Trends in Food Science & Technology 46 (2015) 27–48

Contents lists available at ScienceDirect

Trends in Food Science & Technology

journal homepage: http://www.journals.elsevier.com/trends-in-food-science-and-technology

Review

Analytical methods coupled with chemometric tools for determining the authenticity and detecting the adulteration of dairy products: A review

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ARTICLE INFO

Article history:
Received 29 December 2014
Received in revised form
13 July 2015
Accepted 15 July 2015
Available online 17 July 2015

Keywords:
Dairy products
Authenticity
Adulteration
Spectroscopy
Chemometric

ABSTRACT

Authenticity of dairy products has become an urgent issue for producers, researchers, governments, consumers and so on due to the increase of falsification procedures inducing lost large of money as well as the confidence of consumers. The determination of the authenticity and the detection of adulteration of milk and dairy products have been determined by several analytical techniques (e.g., physico-chemical, sensory, chromatography, and so on). Although these methods are considered as the reference ones, they required sophisticated analytical equipment's and skilled operators; they are also time consuming and need both the purchase and disposal of chemical reagents. Therefore, there is a need to find cheap and fast methods for the determination of the authenticity and the detection of adulteration of these products. Thus, spectroscopic techniques such as fluorescence spectroscopy, near infrared (NIR), mid infrared (MIR), nuclear magnetic resonance (NMR), among others, in combination with multivariate data analysis methods could be considered helpful tools in this domain. The advantages and disadvantages of each technique will be also discussed in this review.

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1. Introduction

Food adulteration has been practiced since a long time ago and becomes increasingly in the last years more sophisticated. Foods and ingredients presenting high-value are the most vulnerable for adulteration (Karoui & De Baerdemaeker, 2007). Determination of food authenticity and detection of adulteration have become an important question in quality control and safety of food. Indeed, consumer awareness has increased about food quality and safety, geographical origin and agricultural practices, mainly after the spread of foodborne diseases around the world (Gonzálvez & de la Guardia, 2013). Non-authentic food products arising from the adulteration and fraud. The replacement of original substance partially or completely with more easily available and cheap substance is the most common procedure performed by defrauders such as the addition of: i) flavors/aromas to improve the value of cheap products; ii) and/or cheap substances to the food products (Hrbek, Vaclavik, Elich, & Hajslova, 2014; Van Leeuwen, Prenzler, Ryan, & Camin, 2014). Indeed, milks could be adulterated by several ways such as mix different types of milk, whey, neutralizing to mask acidity, melamine, salt or sugar to mask extra water or high solid contents, among others (Kartheek, Smith, Muthu, & Manavalan, 2011). For example, Souza et al. (2011) reported that commercial ultra-high temperature milks (UHT) available in the Brazilian market presented at least one adulterant, such as starch, chlorine, formalin, hydrogen peroxide, urine, etc. Other fraudulent means can be used such as the excessive addition of water into milk resulting in the decrease of nutritional quality of the dairy product, or addition of non-milk fat/oil into dairy products (Santos & Pereia-Filho, 2013). The authenticity of dairy products can be also related to their geographical origin and processing technology (Hrbek et al., 2014). Another aspect linked to the authenticity of milk and dairy products concern the need to avoid labeling of conventional milk as a product from organic farming (Molkentin & Giesemann, 2007).

Milk and dairy products are essential foods for human food, where they are considered very important for some consumer groups including children, pregnant women and elderly due to their high nutritional values (Souza et al., 2011). Indeed, milk is one of the seven (7) top foods that could be adulterated (Moore, Spink, & Lipp, 2012). The percentage of adulteration of milk in world varied according to the country. For example, in India, more than

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http://dx.doi.org/10.1016/j.tifs.2015.07.007
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60% of milk is unsafe and adulterated with paint, detergent (Food Safety and Standards Authority of India, 2012), where as in Brasil, it is around 10% (Brazilian Institute of Geography and Statistics, 2012). This fact has been widely registered as the Global ‘melamine scandal’, broken out in 2008 in China, and was related with serious health risks for consumers (Chan, Griffiths, & Chan, 2008; European Food Safety Authority, 2010) due to the use of chemical products to adulterate protein levels in diluted milk. In China, these adulterated products resulted in illness of 294,000 individuals, hospitalization of 50,000, and death of 6 children's (Domingo, Tirelli, Nunes, Guerreiro, & Pinto, 2014).

Nowadays, the determination of the authenticity and the detection of adulteration of dairy products is a major concern in order to: i) assure the traceability system from milk to fork; and ii) ensure that dairy products are correctly labeled in terms of which animals are actually processed for consumption. In order to protect consumers from toxic and harmful compounds in milk and dairy products such as antimicrobials, mycotoxins, residues of cleaners and sanitizers, pesticides residues, heavy metals and so on, requirement for the dairy products traceability has been imposed in developed countries (Jooste, Anelich, & Motarjemi, 2014). Indeed, the traceability system represent a helpful tool in the domain of food safety, where it could effectively trace quality and decrease information asymmetry problems of adverse selection and moral risk in the food system (Peres, Barlet, Loiseau, & Montet, 2007; Zhang, Zhang, Dediu, & Victorn, 2011). Although relevant authenticity issues of dairy products include species, feeding, and farming system, other factors such as processing conditions, packaging, and so on could be considered.

Determination of the authenticity and adulteration detection of dairy products has been performed by using several analytical methods. The physico-chemical methods are the most used ones for authenticating dairy products (Karoui & De Baerdemaeker, 2007; Souza et al., 2011). Although heavy, these techniques are considered among the most interesting ones for the authentication and adulteration purposes. With regard to sensory methods, they have been utilized to determine some attributes (e.g., leakage of whey for appearance, butter or milk flavor, butter taste and rubbery texture in Coalho cheeses) as pointed out by Cavalcante et al. (2007). Other techniques namely liquid and gas chromatography, isotope ratio analysis, and DNA based method, have also been found extremely helpful in dealing with the problems related to adulteration, and authentication of dairy products (Karoui & De Baerdemaeker, 2007; Sharma, Rajput, Poonam, Dogra, & Tomar, 2009; Pajor, Galló1, Steiber, Tasi, & Póti, 2009). As for the physico-chemical methods, liquid and gas chromatographic techniques, which are considered as official ones, for detecting harmful substances are time consuming, high cost, labor intensive and need complex sample pretreatment procedures (Lin, 2009). The isotope ratio analysis and DNA based methods presented also the same drawbacks. For all these reasons, there is a need to develop rapid, inexpensive and efficient analytical methods for the detection of frauds and authentication of milk and dairy products. Recently, more attention has been paid to the development of non-invasive and non-destructive techniques such as infrared, fluorescence, nuclear magnetic resonance (NMR), and so on. These techniques are fast, of relatively low cost, environmentally friendly, and provide a great deal of information with only one test, making them suitable for on-line and/or at-line process control. In addition, spectroscopic techniques often require little or no sample preparation and are relatively easy to operate. Although the advantages of these new analytical techniques, it is sometimes difficult to determine the authenticity and to detect the adulteration of milk and dairy products with high accuracy. This could be explained by the fact that the composition of milk may be related with other factors such as: i) influence of the environment as climate and season; ii) individual differences between the animals; iii) lactation stage, and so on. These factors would have significant impacts on the authenticity of milk and dairy products, and thus might contribute to a difficult authentication of these products.

This review paper will provide a comprehensive overview of the applications of different analytical techniques, in combination with multivariate data analysis, to determine the authenticity and to detect the adulteration of dairy products during the last 7 years. Actual examples illustrating the utilization of these techniques in both laboratory and industrial environments will be discussed as well as their advantages and disadvantages.

2. Chemometric tools

Chemometric can be defined as the science, which based on the application of mathematical and statistical methods in order to process data acquired on a food product in an optimal way. These techniques have proven their abilities as useful tools for the determination of the authenticity and/or the detection of adulteration; indeed, these chemometric tools could be applied when it's not possible to attain the results through the analysis of one single food property but it needs the generation of multivariate data sets (i.e., analysis of more than one sample property) as pointed out by Capuano, Rademaker, van den Bijgaart, & van Ruth, (2014). The multivariate analysis can be applied aims to detect specific hallmarks or fingerprints in a multivariate dataset that might be used to infer about the authentic nature of a food product. . The chemometric tools allow optimal application of the analytical methods, in particular, spectroscopic ones, through the extraction and interpretation of valuable information from large and complex data sets, identification of patterns in the data and development of calibration models in many analytical fields (Souza et al., 2011). The multivariate approach has proven beneficial in analysis of nonselective signals and specifically with respect to handling of interferents and as a diagnostic tool for the detection of deviating samples or outliers (Izenman, 2009). These powerful methods and the computer technology necessary to use them have only become readily available in recent years; their application has become a significant feature for the analytical techniques used to evaluate the quality of dairy products. A large number of scientific studies have been published over the last years wherein chemometrics has been applied to data generated from a variety of analytical methodologies (chromatography, spectroscopy, mass spectrometry, calorimetry, wet chemistry) for the authentication of dairy products (Capuano et al., 2014). A broad range of chemometric tools is now available including data reduction tools, regression techniques, and classification methods (Roggo et al., 2007).

The best-known and most widely used variable-reduction method is the principal component analysis (PCA). PCA is a mathematical procedure, which decomposes the data matrix with n rows (samples) and p columns (variables) into the product of a scores matrix. The scores are the position of the samples in the space of the principal components (PCs) while the loadings are the contributions of the original variables to the PCs. All the PCs are mutually orthogonal, and each successive PC contains less of the total variability of the initial data set.

Another variable-reduction method used to present the data to emphasize the natural groupings in the data set is hierarchical cluster analysis (HCA). The HCA approach involves the assessment of similarity between the samples based on their measured properties (variables). The samples are grouped in clusters in terms of their nearness in the multidimensional space, and the results are presented in the form of dendrograms to facilitate the visualization of the sample relationships (Almeida, Barbosa, Pais, & Formosinho,

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