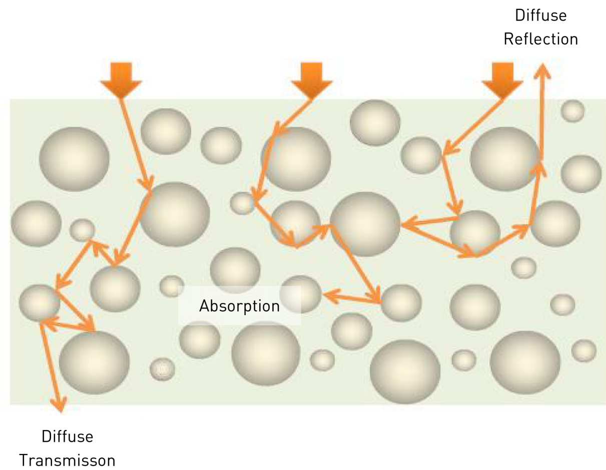
Table 3

Guidelines for model prediction performance indices. [a]

R^2	RPD	Interpretation of R^2
< 0.66	0.75	Not recommended: further research needed
0.66–0.81	< 1.7	Screening and some other ‘approximate’ applications
0.83–0.90	2.3	Usable with caution for many applications
0.92–0.96	3.6	Usable for most applications including quality control
> 0.98	> 5.0	Usable in any application

[a] This table was originally from Phil William who presented in the pre-conference course of ICNIRS 2017 in Copenhagen, Denmark. The authors have received permission from Phil William for the use of the table.


4000 cm-1
400 cm-1
100 cm-1
800 nm
2500 nm
25 µm
100 µm
NIR
MIR
FIR
Gamma waves
X-ray
Ultraviolet
Visible
Infrared
Microwaves
Radio waves
350 nm
800 nm
Wavelength
Light energy (frequency) decreasing


Diffuse
Reflection
Absorption
Diffuse
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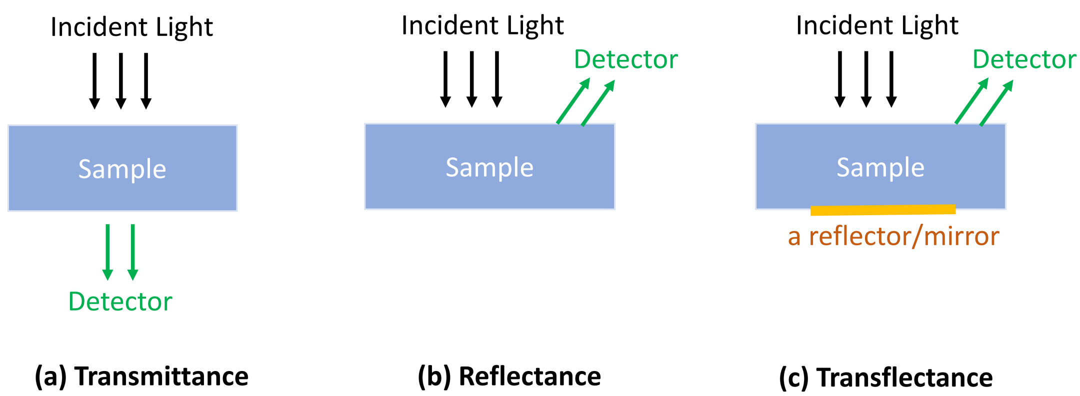
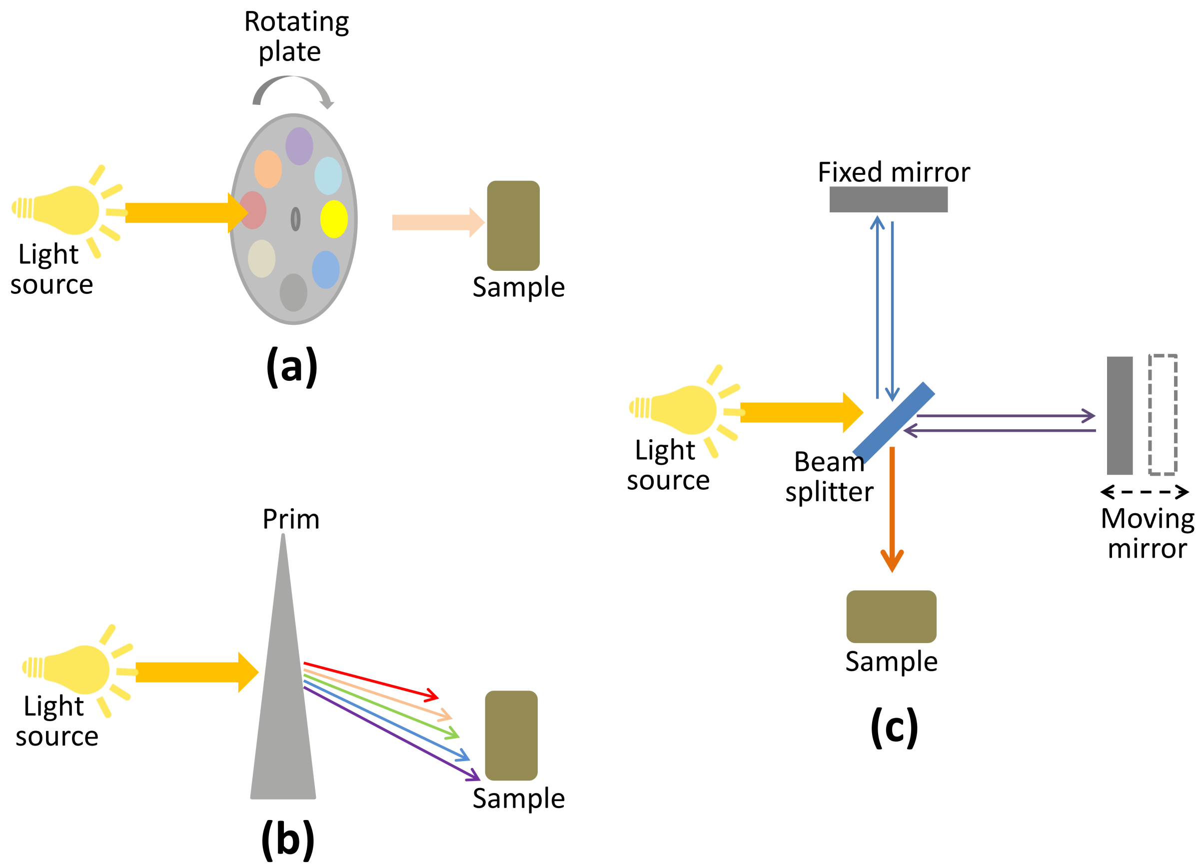
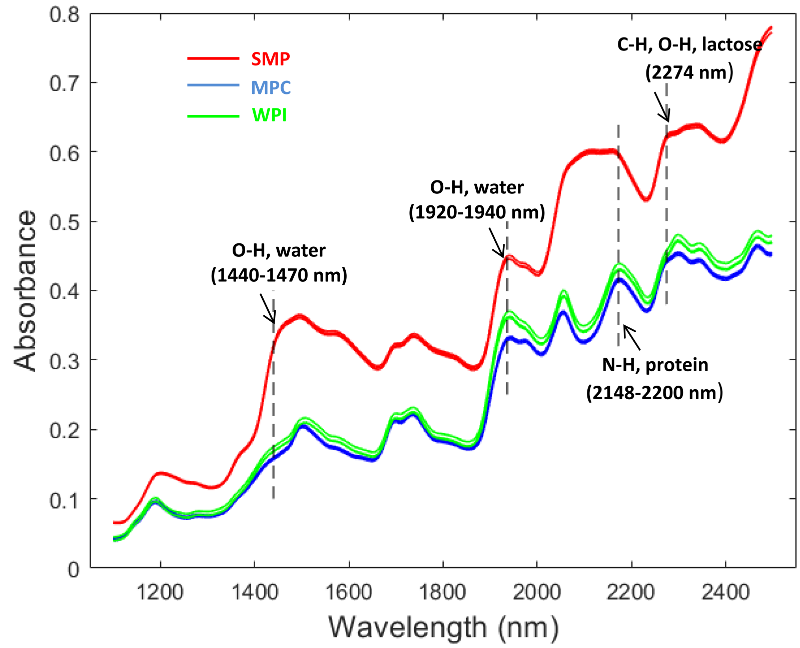
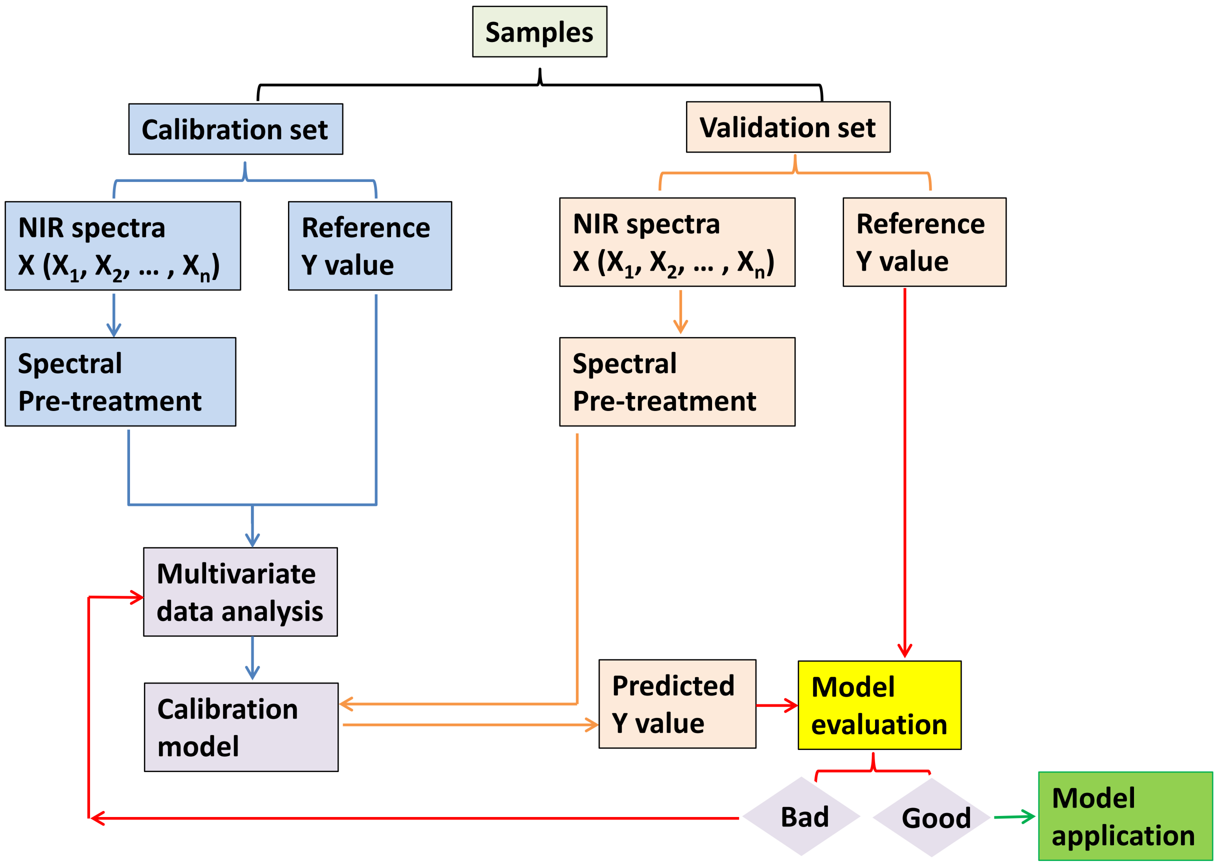

Incident Light
Sample
Detector
(a) Transmittance
Incident Light
Detector
Sample
(b) Reflectance
Incident Light
Detector
Sample
a reflector/mirror
(c) Transflectance


Rotating
plate
Light
source
Sample
(a)
Prim
Light
source
Sample
(b)
Fixed mirror
Light
source
Beam
splitter
Moving
mirror
Sample
(c)


SMP
MPC
WPI
O-H, water
(1440-1470 nm)
O-H, water
(1920-1940 nm)
C-H, O-H, lactose
(2274 nm)
N-H, protein
(2148-2200 nm)
Absorbance
Wavelength (nm)
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
1200
1400
1600
1800
2000
2200
2400


Samples
Calibration set
Validation set
NIR spectra
X (X1, X2, … , Xn)
Reference
Y value
NIR spectra
X (X1, X2, … , Xn)
Reference
Y value
Spectral
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Pre-treatment
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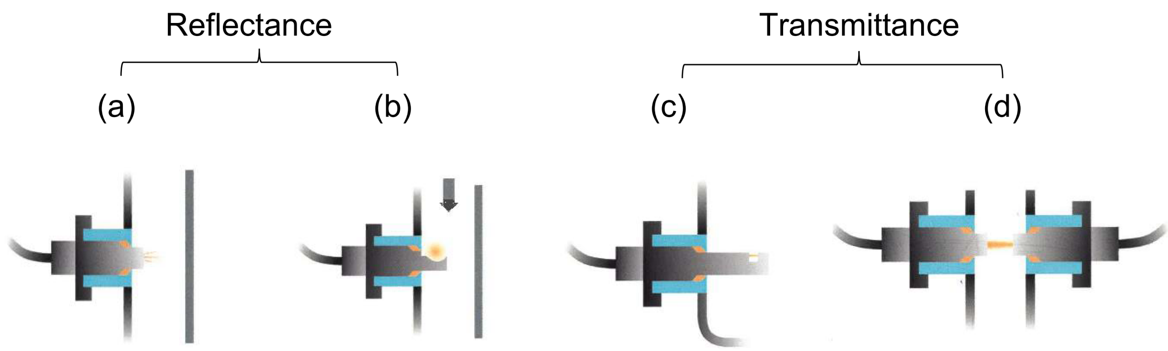
evaluation
Bad
Good
Model
application


Reflectance
Transmittance
(a)
(b)
(c)
(d)