Chapter 4
Infrared Spectroscopy

Colette C. Fagan

4.1 Introduction

Infrared spectra of food products can help to reveal information pertaining to molecular bonds present and hence provide details of their molecular structures. This ultimately can be related to various quality indices. Infrared spectroscopy is an ideal process analytical technology (PAT) tool that can rapidly, accurately and usually non-destructively assess the quality and functional properties of raw, in-process and final product materials. In addition to the need for efficiency, there is an emerging need in food processing for all major compositional and quality parameters to be determined, on-line and in real time. In addition to this, there is a need for food manufacturers to be able to demonstrate the authenticity of their products (Woodcock 2008).

Spectroscopic techniques, other than infrared spectroscopy, have been investigated as potential PAT technologies in the food industry. These include Raman spectroscopy (Chap. 5), fluorescence spectroscopy (Chap. 12) and UV–Vis spectroscopy. UV–Vis has been employed to detect adulterated and authentic spirits (Contreras et al. 2010),discriminate between brands (Barbosa-García et al. 2007), classify coffee (Souto et al. 2010) and quantify β-carotene (Biswas et al 2011). However, the focus of this chapter is infrared spectroscopy, and it will provide an overview of its theory, its instrumentation and its applicability as a PAT tool. Finally, it will review applications of infrared spectroscopy to food products.

C. C. Fagan (✉)
Food and Nutritional Science, Department of Food and Nutritional Sciences,
University of Reading, Reading RG6 6AP, P.O. Box 226, Reading, UK
e-mail: c.c.fagan@reading.ac.uk

C. P. O'Donnell et al. (eds.), *Process Analytical Technology for the Food Industry,*
Food Engineering Series, DOI 10.1007/978-1-4939-0311-5_4,

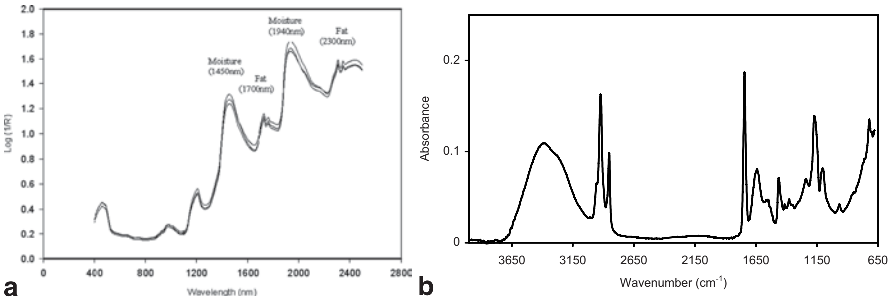

Fig. 4.1 Characteristic **a** NIR spectra and **b** MIR spectra of cheese

4.2 Theory of Near- and Mid-infrared Spectroscopy

Infrared spectroscopy results from the interaction of infrared radiation and matter. The energy provided by the infrared radiations results in transitions between quantized vibrational energy states of molecules, i.e. resulting in molecular vibration. Atoms in a molecule can have a number of vibrational modes. Each mode (i) involves approximately harmonic displacements of the atoms from their equilibrium positions (Griffiths 2010). When atoms vibrate as a simple harmonic oscillator, i.e. according to Hooke's law (Eq. 4.1) where x is the displacement away from equilibrium, k is the proportionality (or force) constant and F is the force in newtons, the vibrational energy states (V_{iv}) can be described according to Eq. 4.2, where h is Planck's constant, v_i is the fundamental frequency of the particular mode and v_i is the vibrational quantum number of the ith mode (0, 1, 2, etc.):

$$F = kx \tag{4.1}$$

$$V_{iv} = hv_i\left(v_i + \frac{1}{2}\right) \tag{4.2}$$

While the energy difference between $v_i = 0$ and $v_i = 1$ of most vibrational modes corresponds to the energy of radiation in the mid-infrared (MIR) range, overtone bands which relate to the transition between $v_i = 0$ and states higher than $v_i = 1$ are located in the near-infrared (NIR) region. Combination bands in the NIR region occur when there is a simultaneous promotion of two modes (Griffiths 2010).

A number of studies have assigned various food constituents (lipids, amides, moisture, sugars) to specific bands in MIR and NIR spectra. A selection of these regions and their associated mode of vibration of some food constituents are given in Tables 4.1 and 4.2. The characteristic broad peaks, resulting from overtone and combination bands, observed in the NIR spectra of a food product are shown in Fig. 4.1a; a corresponding MIR spectrum is shown in Fig. 4.1b. Such infrared spectra (Fig. 4.1) can contain a wealth of information on the molecular make-up of a food product. However, the spectral response of a molecular group can be influenced

Table 4.1 Selected molecular group absorption frequencies in the MIR region

Peak wave number (cm^{-1})	Functional group	Mode of vibration	Constituent
Fingerprint region			
1036, 1088	C–O	Stretch	
1060	C–O	Stretch	Carbohydrates
900–1200	C–O, C–C, O–H	Stretch	Carbohydrates
1115–1170	C–O	Stretch	
1232	C–H	Bend	
1240	C–O	Stretch	
1371	C–H	Bend	
1274, 1372, 1445, 1486	O–C–H, C–C–H, C–O–H	Bend	
1400–1477	C–H	Bend	
Functional group region			
1535–1570	Amide II	Stretch	Protein
1620–1690	Amide I	Stretch	Protein
1640	O–H	Bend	Moisture
1600–1900			Organic acids
1700–1765	C=O	Stretch	Lipids
2869	CH_2	Symmetric stretch	Lipid
2926	CH_3	Anti-symmetric stretch	Lipid
3047–3703	O–H	Stretch	Moisture

Table 4.2 Selected chemical assignments of absorption frequencies in the NIR region

Wavelength (nm)	Functional group	Functional group assignment	Constituent
982	OH	Second overtone; stretch	Water
1458	OH	First overtone; stretch	Water
1940	OH	Combination; asymmetric and scissoring stretch	Water
1210	C–H	Second overtone; stretch	Lipids
1728	C–H	First overtone; stretch	Lipids
1762	C–H	First overtone; stretch	Lipids
2308	C–H, CH_2	Combination; stretch and deformation	Lipids
2348	C–H, $=CH_2$	Combination; stretch and deformation	Lipids
1000–1020	N–H, Amide I	Stretch	Proteins

by neighbouring molecular groups (Reh 2001). The complexity of food substances enhances these difficulties as the presence of various substances can result in peak shifts (Fagan and O'Donnell 2007). Therefore, powerful statistical techniques, for example, principal component analysis (PCA) and partial least squares (PLS) regression, can be used for data compression and model development (Chap. 2).

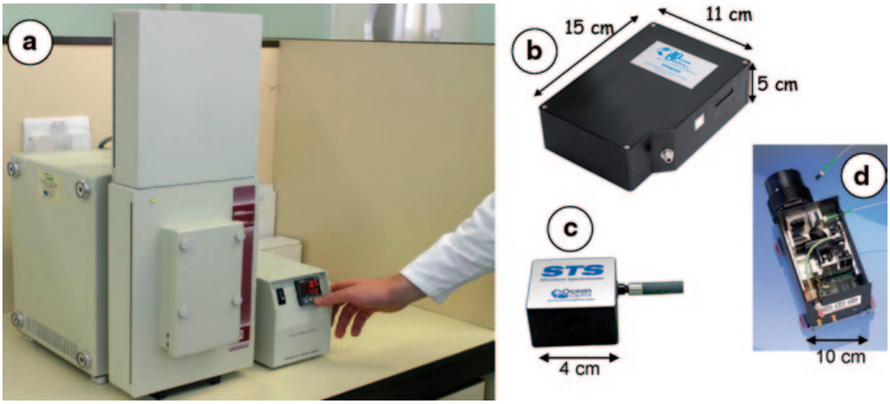

Fig. 4.2 A selection of **a** benchtop and **b–d** miniature, microspectrometer and portable spectrometers

4.3 Instrumentation

There have been significant developments in the field of infrared instrumentation over the past decades. Initially, equipment focused on the use of monochromator or filter (fixed or tunable)-based systems. However, developments in instrumentation, such as Fourier transform infrared (FTIR) spectrometers and polychromators with InGaAs detectors, substantially improved the instrumentation performance, the range of applications and therefore the popularity of such equipment. The principles of such MIR and NIR instrumentation have been reviewed previously and will not be discussed further (Fagan and O'Donnell 2007; Griffiths 2010). However, technical developments in infrared spectroscopy instrumentation will facilitate the transfer of this technology from laboratory to on-line application, thereby enhancing its potential as a PAT tool. Equipment manufacturers have moved from benchtop laboratory instruments (Fig. 4.2a) to the manufacturing of portable miniature-type spectrometers (Fig. 4.2b) to microspectrometers (Fig. 4.2c). These have been driven in part by the requirement of end users who want to have the facility to bring the "spectrometer to the samples" rather than the "samples to the laboratory". This has, for example, opened up opportunities for pre-harvest fruit and vegetable inspection. Such equipment may also include the added functionality of integrated global positioning system (GPS) measurements which are acquired simultaneously with the infrared spectra. Such facilities can allow for the "mapping" of produce quality in situ, thereby allowing the producer to make corrective decisions. In such applications, interference of the environment, such as ambient light and fluctuating temperatures, should be either minimized or accounted for by appropriate data processing (Nicolaï et al. 2007). Another related emerging platform technology is hyperspectral imaging. It has the advantage of acquiring both spectral and spatial information of sample simultaneously. It has shown considerable potential in the pharmaceutical industry in terms of mapping active ingredients in tablets. Its potential as a PAT tool in the food industry is discussed in detail in Chap. 9.

Another significant consideration of the end user must be the development and maintenance of calibration equations. Many portable spectrometers rely on the end user to provide and maintain the calibration equations required. However, this can require a substantial investment in time, labour and cost. Companies are emerging, however, which offer transferable NIR calibration solutions. Such companies have developed calibration models over many years using benchtop spectrometers, and they license them out for transfer to portable spectrometers. A service contract can also be entered into whereby the company maintains, updates and ensures set accuracy levels for the calibration model over time.

Continued research into the development of robust, fast miniature and micro-spectrometers will facilitate the continued adoption of this technology as a PAT tool in the food industry.

4.4 Infrared Spectroscopy as a PAT Technology

Infrared spectroscopy has been widely investigated as a rapid non-destructive assessment tool for food products. Fruit, vegetable, dairy and meat products have been the most widely investigated. However, the majority of these studies have been laboratory based. The greatest advantage in the use of infrared-based technology as PAT tools will be their implementation in the form of on-line/at-line process analysers, which take advantage of rapid analysis times and the minimal sample presentation required. However, it should be noted that the requirements for laboratory-based analysis will differ in comparison with on-line technology. Infrared spectroscopy also has the capacity to predict numerous indices of a material simultaneously. In order to realize the potential of such data-rich tools in the food industry, appropriate data analysis (Chap. 2) and data management strategies (Chap. 3) are required. Food quality, however, cannot be considered as a single, well-defined attribute. In fact, it encompasses a number of properties or characteristics, which are often referred to as quality indices, of the product under test (Abbott 1999). While infrared spectroscopy can offer a solution to this challenge, one must ensure that the basis for the prediction of quality is fully understood, as well as its inherent limitations.

4.5 Applications

4.5.1 Dairy

The dairy industry has seen significant advances towards automation of production processes. For example, the move to closed commercial cheese vats versus the traditional open cheese vat drove the desire for on-line milk coagulation monitoring systems.

4.5.1.1 Raw Material

Milk composition and quality can vary depending on a number of factors, including animal genetics, health, and (in some countries) season. Such variability could significantly impact the quality of the final product. For example, milk fat to protein ratio will significantly affect a number of processing steps (coagulation, syneresis) which ultimately affects cheese quality and quantity. Therefore, it is usual that processors would standardise milk fat and protein content prior to use. Therefore, the use of infrared spectroscopy to facilitate the production of high quality of milk has been investigated in applications ranging from monitoring rumen metabolism through to standardisation of milk in the milk processing plant (Fagan et al. 2009b).

Off-line rapid analysis of milk composition using the FTIR measuring principle has been successfully commercialized with products such as the MilkoScan™ FT 120 (Foss Analytical, Denmark). It utilizes FTIR technology to analyse up to 600 samples/h and can be used for routine analysis, such as fat, protein, lactose, total solids and solids-non-fat, density, freezing point depression, urea and casein analysis, in compliance with International Dairy Federation (IDF) and Association of Analytical Communities (AOAC) standards.

The further development of on-line determination of milk composition and quality would be advantageous as such knowledge is essential for the efficient management of dairy herds. Brandt et al. (2010), however, stated that while a number of sensors are available or in development which can be used for management support in improving mastitis detection, monitoring fertility and reproduction and adapting individual diets, there is still a requirement to adapt these sensors to the particular requirements of on-farm utilization such as robustness, calibration and maintenance, costs, operating cycle duration, and high sensitivity and specificity.

Tsenkova et al. (2001) examined the potential of predicting somatic cell count (SCC) of milk using NIR transflectance spectra obtained using a benchtop spectrophotometer. They stated that the results indicated that NIR spectroscopy would be a suitable screening tool in such an application as the differentiation between healthy and mastitic milk samples was possible. More recently, an NIR spectroscopic sensing system for on-line monitoring of milk quality during milking has been developed (Kawamura et al. 2007). The system was installed between a teatcup cluster and a milk bucket of a milking machine. The authors developed models for the prediction of fat, protein, lactose, SCC and milk urea nitrogen (MUN) during milking with sufficient precision and accuracy (R^2=0.82-0.95), although only four cows were monitored over time. Following this study, the sensing system was installed in an automatic milking system. The system recorded diffusion transmittance spectra (600-1050nm) with a 1-nm interval every 10 s during milking. Seventeen cows were used in this study. The models developed for fat, protein, lactose, SCC and MUN had R^2 values of 0.95, 0.83, 0.72, 0.68 and 0.53, respectively. The authors used the SCC calibration model to discriminate between healthy cow samples and other cow samples. The resulting classification gave a probability, for classifying correctly, of 82%. In both studies, the samples were divided into calibration (2/3) and validation (1/3) sets. Further validation of the models is therefore recommended in conjunction with testing on a wider range of animals.

MIR spectroscopy has also been explored for the offline determination of milk traits (Cecchinato et al. 2009; Dal Zotto et al. 2008; De Marchi et al. 2009). Milk coagulation properties (MCP) will vary depending on a number of factors, including heritable parameters (Cassandro et al. 2008). However, if this information is to be fully exploited, there would be a requirement for a rapid method of determining MCP in milk-recording systems. Dal Zotto et al. (2008) found that MIR spectroscopy could predict the rennet coagulation time (RCT) of milk samples albeit with an R^2 of 0.73, which suggested approximate quantitative predictions were possible. De Marchi et al. (2009) carried out a further examination of this approach. Using a dataset of over a thousand samples, RCT was predicted with an R of 0.79. In both studies, the range error ratio (RER) was similar: 9.2 and 10.6. Cecchinato et al. (2009) investigated the variation of MCP predictions obtained by MIR spectroscopy, as well as estimating the expected response from a breeding program focusing on the enhancement of MCP using MIR predictions as indicator traits. They found that estimated genetic correlations between measure and predictions of RCT were very high.

4.5.1.2 Process Monitoring

NIR technology has been successfully applied at laboratory and commercial scales for monitoring processes during cheese manufacture. In particular, the milk coagulation process during cheese production has received a great deal of attention, and cutting the coagulum either before or after the optimum point results in losses of curd and fat. An increase in cheese moisture also occurs if the gel is too firm when cut. Originally, the determination of the cutting time was established by the cheese maker. Although accurate this method is not feasible in closed commercial vats and, together with an increased desire for automation in the cheese industry, has led to the need for an on-line objective method for the monitoring of milk coagulation. Instruments have been developed based on several technologies to this end. Ideally, a sensor to monitor milk coagulation could be installed on-line to allow for automation of the production process, without causing damage to the forming curd, and NIR sensors meet these requirements. Early methods, which utilized the changes in the optical properties of the milk, were reflection photometry (Hardy and Fanni 1981) and absorbance (McMahon et al. 1984). Although the reflection photometry and absorbance methods were found to monitor coagulation, they found little usage. However, developments in fibre optics have overcome many of the problems associated with these techniques. Light in the NIR spectral region can be transmitted through a fibre optic bundle and diffuse reflectance or transmission monitor. As the gel is formed, reflectance will increase while transmission will decrease. Payne et al. (1993) developed a method based on changes in diffuse reflectance during milk coagulation. Reflectance was measured using a fibre optic probe, utilizing a photodiode light source at a wavelength of 940 nm. The time to the inflection point (t_{max}) was determined from the first derivative and was found to correlate well with Formograph cutting times. Linear prediction equations, which were considered to be of the form required for predicting cutting time, were also developed

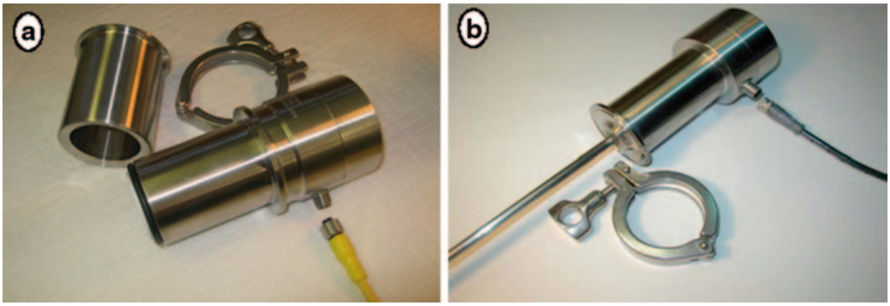

Fig. 4.3 **a** The CoAguLite sensor for predicting the optimal cutting time. **b** The FiberView Dairy Waste Sensor System (Reflectronics Inc, KY)

using t_{max}. This technology has been commercialized as the CoAguLite sensor (Reflectronics Inc, Lexington, KY) (Fig. 4.3a). This technology could also be used in conjunction with other sensors; for example, the FiberView dairy waste sensor system (Fig. 4.3b) could be used to monitor waste streams in dairy facilities. This enables the location, occurrence or concentration of the discharge to be determined. It monitors solids concentration in dairy plant effluents in the range of 0–1 % solids (or higher), and due to its quick response to loss events, it allows operators to take corrective actions.

Syneresis is a critical phase in cheese manufacture, with the rate and extent of syneresis playing a fundamental role in determining the moisture, mineral and lactose content of drained curd and hence that of the final cheese (Lawrence and Gilles 1980; Pearse and Mackinlay 1989). Therefore, research is ongoing into the development of a syneresis control technology. A number of potentially non-invasive technologies have been investigated for such an application, including ultrasound and computer vision (Everard et al. 2007; Fagan et al. 2008a; Taifi et al. 2006; Tellier et al. 1993) and NIR sensing (Castillo et al. 2005a; Fagan et al. 2009a; Fagan et al. 2007a). Initial studies focused on offline optical sensing of whey samples (Castillo et al. 2005b). An adaption of this technology led to the development of a sensor which could be installed in the wall of a cheese vat for on-line continuous monitoring of both coagulation and syneresis (Fagan et al. 2007a). The sensor operated at 980 nm and was sensitive to casein micelle aggregation and curd firming during coagulation and to changes in curd moisture and whey fat contents during syneresis. This sensor was also used to predict whey fat content (i.e. fat losses), curd yield and curd moisture content with standard error predictions (SEPs) of 2.37 g, 0.91 and 1.28 %, respectively (Fagan et al. 2008b). Further work used a wider spectral range (300–1100 nm) in conjunction with PLS regression to predict whey fat and curd moisture with root mean square error of cross-validation (RMSECV) values of 0.094 and 4.066 %, respectively (Fagan et al. 2009a). Mateo et al. (2009) developed another set of models which predicted the yield of whey ($R^2 = 0.83$, error = 6.13 g/100 g) using three terms, namely light backscatter, milk fat content and cutting intensity. These studies were carried out in laboratory-scale cheese vats (7–

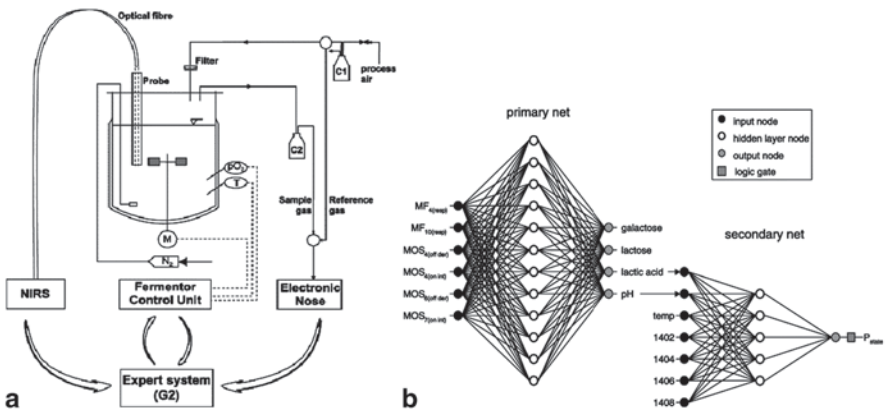

Fig. 4.4 The multi-analyzer setup **a** applied during yogurt fermentations: *C1*, compensator bottle 1, to trap condensating vapour and to compensate for minor flow rate variations; *C2*, compensator bottle 2 and **b** the neural network topology used for sensor fusion. The primary network received six input signals from the electronic nose and was cascaded by the secondary network, which received seven input signals: the output signals from the primary network for pH and lactic acid, four second-derivative NIRS signals (1402–1408 nm) and the first derivative of the reactor temperature signal. A logic gate made the final decision for the state variable. (Cimander et al. 2002)

11 L). Therefore, further scaling up and development under commercial conditions of the technology would be required if it is to become viable at a commercial scale.

NIR spectroscopy has also been investigated as a process control tool in yogurt production. Cimander et al. (2002) studied the potential of NIR spectroscopy to monitor yogurt fermentation in a 4.2-L laboratory-scale vat. A sensor signal fusion approach was adopted with NIR (400–2500 nm), electronic nose, and standard bioreactor sensors installed as part of a multi-analyzer setup (Fig. 4.4a). While the electronic nose followed changes in galactose, lactic acid, lactose and pH, the NIR sensor signal correlated well with the changes in the physical properties during fermentation. Therefore, the signals from the sensors were fused using a cascade artificial neural network (ANN) as detailed in Fig. 4.4b. Results suggested that the accuracy of the neural network prediction was acceptable. This approach was further investigated by Navrátil et al. (2004) under industrial conditions in a 1000-L vat. Signal responses from NIR and electronic nose sensors were subjected to PCA separately. The scores of the first principal component from each PCA were then used to make a trajectory plot for each fermentation batch. PLS regression of the NIR spectra was also used to predict pH and titratable acidity (expressed as Thorner degrees, °Th) during fermentation with reasonable success (SEPs of 0.17 and 6.6 °Th, respectively). MIR spectroscopy has also been employed to monitor the sorghum fermentation process (Correia et al. 2005). They used FTIR spectroscopy to detect differences due to the effect of lactic bacteria on sorghum fermentation. They found it was possible to differentiate between samples which used natural yogurt and *Lactobacillus fermentum* as inocula due to variations in protein and starch structure.

4.5.1.3 Final Product Quality and Authenticity

It is stated in Chap. 12 that “Quality attributes for dairy products can be both the chemical composition of a given product like protein, moisture and fat content, and the sensory quality attributes like taste, smell and consistency.” Therefore, the integration of sensing technologies which provide information on such attributes is critical. Table 4.3 summarizes a number of studies which have examined the potential of NIR and MIR spectroscopies to predict the composition of dairy products. These have primarily been laboratory based. Offline laboratory-based infrared sensing systems which provide rapid compositional analysis of dairy products are available. These systems should conform to relevant standards such as ISO Standard 21543:2006 (ISO 2006). A number of studies have also investigated the prediction of sensory quality attributes. Downey et al. (2005) predicted the maturity and sensory attributes of Cheddar cheese using NIR spectroscopy. Generally, second derivative spectra in the region of 750–1098 nm produced the most accurate models with age predicted with an RMSECV of 0.61 months, while the most successfully predicted sensory texture attributes were rubbery, chewy, mouthcoating and massforming with RER values of 8.8, 6.3, 7.6 and 8.5, respectively. NIR spectroscopy was also employed to predict both the sensory and instrumental attributes of processed cheese using NIR spectroscopy (Blazquez et al. 2006). In general, they found that the models developed for predicting sensory texture in processed cheese were stronger than those for Cheddar cheese, with rubbery, chewy, mouthcoating and massforming predicted with RER values of 9.1, 12.0, 8.1 and 8.1, respectively. Fagan et al. (2007b) compared the NIR models developed by Blazquez et al. (2006) to models developed using MIR spectroscopy, which also predict sensory texture parameters of processed cheese. NIR spectroscopy was better at predicting creamy, chewy, and melting, with the R^2 values of the NIR models indicating excellent predictions as opposed to the good predictions of the MIR models. The RER values for the NIR reflectance models indicated a high utility value, whereas the RER values obtained by Fagan et al. (2007b) had a good practical utility. However, the MIR-derived fragmentable model had better accuracy than the NIR model, with excellent and good predictions, respectively.

The requirement to demonstrate the authenticity and safety of dairy products has also led to research into the use of infrared technology for such applications. Determination of the geographic origin and manufacturing conditions of cheese has received a great deal of attention (Boubellouta et al. 2010; Cattaneo et al. 2008; Karoui et al. 2004, 2005a, b, 2007a, 2008; Kocaoglu-Vurma et al. 2009; Pillonel et al. 2003). For example, Pillonel et al. (2003) studied the potential of MIR and NIR spectroscopies to discriminate between Emmental cheeses ($n=20$) based on geographic origin. Samples were obtained from six regions, and they found that MIR transmission spectra could be used to discriminate (i.e. 100% correct classification) Swiss cheese from the other regions, while NIR spectra classified the samples by the six regions of origin. Karoui et al. (2007a) also examined MIR spectroscopy to determine the authentication of 25 Gruyère “protected designation of origin” (PDO) and L’Etivaz PDO cheeses. They found that the spectral regions

Table 4.3 Application of near- and mid-infrared spectroscopy in cheese and yogurt composition analysis. (Modified from Woodcock (2008))

Composition	Product	Spectral	Mode	Wavelength/ wave number	Prediction	Ref.
Parameter		Region		Range	Error	
Moisture	Cheese	NIR	R	400–2498 nm	SECV = 0.5	Blazquez et al. (2004)
Content	Cheese	NIR	R	900–2500 nm	SEP = 0.429	Čurda and Kukačková (2004)
	Cheese	NIR	R	515–1700 nm	RMSEP = 1.72–2.21	da Costa Filho and Volery (2005)
	Cheese	NIR	R	1900–2320 nm	SEP = 0.889	Lee et al. (1997)
	Cheese	NIR	R	1000–4000 nm	SEP = 0.12–0.35	McKenna (2001)
	Cheese	NIR	T	1000–4000 nm	SEP = 0.12–0.35	McKenna (2001)
	Cheese	MIR	ATR	5000–400 cm^{-1}	SEP = 0.04–0.09	McQueen et al. (1995)
	Cheese	NIR	R	1740–2280 nm	SEP = 0.02–0.05	McQueen et al. (1995)
	Cheese	NIR	R	400–2500 nm	SECV = 0.05–0.92	Pérez-Marín et al. (2001)
	Cheese	NIR	R	400–2498 nm	SEC = 0.412	Rodriguez Otero et al. (1994)
	Cheese	NIR	R	400–2500 nm	RMSEP = 0.58	Wittrup and Nørgaard (1998)
Fat	Cheese	NIR	R	1100–1498 nm	SECV = 0.45	Blazquez et al. (2004)
Content	Cheese	FT-NIR	R	900–2500 nm	SEP = 0.997	Čurda and Kukačková (2004)
	Cheese	NIR	R	1000–2500 nm	RMSEP = 3.61	Karoui et al. (2007b)
	Cheese	NIR	R	1900–2320 nm	SPE = 0.855	Lee et al. (1997)
	Cheese	MIR	ATR	5000–400 cm^{-1}	SEP = 0.12–0.35	McQueen et al. (1995)
	Cheese	NIR	R	1740–2280 nm	SEP = 0.12–0.35	McQueen et al. (1995)
	Cheese	NIR	R	400–2500 nm	SECV = 0.05–0.92	Pérez-Marín et al. (2001)
	Cheese	NIR	R	400–2498 nm	SEC = 0.388	Rodriguez Otero et al. (1994)
	Cheese	NIR	R	400–2500 nm	RMSEP = 0.52	Wittrup and Nørgaard (1998)
Protein	Cheese	FT-NIR	R	900–2500 nm	SEP = 0.303	Čurda and Kukačková (2004)
Content	Cheese	NIR	R	1000–2500 nm	RMSEP = 2.34	Karoui et al. (2006)

Table 4.3 (continued)

Composition Parameter	Product	Spectral Region	Mode	Wavelength/wave number Range	Prediction Error	Ref.
	Cheese	NIR	R	1900–2320 nm	SEP = 0.608	Lee et al. (1997)
	Cheese	MIR	ATR	5000–400 cm^{-1}	SEP = 0.04–0.09	McQueen et al. (1995)
	Cheese	NIR	R	1740–2280 nm	SEP = 0.04–0.09	McQueen et al. (1995)
	Cheese	NIR	R	400–2500 nm	SECV = 0.05–0.92	Pérez-Marín et al. (2001)
	Cheese	NIR	R	400–2498 nm	SEC = 0.397	Rodriguez Otero et al. (1994)
	Yogurt	MIR	ATR	1800–1500 cm^{-1}	REP = 7.25	Khanmohammadi et al. (2009)
	Yogurt	MIR	ATR	1800–1500 cm^{-1}	REP = 3.7	Khanmohammadi et al. (2009)
	Yogurt	MIR	ATR	1515–1800 cm^{-1}	RMSEP = 0.2	Moros et al. (2006)
Sugar	Yogurt	NIR	R	400–1000 nm	RMSEP = 0.2621	Shao and He (2009)
Content	Yogurt	MIR	ATR	1500–900 cm^{-1}	SEP = 0.105–0.05	Khurana et al. (2008)
	Yogurt	NIR	R	400–1000 nm	SEP = 0.389	He et al. (2007)
	Yogurt	NIR	R	400–1000 nm	RMSEP = 0.33–0.36	Shao et al. (2007)
Carbohydrate	Yogurt	MIR	ATR	2850–1083 cm^{-1}	RMSEP = 36	Moros et al. (2006)
Calcium Content	Yogurt	MIR	ATR	1461–1636 cm^{-1}	RMSEP = 9	Moros et al. (2006)

R reflection, *T* transmission, *ATR* attenuated total reflection

3000–2800 cm^{-1} and 1500–900 cm^{-1} were most useful with 90.5 and 90.9 % correct classification results achieved, respectively. MIR spectroscopy (supplemented by partial 16S rDNA sequencing) has also been employed to monitor the population dynamics of microorganisms during cheese ripening (Oberreuter et al. 2003).

4.5.2 Cereal Grains and Seeds

NIR spectroscopy has been widely used in routine quality control analysis in the grain industry since the 1960s (Scotter 1990). This has included the assessment of moisture and protein content (Downey and Byrne 1987; Norris and Williams 1979;

Williams 1979; Williams and Cordeiro 1979, 1981). More recently, developments in this area have focused on assessment of grain quality at harvest, grain quality classification and sorting and grain blending.

Kawamura et al. (2003) developed an automated rice quality inspection system which utilized both visible and NIR technology. The objective was to develop a system which measured not only moisture content but also other rice quality indices in order to grade rough rice according to quality when it arrives at the drying facility. The system they developed consisted of a rice huller, a rice cleaner, an NIR instrument and a Vis segregator. This system enabled rough rice transported to a rice-drying facility to be classified into six qualitative grades.

Grain quality at harvesting is also a critical parameter as there can be significant within-field variability of grain quality parameters, for example, protein and moisture content. Maertens et al. (2004) described some of the requirement for online grain quality assessment at harvest. They included the use of a robust NIR spectrometer, design of a measurement configuration that guarantees a constant grain sample presentation while also avoiding dirt and blockages, that the sensor should be calibrated on the harvester and not under simulated conditions in the laboratory and finally that appropriate signal processing techniques should be employed to filter the spectral data, both in the time and wavelength domain They also studied the potential of an NIR sensor mounted on the bypass of the grain elevator of a combine harvester for online prediction of wheat moisture and protein content. They found that the average prediction errors were 0.56 and 0.31 % for protein and moisture content, respectively, where moisture content was below 18 %.

Detection and removal of internal insects and fungal contamination from seeds (grains, beans and nuts) are important control measures for ensuring storage longevity, seed quality and food safety (Pasikatan and Dowell 2001). NIR spectroscopy has been applied to the detection of infestation of such products. NIR spectroscopy has been used to differentiate among individual wheat kernels that are uninfested, those infested with weevil larvae or pupae, or those that contain a parasitoid pupa (Baker et al. 1999). Wang et al. (2002) recorded single-seed NIR spectra of a total of 1600 soya bean seeds, i.e. 700 sound seeds and 900 seeds damaged by weather, frost, sprout, heat or mould. The regions 750–1690 nm and 450–1690 nm gave the best classification of seeds into "sound" and "damaged" categories. They also found that an optimally developed neural network (parameters: momentum = 0.6, learning rate = 0.7, learning cycles = 150,000, wavelength region = 490–1690 nm) could classify seed according to six categories, i.e. "sound" (100 %) and five damage categories, "weather" (98 %), "frost" (97 %), "sprout" (64 %), "heat" (79 %) and "mold" (83 %), with reasonable success.

Aflatoxin B1 is recognized by the International Agency of Research on Cancer as a group 1 carcinogen for animals and humans, and Fernández-Ibañez et al. (2009) investigated the potential of Fourier transform NIR spectroscopy to detect aflatoxin B1 in cereal grains. They analysed maize and barley samples ($n = 152$) and developed models ($R^2 = 0.82$–0.85) for prediction of the presence of aflatoxin B1, which suggested that NIR spectroscopy could be a suitable alternative for fast detection of aflatoxin B1 in cereals.

4.5.3 Fruit and Vegetables

The application of NIR and MIR to quality assessment of fruit and vegetables has been widely studied (Table 4.4). In terms of infrared spectroscopy's role as a PAT tool in this industry, it could be employed for the optimization of harvesting, defect identification, disease control, process control applications and overall quality classification.

4.5.3.1 Harvest Optimization

Prediction of the optimal harvest time of apples will minimize the occurrence of quality losses. Peirs et al. (2001) predicted the optimal harvest date of apples harvested no more than 8 weeks before the commercial picking date using Vis–NIR spectra collected post harvest in the laboratory (measurements were carried out on the same day or the day after picking). They stated that it was possible to measure apple maturity for harvest of individual cultivars within an orchard and that the number of days before the optimum harvest date was well predicted (R=0.90–0.93). Further work examined the potential of Vis–NIR spectroscopy to estimated apple pre- and post-storage quality indices at harvest (McGlone et al. 2002). The apples were harvested 1–3 weeks before and up to 1 week after the commercial harvest period. Spectral analysis in this case took place between 16 and 24 h after harvest. The authors found that although models were developed to predict quality indices of the apples they were still very poor in terms of prediction accuracies. Therefore, they were unlikely to be useful for sorting or grading due to the high rate of prediction errors that would result. They also stated that the prediction models, with the exception of soluble solids content, may be almost solely dependent on changes in the apple chlorophyll level and not have any direct sensitivity to the constituents or properties of interest.

Clark et al. (2004) examined the potential of Vis–NIR spectroscopy to predict the storage potential of kiwifruit. They employed canonical discriminant analysis (CDA) to optimize the separation between the two categories, i.e. "sound fruit" and "fruit developing a disorder during storage". They estimated that the overall incidence of disorders could have been reduced from 33.9 to 17.9% and 14.7 to 8.5% depending on the harvest or when using all harvests from 13.7 to 6.8%.

A similar approach has also been investigated for mango (Saranwong et al. 2004). Vis–NIR spectra of mango were collected on the day of harvest and models were developed to predict harvest and eating quality using multiple linear regression and PLS regression. They stated that the calibration equations developed were sufficiently accurate to determine the harvest quality, dry matter and starch content of hard green mango fruit non-destructively. Using this information, the soluble solids content of the ripe fruit, which is an eating quality index, could be precisely predicted at the time of harvest.

Table 4.4 Examples of applications of near- and mid-infrared spectroscopy to quality assessment of fruit

Product	Parameter	Technology	Mode	Range	Result	Equipment	Ref.
Avocado	Moisture content	NIR	R		$R^2=0.92$, SEFV = 2 %, RER = 16	NIRS6500 spectrophotometer	Blakey et al. (2009)
Apple, grape, pear; apple-chewy and apple-banana juices	Soluble solids and total solids/total moisture	NIR	R		$R^2=0.90$	Katrina Inc designed NIR sensor	Singh et al. (1996)
Zucchini squash	Chilling injury	FTIR	R		Spectral shift of the maxima to 3400 cm^{-1}	Nicolet 60 SX FTIR spectrometer	Buta et al. (1997)
Fruit concentrates	Total sugar, glucose, fructose and sucrose	NIR	T		Relative standard deviation values obtained vary from 0.4 to 2.3 %	Perkin-Elmer Lambda 9 double beam spectrophotometer	Rambla et al. (1997)
Ginseng	Moisture content	NIR	R		$R^2=0.998$, SEP = 0.12 %	Model 6500, Perstorp Analytical Inc	Ren and Chen (1997)
Kiwifruit	Soluble solids distribution	NIR imaging	R		Prediction error of 1.2 Brixt	Author design	Martinsen and Schaare (1998)
Apple	Soluble solids	NIR	R		$R^2=0.56$, SEP 1.14 Brix	Ocean Optics SD-1000	Ventura et al. (1998)
Fig	Sorting defect and acceptable categories	NIR	R	400–1700 nm	Classification 83 to 100 %	Diode-array Perten Instruments	Burks et al. (2000)
Kiwifruit	Discrimination of pre-harvest fruit management treatment	NIR	R	516–998 nm	Best classifications based on fast Fourier transform features	PS1000 Ocean Optics	Kim et al. (2000)

Table 4.4 (continued)

Product	Parameter	Technology	Mode	Range	Result	Equipment	Ref.
Potato	Elimination of interference from peel	Vis–NIR	R/T	600–1100 nm	RMSEC = 3–4.1 %	Modular spectrophotometer, Bentham Instruments Ltd	Krivoshiev et al. (2000)
Apple	Comparison of two optical configurations for measuring internal apple quality attributes, describe interaction of skin and the incident radiation, determine penetration depth values in apple tissue	Vis–NIR	R	500–1900 nm	Can provide information about the state of the fruit flesh; 0.55 Brix	OSA 6602, Rees Instruments Ltd	Lammertyn et al. (2000)
Mango	Predict physiological properties and quality indices	NIR	R	1200–2400 nm	MLR: SEP = 1.223, 0.161, 17.14, 37.03, R^2 = 0.93, 0.61, 0.82, 0.94 for TSS, acidity, firmness and storage period	Quantum 1200, LTI	Schmilovitch et al. (2000)
Peach	Assess woolliness	NIR (in conjunction with non-destructive impact response (NDIR))	R	900–1400 nm	NIR classified into juicy categories. NIR + NDIR classified woolly peaches at 80 %	OSA 6602, Monolight	Ortiz et al. (2001)

Table 4.4 (continued)

Product	Parameter	Technology	Mode	Range	Result	Equipment	Ref.
Apple	Predict optimal harvest date	Vis/NIR	R	380–2000 nm	Maturity was orchard-dependent; $R = 0.80$–0.90 (soluble solids, acidity)	OSA 6602, Rees Instruments Ltd	Peirs et al. (2001)
Fruit juice	Predict sugar levels	FT-NIR	R/TR	1000–2500 nm	SEP $< 0.10\,\%$; $R^2 = 0.999$	Perkin–Elmer Spectrum Identicheck	Rodriguez-Saona et al. (2001)
Papaya	Methylation level of pectin fractions	FTIR	A	4000–500 cm^{-1}	No significant difference	Bomem MB-100	Manrique and Lajolo (2002)
Apple	Estimation of pre- and post-storage quality indices	Vis/NIR	T	300–1140 nm	Poor predictions (chlorophyll level dependent)	Specially developed laboratory system	McGlone et al. (2002)
Red paprika	Mycotoxin detection	NIR	R	1100–2000 nm	RPD = 5.2, 2.8, 4.4	Foss NIRS system 5000	Hernández-Hierro et al. (2008)

It has also been demonstrated that infrared technology can be used for fruit assessment prior to harvesting. Pérez-Marín et al. (2009) used a handheld micro-electro-mechanical system (MEMS) spectrometer and a diode-array Vis–NIR spectrophotometer to collect the spectra of nectarine during on-tree ripening ($n=144$). They developed models to quantify changes in soluble solids content, flesh firmness, fruit weight and diameter. Both instruments provided good precision for soluble solids content ($R^2=0.89$; SEP = 0.75–0.81 %) and for firmness ($R^2=0.84$–0.86; SEP = 11.6–12.7 N). The diode-array instrument predicted the two other physical parameters well ($R^2=0.98$ and SEP = 5.40 g for fruit weight and $R^2=0.75$ and SEP = 0.46 cm for diameter), while the handheld MEMS instrument proved less accurate in this respect (Pérez-Marín et al. 2009).

A portable non-invasive instrument based on NIR spectroscopy has also been developed to measure the ripeness of wine grapes (Larrain et al. 2008). It was used to predict three ripeness variables with excellent success for Brix and pH ($R^2=0.87$–0.93) and with less accuracy ($R^2=0.56$–0.80) for pH.

4.5.3.2 Defect Identification

Burks et al. (2000) applied NIR spectroscopy to the sorting and classification of figs. They classified the figs according to the number of categories ("passable", "infested", "rotten", "sour", "dirty") with correct classifications ranging from 83 to 100 %. However, 20 PLS factors were required which might limit the robustness to the models. Vis–NIR spectroscopy in both transmission and reflectance modes has been employed to detect brown heart of pears (Fu et al. 2007). They found that, using discriminant analysis, they could discriminate between brown heart pears and non-brown heart pears. Transmission spectra were more successful than reflectance spectra in this classification: a classification rate of 91.2 % using transmission spectra.

A conceptual view of an NIR transmission-based system for apple assessment (Fig. 4.5) has been proposed by McGlone and Martinsen (2004). They employed two prototype on-line NIR transmission systems to determine the percentage of internal tissue browning in apples. One prototype used time-delayed integration spectroscopy (TDIS) in which light transmitted through a moving object was electronically tracked as it moved through the spectrometer's field of view. The other used a large aperture spectrometer (LAS) in which the light from the object is accumulated in a series of one-shot measurements as the fruit progresses through the field of view (McGlone and Martinsen 2004). The systems operated 500 mm s^{-1}. The LAS system gave the best results ($R^2=0.9$) for fast on-line assessment of apples.

Further developments in defect identification have focused on the use of multispectral or hyperspectral imaging (Ariana et al. 2006; Blasco et al. 2007). This emerging platform technology is discussed in Chap. 9.

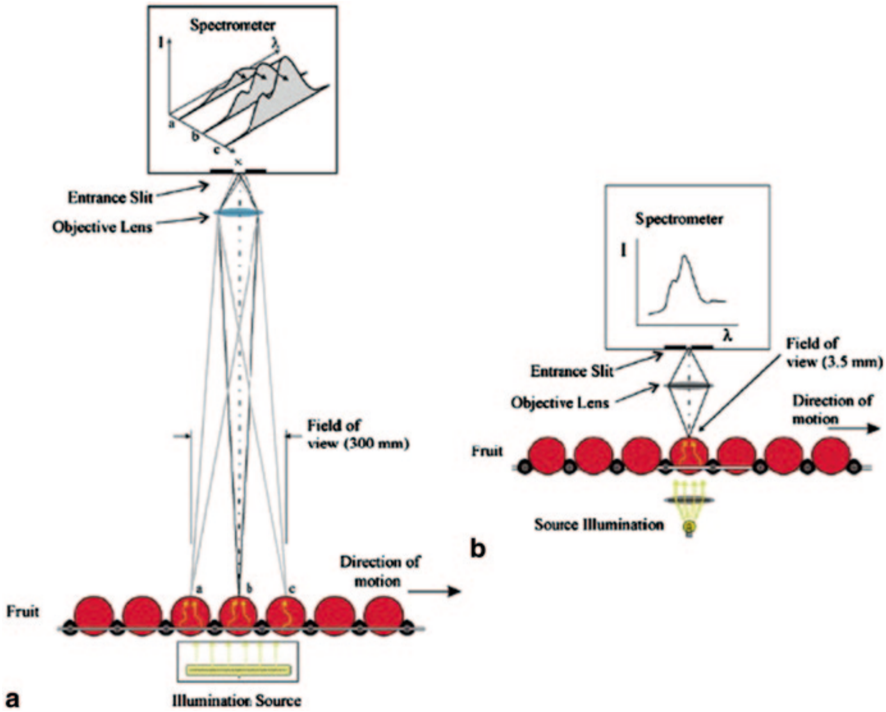

Fig. 4.5 A conceptual view of NIR transmission system. As the fruit passes through a relatively large field-of-view in the TDIS system (**a**), a detector simultaneously accumulates many sequential points over three apples. In contrast, the LAS system (**b**) takes a simple snapshot, like a camera, over a much shorter time for a small portion of one fruit (McGlone and Martinsen 2004). (Reprinted with permission from *Journal of Near Infrared Spectroscopy* 12(1), 37–43 (2004). Copyright: IM Publications LLP 2004)

4.5.3.3 Quality Classification

A key quality characteristic of fruit is SSC. As fruit ripen, there is conversion of insoluble starch into soluble solids, to which the simple sugars (glucose, fructose and sucrose) make the largest contribution (Martinsen and Schaare 1998). Numerous studies have investigated infrared spectroscopy to predict this parameter non-destructively and have been summarized in Table 4.4. The majority of such studies have utilized NIR spectroscopy. A study by Lammertyn (2000) compared two optical configurations, i.e. a bifurcated and a 0°/45° optical configuration. They found that while the former configuration gave slightly better performance for the prediction of SSC, they recommended 0°/45° configuration for commercial applications as it had a lower cost and could be used for non-contact measurements. However, bifurcated reflectance-based instruments have found an array of applications (Fig. 4.6). It should be noted that numerous variables (e.g. cultivar, geographic

Fig. 4.6 A NIR (LabSpec) with bifurcated fibre optic probe for contact reflectance measurement

origin, etc.) can affect the performance of such predictive models, and therefore studies which have independently validated models, for example, over and within seasons, are crucial to an assessment of model robustness (Golic and Walsh 2006). Golic and Walsh (2006) collected NIR spectrum of peaches, nectarines and plums and found that model performance for SSC was acceptable when peaches and nectarines were combined, but it was best if a separate plum model was employed. They also stated that model performance was stable over several seasons in terms of R^2 (typical $R^2 > 0.8$).

4.5.4 *Meat and Poultry*

4.5.4.1 Fresh Meat

A number of studies examined the application of infrared spectroscopy to fat extracts to predict meat quality as fatty acid composition of meat can determine its processing quality. Villé et al. (1995) developed a method for the determination of total fat and phospholipid content in intramuscular pig meat using FTIR spectroscopy. They employed an extraction using chloroform and methanol. FTIR spectra were subsequently recorded in transmission mode, and utilizing selected regions of the FTIR spectra related to the C=O bond (1785–1697 cm^{-1}) developed linear regression equation to predict total fat ($R^2 = 0.99$). A study has also examined the use of FTIR spectroscopy in the NIR and MIR regions of fat extracts and non-processed pork to determine the fatty acid content in fat slices and fat extracts (Ripoche and Guillard 2001). They found that MIR spectra using an attenuated total reflectance samples accessory ($R^2 \sim 0.91$–0.98) and NIR transmission spectra ($R^2 \sim 0.85$–0.96) of fat extracts could be used to predict saturated fatty acids (SFA), monounsaturated fatty acids (MUFA), polyunsaturated fatty acids (PUFA), palmitic acid (C16:0), oleic acid (C18:1) and linoleic acid (C18:2). However, with 9–15 latent variables

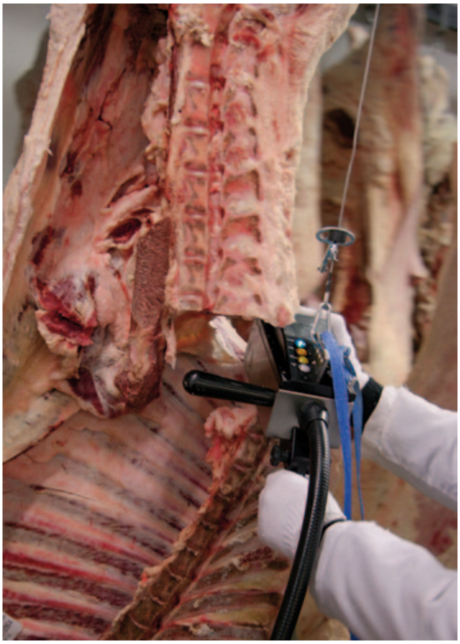

Fig. 4.7 The QualitySpec BT Spectrometer from Analytical Spectral Devices Inc. for measuring meat quality

included in the models, they may not be very robust. While NIR reflectance spectroscopy successfully predicted SFA, PUFA, C18:1 and C18:2 from spectral measurements of the back and breast fat, MUFA and C16:0 could not be predicted. Mitsumoto et al. (1991) used NIR spectroscopy in reflectance and transmittance mode to predict the quality of beef cuts Warner–Bratzler shear value (tenderness) ($R=0.798-0.826$), protein ($R=0.822-0.904$), moisture ($R=0.895-0.941$), fat ($R=0.890-0.965$) and energy content ($R=0.899-0.961$) were successfully predicted using both modes. Park et al. (2001) also developed models for predicting the tenderness, i.e. Warner–Bratzler shear value of beef using NIR reflectance spectra and principal component regression (PCR). The coefficient of determination of the developed models were of a similar order ($R^2=0.612-0.692$). This technology has also been commercially investigated with instruments such as the QualitySpec BT Spectrometer from Analytical Spectral Devices (Fig. 4.7).

NIR spectroscopy has also been investigated at laboratory scale for determination of the maximum temperature to which beef had been subjected to during a heat treatment (Ellekjaer and Isaksson 1992), species identification (Ding and Xu 1999) and authenticity assessment (Fumiere et al. 2000). Other applications of NIR such as the detection of faecal contamination on poultry have been studied. Windham et al. (2003) applied Vis–NIR spectroscopy to discriminate between uncontaminated poultry breast skin and faeces. They found that the developed model could successfully classify faecal-contaminated material due to spectral differences between faecal colour and myoglobin and/or hemoglobin content of the uncontaminated breast skin. However, hyperspectral imaging (Chap. 9) has also been utilized for such an application (Heitschmidt et al. 2007; Liu et al. 2007; Park et al. 2006a, b, 2007).

4.5.4.2 Ground Meat Quality

The quality of ground meat used as a raw material in products such as burgers and sausages is critical as processors must comply with product-type-dependent restrictions, i.e. chemical composition and origin of raw materials (Togersen et al. 1999). Togersen et al. (1999) utilized an on-line NIR sensor to determine the fat, water and protein contents in industrial-scale meat batches (beef and pork) in an industrial environment. The NIR sensor was installed at the outlet of a large meat grinder. The models developed had RMSECV of 0.82–1.49 %, 0.94–1.33 % and 0.35–0.70 % for fat, water and protein, respectively. Togersen et al. (2003) went on to predict the chemical composition of industrial-scale batches of frozen beef using a similar system. The resulting RMSECVs were 0.48–1.11 % (fat), 0.43–0.97 % (moisture) and 0.41–0.47 % (protein).

NIR spectroscopy has also been investigated as a tool for detecting adulteration of hamburgers (Ding and Xu 2000). They found it was possible to predict the level of adulterants in hamburgers with errors of 3.33, 2.99, 0.92 and 0.57 % for the adulterants mutton, pork, skim milk powder and wheat flour, respectively.

4.5.4.3 Meat Emulsion

Optical sensors have also been developed to monitor meat emulsion stability (Alvarez et al. 2007, 2009, 2010a, b). Initial work focused on prediction of meat emulsion stability using reflection photometry (Alvarez et al. 2007). They found that L* values increased at the beginning of chopping associated with reduced cooking losses, following 8 min of chopping there was a reduction in L* and b* values and an associated increase in cooking losses, which suggested the feasibility of an online optical sensor technology to predict the optimum end point of emulsification in the manufacture of finely comminuted meat products. These authors then recorded light backscatter intensity from beef emulsions manufactured with different fat/lean ratio and chopping duration using a dedicated fibre optic prototype (Alvarez et al. 2009). They found several optically derived parameters to be significantly correlated with fat loss during cooking. In subsequent work, they found normalized intensity decreased with increased chopping time as a result of emulsion homogenization, and with increased distance, chopping time had a positive correlation with fat losses during cooking, which in turn had a negative correlation with normalized light intensity and loss of intensity. Therefore, they suggest that light extinction spectroscopy could provide information about emulsion stability (Alvarez et al. 2010).

4.6 Future

Infrared spectroscopy has been demonstrated to be an excellent PAT tool for monitoring critical processes and prediction of quality indices during food processing. Advances in equipment design will assist in the deployment of infrared spectroscopy-based technologies as PAT tools in the food industry. This will include improvements in robustness, cost and advances in microspectrometers. However, where studies have primarily been at laboratory scale, further research is required to ensure appropriate scaling up and transfer of the technology to industry. The combined acquisition of spectral and spatial information through the use of hyperspectral imaging has a number of potential applications. However, further developments are required to reduce the cost and increase the acquisition and processing speed for it to be fully exploited in food quality and safety applications.

References

Abbott JA (1999) Quality measurement of fruits and vegetables. Postharvest Biol Technol 15:207–225

Alvarez D, Castillo M, Payne FA, Garrido MD, Banon S, Xiong YL (2007) Prediction of meat emulsion stability using reflection photometry. J Food Eng 82:310–315

Alvarez D, Castillo M, Payne FA, Xiong YL (2009) A novel fiber optic sensor to monitor beef meat emulsion stability using visible light scattering. Meat Sci 81:456–466

Alvarez D, Castillo M, Xiong YL, Payne FA (2010a) Prediction of beef meat emulsion quality with apparent light backscatter extinction. Food Res Int 43:1260–1266

Alvarez D, Castillo M, Payne FA, Cox RB, Xiong YL (2010b) Application of light extinction to determine stability of beef emulsions. J Food Eng 96:309–315

Ariana DP, Lu R, Guyer DE (2006) Near-infrared hyperspectral reflectance imaging for detection of bruises on pickling cucumbers. Comput Electron Agric 53:60–70

Baker JE, Dowell FE, Throne JE (1999) Detection of parasitized rice weevils in wheat kernels with near-infrared spectroscopy. Biol Control 16:88–90

Barbosa-García O, Ramos-Ortíz G, Maldonado JL, Pichardo-Molina JL, Meneses-Nava MA, Landgrave JEA, Cervantes-Martínez J (2007) UV-vis absorption spectroscopy and multivariate analysis as a method to discriminate tequila. Spectrochim Acta Part A Mol Biomol Spectrosc 66:129–134

Biswas AK, Sahoo J, Chatli MK (2011) A simple UV-Vis spectrophotometric method for determination of [beta]-carotene content in raw carrot, sweet potato and supplemented chicken meat nuggets. LWT—Food Science and Technology. 44:1809–1813

Blakey RJ, Bower JP, Bertling I (2009) Influence of water and ABA supply on the ripening pattern of avocado (Persea americana Mill.) fruit and the prediction of water content using near infrared spectroscopy. Postharvest Biol Technol 53:72–76

Blasco J, Aleixos N, Gómez J, Moltó E (2007) Citrus sorting by identification of the most common defects using multispectral computer vision. J Food Eng 83:384–393

Blazquez C, Downey G, O'Donnell C, O'Callaghan D, Howard V (2004) Prediction of moisture, fat and inorganic salts in processed cheese by near infrared reflectance spectroscopy and multivariate data analysis. J Near Infrared Spectrosc 12:149–157

Blazquez C, Downey G, O'Callaghan D, Howard V, Delahunty C, Sheehan E, Everard C, O'Donnell CP (2006) Modelling of sensory and instrumental texture parameters in processed cheese by near infrared reflectance spectroscopy. J Dairy Res 73:58–69

Boubellouta T, Karoui R, Lebecque A, Dufour E (2010) Utilisation of attenuated total reflectance MIR and front-face fluorescence spectroscopies for the identification of Saint-Nectaire cheeses varying by manufacturing conditions. Eur Food Res Technol 231:873–882

Brandt M, Haeussermann A, Hartung E (2010) Invited review: technical solutions for analysis of milk constituents and abnormal milk. J Dairy Sci 93:427–436

Burks CS, Dowell FE, Xie F (2000) Measuring fig quality using near-infrared spectroscopy. J Stored Prod Res 36:289–296

Buta JG, Qi L, Wang CY (1997) Fourier transform infrared spectra of zucchini squash stored at chilling or non-chilling temperatures. Environ Exp Bot 38:1–6

Cassandro M, Comin A, Ojala M, Zotto RD, De Marchi M, Gallo L, Carnier P, Bittante G (2008) Genetic parameters of milk coagulation properties and their relationships with milk yield and quality traits in Italian Holstein cows. J Dairy Sci 91:371–376

Castillo M, Payne FA, Hicks CL, Lopez MB (2000) Predicting cutting and clotting time of coagulating goat's milk using diffuse reflectance: effect of pH, temperature and enzyme concentration. Int Dairy J 10:551–562

Castillo M, Payne F, Shea A (2005a) Development of a combined sensor technology for monitoring coagulation and syneresis operations in cheese making. J Dairy Sci 88:142–142

Castillo M, Payne FA, Lopez MB, Ferrandini E, Laencina J (2005b) Optical sensor technology for measuring whey fat concentration in cheese making. J Food Eng 71:354–360

Cattaneo TMP, Tornelli C, Erini S, Panarelli EV (2008) Relationship between sensory scores and near infrared absorptions in characterising Bitto, an Italian protected denomination of origin cheese. J Near Infrared Spectrosc 16:173–178

Cecchinato A, De Marchi M, Gallo L, Bittante G, Carnier P (2009) Mid-infrared spectroscopy predictions as indicator traits in breeding programs for enhanced coagulation properties of milk. J Dairy Sci 92:5304–5313

Cimander C, Carlsson M, Mandenius C-F (2002) Sensor fusion for on-line monitoring of yoghurt fermentation. J Biotechnol 99:237–248

Clark CJ, McGlone VA, De Silva HN, Manning MA, Burdon J, Mowat AD (2004) Prediction of storage disorders of kiwifruit (Actinidia chinensis) based on visible-NIR spectral characteristics at harvest. Postharvest Biol Technol 32:147–158

Contreras U, Barbosa-García O, Pichardo-Molina JL, Ramos-Ortíz G, Maldonado JL, Meneses-Nava MA, Ornelas-Soto NE, López-de-Alba PL (2010) Screening method for identification of adulterate and fake tequilas by using UV-VIS spectroscopy and chemometrics. Food Res Int 43:2356–2362

Correia I, Nunes A, Duarte IF, Barros A, Delgadillo I (2005) Sorghum fermentation followed by spectroscopic techniques. Food Chem 90:853–859

Čurda L, Kukačková O (2004) NIR spectroscopy: a useful tool for rapid monitoring of processed cheese manufacture. J Food Eng 61:557–560

da Costa Filho PA, Volery P (2005) Broad-based versus specific NIRS calibration: determination of total solids in fresh cheese. Anal Chim Acta 554:82–88

Dal Zotto R, De Marchi M, Cecchinato A, Penasa M, Cassandro M, Carnier P, Gallo L, Bittante G (2008) Reproducibility and repeatability of measures of milk coagulation properties and predictive ability of mid-infrared reflectance spectroscopy. J Dairy Sci 91:4103–4112

De Marchi M, Fagan CC, O'Donnell CP, Cecchinato A, Dal Zotto R, Cassandro M, Penasa M, Bittante G (2009) Prediction of coagulation properties, titratable acidity, and pH of bovine milk using mid-infrared spectroscopy. J Dairy Sci 92:423–432

Ding HB, Xu RJ (1999) Differentiation of beef and kangaroo meat by visible/near-infrared reflectance spectroscopy. J Food Sci 64:814–817

Ding HB, Xu RJ (2000) Near-infrared spectroscopic technique for detection of beef hamburger adulteration. J Agric Food Chem 48:2193–2198

Downey G, Byrne S (1987) Protein determination of wheat in trade by near-infrared reflectance spectroscopy—calibration and instrument performance over a 4 year period. Sci Des Aliment 7:325–336

Downey G, Sheehan E, Delahunty C, O'Callaghan D, Guinee T, Howard V (2005) Prediction of maturity and sensory attributes of Cheddar cheese using near-infrared spectroscopy. Int Dairy J 15:701–709

Ellekjaer MR, Isaksson T (1992) Assessment of maximum cooking temperatures in previously heat-treated beef.1. Near-infrared spectroscopy. J Sci Food Agric 59:335–343

Everard CD, O'Callaghan DJ, Fagan CC, O'Donnell CP, Castillo M, Payne FA (2007) Computer vision and color measurement techniques for inline monitoring of cheese curd syneresis. J Dairy Sci 90:3162–3170

Fagan CC, O'Donnell CP (2007) Application of mid-infrared spectroscopy to food processing systems. In: Irudayaraj J, Reh C (eds) Nondestructive testing of food quality. Blackwell Publishing, Oxford, pp 119–142

Fagan CC, Castillo M, Payne FA, O'Donnell CP, Leedy M, O'Callaghan DJ (2007a) Novel online sensor technology for continuous monitoring of milk coagulation and whey separation in cheesernaking. J Agric Food Chem 55:8836–8844

Fagan CC, Everard C, O'Donnell CP, Downey G, Sheehan EM, Delahunty CM, O'Callaghan DJ (2007b) Evaluating mid-infrared spectroscopy as a new technique for predicting sensory texture attributes of processed cheese. J Dairy Sci 90:1122–1132

Fagan CC, Du CJ, O'Donnell CP, Castillo M, Everard CD, O'Callagran DJ, Payne FA (2008a) Application of image texture analysis for online determination of curd moisture and whey solids in a laboratory-scale stirred cheese vat. J Food Sci 73:E250–E258

Fagan CC, Castillo M, O'Donnell CP, O'Callaghan DJ, Payne FA (2008b) On-line prediction of cheese making indices using backscatter of near infrared light. Int Dairy J 18:120–128

Fagan C, Castillo M, O'Callaghan D, Payne F, O'Donnell C (2009a) Visible-near infrared spectroscopy sensor for predicting curd and whey composition during cheese processing. Sens Instrum Food Qual Saf 3:62–69

Fagan CC, O'Donnell CP, Rudzik L, Wust E (2009b) Milk and dairy products. In: Sun D-W (ed) Infrared spectroscopy for food quality analysis and control. Elsevier, San Diego, pp 241–273

Fernández-Ibañez V, Soldado A, Martínez-Fernández A, de la Roza-Delgado B (2009) Application of near infrared spectroscopy for rapid detection of aflatoxin B1 in maize and barley as analytical quality assessment. Food Chem 113:629–634

Fu X, Ying Y, Lu H, Xu H (2007) Comparison of diffuse reflectance and transmission mode of visible-near infrared spectroscopy for detecting brown heart of pear. J Food Eng 83:317–323

Fumiere O, Sinnaeve G, Dardenne P (2000) Attempted authentication of cut pieces of chicken meat from certified production using near infrared spectroscopy. J Near Infrared Spectrosc 8:27–34

Golic M, Walsh KB (2006) Robustness of calibration models based on near infrared spectroscopy for the in-line grading of stonefruit for total soluble solids content. Anal Chim Acta 555:286–291

Griffiths PR (2010) Theory and instrumentation for vibrational spectroscopy. In: Chalmers JM, Griffiths P, Li Chan E (eds) Applications of vibrational spectroscopy to food science. Wiley, Chichester

Hardy J, Fanni J (1981) Application of reflection photometry to the measurement of milk coagulation. J Food Sci 46:1956–1957

He Y, Wu D, Feng SJ, Li XL (2007) Fast measurement of sugar content of yogurt using Vis/NIR-spectroscopy. Int J Food Prop 10:1–7

Heitschmidt GW, Park B, Lawrence KC, Windham WR, Smith DP (2007) Improved hyperspectral imaging system for fecal detection on poultry carcasses. Trans ASABE 50:1427–1432

Hernández-Hierro JM, García-Villanova RJ, González-Martín I (2008) Potential of near infrared spectroscopy for the analysis of mycotoxins applied to naturally contaminated red paprika found in the Spanish market. Anal Chim Acta 622:189–194

ISO (2006) 21543:2006 Milk products—guidelines for the application of near infrared spectrometry. International Organization for Standardization, Geneva

Karoui R, Dufour E, Pillonel L, Picque D, Cattenoz T, Bosset JO (2004) Determining the geographic origin of Emmental cheeses produced during winter and summer using a technique based on the concatenation of MIR and fluorescence spectroscopic data. Eur Food Res Technol 219:184–189

Karoui R, Dufour E, Pillonel L, Schaller E, Picque D, Cattenoz T, Bosset JO (2005a) The potential of combined infrared and fluorescence spectroscopies as a method of determination of the geographic origin of Emmental cheeses. Int Dairy J 15:287–298

Karoui R, Bosset JO, Mazerolles G, Kulmyrzaev A, Dufour E (2005b) Monitoring the geographic origin of both experimental French Jura hard cheeses and Swiss Gruyere and L'Etivaz PDO cheeses using mid-infrared and fluorescence spectroscopies: a preliminary investigation. Int Dairy J 15:275–286

Karoui R, Mouazen AM, Dufour É, Pillonel L, Schaller E, Baerdemaeker J, Bosset JO (2006) Chemical characterisation of European Emmental cheeses by near infrared spectroscopy using chemometric tools. Int Dairy J 16:1211–1217

Karoui R, Mazerolles G, Bosset JO, de Baerdemaeker J, Dufour E (2007a) Utilisation of mid-infrared spectroscopy for determination of the geographic origin of Gruyere PDO and L'Etivaz PDO Swiss cheeses. Food Chem 105:847–854

Karoui R, Pillonel L, Schaller E, Bosset JO, De Baerdemaeker J (2007b) Prediction of sensory attributes of European Emmental cheese using near-infrared spectroscopy: a feasibility study. Food Chem 101:1121–1129

Karoui R, De Baerdemaeker J, Dufour E (2008) A comparison and joint use of mid infrared and fluorescence spectroscopic methods for differentiating between manufacturing processes and sampling zones of ripened soft cheeses. Eur Food Res Technol 226:861–870

Kawamura S, Natsuga M, Takekura K, Itoh K (2003) Development of an automatic rice-quality inspection system. Comput Electron Agric 40:115–126

Kawamura S, Kawasaki M, Nakatsuji H, Natsuga M (2007) Near-infrared spectroscopic sensing system for online monitoring of milk quality during milking. Sens Instrum Food Qual Saf 1:37–43

Khanmohammadi M, Garmarudi AB, Ghasemi K, Garrigues S, de la Guardia M (2009) Artificial neural network for quantitative determination of total protein in yogurt by infrared spectrometry. Microchem J 91:47–52

Khurana HK, Jun S, Cho IK, Li QX (2008) Rapid determination of sugars in commercial fruit yogurts and yogurt drinks using fourier transform infrared spectroscopy and multivariate analysis. Appl Eng Agric 24:631–636

Kim J, Mowat A, Poole P, Kasabov N (2000) Linear and non-linear pattern recognition models for classification of fruit from visible-near infrared spectra. Chemom Intell Lab Syst 51:201–216

Kocaoglu-Vurma NA, Eliardi A, Drake MA, Rodriguez-Saona LE, Harper WJ (2009) Rapid profiling of Swiss cheese by attenuated total reflectance (ATR) infrared spectroscopy and descriptive sensory analysis. J Food Sci 74:S232–S239

Krivoshiev GP, Chalucova RP, Moukarev MI (2000) A possibility for elimination of the interference from the peel in nondestructive determination of the internal quality of fruit and vegetables by VIS/NIR spectroscopy. Lebensm-Wiss Technol 33:344–353

Lammertyn J, Peirs A, De Baerdemaeker J, Nicolaï B (2000) Light penetration properties of NIR radiation in fruit with respect to non-destructive quality assessment. Postharvest Biol Technol 18:121–132

Larrain M, Guesalaga AR, Agosin E (2008) A multipurpose portable instrument for determining ripeness in wine grapes using NIR spectroscopy. IEEE Trans Instrum Measurement 57:294–302

Lawrence RC, Gilles J (1980) The assessment of the potential quality of young Cheddar cheese. N Z J Dairy Sci Technol 15:1–12

Lee SJ, Jeon IJ, Harbers LH (1997) Near infrared reflectance spectroscopy for rapid analysis of curds during cheddar cheese making. J Food Sci 62:53–56

Liu YL, Chen YR, Kim MS, Chan DE, Lefcourt AM (2007) Development of simple algorithms for the detection of fecal contaminants on apples from visible/near infrared hyperspectral reflectance imaging. J Food Eng 81:412–418

Maertens K, Reyns P, Baerdemaeker JD (2004) On-line measurement of grain quality with nir technology. Trans ASAE 47:1135–1140

Manrique GD, Lajolo FM (2002) FT-IR spectroscopy as a tool for measuring degree of methyl esterification in pectins isolated from ripening papaya fruit. Postharvest Biol Technol 25:99–107

Martinsen P, Schaare P (1998) Measuring soluble solids distribution in kiwifruit using near-infrared imaging spectroscopy. Postharvest Biol Technol 14:271–281

Mateo MJ, O'Callaghan DJ, Everard CD, Fagan CC, Castillo M, Payne FA, O'Donnell CP (2009) Influence of curd cutting programme and stirring speed on the prediction of syneresis indices in cheese-making using NIR light backscatter. Lwt-Food Sci Technol 42:950–955

McGlone AV, Martinsen PJ (2004) Transmission measurements on intact apples moving at high speed. J Near Infrared Spectrosc 12:37–43

McGlone VA, Jordan RB, Martinsen PJ (2002) Vis/NIR estimation at harvest of pre- and post-storage quality indices for Royal Gala apple. Postharvest Biol Technol 25:135–144
McKenna D (2001) Measuring moisture in cheese by near infrared absorption spectroscopy. J AOAC Int 84:623–628
McMahon DJ, Brown RJ, Ernstrom CA (1984) Enzymic coagulation of milk casein micelles. J Dairy Sci 67:745–748
McQueen DH, Wilson R, Kinnunen A, Jensen EP (1995) Comparison of two infrared spectroscopic methods for cheese analysis. Talanta 42:2007–2015
Mitsumoto M, Maeda S, Mitsuhashi T, Ozawa S (1991) Near-infrared spectroscopy determination of physical and chemical characteristics in beef cuts. J Food Sci 56:1493–1496
Moros J, Inon FA, Khanmohammadi M, Garrigues S, De la Guardia M (2006) Evaluation of the application of attenuated total reflectance-Fourier transform infrared spectrometry (ATR-FT-IR) and chemometrics to the determination of nutritional parameters of yogurt samples. Anal Bioanal Chem 385:708–715
Navratil M, Cimander C, Mandenius CF (2004) On-line multisensor monitoring of yogurt and Filmjolk fermentations on production scale. J Agric Food Chem 52:415–420
Nicolaï BM, Beullens K, Bobelyn E, Peirs A, Saeys W, Theron KI, Lammertyn J (2007) Nondestructive measurement of fruit and vegetable quality by means of NIR spectroscopy: a review. Postharvest Biol Technol 46:99–118
Norris KH, Williams PC (1979) Determination of protein and moisture in hrs wheat by near-infrared reflectance spectroscopy1. Comparative-study of 12 instrumental methods. Cereal Foods World 24:459–459
Oberreuter H, Brodbeck A, von Stetten S, Goerges S, Scherer S (2003) Fourier-transform infrared (FT-IR) spectroscopy is a promising tool for monitoring the population dynamics of microorganisms in food stuff. Eur Food Res Technol 216:434–439
Ortiz C, Barreiro P, Correa E, Riquelme F, Ruiz-Altisent M (2001) PH-Postharvest technology: non-destructive Identification of woolly peaches using impact response and near-infrared spectroscopy. J Agric Eng Res 78:281–289
Park B, Chen YR, Hruschka WR, Shackelford SD, Koohmaraie M (2001) Principal component regression of near-infrared reflectance spectra for beef tenderness prediction. Trans ASAE 44:609–615
Park B, Lawrence KC, Windham WR, Smith DP (2006a) Performance of hyperspectral imaging system for poultry surface fecal contaminant detection. J Food Eng 75:340–348
Park B, Lawrence KC, Windham WR, Smith DP (2006b) Performance of supervised classification algorithms of hyperspectral imagery for identifying fecal and ingesta contaminants. Trans ASABE 49:2017–2024
Park B, Yoon SC, Lawrence KC, Windham WR (2007) Fisher linear discriminant analysis for improving fecal detection accuracy with hyperspectral images. Trans ASABE 50:2275–2283
Pasikatan MC, Dowell FE (2001) Sorting systems based on optical methods for detecting and removing seeds infested internally by insects or fungi: a review. Appl Spectrosc Rev 36:399–416
Payne FA, Hicks CL, Shen PS (1993) Predicting optimal cutting time of coagulating milk using diffuse reflectance. J Dairy Sci 76:48–61
Pearse MJ, Mackinlay AG (1989) Biochemical aspects of syneresis: a review. J Dairy Sci 72:1401–1407
Peirs A, Lammertyn J, Ooms K, NicolaI BM (2001) Prediction of the optimal picking date of different apple cultivars by means of VIS/NIR-spectroscopy. Postharvest Biol Technol 21:189–199
Pérez-Marín MD, Garrido-Varo A, Serradilla JM, Núnez N, Ares JL, Sánchez J (2001) Chemical and microbial analysis of goat’s milk, cheese and whey by near infrared spectroscopy. In: Davies AMC, Cho RK (eds) Near infrared spectroscopy: proceedings of the 10th international conference. NIR Publications, West Sussex, pp 225–228
Pérez-Marín D, Sánchez M-T, Paz P, Soriano M-A, Guerrero J-E, Garrido-Varo A (2009) Nondestructive determination of quality parameters in nectarines during on-tree ripening and postharvest storage. Postharvest Biol Technol 52:180–188

Pillonel L, Luginbuhl W, Picque D, Schaller E, Tabacchi R, Bosset JO (2003) Analytical methods for the determination of the geographic origin of Emmental cheese: mid- and near-infrared spectroscopy. Eur Food Res Technol 216:174–178
Rambla FJ, Garrigues S, de la Guardia M (1997) PLS-NIR determination of total sugar, glucose, fructose and sucrose in aqueous solutions of fruit juices. Anal Chim Acta 344:41–53
Reh C (2001) In-line and off-line FTIR measurements. In: Kress-Rogers E, Brimelow C (eds) Instrumentation and sensors for the food industry. Woodhead & CRC Press, Cambridge & Boca Raton, pp 213–232
Ren G, Chen F (1997) Determination of moisture content of ginseng by near infra-red reflectance spectroscopy. Food Chem 60:433–436
Ripoche A, Guillard AS (2001) Determination of fatty acid composition of pork fat by Fourier transform infrared spectroscopy. Meat Sci 58:299–304
Rodriguez Otero JL, Hermida M, Cepeda A (1994) Determination of fat, protein, and total solids in cheese by near-infrared reflectance spectroscopy. J AOAC Int 78:802–806
Rodriguez-Saona LE, Fry FS, McLaughlin MA, Calvey EM (2001) Rapid analysis of sugars in fruit juices by FT-NIR spectroscopy. Carbohydr Res 336:63–74
Saranwong S, Sornsrivichai J, Kawano S (2004) Prediction of ripe-stage eating quality of mango fruit from its harvest quality measured nondestructively by near infrared spectroscopy. Postharvest Biol Technol 31:137–145
Schmilovitch Ze, Mizrach A, Hoffman A, Egozi H, Fuchs Y (2000) Determination of mango physiological indices by near-infrared spectrometry. Postharvest Biol Technol 19:245–252
Scotter C (1990) Use of near infrared spectroscopy in the food industry with particular reference to its applications to on/in-line food processes. Food Control 1:142–149
Shao YN, He Y (2009) Measurement of soluble solids content and pH of yogurt using visible/near infrared spectroscopy and chemometrics. Food Bioprocess Technol 2:229–233
Shao YN, He Y, Feng SJ (2007) Measurement of yogurt internal quality through using Vis/NIR spectroscopy. Food Res Int 40:835–841
Singh PC, Bhamidipati S, Singh RK, Smith RS, Nelson PE (1996) Evaluation of in-line sensors for prediction of soluble and total solids/moisture in continuous processing of fruit juices. Food Control 7:141–148
Souto UTCP, Pontes MJC, Silva EC, Galvão RKH, Araújo MCU, Sanches FAC, Cunha FAS, Oliveira MSR (2010) UV-Vis spectrometric classification of coffees by SPA-LDA. Food Chem 119:368–371
Taifi N, Bakkali F, Faiz B, Moudden A, Maze G, D., D (2006) Characterization of the syneresis and the firmness of the milk gel using an ultrasonic technique. Meas Sci Technol 17:281–287
Tellier C, Mariette F, Guillement J-P, Marchel P (1993) Evolution of water proton muclear magnetic relaxation during milk coagulation and syneresis:structural implications. J Agric Food Chem 41:2259–2266
Togersen G, Isaksson T, Nilsen BN, Bakker EA, Hildrum KI (1999) On-line NIR analysis of fat, water and protein in industrial scale ground meat batches. Meat Sci 51:97–102
Togersen G, Arnesen JF, Nilsen BN, Hildrum KI (2003) On-line prediction of chemical composition of semi-frozen ground beef by non-invasive NIR spectroscopy. Meat Sci 63:515–523
Tsenkova R, Atanassova S, Kawano S, Toyoda K (2001) Somatic cell count determination in cow's milk by near-infrared spectroscopy: a new diagnostic tool. J Anim Sci 79:2550–2557
Ventura M, de Jager A, de Putter H, Roelofs FPMM (1998) Non-destructive determination of soluble solids in apple fruit by near infrared spectroscopy (NIRS). Postharvest Biol Technol 14:21–27
Villé H, Maes G, De Schrijver R, Spincemaille G, Rombouts G, Geers R (1995) Determination of phospholipid content of intramuscular fat by fourier transform infrared spectroscopy. J Meat Sci 41:283–291
Wang D, Ram MS, Dowell FE (2002) Classification of damaged soybean seeds using near-infrared spectroscopy. Trans ASAE 45:1943–1948
Williams PC (1979) Screening wheat for protein and hardness by near-infrared reflectance spectroscopy. Cereal Chem 56:169–172

Williams PC, Cordeiro HM (1979) Determination of protein and moisture in Hrs wheat by near-infrared reflectance spectroscopy 2. Influence of degrading factors, dockage and wheat variety. Cereal Foods World 24:460–460

Williams PC, Cordeiro HM (1981) Determination of protein and moisture in hard red spring wheat by near-infrared reflectance spectroscopy—influence of degrading factors, dockage, and wheat variety. Cereal Foods World 26:124–128

Windham WR, Lawrence KC, Park B, Buhr RJ (2003) Visible/NIR spectroscopy for characterizing fecal contamination of chicken carcasses. Trans ASAE 46:747–751

Wittrup C, Nørgaard L (1998) Rapid near infrared spectroscopic screening of chemical parameters in semi-hard cheese using chemometrics. J Dairy Sci 81:1803–1809

Woodcock T, Fagan CC, O’Donnell CP, Downey G (2008) Application of near and mid-infrared spectroscopy to determine cheese quality and authenticity. Food Bioprocess Technol 1:117–130