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Use of FTIR for Rapid Authentication and Detection of Adulteration of Food

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Abstract

Fourier transform infrared (FTIR) spectroscopy is an appealing technology for the food industry because simple, rapid, and nondestructive measurements of chemical and physical components can be obtained. Advances in FTIR instrumentation combined with the development of powerful multivariate data analysis methods make this technology ideal for large volume, rapid screening and characterization of minor food components down to parts per billion (ppb) levels. Because of the use of FTIR techniques in quality and process control applications, the food industry is already familiar with the technology and its potential to expand to monitoring for food adulteration. The aim of this review is to compile the current research on applications of near infrared (NIR) and mid-infrared (MIR) spectroscopy for rapid authentication and detection of adulteration in food.

INTRODUCTION

Fourier transform infrared (FTIR) spectroscopy has become an attractive alternative for traditional analytical methods because little sample preparation is needed, analysis is rapid, and the use of hazardous solvents is minimized. These advantages result in time and cost savings and an increase in the number of samples that can be analyzed. Numerous researchers have attempted to utilize these advantages by applying FTIR to food science. The scope of this review is to compile the current research on the applications of FTIR spectroscopy for authentication and detection of adulteration in food.

Knowledge of the basic principles behind spectroscopy facilitates the understanding of how the infrared (IR) technique can be applied to authenticate and detect adulteration in food. IR spectra are produced by recording changes in absorption of IR radiation by molecules, which undergo mechanical motions (vibrational and rotational modes) due to the absorption of energy (Diem 1993, Guillen & Cabo 1997). Within any molecule, a given functional group (i.e. carbonyl group or an amide group) gives characteristic IR absorption at specific, narrow frequency ranges regardless of their relationship with the rest of the molecule. Because of complex interactions of atoms within the molecule, each involved in its own vibrational transitions, the energy of a vibration and, thus, the position of the band in the IR spectrum are sometimes influenced by the atoms surrounding the vibrational group. Thus, the IR spectra can be used to identify or differentiate between samples and also give information about the quantity of functional groups (Diem 1993, Guillen & Cabo 1997). The IR region of the electromagnetic spectrum spans from 14,000–50 cm^{-1} and is divided into three areas: near IR (14,000–4,000 cm^{-1}), mid IR (4,000–400 cm^{-1}), and far IR (400–50 cm^{-1}). This review focuses on the first two regions. Near IR (NIR) and mid IR (MIR) techniques take advantage of the phenomenon occurring when electromagnetic radiation of a specific energy interacts with a molecule.

NIR spectra (**Figure 1*a***) are the result of relatively weak and broad overtones and combination bands of fundamental vibrational transitions associated mainly with C-H, N-H, and O-H functional groups. NIR bands are the result of complex vibrational motion of chemical bonds that tend to deviate from harmonicity. These deviations (anharmonicity) result in bands arising from transitions over two (1st overtone), three (2nd overtone), or higher energy levels (12,500–4,000 cm^{-1}, 800–2,500 nm) of the frequency of fundamental vibrations, leading to a decreasing NIR absorption intensity with increasing rank of overtone. In addition, the NIR spectrum includes combination modes from the interaction of two or more vibrations taking place simultaneously from the absorption of a single photon (Osborne 2000, Barton 2002). Unfortunately, the superposition of many different overtone and combination bands in the NIR region causes a very low structural selectivity of NIR spectra compared to MIR spectra; however, lower NIR absorption intensity can be compensated by increasing the sample thickness for NIR measurements (from millimeters up to centimeters) compared to MIR (micrometers). NIR bands are 10 to 100 times less intense than their corresponding MIR fundamental bands. This can allow for the direct analysis of samples that are highly absorbing or strongly light scattering without dilution or extensive sample preparation (Hall et al. 1996, Shenk et al. 2001). FT-NIR instruments have found a niche in the food industry for chemical quality and process control because of their ruggedness and increased flexibility in handling, such as being able to analyze samples in glass vials. Furthermore, the relatively weak absorption due to water overtones enables high-moisture foods to be readily analyzed (Osborne 2000).

Spectra from the MIR region are commonly used for structural identification (fingerprinting) of organic compounds because the absorption bands are caused by fundamental vibrations of a specific functional group (Guillen & Cabo 1997). The fingerprint region, located within the

MIR region between 1,200 and 700 cm^{-1}, contains bands from lipids, proteins, carotenoids, and polysaccharides, and as a result is rich in structural information (Pare & Belanger 1997). Spectra from the MIR region (**Figure 1*b***) can be used for quantitative analysis applications because the intensities of the bands are proportional to the concentration of their respective functional group (Pare & Belanger 1997).

With the advent of FT instrumentation, the speed and accuracy of IR technology was increased by replacing the use of conventional prism and grating monochromators with an interferometer. FTIR utilizes interferometric modulation of radiation to measure multiple frequencies simultaneously, producing an interferogram that is recalculated using complex algorithms to give the original spectrum. Fourier deconvolution resolves overlapping IR bands, caused by complex samples, by reducing the bandwidth and increasing the peak height (Markovich 1991). Additional advantages of FTIR over traditional dispersive IR instruments include low mechanical wear on equipment because FTIR does not use moving grating parts; simultaneous acquisition of all the wave numbers of light, increasing the signal to noise (S/N) ratio; increased beam intensity going through the sample, leading to higher throughput; superior wavelength resolution; internal wavelength calibration; and advanced wavelength accuracy. Given that the wavelength of the laser is stable and very accurately known, the data can be precisely acquired, allowing for repetitive scans to be well aligned with respect to each other. In FT instrumentation, the resolution is not determined by the size of the beam, but by the stroke (travel) of the movable mirror and the number of data collected during a stroke. These optical advantages result in a significantly reduced data acquisition time compared with a spectrum obtained with similar resolution on a dispersive instrument (Diem 1993), making the technology an excellent tool for qualitative and quantitative analyses of food matrices.

The use of attenuated total reflectance (ATR) with FTIR allowed the spectral collection from solids, liquids, semisolids, and thin films. ATR IR spectroscopy provides a fast analytical tool as compared with traditional IR transmission spectroscopy, requiring less sample preparation, improving the sample-to-sample reproducibility, minimizing user-to-user spectral variation, and giving high throughput relative to the available energy in the FTIR sample compartment, which resulted in better quality data for more precise material verification and identification (Pike Technologies 2010). ATR is a reflection technique in which the IR light is reflected internally off the back surface of an internal reflection element with a high index refraction, which is in contact with the sample (PerkinElmer 2004). The IR beam travels inside the crystal and a standing wave of radiation, called the evanescent wave, is created (PerkinElmer 2004). A sample in contact with the crystal can interact with the evanescent wave, absorb IR radiation, and have its IR spectrum detected. The evanescent wave is attenuated by the sample's absorbance, which gives rise to the name ATR (PerkinElmer 2004). The high refractive index crystals typically are made of diamond, zinc selenide, KRS-5 (thallium iodide/thallium bromide), or germanium. The number of reflections at the surface of the crystal will vary depending on length and thickness of the crystal and the angle of incidence (PerkinElmer 2004). This provides the ATR with a multiple-fold increase in the sample's response compared with singe-reflection crystals (Pike Technologies 2010).

Several excellent books cover aspects of fundamental theory, instrumentation, chemometric methods, and applications of vibrational spectroscopy and should be referenced if more detailed information is desired (Chalmers & Griffiths 2001, Osborne 2000, Robinson 1991, Sielsler 2002, Williams & Norris 2001). Chemometrics is the science of extracting chemically relevant information from complex multidimensional data produced in chemical experiments by using multivariate analysis techniques to reduce the dimensionality of the data set.

APPLICATIONS OF FTIR FOR AUTHENTICATION AND DETECTION OF ADULTERANTS

Authentication of products by commodity, variety, and geographical origin is important for regulatory agencies, food processors, retailers, and consumers because expensive ingredients have the potential for adulteration and fraudulent or accidental mislabeling. There is a need for a rapid technique to validate these claims, and the potential application of FTIR has been explored in recent years. Combining FT-NIR and FT-MIR with multivariate statistical methods has been applied for authentication of herbal products, fruit juices, agricultural products, edible oils, dairy, and numerous other food products. These efforts have had varying degrees of success at classifying products as authentic or unauthentic depending on the region of the electromagnetic spectrum employed and chemometric techniques used on the spectra. Fingerprints of authentic commodities may be considered to represent their overall chemical composition and therefore have the potential to detect adulteration. This method of detection possesses various benefits as an authenticity screening tool; it is fast (tests can be carried out in 1–2 min) and simple to use. A large number of potential adulterants may be searched from a single spectrum, no sample preparation is required, and little waste material is generated. From a regulatory perspective, it has the additional benefit of not destroying the sample being tested (Kelly & Downey 2005). **Table 1** summarizes the applications of NIR spectroscopy in monitoring authentication of foods.

NIR reflectance spectroscopy has been used to develop a fast authentication system for herbal supplements. *Echinacea* species *E. purpurea* (L.) Moench, *E. angustifolia*, and *E. pallida* are widely used as immunostimulant herbal preparations, and commercial preparations are frequently adulterated or substituted with roots of *Parthenium integrifolium* L. or different *Echinacea* species that negatively affect the reliability and efficacy of *Echinacea* commercial products (Laasonen et al. 2002a). NIR spectroscopy has been reported for the fast identification of *E. purpurea* roots (Laasonen et al. 2002b) and the determination of echinacoside content (Schulz et al. 2002). The presence of other *Echinacea* species can be detected at a minimum of 10% adulteration by using FT-NIR spectroscopy (Laasonen et al. 2002b). Owing to the low content of echinacosides in the most valuable *E. purpurea* roots as compared with *E. pallida* and *E. angustifolia* (Laasonen et al. 2002b), partial least-squares (PLS) algorithms using NIR spectra produce robust models for the fast and reliable screening of *E. purpurea* in herbal preparations.

Adulteration of dietary supplement oils (DSOs) such as grapeseed oil, flax oil, burageseed oil, and evening primrose oil with cheaper and less beneficial oils has become a food quality/safety issue. Variations between different brands of the same oil due to plant origin, variety, and processing conditions, as well as oil types having very similar compositions, can result in possible misclassifications of authentic oils. A detection limit of 2% for DSOs adulterated (2%–20% v/v) with common foods oils has been reported (Ozen et al. 2003).

A noteworthy application of NIR spectroscopy has been for the detection of adulterants in juices, purees, and syrups. These products are often adulterated with cheaper juice concentrates, cane, corn, or beet sugars, and syrups for economic gain. Twomey et al. (1995) reported the use of NIR and factorial discriminant analysis for the detection of adulteration of orange juice with orange pulpwash, grapefruit juice, and synthetic sugar/acid mixture. Accurate classification rates >90% were determined for adulterated orange juice at 50 g kg^{-1} or higher levels, with no adulterated orange juice being predicted as authentic. Contal et al. (2002) showed that adulteration of strawberry or raspberry juice with apple juice could be detected at levels >10% by using PLS-NIR models. Transmittance NIR spectra can accurately and precisely predict the sugar levels in non-scattering juices (Rodriguez-Saona et al. 2001), whereas NIR transflectance data improve the prediction errors for scattering juice samples (Segtnan & Isaksson 2000). Furthermore, the

Table 1 Application of dispersive NIR for food authenticity

Sample	Method	Multivariate model	Results	Source
Echinacosides in Echinacea roots	Reflectance	PLSR	$R^2 = 0.94$, RMSECV = 0.23 g 100 g^{-1}	Schulz et al. 2002
Adulteration of orange juice	Reflectance	FDA	94% accuracy at levels >50 g kg^{-1} adulterants	Twomey et al. 1995
Cocoa procyanidins	Reflectance	PLSR	$R^2 = 0.98$, SECV = 6.20	Whitacre et al. 2003
Phenolic substances and alkaloids in green tea leaves	Reflectance	PLSR	Gallic acid, SECV: 0.2 g kg^{-1}, R^2: 0.89; epicatechin, SECV: 2.6 g kg^{-1}, R^2: 0.97; caffeine, SECV: 1.7 g kg^{-1}, R^2: 0.97	Schulz et al. 1999
Perseitol in avocado honey	Reflectance	PLSR, PCR	$R^2 = 0.87$, SEP = 0.13	Dvash et al. 2002
Citrus oils	Transflectance	PCA, PLSR	$R^2 = 0.79$–1.00, SEC = 0.03–1.09	Steuer et al. 2001
Apple adulteration in strawberry and raspberry purees	Reflectance	SIMCA, PLSR	Most accurate models produced prediction errors of 3.4% apple (in raspberry) and 5.5% (in strawberry)	Contal et al. 2002
Vegetable proteins in milk powder	Reflectance	MLR	$R^2 = 0.99$, SEP = 0.23	Maraboli et al. 2002
Authentication of green asparagus	Reflectance	PLSR	$R^2 = >0.96$, SEP = 0.07	Perez et al. 2001
Adulteration in alcoholic beverages	Transmittance	PCA, SIMCA	Correct classification of 100%	Pontes et al. 2006
(Online) acrylamide adulteration in chips	Reflectance	PLSR	$R^2 = 0.83$; prediction error 266.6 μg kg^{-1} (using low resolution equipment)	Pedreschi et al. 2010
Acrylamide adulteration in chips	Reflectance	PLSR	$R^2 = 0.95$, prediction error 256.6 μg kg^{-1}	Segtnan et al. 2006
Whey adulteration in cow milk	Reflectance	DPLS, SIMCA	$R^2 = 0.999$, RMSEP = 0.264	Kasemsumran et al. 2007

Abbreviations: PLSR, partial least squares regression; FDA, factorial discriminant analysis; PCA, principal component analysis; SIMCA, soft independent model class analogs; PCR, principal component regression; LDA, linear discriminant analysis; MLR, multiple linear regression.

interference from the strong and broad vibrational bands of water (Fischer et al. 1994) in NIR measurements of aqueous systems can be minimized by rapid solvent elimination and measurement of the dry extract by using diffuse reflectance spectroscopy (Alfaro et al. 1990, Li et al. 1996).

Table 2 summarizes the applications of MIR spectroscopy in monitoring authentication of foods. MIR has been used in juice authentication of high-value ingredients adulterated with inferior sources. Pomegranates have been praised for their antioxidant activity and for potential chemopreventative effects against prostate cancer (Vardin et al. 2008). MIR spectra have been used to differentiate pure pomegranate juice concentrate from juice adulterated with grape juice concentrate (2%–14% v/v) using principle component analysis (PCA) and the 1,780–1,685 cm^{-1} (C = O stretching) IR region (Vardin et al. 2008). Similarly, MIR has been successful in the differentiation of fruit varieties and geographical origins. An important parameter for monitoring authenticity in fruit purees, preps, and jams is the percent fruit content, and minimum requirements for each product type have been established. A partial least-squares regression (PLSR) correlating fruit content and FTIR spectra centered on a band at 1729 cm^{-1} and provided good calibration statistics ($R^2 = 0.94$) when applied to strawberry jam (Fugel et al. 2005). He et al. (2007) looked at cranberries, blueberries, concord grapes, plum nectar blend, and apple juices from

Table 2 Application of MIR for food authenticity

Sample	Multivariate model	Results	Source
Lard adulteration			
in cake	PLSR	$R^2 = 0.9790$; SEC = 1.75	Syahariza et al. 2005
in chocolate	PLSR	$R^2 = 0.99$; SE = 1.30	Che Man et al. 2005
Extra virgin olive oil			
Adulteration with vegatable oils		$R^2 = 0.99$; detection limit 6%	Vlachos et al. 2006
	PCA	Detection limit of 5% for binary mixture; error limit 1.04	Gurdeniz & Ozen 2009
Adulterated with palm oil	PLS and PCR	$R^2 = 0.999$; SECV = 0.285 (first derivation)	Rohman & Che Man 2010
Evaluating origin	PLS	Was able to correctly classify 80% (mean centered and first and second derivation)	Hennessy et al. 2009
Others			
Juice concentrate adulteration	PLSR	$R^2 = 0.9751$; could also predict total solids ($R^2 = 0.9916$) and titratable acidity ($R^2 = 0.9114$)	Vardin et al. 2008
Authentication of fruits	SIMCA	Extraction improved SIMCA; 100% correct classification at commodity level	He et al. 2007
Classifying honey adulterants as simple and complex sugars	LDA, PCA, LDS	100% classification of simple and complex sugars using PLS and LDA; combining honey varieties lowered it to 95.5%	Sivakesava & Irudayaraj 2002a
Artisianal honeys adulterated with sugar solutions	SIMCA, PLSR	Classification over 95% for beet sucrose and dextrose; could not unambiguously detect HFCS or invert beet	Kelly et al. 2006
Butter adulterated with margarine	PLSR	Using calibration models of selected ranges (0–5%, 0–25, 20–60, etc) $R^2 = 0.99$; SECV = 1.2% (second derivation)	Koca et al. 2010
Classifying wines as organic versus nonorganic	PCA, DPLS, LDA	DPLS correctly classified 85%; LDA correctly classified 75%	Cozzolino et al. 2009

Abbreviations: PLSR, partial least squares regression; SIMCA, soft independent model class analogy; PCA, principal component analysis; LDA, linear discriminant analysis; PCR, principal component regression.

various manufacturers. Spectral data collected after solid phase extraction of juices improved the pattern recognition [soft independent analysis of class analogy (SIMCA)] modeling power compared with using pure juice and allowed for differentiation of juices with varying origins. Solid phase extraction minimized the interference of sugar on the spectra and isolated the phenolic components that provided a unique fingerprint for juice authentication. The authors acknowledged the limitations of this method, including the extraction procedure and the need for a broader range of samples to improve the robustness of the model, but noted that this method was an improvement over previous attempts (He et al. 2007). Traditional methods for authenticating fruit juices include chromatography or carbon isotope ratio analysis, neither of which is very practical because they are time-consuming, not practical for quality control settings, use harmful solvents, and monitor only one parameter at a time.

Consumers are becoming more interested in organic products and are willing to pay a premium price for these items. The wine industry has acknowledged this trend, and vineyards around the world are currently producing many organic wines. As of 2009, there was no standardized

wine-industry method that would enable organic wine composition and authenticity to be easily and efficiently determined (Cozzolino et al. 2009). Cozzolino et al. (2009) was the first to look at using the technology to classify commercial wines from organic versus nonorganic production systems. Nearly 200 samples of red and white wines from 13 regions in Australia were analyzed using MIR combined with PCA, discriminant PLS (DPLS) regression, and linear discriminant analysis (LDA). DPLS correctly classified 85% of organic wines, whereas LDA was able to classify 75%. In general, the PCA score plot separated the organic and nonorganic wine classes, but there was a slight overlap (Cozzolino et al. 2009). The authors reported that MIR combined with chemometric techniques allowed good discrimination between samples produced under organic and nonorganic production systems. Exploration of the PLS loadings did not show any particular individual chemical parameter that explained the separation between wines, rather that many chemical compounds, including phenolics and volatile or non-volatile compounds, could contribute to the discrimination between wines.

Maple syrup and honey products are also targets for unscrupulous manufacturers to make profit by adding cheaper cane and beet sugars. NIR and FTIR spectroscopy in combination with discriminant (LDA and canonical variate analysis) and quantitative (PLSR and principle component regression) analysis have been successfully applied for the classification of adulterants in maple syrup (Paradkar et al. 2003). Models developed with NIR measurements were suited for quantitative analysis of the presence of adulterants. Models developed from FTIR spectra using the fingerprint region resulted in models with superior quantitative and discriminative performances (**Table 3**, **Figure 2**) for detecting adulterants as compared with those obtained from dispersive NIR spectra. Similar results have been reported for the analysis of thyme, oregano, and chamomile essential oils by dispersive NIR and ATR-IR spectroscopy (Schulz et al. 2003).

Honey has been defined as a natural substance produced by honeybees and the addition of sugars voids this definition (Kelly et al. 2006). Diluting honey with simple and complex sugars is the most common way honey is adulterated. This is very difficult to detect because the adulterants mimic the natural sugar profile of honey (38.2% fructose and 31.2% glucose) (Sivakesava & Irudayaraj 2002a). Honey adulteration is also difficult to detect because of the large variability in the product due to flower and bee species, maturity, environment, and processing or storage conditions. Purity of honey is currently tested by carbon isotope ratio analysis, which is expensive and time consuming. In order to use MIR to authenticate honey, the spectra need to be corrected against a background of water to correct for water overlapping the signal from solutes. Analysis was focused on the region that corresponds to sugars (800–1,500 cm^{-1}). Pure honey was adulterated with 7%–25% glucose, fructose, sucrose, and invert sugar. LDA achieved 100% classification of simple sugars and PLS data compression achieved 100% classification for complex sugars. Combining varieties of honey required more factors and lowered the success rate to 95.5%. Further work must be conducted to include honey of many origins and with many adulterants (Sivakesava & Irudayaraj 2002a).

Another study looked at artisanal honey adulterated with different sugar syrups [invert beet syrup, high fructose corn syrup (HFCS), partial invert cane syrup, dextrose syrup, and beet sucrose]. Models were able to correctly classify 95% of authentic honey, beet sucrose, and dextrose samples but were not able to confidently detect adulteration with HFCS or invert beet syrup (Kelly et al. 2006). Iglasias (2006) used MIR to evaluate the botanical origin of honey when correlated with pollen analysis. As with many food products, honey produced in certain regions is prized for its outstanding sensory qualities and is sold for a high price. MIR could be used as a screening tool but would need to be combined with additional testing to confidently identify the origin of the product. Furthermore, MIR did not allow for a quantitative determination of hydroxymethyl furan (HMF) as an indicator of heat damage to the product (Iglasias 2006).

Table 3 Model validation of FT-NIR and FT-MIR methods for the determination of food components

Sample	Method	Factors	SECV	R^2	Source
Vitamin C in foods and pharmaceuticals	FTIR-attenuated total reflection	11	0.28%	0.998	Yang & Irudayaraj 2002
	NIR diffuse reflectance DRIFTS	9	1.93%	0.975	
	FTIR purged photoacustic detector	9	1.45%	0.985	
	NIR reflectance fiber optic probe	5	1.23%	0.973	
	FT-NIR reflectance	3	1.64%	0.980	
Cholesterol in dairy products	FT-NIR	2	1.44%	0.99	Paradkar & Irudayaraj 2002a
	FT-MIR; first derivative	1	0.68%	1.00	Paradkar & Irudayaraj 2002b
Tetracycline in milk (range 4–2000 ppb)	FT-NIR	4	450 ppb	0.72	Sivakesava & Irudayaraj 2002b
	FT-MIR	15	382 ppb	0.85	
Tetracycline in milk (range 4–520 ppb)	FT-NIR	4	110 ppb	0.87	
	FT-MIR	11	101 ppb	0.87	
Extra virgin olive oil adulteration	FT-NIR	13	3.48%	0.99	Yang & Irudayaraj 2001
	FT-MIR	11	4.74%	0.98	
Infant formula adulterated with melamine	FT-NIR	6	(RMSECV) 0.62	0.99	Mauer et al. 2009
	FT-MIR	10	(RMSECV) 1	0.95	
Maple syrup	FT-NIR	11	3.872	0.95	Paradkar et al. 2003
	FT-MIR (region 800–1200 cm^{-1})	6	2.091	0.97	

Abbreviations: SECV, standard error cross validation; FT, Fourier transform; IR, infrared; NIR, near-infrared; MIR, mid-infrared.

FTIR spectroscopic analysis has shown potential to determine lard adulteration. Syahariza et al. (2005) evaluated lard adulteration in shortening at levels of 0%–100% combined with PLSR models in the regions 1,117–1,097 cm^{-1} and 990–950 cm^{-1}, producing a model with an $R^2 = 0.9790$ and a standard error of calibration of 1.75. Also, ATR-IR combined with PLSR regression was able to detect cocoa butter spiked with lard (0%–15%) with an $R^2 = 0.99$ and a standard error of 1.305 (Che Man et al. 2005). Adulteration of olive oil dates back at least to the time of the Roman Empire (Ulberth & Buchgraber 2000). Applying spectroscopy for olive oil authentication emerged in the mid-1990s, and a considerable amount of work has been devoted to using the technique for detection of extra virgin olive oil (EVOO) adulteration, specifically for improving limits of detection. The band at 3,009 cm^{-1} has been identified for quantification of adulteration because the height of this band for EVOO is smaller than it is for other types of oils and changes according to the extent of adulteration. A high correlation coefficient (above 0.99) was established with a detection limit of approximately 6% (Vlachos et al. 2006). Wavelet compression prior to PCA produces a detection limit of 5% for binary oil mixtures (Gurdeniz & Ozen 2009). EVOO adulteration with palm oil can be detected using the first derivative of the FTIR spectra in the fingerprint region and PLSR ($R^2 = 0.999$, SECV = 0.285) (Rohman & Che Man 2010). FTIR spectra reflect different substitution patterns of triglycerides, differences in chain length of acyl moieties, and differing degrees of unsaturation (Guillen & Cabo 1997).

EVOO has also been the subject of studies to confirm geographical origin claims. The European Union supplies EVOO manufacturers with labels assigning protected designation of origin.

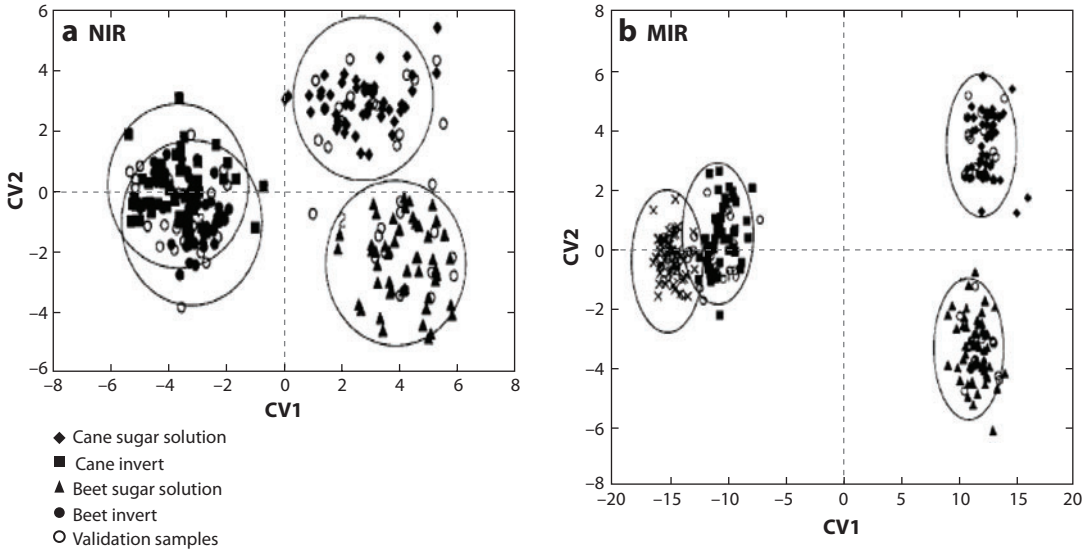

Figure 2

Classification of adulterants in maple syrup by (*a*) near-infrared (NIR) and (*b*) mid-infrared (MIR) using partial least squares (PLS)/canonical variate analysis (CVA) (Paradkar et al. 2003).

Nuclear magnetic resonance (NMR) has been used to classify samples with up to 90% success, but this method is expensive and time consuming. Mean-centered first and second derivative spectra combined with PLSR correctly classified 80% of samples on the basis of origin being ideal for screening because of the high throughput of the method (Hennessy et al. 2009).

Traditional methods for monitoring milk authenticity rely on wet chemistry to determine the amount of a marker compound in a suspect material and a subsequent comparison of values obtained with those from an equivalent material (Karoui & De Baerdemaeker 2007). Woodcock et al. (2008) reviewed the current state of development of both NIR and MIR in cheese authenticity with ATR-IR spectroscopy being widely employed. These techniques have the potential to assist food processors to adhere to increasingly stringent food authenticity legislation. Picque et al. (2002) reported the discrimination of Emmental cheeses from different regions by IR spectroscopy. Data from ATR-IR spectroscopy of a water-soluble fraction enabled the classification of the different types of Emmental cheeses with 78% accuracy, whereas 87% accuracy was obtained by using transmission spectra of dried extracts. The authors highlighted the ability of IR to discriminate according to the source of the milk used for the manufacturing process. In another study, Pillonel et al. (2002) reported the results of a broad screening test into the authenticity of Emmental cheese and its geographic traceability. NIR diffuse reflectance gave 100% discrimination by grouping into the six regions of cheese origin, whereas MIR transmittance achieved 100% correct classification when comparing Switzerland with the other regions pooled as one group.

The standard of identity of butter requires that the product contain no vegetable oil. Therefore, the addition of margarine violates this claim. Current techniques for authenticating butter include gas chromatography (GC), mass spectrometry (MS), NMR, and ultraviolet-visible (UV/Vis) spectroscopy. These methods have detection limits of 2%–5%, but they are expensive and time consuming. IR spectroscopy combined with PLSR-generated models that

estimated adulteration of butter with levels of margarine ranging from 0% to 100% v/v with an $R^2 > 0.99$ and SECV < 1.2%. Development of chemometric models using smaller ranges of adulteration levels (0%–5%, 0%–25%, and 20%–60%) improved the robustness of the models (Koca et al. 2010).

APPLICATIONS OF FTIR FOR DETECTION OF POTENTIALLY HARMFUL ADULTERANTS

Food products are most commonly adulterated with materials that are of a lower quality and, as a result, are typically less expensive. A smaller segment of adulteration includes products contaminated with potentially harmful compounds. This includes compounds that unintentionally occur as a result of a production process (such as *trans* fats and acrylamide) and intentional chemical contaminants (e.g., melamine). Advances in FTIR spectroscopic instrumentation and multivariate data analysis techniques show significant potential for determining changes in food composition that may be indicative of the addition of harmful extraneous material.

The *trans*-fat content of food has recently been identified as a health concern for the public. Although small amounts of *trans* fats are found naturally in dairy and meat products, the major source is partially hydrogenated vegetable oils used in commercial food products (Mossoba et al. 2009). The hydrogenation process is beneficial in that it enables oils that are low in saturated fat to be used in place of saturated fats, but partial hydrogenation produces *trans* fats. *Trans*-fatty acids have been documented to increase low density lipoprotein (LDL) cholesterol and lower high density lipoprotein (HDL), increasing the risk for coronary heart disease (McCarthy et al. 2008). Beginning in 2006, the amount of *trans* fat present in food and dietary supplements is required on the nutrition label and is to be expressed as grams/serving (Mossoba et al. 2009). Amounts below 0.5 g are recorded as *trans* free. It is the FDA's policy that it is the manufacturer's responsibility to ensure the validity of a product's stated nutrition information. Further, they define a product as misbranded if the amount of *trans* fat found during FDA analysis is greater than 120% of what is presented on the nutrition label (Mossoba et al. 2009). The basic procedure for analysis via GC involves extracting the fat, preparing volatile fatty acid methyl ester (FAME) derivatives, resolving the mixture with a column that is capable of separating all the FAME components, summing all individual *trans* FAME, correcting the detector's response, and finally converting to triacylglycerol equivalents (Mossoba et al. 2009). Because each step needs to be quantitative or representative, these methods require highly trained technicians.

IR methods have been used since the 1950s to determine the amount of isolated *trans* double bonds in fats and oils. This method is based on the IR band at 966 cm^{-1} corresponding to CH-out-of-plane deformation (**Figure 3*a***). This band is unique to isolated *trans* double bonds. The challenge with using this technique includes resolving the *trans* band from those due to conjugated double bonds or due to interferences attributed to other functional groups. It has been found that fats and oils with low levels of *trans*-fatty acids are affected the most by overlapping bands. The *trans* band is found on an elevated sloping baseline, decreasing the accuracy of area or height measurements as the level of *trans* fat decreases (Mossoba et al. 2009). Two ATR-FTIR official methods (AOCS Cd 14d-99 and AOAC method 2000.10) incorporate a background of *trans* fat–free oil to flatten the sloping baseline. Reference standards are created using trielaidin added to a *trans*-free reference oil. The standards are scanned on a 65°C single or multibounce ATR cell to generate a calibration curve. Using the curve, unknown *trans* levels, expressed as percent of total fat, can be calculated. It should be noted that using reference oil that differs considerably from the composition of the unknown sample can have an adverse affect on the accuracy of the model, especially below 5% total fat (Mossoba et al. 2009). A new negative second derivative ATR-FTIR

method claims to improve accuracy and precision of quantitating levels of *trans* fat in a food sample and is currently being validated by an international study. This method measures the height of the negative second derivative of *trans* absorption relative to air (**Figure 3*b***). Reference standards are generated from *trans* monoene trielaidin diluted in tripalmitin. This rapid method is ideal for determining total *trans* content that is needed for current labeling requirements (Mossoba et al. 2009).

NIR spectroscopy can also be used to determine *trans* content in edible fats and oils. Li et al. (1999, 2000a,b) and Cox et al. (2000) have done extensive work on the use of FT-NIR for the rapid determination of important quality parameters of fats and oils such as peroxide value, iodine value, *cis* and *trans* content, and saponification number. Li et al. (2000c) developed a PLS calibration model from FT-NIR spectra for the rapid determination of *trans* fats and oils. The calibration model was correlated to *trans* values determined by using MIR with single-bounce IR horizontal attenuated total reflectance (IR-HATR) reference method (American Oil Chemist's Society official method). There was no discernible *trans* absorption band in the FT-NIR spectrum as compared with the strong *trans* signal at 966 cm^{-1} in the MIR spectrum. Nevertheless, the PLS-FT-NIR model was able to estimate the *trans* content of edible oils. By using a training set that included a wide variety of oil types, the calibration model predicted the *trans* content with an accuracy of $\pm 1.1\%$. It was possible to obtain more accurate and reproducible predictions ($\pm 0.5\%$) by calibrating a more limited training set that had specific characteristics. It is important to note that the reproducibility of the IR-HATR method is $\pm 0.4\%$. The product-specific calibration produced serious predictive errors when nonrepresentative samples were analyzed (Li et al. 2002b).

Acrylamide is a Maillard reaction product formed during baking, frying, and roasting foods such as potatoes and has been identified as a potential carcinogen. Standard procedures for acrylamide determination are based on chromatography and mass spectroscopy that are challenging to implement at manufacturing facilities for routine analysis. One acrylamide precursor, asparagine, is present in high levels in potatoes. High temperature/short time frying results in potato chips having one of the highest known levels of acrylamide (Segtnan et al. 2006). NIR spectral analysis has focused on the bands originating from carbohydrates, with a starch band at 1,934 nm found to be most significant. PLSR models using the spectra of ground chips in the region 400–2,498 nm correlated against predetermined quantities of acrylamide ($R^2 = 0.95$, prediction error of 256.6 $\mu g\ kg^{-1}$). NIR spectral models are accurate enough for screening of acrylamide contents in processed potato chips (Segtnan et al. 2006). However, the method needs to be tested and calibrated for each specific production process. Evaluation of the feasibility of using online monitoring of acrylamide in chips using NIR gave a model with a $R^2 = 0.83$ and prediction error of 266 $\mu g\ kg^{-1}$. The lower correlation was attributed to lower spectral resolution of the online instrument. Online NIR monitoring could be used to separate samples with very high levels of acrylamide from samples with average to low content (Pedreschi et al. 2010).

FT-NIR and multivariate analysis for the detection of food tampering with threat agents (Rodriguez-Saona et al. 2000) were developed and evaluated for the rapid detection of castor bean meal (CBM). The seeds of the castor plant (*Ricinus comunis*) contain the extremely toxic protein ricin that specifically and irreversibly inactivates eukaryotic ribosomes, promoting cell death by inhibiting protein synthesis. CBM is a byproduct of the production of castor oil and is readily available and could easily be used to deliberately contaminate the food supply, thus making it a potential threat (Wellner et al. 1995). Analysis of spiked food matrices (bleached flour, wheat flour, and blueberry pancake mix) with different CBM (0.5%–8% w/w) levels by diffuse reflectance FT-NIR predicted the CBM contamination with standard error of cross-validation (SECV) <0.6% and coefficient of correlation greater than 94%. Prediction of the CBM content by the calibration models was largely influenced by the spectral bands characteristic of amides

(4,880 and 4,555 cm^{-1}) and lipids (5,800, 5,685, 4,340, and 4,261 cm^{-1}). PLSR models accurately predicted the content of CBM in contaminated samples with no false positives for samples containing the placebo contaminants (egg white, soybean meal, tofu, and infant formula) (Rodriguez-Saona et al. 2000).

The rapid determination of tetracycline in milk was evaluated by FT-NIR spectroscopy (Sivakesava & Irudayaraj 2002b). Tetracycline antibiotics are widely used in animal husbandry for treatment of bacterial infections, suggesting a potential for tetracycline residues to be transferred to milk. The FDA has established a tolerance of 300 ppb for the sum of residues of tetracyclines in milk. The tetracycline concentration (ppb) range used in the calibration model drastically affected the performance of the chemometric models. Thus, by using separate ranges, the accuracy and predictive ability of the calibration model was significantly improved. Models developed by FT-MIR showed slightly better performance (lower SEP and higher R^2) than FT-NIR models but the repeatability of the FT-NIR was better than the FT-MIR procedure (Sivakesava & Irudayaraj 2002b). Similarly, Schulz et al. (2003) reported the reliable prediction of low concentrations of two carcinogenic compounds: methyleugenol (range 2–235 $\mu g\ 100\ g^{-1}$) and estragole (range 34–138 $\mu g\ 100\ g^{-1}$) in air-dried basil leaves by PLS calibration model based on NIR spectral data. The performance of the NIR calibration models gave values of SECV of 19.1 and 12.8 $\mu g\ 100\ g^{-1}$ and coefficient of correlation of 0.95 and 0.89 for methyleugenol and estragole, respectively.

Melamine (2,4,6-triamino-1,3,5-triazine) is used industrially in the production of plastics and glue, and also as a plant fertilizer. The compound's high nitrogen content increases the apparent protein content as measured by traditional protein analysis methods, which measure total nitrogen content as an indicator of protein levels. This makes melamine a potential adulterant in protein-rich foods such as milk and infant formula. Melamine adulteration has been reported in these products as well as in pet food, candy, coffee drinks, and others. Contaminated milk in China was likely the source of 300,000 cases of renal complications in children and at least six deaths (Mauer et al. 2009). Currently, the FDA uses an liquid chromatography–mass spectrometry (LC-MS)/MS method to detect melamine in infant formula. The detection limit for this method is 250 ppb, but it is time consuming and labor intensive. As a result, it is not efficient for screening large numbers of samples. Detection methods in other food products are also time consuming with varying levels of detection (Mauer et al. 2009).

NIR and MIR combined with multivariate statistical analysis has allowed classification of adulterated and unadulterated infant formulas with high confidence. FTIR-ATR analysis was done using the regions 3,330–2,993 cm^{-1} and 1,321–983 cm^{-1}, corresponding to the stretching vibration of amino groups and the fingerprint region generating a PLSR model with an $R^2 \geq 0.95$ and RMSECV ≤ 1. The NIR model (12,497–6,098 cm^{-1} and 5,450–4,248 cm^{-1}) performed slightly better based on a unique signal with an $R^2 = 0.999$ and RMSECV $= 0.62$ (Mauer et al. 2009). The FDA has established a threshold of 1 ppm for melamine in infant formula and 2.5 ppm in other foods. IR spectroscopy combined with chemometrics has been reported as a rapid method for detecting melamine in milk powder with detection limits of ~75–100 ppm (FOSS 2009). The technique has potential for use as a tool for screening adulteration in milk with unsuspected adulterants or contaminants at detection levels of 250–500 ppm (FOSS 2009). Factorization analyses of NIR and MIR spectra were able to distinguish between adulterated (1 ppm) and unadulterated infant formula samples (Mauer et al. 2009). NIR and FTIR methods for melamine detection are rapid and sensitive but are dependent on the food matrix, requiring new calibration models for different brands of infant formula or food products (Mauer et al. 2009).

A quality issue was identified by a dairy company regarding the safety of their products. Using a technique very similar to that used for melamine detection, a methodology was developed for the identification and quantitation of a foreign material found in the milk intake filter. The IR spectra of the foreign material was collected (**Figure 4**) and matched with commercial block rodent bait TOM CAT®, providing identical IR absorption patterns. Once the foreign material had been identified, the concern shifted to the quality of the milk. The dairy company was interested in evaluating milk samples for possible contamination with the foreign material. A PLSR calibration model was developed by spiking uncontaminated milk with known levels of the bait (100–2,400 ppb), which gave performance statistics with $R^2 > 0.99$ and SECV of ~100 ppb (7 factors) in spiked milk samples at 850–1,500 cm^{-1}, showing potential for the estimation of the bait contaminant levels in few minutes (~2 min). Classification of potential contamination of the milk samples with the bait showed detection limits <1.2 ppm of the bait (L.E. Rodriguez-Saona, unpublished observations).

The development of IR microspectroscopy (IRMS) has allowed for the acquisition of spectra from a sample area measuring only a few microns (Baeten & Dardenne 2002). IRMS combines two analytical technologies for biological analysis by coupling an infinity-corrected microscope to a high-performance IR spectrometer equipped with a mercury-cadmium-telluride (MCT) detector that will produce a spectrum with a noise level 10 to 100 times lower than the noise from the commonly used deuterated triglycine sulfate (DTGS) detector. IRMS significantly improves the sensitivity, reproducibility, differentiation, and speed capabilities of IR spectroscopy and has permitted the acquisition of spectra from samples as small as 100 pg (10^{-10} g), promoting its application in the medical and biological fields (Ozen et al. 2003). IRMS provides capabilities for high-throughput screening of chemical contaminants and the ability to resolve spectral profiles within desired regions of the target. The new generation of powerful MIR spectroscopic chemical imaging techniques combines step-scan Fourier transform Michelson interferometry with indium antimonide focal-plane array (FPA) image detection. The IR focal-plane array detector provides an instrumental multiplex/multichannel advantage, enabling spectra at all pixels to be collected simultaneously, while the interferometer portion of the system allows all the spectral frequencies to be measured concurrently. This high-definition technique represents the future of IR chemical imaging analysis, which combines the capability of spectroscopy for molecular analysis with the power of visualization. IR imaging allows the precise characterization of the chemical composition, domain structure, and chemical architecture of a variety of substances, information often crucial to the understanding of complex samples (Diem 1993).

CONCLUSION

Vibrational spectroscopic methods such as FT-NIR and FT-MIR spectroscopy are emerging as powerful techniques in monitoring adulteration and authenticity of foods. In recent years, the food industry and consumers have experienced several new or unsuspected contamination problems such as acrylamide, organic pollutants, Sudan dyes, and recently melamine in dairy products. Analysis of chemical food contaminants and toxins requires the development and validation of analytical methods and their implementation as quality control programs and risk management systems by food producers and authorities. This review shows the ability of IR combined with chemometrics to achieve resolution of unique spectral markers for differentiation (unexpected agents) and quantitation (identified agent) of food contaminants. FTIR spectroscopy is a well-established analytical technique for rapid, high-throughput, nondestructive analysis of a wide range of sample types, providing a fingerprint characteristic of chemical or biochemical substances present in the sample. Advances in FTIR instrumentation and multivariate techniques have shown

potential for analysis of complex multispectral information for the discrimination, classification, quantification, and identification of biological systems.

The ability of IR spectroscopy to reveal qualitative and quantitative characteristics about the nature of chemicals, their structure, interactions, and molecular environments provide unparalleled capabilities for detection of contaminants and adulterants in foods. Advantages of approaches based on vibrational spectroscopy include low operational cost, small size, compactness, robustness, high throughput, ease of use, and minimum background training to operate. Thus, FTIR spectroscopy can provide the food industry with rapid and specific tools for analysis of food chemical contaminants and for the reliable assessment of quality and safety. It will enable the food manufacturer to rapidly evaluate the quality of their food, allowing for timely correction measures during manufacture.

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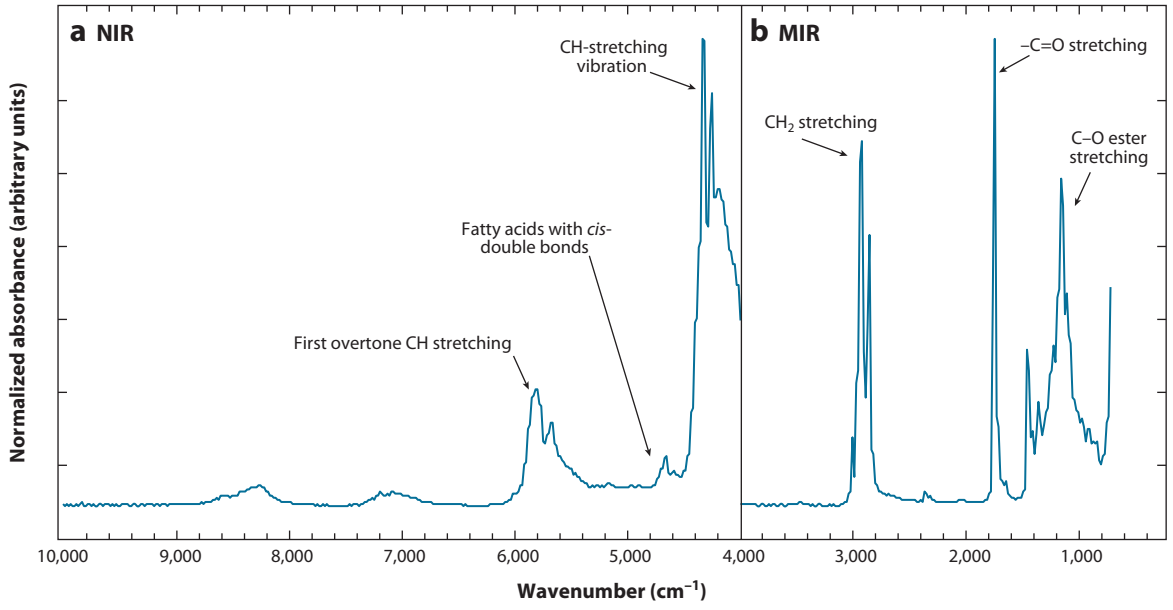

Figure 1

(*a*) Near-infrared (NIR) and (*b*) mid-infrared (MIR) raw spectra of grapeseed oil with peak identification.

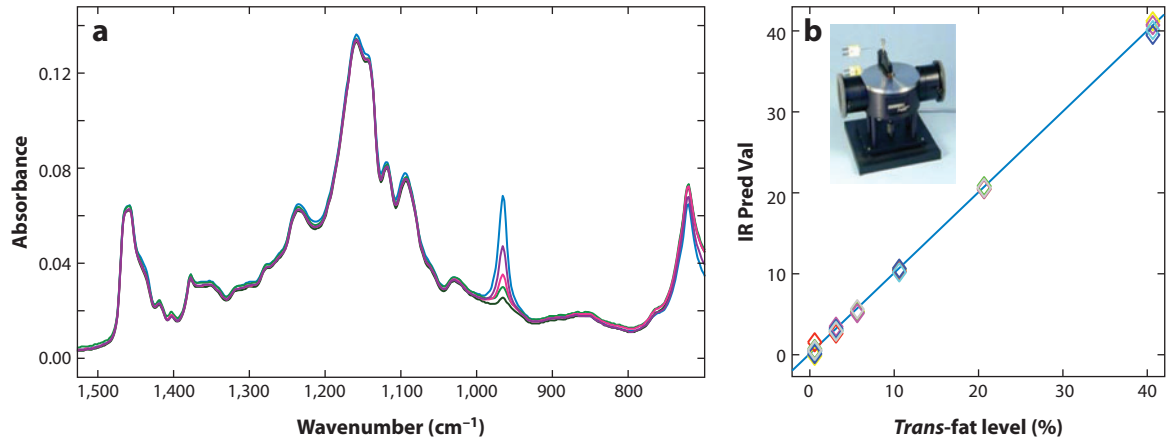

Figure 3

(*a*) Fourier transform infrared (FTIR) spectra of edible oil collected on a FatIR accessory highlighting the characteristic *trans* band at 966 cm^{-1}. (*b*) Partial least squares regression model of % *trans* fat using FTIR spectra.

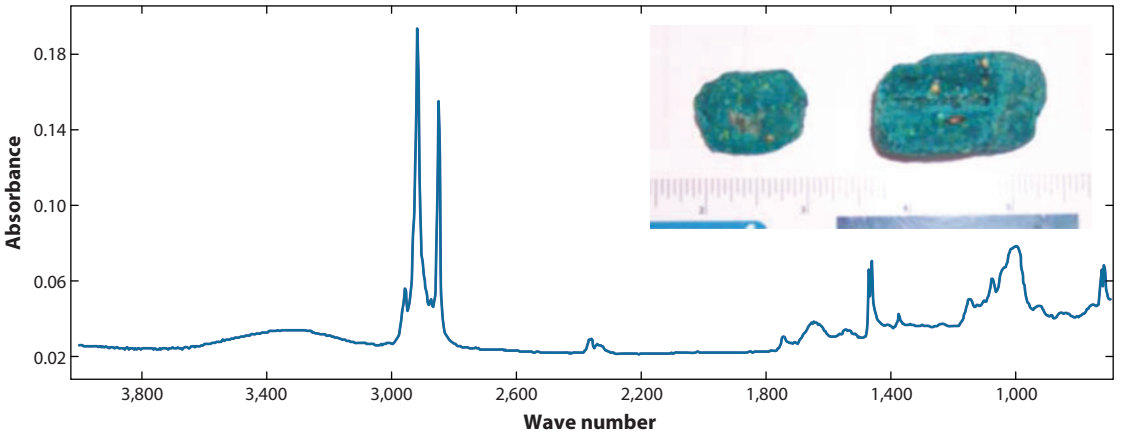

Figure 4

Attenuated total reflectance (ATR) spectra of the foreign material contaminant (block bait) found in the milk intake filter. Picture of the material is shown in the insert.

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Errata

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