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Identifying cheese whey an adulterant in milk: Limited contribution of a sensometric approach

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ABSTRACT

A sensometric approach for the identification of sensory descriptors that characterize milk samples added with whey was investigated. Sweet cheese whey (pH = 6.59, nonfat dry = 8.06% w/w) was added to raw milk in increasing concentrations (0, 5, 10, 15 and 20% v/v), and then submitted to quantitative descriptive analysis. The data treated used multivariate statistical methods, principal component analysis (PCA) and hierarchical cluster analysis (HCA). Some inconsistent results were reported during the evaluation of the samples, suggesting problems along the panel member training and/or lack of concentration, motivation along the test. Our results suggest chemometric methods allied to descriptive sensory tests present limited contribution to investigate authenticity of milk due the presence of cheese whey. This aspect compromising the performance the multivariate analysis, which findings should be face as tendency.

1. Introduction

The authenticity of foods is currently of major concern for researchers, consumers, industries and policy makers at all levels of the production process. Milk is a typical example as, although this product provides several benefits to the human health (Napgal et al., 2012), it is a fairly expensive raw material. Therefore, from an economic standpoint, modifying the milk composition and replacing part of it with other dairy or non-dairy ingredients could seem as an attractive practice (De La Fuente & Juarez, 2005). Indeed, milk is one of the seven top foods that are adulterated, and this fact has been widely recorded (Moore, Spink, & Lipp, 2012). Recently, Souza et al. (2011) reported that commercial ultra-high temperature milks available in the Brazilian market presented at least one adulterant, such as starch, chlorine, formalin, hydrogen peroxide and urine. Moreover, the addition of cheese whey in fluid milk has already been reported elsewhere (Lasmar et al., 2011), and it is very difficult to detect such an alteration by official analytical procedures (AOAC, 2005), making it necessary to implement new experimental procedures/assays, such as Fourier transform infrared spectroscopy (Cassoli, Sartori, Zampar, & Machado, 2011).

However, equipment and accessories involved in implementing these analytical methodologies may represent a barrier to health agencies and sectors of surveillance, mainly in developing countries, which typically have limited financial and human resources. In this sense, the use of chemometric techniques (Almeida, de Sá Oliveira, Stephani, & Cappa de Oliveira, 2013; Finete, Gouvêa, Marques, & Netto, 2013; Santos, Pereira-Filho, & Rodriguez-Saona, 2013a) allied to sensory methods, particularly sensory descriptive techniques – we named sensometric approach – as a tool in identifying this type of fraud can be interesting and useful, as the time involved in training the panel can be readily compensated by the speed of obtaining the results.

In this sense, this study aimed to evaluate the potential use of sensometric approach (quantitative descriptive analysis allied to chemometric methods) to identify attributes that allow detecting adulteration of raw fluid milk intentionally added with cheese whey at different concentrations.

2. Material and methods

2.1. Sampling

The raw milk samples (18 L) were collected on a weekly basis on a private property in the city of Areal, Rio de Janeiro. The selection criterion of the property was based on a previous analysis of milk quality and permission to follow up the milking. Such monitoring was performed to avoid the addition of substances to the raw milk. After milking, the milk was immediately cooled (4 ± 1 °C) and taken to the Veterinary Faculty of the Universidad Federal Fluminense, in Niteroi city, in the state of Rio de Janeiro.

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2.1.1. Cheese whey acquisition

The cheese whey used in the experimental analyses was obtained by the manufacture of Minas fresh cheese. For this purpose, an enzymatic clotting (Chr Clerici Power Rennet, Caglificio Clerici, Sao Paulo, Brazil) was performed before the cutting of milk gel, followed by filtration (Gomes et al., 2011). The Sweet cheese whey (pH = 6.59, nonfat dry = 8.06% w/w) obtained from this process was added to raw milk (500 mL) in increasing concentrations (0, 5, 10, 15 and 20% v/v, coded M0, M5, M10, M15 and M20, respectively) being the mix cheese whey/milk pasteurized for 65 °C/30 min. The temperature of 65 °C was obtained in the cooker and was maintained by a water bath. The samples were stored in glass bottles, kept under refrigeration (4 ± 1 °C) and subsequently offered to the panelists throughout the training period. There was no addition of salt during the processing. Table 1 shows the description of samples and their encodings.

Table 1
Samples description.

Code	Sample
M0	Milk without addition of whey
M5	Milk with 5% cheese whey
M10	Milk with 10% cheese whey
M15	Milk with 15% cheese whey
M20	Milk with 20% cheese whey

2.2. Sensory profiling

Tests were conducted in a standard room equipped with three individual taste booths. Samples (about 15 mL) were served at 4 ± 1 °C in white plastic vessels coded with three random digit numbers. Still mineral water and unsalted crackers were used as palate cleanser.

The sensory profile of each product was determined by thirteen assessors (5 women, 8 men, aged 24–32 years) who were selected and trained according to the methodology of quantitative descriptive analysis (QDA, Lawless & Heymann, 2010). All of them had prior experience with quantitative descriptive analysis, and are regular consumers of fluid milk. This methodology and its established procedures have been successfully used for fluid milk products like milk with low lactose content (Adhikari, Dooley, Chambers, & Bhumiratana, 2010), cheeses (Albenzio et al., 2013; Santillo et al., 2012; Wadhwani & McMahon, 2012), ice cream (Cadena, Cruz, Faria, & Bolini, 2012) and functional dairy products such as probiotics, prebiotics and symbiotic yogurts (Allgeyer, Miller, & Lee, 2010; Cliff et al., 2013; Desai, Shepard, & Drake, 2013; Gonzalez, Adhikari, & Sancho-Madriz, 2010; Pimentel, Cruz, & Prudencio, 2013).

For descriptor selection, an initial list of terms was prepared with the information obtained from the literature (Leonardi, Caniatti-Brazacca, & Arthur, 2011), as the overall there is not a notable change in the whey composition in dairy factories around the world. A group of 13 assessors were asked to evaluate the suitability of these descriptors to describe the sensory characteristics of the samples according to the checklist method (Damasio & Costell, 1991). They could propose new terms after performing the repertory grid (Moskowitz, 1983), where the samples were presented in pairs, along with the sensory plug, and each taster described similarities and differences between them in relation to appearance, odor, flavor and texture. Then, two sessions of 2 h were held. In these sessions, the assessors tested the samples and discussed the most suitable attributes. A list, composed of nine terms regarding appearance, odor, flavor and texture of the samples, was finally selected. The final list of descriptors, their definitions and some reference products are shown in Table 2. The intensity of each attribute was scored on a non-structured 9 cm line scale anchored as "weak" (1) at the lower end and "intense" (9) at the higher end.

The same group of 13 assessors was trained in six 1 hour training sessions, twice weekly according to the ISO 8586-1 (1993) guidelines. The first session was held with the panel leader and with all the assessors and was aimed at defining the descriptors, determining the sample evaluation procedures, and establishing the definitive scorecard. In the following sessions, each assessor evaluated the intensity of the nine previously selected attributes in separate booths on five different samples. At the end of each of these sessions the panel leader and the assessors discussed the individual results obtained in order to establish consensus criteria for evaluation.

Final evaluation of the descriptive analysis of the five samples was carried out in triplicate with each assessor evaluated all samples. Three different sessions were performed, during three consecutive days. Discriminating capability and repeatability using the data collected during the training sessions were used of the panelists' evaluation (Morais, Cruz, Faria, & Bolini, 2014). The reference sample together with the scorecard was presented at the beginning of each session. This process allowed the panelists to create the appropriate context for each scale. The reference sample was removed before sample evaluation. The panelists were given 30 mL of each sample in disposable cups coded with three-digit numbers in the temperature of 4 ± 1 °C. For each sample, odor attributes were evaluated first. Then, assessors were asked to evaluate visual texture, flavor, and finally, in mouth textural attributes. To reduce the influence of serving order, the samples evaluated in each session were served according to a balanced design (MacFie, Bratchell, Greenhoff, & Vallis, 1989).

2.3. Statistical analysis

Two-way analysis of variance (ANOVA) (samples and assessors) with interaction was applied to the sensory data obtained for each attribute. Individual differences among assessors were analyzed by a fixed model, considering samples as fixed factor. When a significant interaction between assessors and sample was observed for a descriptor, a mixed model ANOVA was performed, considering samples as fixed effect and assessors as random effect (Bayarri, Carbonell, Barrios, & Costell, 2011; González-Tomás, Bayarri, & Costell, 2009). F_{sample} values were then recalculated taking the average square of the interaction as denominator. Least significant differences (LSD) between samples were determined by Fisher test ($\alpha = 0.05$). Descriptive measures (mean and standard deviation values) were provided for each sensory attribute (Granato, Calado, & Javis, 2014), with the latter being calculated using the results obtained in the three sessions performed in the final evaluation of the samples.

Chemometric methods were also used to data treatment. Principal component analysis (PCA) was also applied to the mean values of attribute intensity (Cruz et al., 2013a). Additionally, performed hierarchical cluster analysis (HCA) was also applied to the first two dimensions obtained in PCA. This analysis was performed on a 5 × 9 matrix, which lines the samples 5 samples and the columns 9 sensory descriptors as will be shown later. All calculations were performed with the software version XLSTAT for Windows 2012.5 (Addinsoft, Paris, France). As the final evaluation of the samples in QDA was performed three times, each result was considered as independent trial. Furthermore, three PCA and HCA were generated showing three independent maps, showing the graphical position of the samples and the sensory descriptors associated to them. These figures were used to check the stability of the technique, and in this sense, this procedure constituted a way to validate the results of the methodology.

3. Results and discussion

Table 3 shows the results obtained by a fixed model of two-way ANOVA with interaction, considering sources of variation samples and assessors applied to all sensory descriptors raised by the sensory panel for the QDA. The panelists reported the existence of nine sensory