Recent technological advances for the determination of food authenticity

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The relative potential of various technologies for the confirmation of food authenticity and quality are discussed. Techniques that have found new applications in the field of quality assurance since 2001 are discussed in terms of their potential ease of application in an industrial setting. The use of specific techniques with chemometric analysis for the classification of food samples based on quality attributes is also included in this review. The techniques discussed are spectroscopy (UV, NIR, MIR, visible, Raman), isotopic analysis, chromatography, electronic nose, polymerase chain reaction, enzyme-linked immunosorbent assay and thermal analysis.

Introduction

The development of new and increasingly sophisticated techniques for the authentication of food products continues apace with increasing consumer awareness of food safety and authenticity issues. Food authentication is also of concern to food processors that do not wish to be subjected to unfair competition from unscrupulous processors who would gain an economic advantage from the misrepresentation of the food they are selling.

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 (Official Journal of EC, 2002). The various safeguards and examples of foodstuffs covered by them are detailed in Table 1.

Abbreviations

DA, discriminant analysis; DSC, differential scanning calorimetry; ELISA, enzyme-linked immunosorbent assay; FT-IR, Fourier-transform infrared; FT-MIR, Fourier-transform mid-infrared; GC, gas chromatography; GC-MS, gas chromatography-mass spectroscopy; GC-TOFMS, gas chromatography-time of flight mass spectroscopy; HPLC, high performance liquid chromatography; HR-NMR, high-resolution mass spectroscopy; IR, infrared; IRMS, isotopic ratio mass spectroscopy; LDA, linear discriminant analysis; LR-NMR, low-resolution NMR spectroscopy; MIR, mid-infrared; MS, mass spectroscopy; NIR, near-infrared; NMR, nuclear magnetic resonance; PCA, principal components analysis; PCR, principal components regression; PDO, protected designation of origin; PLS, partial least squares; SNIF-NMR, site-specific nuclear isotopic fractionation NMR spectroscopy; UV–vis, ultraviolet–visible.

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Spectroscopic techniques

**MIR and NIR spectroscopy**

IR spectroscopy is a rapid and non-destructive technique for the authentication of food samples. Analysis of a food sample using the MIR spectrum (4000–400 cm\(^{-1}\)) reveals information about the molecular bonds present and can therefore give details of the types of molecules present in the food. NIR spectroscopy utilises the spectral range from 14,000 to 4000 cm\(^{-1}\) and provides much more complex structural information related to the vibrational behaviour of combinations of bonds. These techniques are suited for use in an industrial setting due to their ease of use and the relatively low financial cost of obtaining and running the equipment. The IR studies discussed here all employed some form of chemometric analysis, resulting in powerful analytical techniques, which have been successfully employed in classification studies for a wide variety of food products.

Foods that have recently been effectivley tested for adulteration using NIR spectroscopy include fruit purées and juices (Contal, Leon, & Downey, 2002; Rodriguez-Saona, Fry, McLaughlin, & Calvey, 2001), maple syrup (Paradkar, Sakhamuri, & Irudayaraj, 2002), honey (Downey, Fouratier, & Kelly, 2004), Echinacea root (Laasonen et al., 2002), milk powder (Maraboli, Cattaneo, & Giangiacomo, 2002) and fishmeal (Murray, Aucott, & Pike, 2001). Differentiation of wines on the basis of grape variety (Cozzolino, Smyth, & Gishen, 2003) yielded correct classification levels of up to 100%. The use of MIR for authentication of red wines on the basis of vintage year enabled correct classification levels of up to 100%, while correct geographical classification of the same wines achieved average levels of 85% (Pique, Cattenoz, Corrieu, & Berger, 2005). The adulteration of olive oils with a variety of common adulterants was detected using NIR analysis with very low error limits (Christy, Du, & Ozaki, 2004). The differentiation of meat from different animals sources (beef, pork, lamb and chicken) using visible and NIR analysis with PCA (Cozzolino & Murray, 2004) gave correct classification levels of 80%. Apple juice samples were differentiated on the basis of apple variety using NIR with LDA and PLS (Reid, Woodcock, O’Donnell, Kelly, & Downey, 2005). The results showed correct classification of samples on the basis of apple variety as high as 100%.

With regards to MIR, wine samples have been differentiated on the basis of geographical and varietal origin (Roussel, Bellon-Maurel, Roger, & Grenier, 2003). The use of MIR and chemometrics to detect adulteration of apple juice with beet syrup and cane syrup gave correct classifications of 100 and 96.2% respectively (Sivakava, Irudayaraj, & Korach, 2001). Adulteration of honey samples with sugar solutions at levels of 14% w/w has also been detected using MIR and PLS (Kelly, Downey, & Fouratier, 2004). Quantification of saccharides present in honey samples using MIR has been found to be capable of classifying apple juice beverages on the basis of the percent of pure apple juice present (Gomez-Carracedo, Andrade, Fernandez, Prada, & Muniategui, 2004).

**Raman spectroscopy**

Raman spectroscopy is the measurement of the wavelength and intensity of inelastically scattered light from molecules, some of which is scattered at a different wavelength. This inelastically scattered light is called Raman scatter. The particular molecule and its environment will determine what Raman signals will be observed. Raman spectroscopy possesses advantages for the analysis of food samples such as high sensitivity to C=C, C≡C and C≡N bonds, low sensitivity to water and high selectivity to inorganic substances (salts). These advantages result in its potential use for niche applications in the food industry.

Raman spectroscopy has been employed in edible oil authentication. By combining Raman spectroscopy and chemometric analysis, the detection of adulteration of olive oil samples with different levels of hazelnut oil...
NMR spectroscopy

NMR spectroscopy involves the analysis of the energy absorption by atomic nuclei with non-zero spins in the presence of a magnetic field. The energy absorptions of the atomic nuclei are affected by the nuclei of surrounding molecules, which cause small local modifications to the external magnetic field. NMR spectroscopy can therefore provide detailed information about the molecular structure of a food sample, given that the observed interactions of an individual atomic nucleus are dependent on the atoms surrounding it.

High-resolution NMR (HR-NMR; utilises frequencies above 100 MHz) has been applied in many more food authenticity studies than low-resolution NMR (LR-NMR; uses frequencies of 10–40 MHz). The advantage of HR-NMR over LR-NMR is that it is possible to obtain much more detailed information regarding the molecular structure of a food sample using HR-NMR. The major disadvantage of HR-NMR is that it is one of the most expensive analytical techniques to employ, both in terms of the initial capital outlay and running costs.

Italian olive oil samples were differentiated using H-1 NMR on the basis of their geographical origin in Italy (Mannina, Patumi, Proietti, Bassi, & Segre, 2001) and from different areas within Tuscany, itself a relatively small geographical area (Mannina, Patumi, Proietti, & Segre, 2001).

Chemometrics in conjunction with C-13 NMR was capable of differentiating oils on the basis of botanical origin and, in the case of olive oils, on the basis of processing (Brescia, Alvitti, Liuuzzi, & Sacco, 2003; Mannina et al., 2003; Zamora, Alba, & Hidalgo, 2001; Zamora, Gomez, & Hidalgo, 2002). The detection of adulteration of olive oil samples with seed oil was possible using P-31 and H-1 NMR with multivariate discriminant analysis and enabled adulteration levels as low as 5% v/v to be detected (Vigli, Philippidis, Spyros, & Dais, 2003).

Wines from the Apulia region of Italy were differentiated from wines from Slovenia using H-1 NMR and chemometric analysis (Brescia, Kosir, Caldarola, Kidric, & Sacco, 2003). The addition of pulpwash is a serious quality control issue in the production of orange juice and its detection was possible using a combination of H-1 NMR and principal components analysis (Le Gall, Puaud, & Colquhoun, 2001). Coffee samples were differentiated according to manufacturer using H-1 NMR and a combination of principal components analysis and linear discriminant analysis (Charlton, Farrington, & Brereton, 2002).

SNIF-NMR and IRMS

The specific proportions of the particular isotopes of hydrogen and oxygen present in molecules are dependent mainly on climatic and geographical conditions and, to a lesser extent, the photosynthetic metabolism of plants. The effect of these conditions on the final isotopic composition of a molecule is known as isotopic fractionation. This natural phenomenon is exploited by two particular analytical techniques—SNIF-NMR and IRMS—which are perhaps the most sophisticated and specific techniques for determining food authenticity. Both techniques are capable of determining the exact proportion and location of specific isotopes within a food sample. However, the financial cost of purchasing and operating such high-specification NMR and MS instruments is quite high and time-consuming sample preparation is required before analysis can take place.

SNIF-NMR has found widespread acceptance as a definitive technique for the authentication of wine samples and the EU adopted it in 1990 as the official method for controlling chaptalisation (addition of sugars prior to fermentation) in wines (The Commission of the EC, 1990). Recently, the use of chemometrics with both SNIF-NMR and IRMS has received attention as a means of determining chaptalisation and the varietal and geographic origin of wines (Kosir, Kocjancic, Ogrinc, & Kidric, 2001; Martinelli et al., 2003; Ogrinc, Kosir, Kocjancic, & Kidric, 2001). Tequila has also been authenticated using SNIF-NMR and IRMS (Aguilar-Cisneros, Lopez, Richling, Heckel, & Schreier, 2002; Bauer-Christoph et al., 2003).

It has been demonstrated that adulteration of orange juices can be detected using IRMS of delta 13C values for sugar adulteration (Antolovich & Robards, 2001). A combination of SNIF-NMR sample preparation and IRMS analysis of the 18O/16O ratio was able to detect adulteration of pure orange juice with orange juice made from concentrated at levels of 25% and above (Jamin, Guerin, Retif, Lees, & Martin, 2003). Apple juices have been successfully authenticated using IRMS analysis of the C12/C13 and H2/H1 ratios (Kelly et al., 2003) for detection of the addition of low-cost sugar syrups to the juice samples. Milk samples have been authenticated in terms of their geographic origin within
the Apulia region in southern Italy with correct classification levels of 100% (Brescia, Caldarola, Buccolieri, Dell’Attì, & Sacco, 2003). Characterisation of animal products (milk from dairy cows and meat from steers) according to geographic origin and feeding diet was successfully carried out using nuclear magnetic resonance and isotope ratio mass spectrometry (Renou, Bielicki, et al., 2004; Renou, Deponge, et al., 2004). The determination of the geographic origin of specific types of cheeses (Manca et al., 2001; Pillonel, Badertscher, et al., 2003), cereal products (Brescia, Sgarrella, Ghelli, & Sacco, 2003) and lamb (Piasentier, Valusso, Camin, & Versini, 2003) were also successfully carried out.

Fluorescent and UV–vis spectroscopy

While there has been a steady growth in food authentication applications for NIR, MIR and Raman spectroscopy, there has been very little research carried out using either fluorescent or UV–vis spectroscopy for this purpose. Recent work using fluorescence spectroscopy has dealt with discriminating virgin olive oils from other types of olive oil (Guimet, Ferre, Boque, & Rius, 2004). UV–vis spectroscopy has been applied to detecting the presence of extraneous colourants in cochineal food colouring (González, Lobo, Méndez, & Carnero, 2005). Despite these examples, there has been a dearth of research involving fluorescent and UV–vis spectroscopy to date. However, the recent development of a portable UV-spectroscopic testing device for the authentication of Scotch whisky illustrates the potential of UV spectroscopy for industrial use (Diageo, in press). This spectroscopic device can be used in the field and cuts the time for the screening of Scotch whisky down to less than 1 min. It is clear that the potential of UV, and fluorescent, spectroscopy for food authentication has not been fully investigated and that future research may lead to the development of more applications for these techniques, which possess the advantages of being quick and relatively inexpensive to carry out.

Chromatographic techniques

Liquid and gas chromatography are capable of separating and enabling identification of almost any type of molecule present in a food sample. Liquid chromatography, in particular HPLC, can detect compounds such as proteins, amino acids, phenolic compounds and carbohydrates, while GC is more suited to the analysis of naturally volatile or semi-volatile molecules. The principal disadvantage of the two techniques relates to their use in conjunction with chemometrics. There is often a need to extract the specific analytical data relating to individual compounds and this adds to the time and labour required.

Gas chromatography

The detection of adulteration of olive oil samples using GC has focussed on comparison of their fatty acid composition (Cercaci, Rodriguez-Estrada, & Lercker, 2003; Gamazo-Vasquez, Garcia-Falcon, & Simal-Gandara, 2003) and chemometric analysis of their fatty acid contents (Dourtoglou et al., 2003). Chemometric analysis of the triacylglycerol and fatty acid composition of French olive oil samples was employed to determine their varietal and geographical authenticity (Ollivier, Artaud, Pinatel, Durbec, & Guerere, 2003). Similar analyses of the triacylglycerol component of Italian olive oils enabled their classification as Italian as opposed to Argentinian (Mannina, Fontanazza, Patumi, Ansanelli, & Segre, 2001). Differentiation of mono-varietal Sicilian olive oil samples was carried out using LDA analysis of 10 GC peaks and resulted in 95% confidence levels (Mannina et al., 2003). In fact, it was found in this study that GC analysis in conjunction with chemometrics gave clearer separation between cultivars than NMR analysis of the same samples in conjunction with chemometrics.

Chemometric analysis has been successfully applied to gas chromatographic data to authenticate several other food types including coffee (Alves, Casal, Oliveira, & Ferreira, 2003; Casal, Alves, Mendes, Oliveira, & Ferreira, 2003) and fruit juices (del Castillo, Caja, Blanch, & Herraiz, 2003; del Castillo, Caja, & Herraiz, 2003). GC was used in conjunction with LDA and PLS to differentiate apple juice samples on the basis of apple varieties and also applied heat-treatment (Reid, O’Donnell, Kelly, Daniel, & Downey, 2004). The adulteration of strawberry purées with different levels of apple purée was detected using SPME-GC and PLS analysis at levels of 25% and above (Reid, O’Donnell, & Downey, 2004).

HPLC

The number of new applications of HPLC in food authenticity is low for recent years, nevertheless there have been some applications reported for the technique (Table 2).

HPLC analysis of the whey protein β-lactoglobulin has enabled detection of the adulteration of ovine and caprine cheese with bovine milk (Ferreira & Cacote, 2003) at levels as low as 2% v/v, and of caprine milk with bovine milk (Chen, Chang, Chung, Lee, & Ling, 2004). Wines from different designated areas of origin in the Canary Islands were correctly classified at levels as high as 100% using HPLC analysis of selected polyphenol compounds combined with PCA and LDA of the concentration of these compounds (Rodriguez-Delgado, Gonzalez-Hernandez, Conde-Gonzalez, & Perez-Trujillo, 2002).
Spanish table wines were correctly differentiated at levels of 83–86% using PCA and LDA of the HPLC data obtained for selected biogenic amine compounds in the samples (Romero, Sanchez-Vinas, Gazquez, & Bagur, 2002).

HPLC analysis of the triglyceride and tocopherol composition of coffee samples was combined with PCA and LDA to differentiate coffee samples on the basis of variety (Gonzalez, Pablos, Martin, Leon-Camacho, & Valdenebro, 2001). Olive oil cultivars from Spain were classified using PCA and DA analysis of their HPLC triglyceride composition (Aranda, Gomez-Alonso, del Alamo, Salvador, & Fregapane, 2004). Adulteration of olive oil with hazelnut oil could be detected by HPLC analysis of the polar compounds of the oil samples (Gordon, Covell, & Kirsch, 2001). Using this technique, it was possible to identify the adulteration of durum wheat cultivars and wheat cultivars commonly used as adulterants in durum wheat (Bonetti et al., 2004). Using this technique, it was possible to identify the adulteration of durum wheat cultivars, which are the wheat cultivars of choice for use in high-quality pasta, at levels as low as 5% w/w.

Advances in recent years that may increase the ease with which these chromatographic techniques can be applied to industrial food authentication. The time of analysis for GC can be greatly reduced by using GC-TOFMS but this technique has found limited applications to date in the area of food authenticity, principally due to the cost of acquiring and running a GC-MS system.

### Electronic nose

Electronic nose technology is based on the detection by an array of semi-selective gas sensors of the volatile compounds present in the headspace of a food sample. Advantages of electronic nose technology include the relatively small amount of sample preparation that is involved and the speed of analysis. However, this technique employs sensors that are not very selective for particular types of compounds thus preventing any real identification or quantitation of individual compounds present in a food sample. Such a drawback has obvious implications for food authentication, as an adulterant could not be definitively identified.

There has been some success using electronic nose technology for the differentiation of olive oils on the basis of geographical origin (Guadarrama, Rodriguez-Mendez, Sanz, Rios, & de Saja, 2001) and the adulteration with either sunflower oil or olive-pomace oil (Martin, Oliveros, Pavon, Pinto, & Cordero, 2001). Oliveros et al. (2002) successfully applied electronic nose and chemometric analysis for the detection of adulteration of olive oil samples with sunflower and olive-pomace oil at levels as low as 5%. Electronic nose analysis of sunflower oil and different grades of olive oil demonstrated that it was also possible to differentiate extra virgin olive oil, non-virgin olive oil and sunflower oil (James, Scott, O’Hare, Ali, & Rowell, 2004).

Electronic nose technology in conjunction with chemometric analysis has also been successfully applied to differentiation studies on wine samples. Spanish white, red and rosé wines were differentiated using a combination of electronic nose and principal components analysis (Guadarrama, Fernandez, Inguez, Souto, & de Saja, 2001). Italian wines of different geographical and varietal origin were also successfully separated with correct classification rates as high as 100% (Penza & Cassano, 2004). Authenticity studies with electronic nose have also been successfully carried out for the differentiation of unifloral honey samples (Ampuero, Bogdanov, & Bosset, 2004), the determination of the geographical origins of Valencia orange juices (Steine, Beaucousin,
Despite its drawbacks, electronic nose technology remains an area of research that holds much potential for future development. It is a rapid means of analysis, can be easily used in conjunction with chemometrics and, as shown above, has had a good degree of success in the authentication of a wide range of food types.

DNA-based technology

The growth in recent years of research into food authentication methods based on the analysis of the DNA present has been due in no small part to the public health concern associated with the BSE crisis. The majority of work related to exploiting DNA analysis has focussed on using PCR to amplify the specific areas of DNA of interest. The principle of PCR is that specific lengths of DNA can be copied enough times to provide a sufficient amount of that area of DNA to be analysed using a variety of methods with electropheretic techniques being the most frequently used.

The greatest amount of research on the application of PCR for the authentication of food samples involves the analysis of meat and meat-based products, including fish. It has been shown that it is possible to detect adulteration of minced beef with chicken and pork meat (Calvo, Osta, & Zaragoza, 2002). Differentiation between meat samples from different breeds of cattle (Vascconcellos, Tambasco-Talhari, Pereira, Coutinho, & Regitano, 2003) has also been successfully demonstrated using the technique. The analysis of minced lamb using real-time PCR enabled the detection of the addition of minced beef at a level of 2% (w/w) (Sawyer, Wood, Shanahan, Gout, & McDowell, 2003). DNA technology was also capable of detecting adulteration of duck and goose foie gras with chicken (Rodriguez et al., 2003) and adulteration of poultry pate with pork (Calvo, Zaragoza, & Osta, 2001). Species identification in meat, bone and fishmeal destined for use as animal feed has also been carried out using PCR (Bellagamba, Moretti, Comincini, & Valfre, 2001; Bellagamba, Valfre, Panseri, & Moretti, 2003).

The use of PCR technology to differentiate between different species of fish has received much attention. Frozen samples of flatfish have been differentiated according to species (Comesana, Abella, & Sanjuan, 2003). The identification of fish species in tinned tuna and sardine has also been successfully carried out using PCR analysis (Jerome, Lemaire, Verrez-Bagnis, & Etienne, 2003; Terol, Mascarell, Fernandez-Pedrosa, & Perez-Alonso, 2002). The authentication of Messolonghi fish roe, which possesses protected designation of origin status (PDO), was also demonstrated using PCA, enabling differentiation of higher-value Messolonghi fish roe and fish roe from other areas of Greece (Klossa-Kilia, Papasotiropoulos, Kilias, & Alahiotis, 2002).

It has also been shown that pasta made using high-quality durum can be tested for adulteration using PCR (Alary, Serin, Duviau, Joudrier, & Gautier, 2002). The adulteration of spelt flour products and non-wheat semolina and pasta with wheat was also possible using PCR (Terzi, Malnati, Barbarana, Stanca, & Faccioli, 2003; von Buren, Stadler, & Luthy, 2001).

Immunological technology

The majority of reported studies on immunological techniques for food authentication concern the use of ELISA. This technique involves the cultivation of antibodies or antisera that are capable of binding to a protein of interest, thereby enabling the detection of that protein, both qualitatively and quantitatively. The major advantage of this approach is that antibodies or antisera can be manufactured to respond specifically to the protein of interest, thereby enabling recognition and quantitation of that protein exclusively. The disadvantages of the ELISA approach include the initial difficulty in producing an antibody specific to a particular protein. However, this is a relatively minor difficulty to overcome when the selectivity of the technique is taken into account.

Recent research using ELISA-based techniques include detecting the presence of meat from different species in food products (Jha, Kumar, & Mandokhot, 2003) and the presence of vegetable proteins in milk powder (Sanchez, Perez, Puyol, Calvo, & Brett, 2002). There have also been promising results for the use of ELISA to differentiate milk from different species (Bania, Ugorski, Polanowski, & Adamczyk, 2001; Moatsou & Anifantakis, 2003), as well as to detect the adulteration of sheep and goat milk with cow milk at levels as low as 0.1% (Hurley, Coleman, Ireland, & Williams, 2004). This technique holds much potential for the authentication of food products but, to date, limited advances have been made in extending its authentication capabilities.

Thermal techniques

Differential scanning calorimetry (DSC) allows the physical changes that occur upon heating a food sample to be determined. Such changes include the glass transition point. The technique possesses the advantages of being relatively quick and simple to carry out, with relatively little sample preparation necessary. In terms of food authentication, the majority of work involving DSC analysis has focussed on fats and oils. DSC has proven successful at the detection of adulteration of lard and beef tallow in canola oil (Mariikkar, Ghazali, Man, & Lai, 2002) and of lard in palm oil (Mariikkar, Ghazali, Man, & Lai, 2003). Apart from fats and oils, the...
Differentiation of honey samples on the basis of floral origin and adulteration was possible using DSC (Cordella, Faucon, Cabrol-Bass, & Sbirrazzuoli, 2003). It is clear that DSC holds much potential for the authentication of food products although reported applications to date are limited.

Conclusions
There has been a great deal of research into the development of new applications for existing analytical and chemometric techniques for food authentication since 2001. However, it must be stated that, despite this ongoing research, those involved in carrying out food fraud are also continuing to develop new ways of circumventing accepted techniques for food authentication.

The majority of recent applications have occurred within the areas of newer technologies such as SNIF-NMR, IRMS and DNA-based technologies. The techniques of SNIF-NMR and IRMS have the principal advantage of being practically impossible to outlaw, due to the dual specificity of the atoms analysed and the location of those atoms in molecules within the food sample. On the other hand, these techniques are not likely to find widespread application in the food industry unless the instrument and running costs are lowered. The DNA-based techniques have particular potential for use in the authentication of food samples of animal origin, such as species determination in meat products. Yet, DNA analysis has its pitfalls with regards to food authentication. An example of this is the removal by DNA digestion techniques of pork protein before injection of pork into chicken products, thus rendering protein and DNA analysis obsolete in the authentication of products adulterated in this way.

IR spectroscopy has been shown to be a sensitive and rapid technique in the authentication of a wide variety of food samples, with the advantage of being easy to use in conjunction with chemometric analysis for more definitive classification of food samples. NMR spectroscopy, despite it very high level of specificity and accuracy in food characterisation, has similar impediments in terms of financial costs associated with it to SNIF-NMR and IRMS for online applications. The use of immunological techniques could possibly provide a useful alternative to DNA analysis in such cases.

Developments in GC technology, such as GC-TOFMS, have led to great reductions in the analysis times for food samples, with chromatographic runs lasting only a few minutes. This technique has possible industrial potential, although the use of MS detection would vastly increase the cost. Electronic nose technology has the advantage of being relatively cheap, quick and easy to operate. This technique is not very well developed yet and problems still exist regarding the sensors used in the electronic nose instrumentation, such as sensor poisoning and a lack of specificity. If these obstacles can be overcome, it is possible that electronic nose technology could find many industrial applications in the area of food authentication and quality assurance. Thermal analysis for food authentication has not received much research attention and therefore remains an area for future development.

The outlook for food authentication indicates that research into relatively novel techniques such as SNIF-NMR, IRMS and electronic nose offer the greatest potential for the development of new food authentication protocols. The continuing development of applications for more established techniques should focus on the potential of these techniques for use with chemometric analysis.

References


Official Journal of the European Communities; Article 8, Regulation (EC) No. 178/2002D.


