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Application of Near and Mid-Infrared Spectroscopy to Determine Cheese Quality and Authenticity

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Abstract This paper reviews the current state of development of both near-infrared (NIR) and mid-infrared (MIR) spectroscopic techniques for process monitoring, quality control, and authenticity determination in cheese processing. Infrared spectroscopy has been identified as an ideal process analytical technology tool, and recent publications have demonstrated the potential of both NIR and MIR spectroscopy, coupled with chemometric techniques, for monitoring coagulation, syneresis, and ripening as well as determination of authenticity, composition, sensory, and rheological parameters. Recent research is reviewed and compared on the basis of experimental design, spectroscopic and chemometric methods employed to assess the potential of infrared spectroscopy as a technology for improving process control and quality in cheese manufacture. Emerging research areas for these technologies, such as cheese authenticity and food chain traceability, are also discussed.

Keywords Spectroscopy · Cheese · Authenticity · Quality · Near-infrared · Mid-infrared · Process analytical technology

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Introduction

In common with the processed food industry at large, the dairy industry has come under increasing pressure to deliver products of high and constant quality into the market place (Downey et al. 2005). Globally, cheese represents about 30% of total dairy product sales with a forecast of 9.8% sales growth between 2003 and 2007 (Farkye 2004). It is important to determine cheese quality in a rapid and cost-effective manner.

The chemical characteristics of cheeses have been traditionally undertaken by different physico-chemical methods to determine pH, fat content, nitrogen fractions, volatile fatty acids, organic acids, and so on (Karoui et al. 2007). These methods can be labour-intensive and expensive. 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 online and in real-time. The potential of techniques such as infrared spectroscopy, ultrasound, and computer vision to fulfil these needs have been examined. Food manufacturers are also required to demonstrate the authenticity of their products. The rights of consumers and genuine food processors in terms of food adulteration and fraudulent or deceptive practices in food processing are set out in a recent European Union regulation regarding food safety and traceability (European Commission 2002). The European Union systems for the promotion and protection of food products of recognized quality and origin and selected examples of protected cheeses are listed in Table 1.

Considerable work has been carried out in the area of cheese quality and authenticity determination using near-infrared (NIR) and mid-infrared (MIR) spectroscopic techniques at laboratory scale. Recent technical developments in NIR and MIR spectroscopy and chemometrics will facilitate

Table 1 European Commission systems for the promotion and protection of food products of recognized quality and origin and selected examples of protected cheeses

System	Legislation	Types of food protected	No of cheeses protected	Selected examples of protected cheeses
Protected designation of origin (PDO)	European Council Regulation (EC) No 2081/92	Foodstuffs which are produced, processed, and prepared in a given geographical area using recognized know-how	139	Fromage de Herve (Belgium) Feta (Greece) Manouri (Greece) Cabrales (Spain) Beaufort (France) Salers (France) Imokilly Regato (Ireland) Bitto (Italy) Buxton Blue (United Kingdom)
Protected geographical indication (PGI)	European Council Regulation (EC) No 2081/92	Foodstuffs in which a common geographical link occurs in at least one of the stages of production, processing, or preparation	12	Danablu (Denmark) Esrom (Denmark) Queso de Valderón (Spain) Emmental de Savoie (France) Tomme de Savoie (France) Tomme de Pyrénées (France) Svecia (Sweden) Teviotdale cheese (United Kindom)
Traditional speciality guaranteed (TSG)	European Council Regulation (EC) No 2082/92	Foodstuffs possessing a traditional character, either n the composition or means of production	2	Mozzerella (Italy) Hushållsost (Sweden)

the transfer of this technology from laboratory to online application, which, in addition to the rapid, non-destructive and relatively low-cost nature of infrared spectroscopy, make it an ideal process analytical technology (PAT) tool.

PAT is a system for designing, analyzing, and controlling manufacturing through timely measurements of critical quality and performance attributes of raw and in-process materials and processes (Balboni 2003). The implementation of a PAT system in cheese manufacture would assist in achieving the production goal of a consistently high-quality product. However, this requires the control of the manufacturing process through real-time analysis of critical quality parameters. Therefore, PAT tools, i.e., techniques and technologies, which can rapidly, accurately, and preferably non-destructively assess the quality and functional properties of cheese, such as infrared spectroscopy, are essential for the modern cheese industry. This will allow for increased process monitoring and control of cheese manufacturing.

The majority of published research focuses on one particular type of cheese, one quality parameter or one sensing technology. The objective of this review was to present a comprehensive overview of recent developments in the area of cheese quality and authenticity determination using NIR and MIR spectroscopy. This will facilitate a full assessment of the potential of these techniques as PAT tools in the areas of process monitoring, quality control and authenticity determination of cheese. The review will be divided into sections according to the parameter of the cheese being measured, i.e., process monitoring of coagulation, syneresis, and ripening and determination of authenticity, composition, sensory, and rheological parameters. A brief overview of NIR and MIR spectroscopy and chemometric analysis will also be provided.

Overview of NIR and MIR Spectroscopy

NIR radiation is defined as that wavelength region from 750 to 2,500 nm lying between the visible light and the infrared light (Büning-Pfaue 2003). NIR spectroscopy is a physical, non-destructive high-precision technology requiring minimal or no sample preparation. It is also well suited to online use. Once calibrated, an NIR spectrometer may be operated with minimal training. A typical NIR food spectrum has two dominant and broad peaks located near 1,440 and 1,930 nm. These peaks are due to water and are responsible for some typical complications encountered in chemometric analysis. Effects of hydrogen bonding and sample temperature are also found to affect the reliability of NIR spectroscopic results (Büning-Pfaue 2003). The main disadvantage of NIR spectroscopy is its weak sensitivity to minor constituents such as salt and water-soluble nitrogen. The sensitivity limit is about 0.1% for most constituents

(Iwamoto and Kawano 1992). The development of instrumentation, measurement techniques, and chemometrics applicable to the food industry has been widely reviewed in a number of recent publications (Benson 2003; Penner 2003; Wehling 2003; Millar 2004; Sayago et al. 2004). The first NIR spectra of casein, fat, lactose, and powdered milk were obtained by Goulden (1957). NIR spectroscopy has traditionally been applied for the measurement of compositional parameters of food products. However, it can also be used for the determination of complex quality properties such as texture and sensory attributes. Figure 1 shows examples of NIR spectra of processed cheese.

MIR Spectroscopy is the measurement of the wavelength and intensity of the absorption of the mid-infrared range (4,000–200 cm^{-1}, 2,500–50,000 nm) by a sample. The MIR range is sufficiently energetic to excite molecular vibrations to higher energy levels. The wavelength of MIR absorption bands are characteristic of specific types of chemical bonds, and the main application of MIR spectroscopy is the identification of organic and organometallic molecules. This analytical technique is employed worldwide in industrial quality control. Its application to quantitative studies has increased during the last decade, propelled by its straightforward and powerful linkage to chemometric (multivariate) methods (Andrade et al. 2003). Particular advantages of MIR spectroscopy are speed of measurement, moderate instrument cost, and relative ease of sample presentation (Downey 1998). MIR spectroscopy has become more accessible to food samples after the introduction of attenuated total reflection (ATR) sample presentation systems (Downey et al. 2005). Examples of MIR spectra of Cheddar cheese can be seen in Fig. 2.

Chemometrics

Chemometrics can be described as going beyond the limitations of univariate statistics using multivariate methodologies derived from mathematics, statistics, and computer science (Geladi 2003). Multivariate calibration and multivariate classification are two of the most common multivariate methodologies. Multivariate approaches can be used to overcome problems in spectroscopy such as collinearity, i.e., where variables in the calibration have high correlations between them or where it is difficult to select a specific wavelength for calibration, as infrared spectrum frequently contain data points carrying overlapping information. Multivariate calibration approaches such as principal component analysis (PCA) and partial least-squares regression may be used to remove these redundancies from the data. These techniques transform the spectral data into a more useful form from which relevant information can be extracted. These techniques typically condense the original data points into as few as 10 principal component (PC) scores. These PC can then be used to develop calibration equations for the predication of the desired parameter, i.e., multivariate regression.

Classification methods can be divided into two groups, unsupervised and supervised classification. Unsupervised classification is usually called cluster analysis. It is an attempt to group samples into clusters based on specific distance measurements and is used when little or no information is available about group structures in the data. Cluster analysis can be preformed using either PCA or more formally using hierarchical methods which produce dendrograms (Næs et al. 2002). Using plots of PC scores

Fig. 1 NIR spectra of processed cheese samples between 400 and 2,600 nm

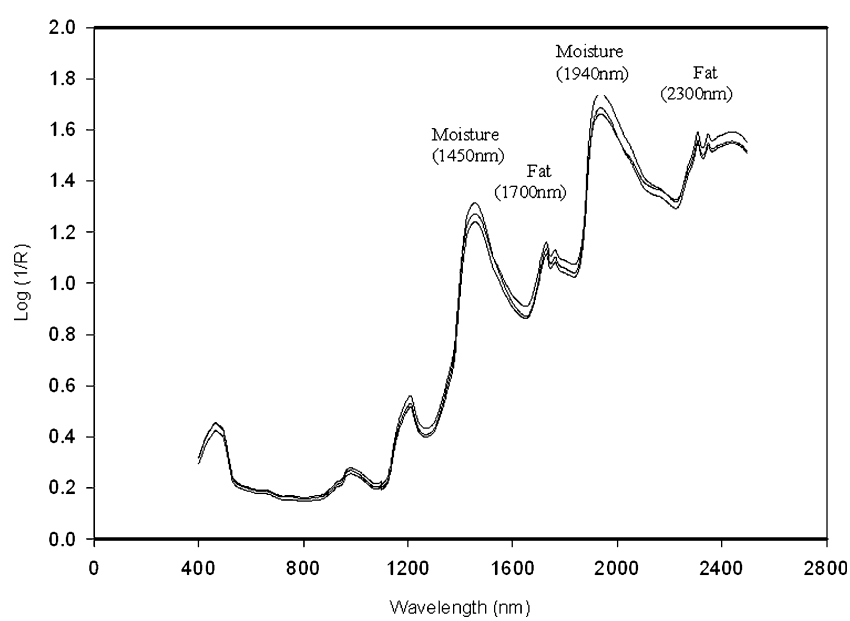
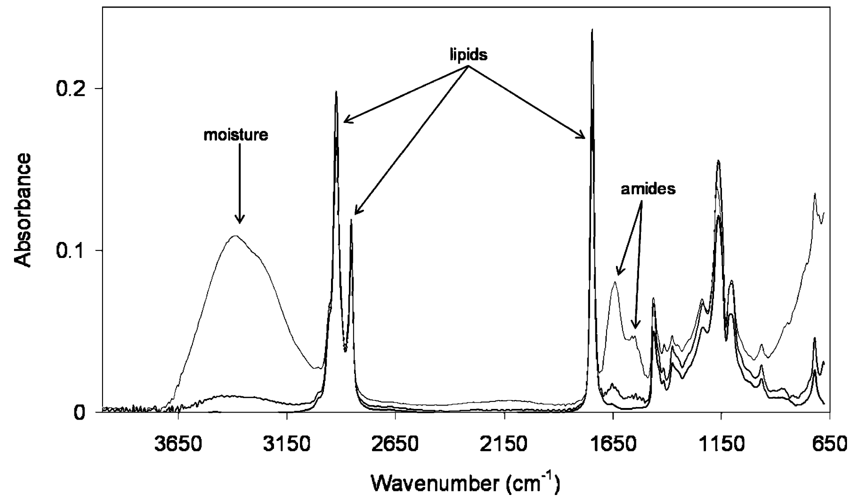


Fig. 2 MIR spectra of Cheddar cheese samples between 650 and 4,000 cm^{-1}



against one another can often reveal patterns or clustering within a data set (Wilson et al. 2001). Discriminant analysis, or supervised classification, is primarily used to build classification rules for a number of known subgroups. New samples are then assigned to the most likely subgroup based on these rules. There are many techniques used to carry out discriminant analysis, and they are detailed by Næs et al. (2002).

Recent results have shown that spectroscopic methods in combination with multivariate statistical analysis have broad applications for the investigation of food properties and structures (Karoui and Dufour 2003).

Control of Coagulation and Syneresis During Cheese Processing

The first step in cheese manufacture is the transformation of milk into a gel. This is often achieved by adding an enzyme to destabilize the casein micelles. When sufficient micelles are destabilized, they aggregate together, forming a gel network (coagulum); the time taken to form this network is called the gel time. Over time, the firmness of the coagulum continues to develop until it is sufficiently firm to cut—this time is known as the cutting time. Several studies have indicated the importance of obtaining objective online measurements for monitoring gel time, coagulum firmness, and cutting time during cheese manufacture to obtain high quality and consistent cheese products (Hori 1985; Payne et al. 1993b). Originally, the determination of the cutting time was established by the cheese maker. Although accurate, this method is not feasible in closed commercial vats. This, together with an increased desire for automation in the cheese industry, has led to several online coagulation monitoring systems being developed. During milk coagulation, the time at which the gel is cut directly affects the quality of the resulting curd, and hence, the finished cheese. If the gel is too firm when cut, the result will be higher losses of curd and fat. An increase in cheese moisture will also occur if the gel is cut before the optimum time. This has resulted in the development of a number of online sensors which can be used to successfully monitor milk coagulation. O'Callaghan et al. (2002) comprehensively reviewed a number of systems (optical, thermal, mechanical, and vibrational) for monitoring curd setting during cheesemaking. NIR optical techniques can offer online monitoring of coagulation without causing damage to the forming curd (O'Callaghan et al. 2002).

A selection of critical studies which have advanced the application of NIR radiation to monitoring coagulation are reviewed.

One of the earliest methods which utilized the changes in the optical properties of the milk included monitoring absorbance (McMahon et al. 1984). A Beckman DU-8B UV/Vis spectrophotometer was used to monitor apparent absorbance of the coagulating milk. The resulting measurements were a direct result of light scattering changes caused by changes of molecular weight, size, and a number of colloidal casein micelle aggregates. Although such methods were found to monitor coagulation, they found little usage. However, developments in fiber optics have overcome many of the problems associated with these techniques. Radiation in the NIR region can be transmitted through a fiber-optic bundle and diffuse reflectance or transmission monitored. As the gel is formed, reflectance will increase while transmission will decrease.

Payne et al. (1993a, b) developed methods based on changes in diffuse reflectance during milk coagulation utilizing photodiode light sources at 940 and 950 nm. In both cases, cutting time was predicted (Table 2) using parameters derived from the NIR reflectance profiles recorded during coagulation. However, these studies only monitored coagulation at a single wavelength. Laporte et al. (1998) used full spectrum information and multivariate

Table 2 Application of NIR spectroscopy in control of coagulation and syneresis during cheese processing

Parameter predicted	Measurement mode	Wavelength range (nm)	Prediction error	Reference
Coagulation				
K20[a]	*R*	940	SE=2.45 min	Payne et al. 1993b
t_c[b]	*R*	950	SE=1.5–2.4 min	Payne et al. 1993a
% Coagulation	*R*	1,100–2,500	SEP=0.26%	Laporte et al. 1998
t_{cr}[c]	*R*; *T*; *T*	880; 680; 850	SE=~2 min	O'Callaghan et al. 1999
Syneresis				
CPVF[d]	R	700	SE=23%	Guillemin et al. 2006
CPSD[e]	R	700	SE=7.5%	Guillemin et al. 2006
Whey fat losses	R	980	SEP=2.37 g/100 g	Fagan et al. 2007e
Curd Yield	R	980	SEP=0.91%	Fagan et al. 2007e
Final curd moisture	R	980	SEP=1.28%	Fagan et al. 2007e
Curd moisture[f]	R	980	SEP=1.27%	Fagan et al. 2007e

[a] Time taken for development of 20-mm spread on the Formagraph (Foss Food Technology, Minneapolis, MN)
[b] Time when the gel could be pulled from wall of container for a distance of ~5 mm using a spatula
[c] Time taken for the storage modulus to reach 20 Pa
[d] Casein particle volume fraction
[e] Casein particle size distribution
[f] Curd moisture as a function of syneresis time, i.e., time from gel cutting

partial least-squares modeling. Reflectance was monitored during coagulation and spectra collected between 1,100 and 2,500 nm with a resolution of 2 nm using a NIRSystems scanning spectrophotometer (Perstorp Analytical, Silver Springs, MD) equipped with a coaxial fiber optic probe fixed in the cheese vat and used to predict the percentage of coagulation (Table 2).

Table 2 also summarizes the results of O'Callaghan et al. (1999) in which a range of online sensors, including a fiber-optic NIR reflectance probe (CoAguLite, Reflectronics, Lexington, KY) operating at a wavelength of 880 nm and two NIR transmission probes, a food grade sensor operating at a wavelength of 680 nm (TrxPro, Zellweger Analytics, Sutton-in-Ashfield, UK) and a Gelograph NT (Gel Instrumente, Thalwil, Switzerland) operating at 850 nm were compared. Further work by O'Callaghan et al. (2000) compared the response of three of these NIR sensors, the Gelograph NT, TxPro, and CoAguLite, i.e., two transmission and one reflectance sensors, to the response of thermal (Hot-Wire sensor, INRA Thiverval-Grignon, France) and vibrational sensors where protein and enzyme concentration was varied. While they found that all sensors were sensitive to changes in enzyme concentration, the NIR sensors were more sensitive to changes in the rate of curd firming because of varying protein levels than the hot wire and torsional vibrational systems. Indeed, the NIR sensors predicted the curd firming time with a standard error less than 100 s. However, O'Callaghan et al. (2000) also stated that an algorithm combining the output of an NIR sensor as well as protein level was required to accurately predict curd firming times. This was also confirmed by O'Callaghan et al. (2001) who found that while the hot wire sensor displayed poor sensitivity to the rate of curd firming, an optical sensor detecting reflection at 660 nm could detect different rates of curd firming under varied condition of enzyme level, pH, and temperature. Mertens et al. (2002) have further examined milk coagulation NIR reflectance profiles and introduced the use of a model to study shape variability of the NIR signals for cutting time prediction.

Cutting of the coagulum initiates syneresis, which is the expulsion of whey from curd particles. Once the whey is drained, the curd is processed into cheese. The rate and extent of syneresis significantly impacts final product quality and yield. However, current practice in industry is to empirically control syneresis by varying process parameters such as temperature. The use of an online sensor technology for monitoring syneresis would facilitate the control of curd moisture content, and hence, the production of a high-quality and consistent cheese product. Hence, the development of a syneresis control technology is another emerging area of research (Guillemin et al. 2006; Fagan et al. 2007e, f). Currently, only regions of the NIR spectra have been investigated as a technology for monitoring syneresis (Guillemin et al. 2006; Fagan et al. 2007c, e, f; Table 2).

Fagan et al. (2007c) proposed that a sensor detecting near-infrared light backscatter (300–1,100 nm) in a cheese vat and with a large field of view (LFV) relative to curd particle size would have potential for monitoring both milk coagulation and curd syneresis. Figure 3 shows an example of the response of the LFV sensor during both coagulation and syneresis at wavelengths between 515 and 900 nm. Fagan et al. (2007c) reported that the response of the prototype sensor was affected by temperature and that the sensor showed potential for predicting whey fat content,

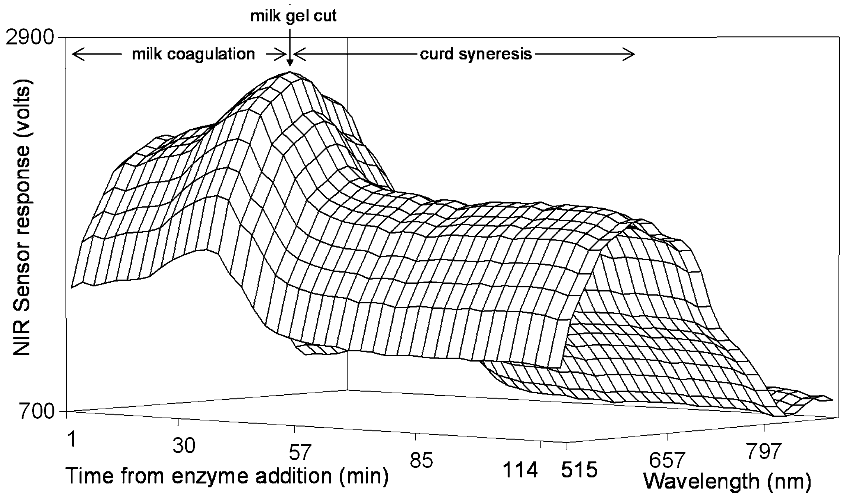


Fig. 3 Spectral response of the NIR large field of view sensor between 515 and 900 nm during milk coagulation and curd syneresis

curd moisture content, and curd yield. However, the preliminary predictions presented in that study were limited in their use because of the small data set used and the reduced strength of the predictions. Further work by Fagan et al. (2007e) found that the LFV optical sensor provided the information on gel assembly and curd shrinkage kinetics required for accurate predictions of whey fat losses and curd yield as well as for curd moisture control (Table 2). Guillemin et al. (2006) modified a NIR milk coagulation sensor (700 nm) to investigate its potential for the online determination of casein particle size distribution and of the volume fraction relative to the whey as a function of time. It was found that utilizing multiple thresholds of the optical signal associated with data processing using neural networks provided useful results (Table 2). These studies highlight the growing number of potential areas in which infrared spectroscopy may be applied for coagulation and syneresis monitoring.

Monitoring of Cheese Ripening

During the maturation and ripening phases, cheese undergoes a complex series of chemical, bacterial, and enzymatic reactions which are responsible for breakdown of the protein matrix and ultimately the development of the texture and sensory characteristics that are typical of mature cheese. Therefore, determination of the degree of ripening or maturation of cheese is important, and as stated by Martín-del-Campo et al. (2007), having information available regarding the evolution of lipolysis, glycolysis, and proteolysis may help the cheese-maker to better understand the biochemical kinetics of ripening to improve the ripening process. Considerable interest exists in the development of instrumental techniques to enable more objective, faster, and less expensive assessments to be made in this area (Downey et al. 2005). Published material relating to the application of NIR and MIR spectroscopy to this end for a range of cheese types is outlined below.

NIR vs MIR Spectroscopy for Determining Ripening Stage

The main papers reviewed in this area use MIR spectroscopic techniques in analyzing the cheese (Chen et al. 1998; Kulmyrzaev et al. 2005; Lucia et al. 2001). It should be noted that Fourier transform infrared (FTIR) spectroscopy is the preferred method of MIR spectroscopy, and it will be used in this review to indicate when MIR spectra were obtained by FTIR spectroscopy. An FTIR instrument comprises of an interferometer which contains a beam splitter, a fixed mirror, and a movable mirror. When light from the source hits the beam splitter, the light is split in two, and each is directed towards one of the mirrors. One beam is reflected off the fixed mirror and the other off the movable mirror and they recombine back at the beam splitter. Because the path that one beam travels is a fixed length and the other is constantly changing as its mirror moves, the signal which exits the interferometer is the result of these two beams "interfering" with each other. This beam is directed through the sample compartment to the detector, producing an interferogram containing spectral information on the sample. The interferogram is then transformed digitally, from detector response vs optical path difference using a fast Fourier transform algorithm, providing a typical absorbance vs wavelength spectra. Chen et al. (1998) maintained that MIR has a distinct advantage over NIR because specific bands may be assigned to specific chemical entities. While it is acknowledged that NIR spectroscopy has shown great potential in applications which involve direct, rapid, and non-destructive quantification of major components in solid foods such as cheese, NIR requires large calibration sets and correlation methods, and it is difficult to use these methods to monitor biochemical events and secondary catabolic changes such as those which take place during cheese ripening. The R^2 values of 0.77 and 0.86 obtained for correlations between fat and amide (protein) groups with the proximate analysis indicated that this technique could provide a basis for a rapid analysis of fat and protein contents. It was concluded that it may be possible to obtain information on the protein secondary structure by proper masking of the water vapor and by using spectra enhancement techniques. Because the rate and products of lipolysis and proteolysis are important for flavor and texture development, this information could help dairy scientists understand the biochemical reactions as they monitor the maturation of cheese.

Dufour et al. (2000) demonstrated the potential of MIR spectroscopy to discriminate between semi-hard cheeses cheese during the first 3 months of ripening. The level of water-soluble nitrogen (WSN) in cheese is known to increase during ripening and can be taken as an indicator of cheese ripening. Karoui et al. (2006c) successfully predicted the WSN content of Emmental cheese using FTIR spectroscopy and a polyethylene card sample presentation technique (R^2=0.80), whereas Fagan et al. (2007d) successfully predicted the WSN content (R^2=0.88) and age (R^2=0.62) of Cheddar cheese using FTIR spectroscopy and an ATR sample presentation technique. Kulmyrzaev et al. (2005) also used FTIR spectroscopy, in this case, to monitor the rheological properties of soft cheese samples during ripening. It was concluded that FTIR spectroscopy combined with chemometric tools has the potential to evaluate structure at the molecular level, and hence, allows information on the molecular structure and interactions of the cheese matrix to be derived.

Wavelength Selection for Determining Ripening Stage

Chen et al. (1998) recorded spectra between 4,000 and 400 cm^{-1} with a resolution of 4 cm^{-1}. It was noted that water has a very strong broad band that absorbs highly in the range between 3,700 and 3,100 cm^{-1}, a weaker band around 2,000 cm^{-1}, and another strong band at 1,640 cm^{-1}; at about 800 cm^{-1}, water does not absorb. The moisture content of the cheeses affected the recorded spectra and the multiple N–H bonds in the 3,330- to 3,060-cm^{-1} region. This effect was eliminated by collecting the spectra for at least 10 min in the film holder of the FTIR under ambient conditions after equilibrium of the sample. This procedure gives enough time for free water in the sample to evaporate and produce a stable spectrum. Kulmyrzaev et al. (2005) recorded spectra between 3,000 and 900 cm^{-1} and used three separate sections of the MIR spectrum in their analysis. Common components and specific weights analysis showed that the common component 1 discriminating young and ripened cheeses explained 95, 92, and 73% of the inertia of the 900 to 1,500, 2,800 to 3,000, and 1,500 to 1,700 cm^{-1} infrared regions, respectively. For this study, therefore, the 900–1,500 cm^{-1} yields the best results. Lucia et al. (2001) recorded the MIR spectral region from 700 to 4,000 cm^{-1} for the evaluation of the proteolytic activity of *Yarrowia lipolytica* and its contribution to cheese ripening.

Cheese Composition Measurement

Cheeses are produced with a wide range of texture and compositional parameters. While variety of cheese type is deliberate and desirable, the quality of any given type of cheese is greatly determined by its texture, which, in turn, is influenced by moisture and other composition components and processing conditions. Manufacturers have traditionally depended on a wide range of chemical analysis techniques to quantify major food components such as moisture, protein, and fat. The potential of NIR and MIR spectroscopy to determine cheese compositional parameters is outlined below. An overview of the experimental designs, the spectroscopic techniques used, and the chemometric approaches employed will be presented. Applications of NIR and MIR spectroscopy to determine a range of compositional parameters for a range of cheese types are outlined in Table 3.

It should be noted that composition determination is one of the older and most established applications of infrared spectroscopy. This is clearly demonstrated in Table 3 with reference citing the prediction of cheese composition spanning from 1994 to 2005. This is in contrast to Table 4, which has references reporting on the prediction of cheese sensory and rheological properties ranging from 1998 to 2007, and in the case of authenticity determination, an emerging area of research, references are from 2003 to 2006 (Table 5).

Transmittance vs Reflectance Spectroscopy for Determining Composition

A novel study comparing NIR reflectance and transmittance spectroscopy in the area of cheese analysis was published by McKenna (2001). He stated that data in the literature indicate that the measurement of moisture in cheese by NIR transmittance spectroscopy is more accurate than by reflectance. The accuracy of NIR transmission measurement was calculated for Edam, Gouda, Brie, Colby, and Cheddar and a range of SEP values (0.12–0.35) were obtained using different methods of calibration for a number of types of cheese types. Close agreement between these results as they related to the precision of the reference method was observed. Despite this study stating that transmittance spectroscopy is more accurate than reflectance, it can be clearly observed from Table 3 that the

Table 3 Application of NIR and MIR spectroscopy in cheese composition analysis

Composition parameter	Spectral region	Data pretreatment	Mode[a]	Wavelength range	Prediction error	Reference
Moisture Content	NIR	SC[b] and 2nd D[c]	*R*[d]	400–2,498 nm	SECV=0.5	Blazquez et al. 2004
	NIR	Outliers identified	*R*[d]	900–2,500 nm	SEP=0.429	Cûrda and Kukačková 2002
	NIR	Smooth[e] and SNV[f]	*R*[d]	515–1,700 nm	RMSEP=1.72–2.21	da Costa Filho and Volery 2005
	NIR	Smooth[e]	*R*[d]	1,900–2,320 nm	SEP=0.889	Lee et al. 1997
	NIR	SC[b]	*R*[d]	2,500–10,000 cm^{-1}	SEP=0.12–0.35	McKenna 2001
	NIR	SC[b]	*T*[g]	2,500–10,000 cm^{-1}	SEP=0.12–0.35	McKenna 2001
	MIR	RD[h]	*R*[d]	5,000–400 cm^{-1}	SEP=0.04–0.09	McQueen et al. 1995
	NIR	RD[h]	*R*[d]	1,740–2,280 nm	SEP=0.02–0.05	McQueen et al. 1995
	NIR	RD[h], 1st and 2nd D[c]	*R*[d]	400–2,500 nm	SECV=0.05–0.92	Pérez–Marín et al. 2001
	NIR	1st, 2nd and 3rd D[c]	*R*[d]	400–2,498 nm	SEC=0.412	Rodriguez–Otero et al. 1994
	NIR	SC[b]	*R*[d]	400–2,500 nm	RMSEP=0.58	Wittrup and Nørgaard 1998
Fat Content	NIR	SC[b] and 2nd D[c]	*R*[d]	1,100–1,498 nm	SECV=0.45	Blazquez et al. 2004
	FT–NIR	Outliers identified	*R*[d]	900–2,500 nm	SEP=0.997	Čurda and Kukačková 2002
	NIR	Norm[i] and smooth[e]	*R*[d]	1,000–2,500 nm	RMSEP=3.61	Karoui et al. 2007
	NIR	Smooth[e]	*R*[d]	1,900–2,320 nm	SPE=0.855	Lee et al. 1997
	MIR	RD[h]	*R*[d]	5,000–400 cm^{-1}	SEP=0.12–0.35	McQueen et al. 1995
	NIR	RD[h]	*R*[d]	1,740–2,280 nm	SEP=0.12–0.35	McQueen et al. 1995
	NIR	RD[h], 1st and 2nd D[c]	*R*[d]	400–2,500 nm	SECV=0.05–0.92	Pérez–Marín et al. 2001
	NIR	1st, 2nd, and 3rd D[c]	*R*[d]	400–2,498 nm	SEC=0.388	Rodriguez–Otero et al. 1994
	NIR	SC[b]	*R*[d]	400–2,500 nm	RMSEP=0.52	Wittrup and Nørgaard 1998
Protein Content	FT–NIR	Outliers identified	*R*[d]	900–2,500 nm	SEP=0.303	Čurda and Kukačková 2002
	NIR	Norm[i], 1st D[c], smooth[e]	*R*[d]	1,000–2,500 nm	RMSEP=2.34	Karoui et al. 2006a
	NIR	Smooth[e]	*R*[d]	1,900–2,320 nm	SEP=0.608	Lee et al. 1997
	MIR	RD[h]	*R*[d]	5,000–400 cm^{-1}	SEP=0.04–0.09	McQueen et al. 1995
	NIR	RD[h]	*R*[d]	1,740–2,280 nm	SEP=0.04–0.09	McQueen et al. 1995
	NIR	RD[h], 1st and 2nd D[c]	*R*[d]	400–2,500 nm	SECV=0.05–0.92	Pérez–Marín et al. 2001
	NIR	1st, 2nd, and 3rd D[c]	*R*[d]	400–2,498 nm	SEC=0.397	Rodriguez–Otero et al. 1994

[a] Measurement mode
[b] derivative
[c] Scatter correction
[d] Reflectance
[e] Smoothing
[f] Standard normal variant
[g] Transmittance
[h] Raw data
[i] Normalisation

spectroscopic mode most commonly used in the literature, in the area of cheese compositional analysis, is reflectance mode (Blazquez et al. 2004; Lee et al. 1997; Pérez-Marín et al. 2001; Rodriguez-Otero et al. 1994; da Costa Filho and Volery 2005; Karoui et al. 2006a; Skeie et al. 2006; Downey et al. 2005). These authors report predictions with varying degrees of accuracy, but overall, the results are extremely accurate.

NIR vs MIR Spectroscopy for Determining Composition

Just as it can be seen from Table 3 that reflectance spectroscopy has been used most often in the area of cheese compositional analysis, it can also be quickly noticed that NIR spectroscopy has been used as a tool in this analysis much more frequently than MIR spectroscopy (Blazquez et al. 2004; Čurda and Kukačková 2002; Lee et al. 1997; Pérez-Marín et al. 2001; Rodriguez-Otero et al. 1994; Wittrup and Nørgaard 1998; da Costa Filho and Volery 2005; Karoui et al. 2006a; Skeie et al. 2006; Downey et al. 2005). McQueen et al. (1995) carried out a comparison of NIR and MIR spectroscopic techniques in obtaining protein, fat, and moisture contents of 24 cheese samples. Reference values were obtained using standard wet chemistry techniques. It was concluded that NIR spectroscopy was a more attractive technique. Prediction correlation coefficients between 0.93 and 0.96 and standard errors of prediction of between 2 and 5% compared favorably with the corresponding values of 0.81–0.92 and 4–9 % for the MIR results. It was also noted that the superior results of NIR spectroscopy are more attractive because the instrument is easier to use than the MIR instrument and provides faster results with simpler statistical analysis and is more compact and robust. These findings demonstrate why the NIR

Table 4 Application of NIR and MIR spectroscopy to predict sensory and rheological properties of cheese

Parameter	Spectral region	Data pretreatment	Mode[a]	Wavelength range	Error of prediction	Reference
Texture	MIR	1st and 2nd D[b]	*R*[c]	4,000–640 cm^{-1}	RMSECV=0.08–68.5	Fagan et al. 2007a
	MIR	1st and 2nd D[b], SC[d]	*R*[c]	4,000–2,839; 1,767–930 cm^{-1}	RMSECV=4.6–7.4	Fagan et al. 2007b
	NIR	2nd D[b]	*R*[c]	12,000–4,000 cm^{-1}		Cattaneo et al. 2005
	MIR	2nd D[b]	*R*[c]	4,000–700 cm^{-1}		Cattaneo et al. 2005
	NIR	1st and 2nd D[b], SC[d]	*R*[c]	1,100–2,498 nm	RMSECV=0.1–16.5	Blazquez et al. 2006
	NIR	2nd D[b]	*R*[c]	750–2,498 nm	RMSECV=2.3–5.0	Downey et al. 2005
	NIR	2nd D[b]	*R*[c]	1,100–2,500 nm	RMSEP=0.248	Cattaneo et al. 2002
	NIR		*R*[c]	750–1,098 nm		Downey 1998
	NIR	1st and 2nd D[b], SNVD[e]	*R*[c]	1,110–2,490 nm	SE=0.2–1.0	Sørensen and Jepsen 1998
	NIR	1st and 2nd D[b], SNVD[e]	*T*[f]	850–1,050 nm	SE=0.2–1.0	Sørensen and Jepsen 1998
Flavour	MIR	na[g]	*R*[c]	4,000 to 700 cm^{-1}	All samples classified	Subramanian et al. 2007
Structure	NIR	Norm[h]	*R*[c]	1,000–2,500 nm	RMSECV=0.11–0.28	Karoui et al. 2007
	NIR	2nd D[b]	*R*[c]	1,100–2,500 nm	RMSEP=0.248	Cattaneo et al. 2002

[a] Measurement mode
[b] Derivative
[c] Reflectance
[d] Scatter correction
[e] Standard normal variant and detrend
[f] Transmittance
[g] Data not available
[h] Normalisation

technology has been selected over MIR by the majority of researchers in this area.

Wavelength Selection for Determining Composition

It is worth noting that the wavelength used in the compositional analysis of cheese varies widely. In the literature where NIR spectroscopy was used, many authors used the full NIR spectra of 400–2,500 nm (Blazquez et al. 2004; Pérez-Marín et al. 2001; Rodriguez-Otero et al. 1994; Wittrup and Nørgaard 1998). Some authors such as Adams et al. (1999) used a low range in the NIR spectrum for their statistical analysis (700–1200 nm), whereas others such as Lee et al. (1997) used a higher range (1,900–2,320 nm). Although results depended on a number of parameters and they varied from study to study, the general trend in the papers studied in this review showed that the best results were found when the full NIR spectrum (400–2,500 nm) was used for the compositional analysis of cheese.

Measurement of Cheese Sensory and Rheological Attributes

Reported applications of NIR and MIR spectroscopy to determine sensory and rheological parameters for a range of cheese types are outlined in Table 4. A brief outline of the reported studies is described below.

Table 5 Application of NIR and MIR spectroscopy in cheese authenticity determination

Parameter	Spectral region	Data pretreatment	Wavelength range (cm^{-1})	% Correctly classified	Reference
Manufacturing process	MIR	2nd Deriv.	1,050–1,800	93%	Picque et al. 2004
	MIR	Normalisation	3,000–900 cm^{-1}	64.8 and 33.3% for cal and val[b]	Karoui et al. 2006c
	NIR	Normalisation	315–1,700 nm	85.2 and 63.2% for cal and val[b]	Karoui et al. 2006c
Geographical origin	MIR[a]	Normalisation	4,000–900	89 and 76.7% for cal and val[b]	Karoui et al. 2004
	NIR	Normalisation	4,000–10,000	89 and 86.8% for cal and val[b]	Karoui et al. 2005a
	MIR	Normalisation	3,000–2,800	84.1 and 85.7% for cal and val[b]	Karoui et al. 2005a
	MIR[a]	Normalisation	1,700–1,500		Karoui et al. 2005b
	MIR	2nd Deriv.	1,050–1,800	71 and 68% for cal and val[b]	Picque et al. 2004
	NIR	Normalisation or 2nd deriv.	4,000–10,000	85–100%	Pillonel et al. 2003
	MIR	Normalisation or 2nd deriv.	3,500–700	90–93%	Pillonel et al. 2003

[a] Combined with fluorescence spectroscopy
[b] Calibration and validation spectral sets

Transmittance vs Reflectance Spectroscopy for Determining Sensory and Rheological Attributes

The literature shows that reflectance spectroscopy is the most common mode used in the area of cheese sensory and rheological testing (Downey 1998; Cattaneo et al. 2002, 2005; Čurda and Kukačková 2002; Downey et al. 2005; Blazquez et al. 2006; Karoui et al. 2007). Sørensen and Jepsen (1998), who used both reflectance and transmittance spectroscopy, found that the accuracy of the results obtained using reflectance spectroscopy was better than those obtained using transmittance spectroscopy. In this paper, transmittance mode was used in the 850- to 1,050-nm region, and reflectance mode was used in the 1,110- to 2,490-nm region. The study demonstrated that NIR spectroscopy can predict consistency properties of semi-hard cheese such as springiness and hardness with a prediction error that may be less than the imprecision of average results obtained by two or more assessors.

NIR vs MIR Spectroscopy for Determining Sensory and Rheological Attributes

The majority of the papers listed in Table 4 use NIR spectroscopy to investigate the sensory and rheological attributes of cheese samples. Fagan et al. (2007a, b) applied MIR spectroscopy to the prediction of sensory and instrumental texture of processed cheese, whereas Cattaneo et al. (2005) used both NIR and MIR spectroscopic techniques to evaluate the shelf-life period in which Crescenza cheese “freshness” is maintained, allowing a comparison to be drawn between the techniques. The main advantage of using MIR spectroscopic techniques was to rapidly draw a profile of the product relating to its total composition and quality. More recently, Subramanian et al. (2007) developed a sample preparation method and FTIR technique to rapidly predict the flavor quality of Cheddar cheese. They suggested that cheese samples could be classified based on their flavor quality using FTIR spectroscopy and that the discrimination was due to organic and fatty acids and their esters.

Wavelength Selection for Determining Sensory and Rheological Attributes

The majority of the papers in this area use NIR spectroscopy in their data acquisition, with just three studies reporting on the application of MIR spectroscopy. While two of these studies utilized almost the entire MIR spectral range (Cattaneo et al. 2005; Fagan et al. 2007a), Fagan et al. (2007b) found that improved prediction of sensory texture was possible if only certain regions of the MIR spectra were employed (4,000–2,839 and 1,767–930 cm^{-1}). From Table 4, it is noticeable that none of the papers mentioned use the full NIR spectroscopic range. The majority of the NIR spectrum was used by Downey et al. (2005) where 750–2,498 nm was utilized for data analysis. The root mean square errors of cross validation for the rheological properties being predicted in this paper were 2.3–5.0. Although the set of experimentally produced cheese samples is limited, these results demonstrated the ability of NIR spectroscopy to predict cheese sensory and rheological properties with sufficient accuracy to be industrially useful. Other papers (Sørensen and Jepsen 1998; Cattaneo et al. 2002; Blazquez et al. 2006) use roughly the second half of the NIR spectrum (1,100–2,500 nm). Some of these papers had slightly better error values than Downey et al. (2005), suggesting either that this region shows more information in the area of cheese rheology, or simply that it is prudent to try different wavebands before deciding on a wavelength range to use for model development. This is often achieved by examining the loading scores of the entire wavelength range and using sections for the analysis which show particular influence.

Cheese Authenticity Determination

Cheese authenticity is an emerging research area, which is becoming increasingly important to the dairy sector (see Table 5). The introduction of recent legislation (European Commission 2002) and the requirement for manufacturers and producers to be able to demonstrate food chain traceability, together with a rise in consumer awareness in food products, has focused renewed interest in food authenticity determination. Rapid and cost-effective technologies such as NIR and MIR spectroscopy are presently being evaluated for food authenticity determination.

NIR vs MIR Spectroscopy for Determining Cheese Authenticity

Although for most of the applications mentioned in this review thus far the use of NIR spectroscopy is more prevalent than MIR spectroscopy, in the area of cheese authenticity, which encompasses geographical origin and adulteration detection, there is a more even balance between the technologies utilized in the literature. MIR spectroscopy was widely employed (Pillonel et al. 2003; Pillonel and Bosset 2004; Karoui et al. 2004, 2005a, b; Picque et al. 2004). Karoui et al. (2006b) outlined the feasibility of discriminating the manufacturing process and sampling zone in ripened soft cheeses using ATR MIR (3,000–900 cm^{-1}) and reflectance NIR (315–1,700 nm) spectroscopy. This paper gives a good comparison of the two spectroscopic techniques used on the same cheese samples. Regarding the MIR spectra, the percentage of samples correctly classified into six groups (three for external and three for central zones) by factorial discriminant analysis was 64.8 and 33.3 %, respectively, for the calibration and validation sets, respectively. Better classification was obtained from the NIR spectra where the corresponding results were 85.2 and 63.2%. This paper would suggest that although many papers have been published in the area of cheese authenticity using MIR spectroscopic techniques, NIR spectroscopy is still a more accurate tool in this area. Karoui et al. (2005a) overcame some of these difficulties of the MIR technique by concentrating on a particular wavelength range. In assessing the potential of infrared spectroscopies for the determination of the geographical origin of Emmental cheeses, NIR spectroscopy was found to give 89 and 86.8% correct classification for the calibration and validation spectral data sets, respectively. The MIR results were comparable to the NIR results, giving corresponding correct classification figures of 84.1 and 85.7% within the 3,000- to 2,800-cm^{-1} region. NIR results were still superior to the MIR results, but by concentrating on this section of the wavelength range, a closer comparison was achieved. A good example of where wavelength selection leads to favorable results is outlined by Picque et al. (2004) who obtained a correct classification of 93% by using the reduced wavelength range of 1,050–1,800 cm^{-1} in the MIR region.

Future Developments

In the future, hyperspectral imaging (HSI), which is an emerging platform technology that integrates conventional spectroscopy as well as imaging, should find application in process control during cheese manufacture. HSI is a powerful technology employed to attain both spatial and spectral information from an object (Gowen et al. 2007). It can combine images acquired at a number of narrow wavebands which are sensitive to product features of interest and may allow for enhanced identification of quality problems during manufacture.

Conclusions

Quality control measurement is the main reported application of both NIR and MIR spectroscopy with respect to cheese processing. Reflectance spectroscopy has been demonstrated to be more suited to industrial applications than transmittance spectroscopy. The potential of these technologies for compositional, ripening stage, rheological, and process monitoring applications at laboratory scale is also well documented. The application of NIR and MIR spectroscopy for cheese authenticity is an emerging research area and represents an important future application area for this technology. These techniques also have the potential to assist food processors to adhere to increasingly stringent European Union food authenticity legislation.

The majority of the studies detailed in this review were carried out at laboratory scale with limited sample sizes. Larger plant scale studies using a wider range of commercially produced cheeses are required before the full potential of these technologies may be exploited by cheese manufacturers. Equipment manufacturers are likely to put renewed emphasis on developing online instrumentation to facilitate improved process monitoring of cheese quality during manufacture.

References

Adams, M. J., Latham, K., Barnett, N. W, & Poynton, A. J. (1999). Calibration models for determining moisture and fat content of processed cheese using near-infrared spectroscopy. *Journal of the Science of Food and Agriculture, 79*, 1232–1236.

Andrade, J. M., Gómez-Carracedo, M. P., Fernández, E., Elbergali, A., Kubista, M., & Prada, D. (2003). Classification of commercial apple beverages using a minimum set of mid-IR wavenumbers selected by Procrustes rotation. *Analyst, 128*, 1193–1199.

Balboni, M. L. (2003). Process analytical technology concepts and principles. *Pharmaceutical Technology, 27*, 54–66 (October).

Benson, I. B. (2003). Near infra-red absorption technology for analysing food composition. In M. Lees (Ed.), *Food Authenticity and Traceability* (pp 101–130). Cambridge, England: Woodhead Publishing Ltd.

Blazquez, C., Downey, G., O'Callaghan, D., Howard, V., Delahunty, C., Sheehan, E., et al. (2006). Modelling of sensory and instrumental texture parameters in processed cheese by near infrared reflectance spectroscopy. *Journal of Dairy Research, 73*, 58–69.

Blazquez, C., Downey, G., & O'Donnell, C. (2004). Prediction of moisture, fat and inorganic salts in processed cheese by near infrared reflectance spectroscopy and multivariate data analysis. *Journal of Near Infrared Spectroscopy, 12*, 149–158.

Büning-Pfaue, H. (2003). Analysis of water in food by near infrared spectroscopy. *Food Chemistry, 82*, 107–115.

Cattaneo, T. M. P., Giardina, C., Sinelli, N., Riva, M., & Giangiacomo, R. (2005). Application of FT-NIR and FT-IR spectroscopy to study the shelf-life of Crescenza cheese. *International Dairy Journal, 15*, 693–700.

Cattaneo, T. M. P., Maraboli, A., & Giangiacomo, R. (2002). Near infrared spectroscopy applied to pasta filata cheese in relation to textural analysis. In A. M. C. Davies & R. K. Cho (Eds.), *Proceedings of 10th International Conference* (pp 235–238). UK, Chichester: NIR Publications.

Chen, M., Irudayaraj, J., & McMahon, D. J. (1998). Examination of full fat and reduced fat Cheddar cheese during ripening by Fourier transform infrared spectroscopy. *Journal of Dairy Science, 81*, 2791–2797.

Čurda, L., & Kukačková, O. (2002). NIR spectroscopy: a useful tool for rapid monitoring of processed cheese manufacture. *Journal of Food Engineering, 61*, 557–560.

da Costa Filho, P. A., & Volery, P. (2005). Broad-based versus specific NIRS calibration: Determination of total solids in fresh cheese. *Analytica Chimica Acta, 544*, 82–88.

Downey, G. (1998). Food and food ingredient authentication by mid-infrared spectroscopy and chemometrics. *Trends in Analytical Chemistry, 17*, 418–424.

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. *International Dairy Journal, 15*(6–9), 701–709.

Dufour, E., Mazerolles, G., Devaux, M. F., Duboz, G., Duployer, M. H., & Riou, N. M. (2000). Phase transition of triglycerides during semi-hard cheese ripening. *International Dairy Journal, 10*, 81–93.

European Commission (2002) Article 8, Regulation (EC) No. 178/2002D. Laying down the general principles and requirements of food law, establishing the European Food Safety Authority and laying down procedures in matters of food safety. Retrieved 27 July 2007 from http://europa.eu.int/comm/food/food/foodlaw/traceability/index_en.htm.

Fagan, C. C., Everard, C., O'Donnell, C. P., Downey, G., Sheehan, E. M., Delahunty, C. M., et al. (2007a). Evaluating mid-infrared spectroscopy as a new technique for predicting sensory texture attributes of processed cheese. *Journal of Dairy Science, 90*, 1122–1132.

Fagan, C. C., Everard, C., O'Donnell, C. P., Downey, G., Sheehan, E. M., Delahunty, C. M., et al. (2007b). Prediction of processed cheese instrumental texture and meltability by mid-infrared spectroscopy coupled with chemometric tools. *Journal of Food Engineering, 80*, 1068–1077.

Fagan, C. C., Leedy, M., Castillo, M., Payne, F. A., O'Donnell, C. P., & O'Callaghan, D. J. (2007c). Development of a light scatter sensor technology for on-line monitoring of milk coagulation and whey separation. *Journal of Food Engineering, 83*, 61–67.

Fagan, C. C., O'Donnell, C. P., O'Callaghan, D. J., Downey, G., Sheehan, E. M., Delahunty, C. M., et al. (2007d). Application of mid-infrared spectroscopy to the prediction of maturity and sensory texture attributes of Cheddar cheese. *Journal of Food Science, 72*, E130–E137.

Fagan, C. C., Castillo, M., O'Donnell, C. P., O'Callaghan, D. J., & Payne, F. A. (2007e). On-line prediction of cheese making indices using backscatter of near infrared light. *International Dairy Journal* (in press) DOI 10.1016/j.idairyj.2007.09.007.

Fagan, C. C., Castillo, M., Payne, F. A., O'Donnell, C. P., Leedy, M., & O'Callaghan, D. J. (2007f). Novel on-line sensor technology for continuous monitoring of milk coagulation and whey separation in cheese making. *Journal of Agricultural and Food Chemistry, 55*(22), 8836–8844.

Farkye, N. Y. (2004). Cheese technology. *International Journal of Dairy Technology, 57*(2–3), 91–98.

Geladi, P. (2003). Chemometrics in spectroscopy. Part 1. Classical chemometrics. *Spectrochimica Acta Part B, 58*, 767–782.

Goulden, J. D. S. (1957). Diffuse reflexion spectra of dairy products in the near infra-red region. *Journal of Dairy Research, 24*, 242–251.

Gowen, A. A., O'Donnell, C. P., Cullen, P. J., Downey, G., & Frias, J. M. (2007). Hyperspectral imaging—An emerging process analytical tool for food quality and safety control. *Trends in Food Science & Technology, 18*(12), 590–598.

Guillemin, H., Trelea, I. C., Picque, D., Perret, B., Cattenoz, T., & Corrieu, G. (2006). An optical method to monitor casein particle size distribution in whey. *Lait, 86*, 359–372.

Horik, T. (1985). Objective measurements of the process of curd formation during rennet treatment of milks by the hot wire method. *Journal of Food Science, 50*, 911–917.

Iwamoto, M., & Kawano, S. (1992). Advantages and disadvantages of NIR applications for the food industry. In I. Murray & I. A. Cowe (Eds.), *Making light work: Advances in near infrared spectroscopy*, (pp 367–375). Germany, Weinhiem: Wiley-VCH.

Karoui, R., Bosset, J.-O., Mazerolles, G., Kulmyrzaev, A., & Dufour, É. (2005b). Monitoring the geographic origin of both experimental French Jura hard cheeses and Swiss Gruyère and L'Etivaz PDO cheeses using mid-infrared and fluorescence spectroscopies: A preliminary investigation. *International Dairy Journal, 15*, 275–286.

Karoui, R., & Dufour, E. (2003). Dynamic testing rheology and fluorescence spectroscopy investigations of surface to centre differences in ripened soft cheeses. *International Dairy Journal, 13*, 973–985.

Karoui, R., Dufour, É., Pillonel, L., Emmanuelle, S., Picque, D., Cattenoz, T., et al. (2005a). The potential of combined infrared and fluorescence spectroscopies as a method of determination of the geographic origin of Emmental cheeses. *International Dairy Journal, 15*, 287–298.

Karoui, R., Dufour, É., Pillonel, L., Picque, D., Cattenoz, T., & Bosset, J. O. (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. *European Food Research and Technology, 219*, 184–189.

Karoui, R., Mouazen, A. M., Dufour, I., Pillonel, L., Picque, D., De Baerdemaeker, J., et al. (2006c). Application of the MIR for the determination of some chemical parameters in European Emmental cheeses produced during summer. *European Food Research and Technology, 222*, 165–170.

Karoui, R., Mouazen, A. M., Dufour, É., Pillonel, L., Schaller, E., De Baerdemaeker, J., et al. (2006a). Chemical characterization of European Emmental cheeses by near infrared spectroscopy using chemometric tools. *International Dairy Journal, 16*, 1211–1217.

Karoui, R., Mouazen, A. M., Ramon, H., Schoonheydt, R., & De Baerdemaeker, J. (2006b). Feasibility study of discriminating the manufacturing process and sampling zone in ripened soft cheeses using attenuated total reflectance MIR and fiber optic diffuse reflectance VIS–NIR spectroscopy. *Food Research International, 39*, 588–597.

Karoui, R., Pillonel, L., Schaller, E., Bosset, J.-O., & De Baerdemaeker, J. (2007). Prediction of sensory attributes of European Emmental cheese using near-infrared spectroscopy: A feasibility study. *Food Chemistry, 101*, 1121–1129.

Kulmyrzaev, A., Dufour, É., Noël, Y., Hanafi, M., Karoui, R., Qannari, E. M., et al. (2005). Investigation at the molecular level of soft cheese quality and ripening by infrared and fluorescent spectroscopies and chemometrics—Relationships with rheology properties. *International Dairy Journal, 15*, 669–678.

Laporte, M. F., Martel, R., & Paquin, P. (1998). The near-infrared optic probe for monitoring rennet coagulation in cow's milk. *International Dairy Journal, 8*, 659–666.

Lee, S. J., Jeon, I. J., & Harbers, L. H. (1997). Near infrared reflectance spectroscopy for rapid analysis of curds during Cheddar cheese making. *Journal of Food Science, 62*(1), 53–56.

Lucia, V., Daniela, B., & Rosalba, L. (2001). Use of Fourier transform infrared spectroscopy to evaluate the proteolytic activity of *Yarrowia lipolytica* and its contribution to cheese ripening. *International Journal of Food Microbiology, 69*, 113–123.

Martín-del-Campo, S. T., Picque, D., Cosío-Ramírez, R., & Corrieu, G. (2007). Middle infrared spectroscopy characterization of ripening stages of Camembert-type cheeses. *International Dairy Journal, 17*, 835–845.

McKenna, D. (2001). Measuring moisture in cheese by near infrared absorption spectroscopy. *Journal of AOAC International, 84*(2), 623–628.

McMahon, D. J., Brown, R. J., & Ernstrom, C. A. (1984). Enzymic coagulation of milk casein micelles. *Journal of Dairy Science, 67*, 745–748.

McQueen, D. H., Wilson, R., Kinnunen, A., & Jensen, E. P. (1995). Comparison of two infrared spectroscopic methods for cheese analysis. *Talanta, 42*, 2007–2015.

Mertens, B. J. A., O'Donnell, C. P., & O'Callaghan, C. P. (2002). Modelling near-infrared signals for on-line monitoring in cheese manufacture. *Journal of Chemometrics, 16*, 89–98.

Millar, S. (2004). Near infrared diffuse reflectance in texture measurements. In D. Kilcast (Ed.), *Texture in food, vol. 2: Solid foods* (pp 167–183). Cambridge, England: Woodhead Publishing Ltd.

Næs, T., Isaksson, T., Fearn, T., & Davies, T. (2002). *A user-friendly guide to multivariate calibration and classification*. Chichester, UK: NIR Publications.

O'Callaghan D. J, Mulholland E. P., Duffy A. P., O'Donnell C. P., & Payne F. A. (2001) Evaluation of hot wire and optical sensors for on-line monitoring of curd firmness during coagulation. *Irish Journal of Agricultural and Food Research, 40*, 227–238.

O'Callaghan, D. J., O'Donnell, C. P., & Payne, F. A. (1999). A comparison of on-line techniques for determination of curd setting time using cheesemilks under different rates of coagulation. *Journal of Food Engineering, 41*, 43–54.

O'Callaghan, D. J., O'Donnell, C. P., & Payne, F. A. (2000). On-line sensing techniques for coagulum setting in renneted milks. *Journal of Food Engineering, 43*, 155–165.

O'Callaghan, D. J., O'Donnell, C. P., & Payne, F. A. (2002). Review of systems for monitoring curd setting during cheesemaking. *International Journal of Dairy Technology, 55*, 65–74.

Payne, F. A., Hicks, C. L., Madangopal, S., & Shearer, S. A. (1993a). Fibre optic sensor for predicting the cutting time of coagulating milk for cheese production. *Transactions of the ASAE, 36*, 841–847.

Payne, F. A., Hicks, C. L., & Sheng, P. (1993b). Predicting optimal cutting time of coagulating milk using diffuse reflectance. *Journal of Dairy Science, 76*, 48–61.

Penner, M. H. (2003). Basic principles of spectroscopy. In S. S. Nielsen (Ed.), *Food analysis* (3rd ed., pp 359–369). NY, USA: Kluwer.

Pérez-Marín, M. D., Garrido-Varo, A., Serradilla, J. M., Núnez, N., Ares, J. L., & Sánchez, J. (2001). Chemical and microbial analysis of goat's milk, cheese and whey by near infrared spectroscopy. In A. M. C., Davies & R. K., Cho (Eds.), *Near infrared spectroscopy: Proceedings of the 10th International Conference* (pp 225–228). West Sussex, UK: NIR Publications.

Picque, D., Cattenoz, T., Pillonel, L., Bosset, J. O., & Corrieu, G. (2004). Discrimination of European Emmental cheese by mid-infrared spectroscopy. Proceedings of IDF Symposium on Cheese: Ripening, Characterisation and Technology, Prague, Czech Republic, 21–25 March 2004.

Pillonel, L., & Bosset, J. O. (2004). Cheese authenticity and traceability: An analytical challenge. Proceedings of IDF Symposium on Cheese: Ripening, Characterisation and Technology, Prague, Czech Republic, 21–25 March 2004.

Pillonel, L., Luginbuhl, W., Picque, D., Schaller, E., Tabacche, R., & Bosset, J. O. (2003). Analytical methods for the determination of the geographic origin of Emmental cheese: Mid- and near-infrared spectroscopy. *European Food Research and Technology, 216*(2), 174–178.

Rodriguez-Otero, J. L., Hermida, M., & Cepeda, A. (1994). Determination of fat, protein, and total solids in cheese by near-infrared reflectance spectroscopy. *Journal of AOAC International, 78*(3), 802–806.

Sayago, A., Navas, M. J., & Asuero, A. G. (2004). Spectrophotometric determination of organic compounds: Applications in food analysis. *Alimentaria, 353*, 55–63.

Skeie, S., Feten, G., Almøy, T., Østlie, H., & Isaksson, T. (2006). The use of infrared spectroscopy to predict selected free amino acids during cheese ripening. *International Dairy Journal, 16*, 236–242.

Sørensen, L., & Jepsen, R. (1998). Assessment of sensory properties of cheese by near-infrared spectroscopy. *International Dairy Journal, 8*, 863–871.

Subramanian, A., Harper, J. W., & Rodriguez-Saona, L. E. (2007). Classification of cheddar cheese based on flavor quality using Fourier transform infrared spectroscopy. *Journal of Dairy Science, 90*(Suppl 1), 114.

Wehling, R. (2003) Infrared spectroscopy. In S. S., Nielsen (Ed.), *Food analysis* (3rd ed., pp 387–399). NY, USA: Kluwer.

Wilson, R. H., Colquhoun, I. J., & Kemsley, E. K. (2001). Screening food products for authenticity using infrared spectroscopy. Application note 54-55. The Institute of Food Research, Norwich Research Park, Colney, Norwich, UK.

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