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Monitoring of mild heat treatment of camel milk by front-face fluorescence spectroscopy

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ABSTRACT

The potentiality of the front-face fluorescence spectroscopy coupled with chemometric tools to characterize changes in camel milk following thermal treatments in the 55–75 °C temperature range from 0.5 min up to 30 min was studied. Nicotinamide adenine dinucleotide (NADH), fluorescent Maillard reaction products (FMRP) and vitamin A fluorescence spectra were collected on camel milk samples. Using principal component analysis (PCA), the vitamin A spectra allowed some discrimination between milk samples according to heat treatment intensity and holding time. The best results was obtained by applying common components and specific weights analysis (CCSWA) to the three spectral data sets, since a clear differentiation between camel milk samples preheated at 70 and 75 °C from the others was achieved. Apparent activation energy of camel milk determined from NADH, FMRP and vitamin A spectra was of 85.1, 84.2 and 47.3 kJ mol^{-1}, respectively. The spectral patterns of the CCSWA model enabled us to get information about the molecular changes occurring in camel milk fluorophores during heat treatment.

1. Introduction

Milk plays an important role in the human diet thanks to its balanced composition insuring the essential nutriments including proteins, carbohydrates, fats, vitamins, and so on (Haug, Hostmark, & Harstad, 2007). Camel milk represents one of the main sources of nutrients in many parts of the world, especially, in the arid and semi-arid areas (Al-haj & Al-kanhal, 2010). From a nutritional point of view, it has been reported that the level of vitamin C in camel milk is three to five times higher than that in bovine milk (Al-haj & Al-kanhal, 2010). Additionally, camel milk has many therapeutic properties since it has been used as anti-carcinogenic (Magjeed, 2005) and anti-diabetic (Agrawal, Budania, Sharma, Gupta, & Kochar, 2007) agents.

Thermal processing of milk is considered as one of the essential steps of milk production. The purpose of milk heat treatment is to improve the quality of this biological fluid and prolong its shelf life by either partial destruction of microorganisms or complete sterilization of milk (Hattem, Manal, Hanna, & Elham, 2011; McKinnon, Yap, Augustin, & Hermar, 2009; Mohamed & El Zubeir, 2014). However, thermal treatment affect functional properties of milk proteins and induce changes in their physical, chemical, and sensorial properties such as the degradation of lactose, denaturation of whey proteins, destruction of some vitamins and enzymes, hydrolysis of proteins and lipids, and so on (Donato & Guyomarc'h, 2009; Elagamy, 2000; Farah, 1986; Sakkas, Moutafi, Moschopoulou, & Moatsou, 2014).

In the literature, various conventional methods has been employed to monitor the quality of heated milk such as physico-chemical, chromatographic, microbiological, and sensory measurements (Christensen & Reineccius, 1992; Clare et al., 2005; Elagamy, 2000; Elliott, Dhakal, Datta, & Deeth, 2003; Hattem et al., 2011; Mayer, Raba, Meier, & Schmid, 2010; Sakkas et al., 2014; Ul Haq et al., 2013, 2014). Although the above mentioned techniques are considered as reference ones, they are destructive, costly, time-consuming, and require sophisticated analytical equipment and skilled operators (Dufour & Riaublanc, 1997). Thereby, more attention has been paid to the development of non-invasive and non-destructive techniques to be used as rapid

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screening tools for the evaluation of the quality of milk (Kamal & Karoui, 2015). Front-face fluorescence spectroscopy (FFFS) is a rapid method, relatively inexpensive, and gives a great deal of information with only one test. This technique is known to be sensitive and non-destructive, and enable to provide information on the presence of fluorescent molecules and their environment in all types of biological samples containing double conjugated bonds (Christensen, Becker, & Frederiksen, 2005; Karoui et al., 2005b; Karoui, Dufour, Schoonheydt, & De Baerdemaeker, 2007a). In dairy products, several intrinsic fluorescent components including tryptophan, vitamin A, aromatic amino acids and nucleic acids, fluorescent Maillard reaction products (FMRP), NADH, and so on were found to be sensitive to their environment (Andersen & Mortensen, 2008; Dufour & Riaublanc, 1997; Strasburg & Ludescher, 1995).

The application of FFFS in the research fields of dairy products is usually related with the use of multivariate statistical analyses. For example, this technique coupled with chemometric tools has been used to: i) monitor the structural changes occurring in milk proteins and their physicochemical environment during milk coagulation and heat treatment (Blecker, Habib-Jiwan, & Karoui, 2012; Boubellouta & Dufour, 2008; Dufour & Riaublanc, 1997; Hougaard, Lawaetz, & Ipsen, 2013; Kulmyrzaev, Levieux, & Dufour, 2005); ii) determine the quality of different varieties of cheeses during ripening (Karoui, Dufour, & De Baerdemaeker, 2007b; and iii) authenticate milk (Karoui, Martin, & Dufour, 2005a) and Emmental cheeses according to their geographic origin (Botosoa & Karoui, 2013).

Compared to other milk types, camel milk is known to have high biological value: absence of β-lactoglobulin, high percentage of vitamin C which is three to five times higher than that of cow's milk, high contents of anti-microbiologic agents, due to the presence of lysozyme, lactoperoxydase, lactoferrin, immunoglobulin, and bacteriocins produced by lactic acid bacteria (Elagamy, Nawar, Shamsia, Awad, & Haenlein, 2009).¶All these characteristics contributed to the stability of camel milk, since its stability is maintained for 5 days at 30 °C, while cow's milk is completely destabilized after 48 h at the same temperature.

At our best knowledge, only limited studies were carried on the effect of heat treatment of camel milk constituents. For example, Elagmy (2000) compared by using electrophoresis method, the impact of heat treatment in the 65–100 °C range for 10, 20 and 30 min on the quality of whey proteins, similar to the study conducted by Farah (1986). Recently, Mohamed and El Zubeir (2014) determined the impact of two heat treatments (63 °C and 72 °C for 30 min and 15 s, respectively) on the microbiological quality of camel milk and reported that heat treatment improves the microbial quality and extends the shelf life of camel milk.

No data are present in the literature on the effect of camel milk heat treatment on its structure. Thus, the objective of this study was to explore the potential of FFFS combined with multivariate statistical methods to characterize changes occurring in milk by the application of mild heat treatment (55–75 °C temperature range from 0.5 min up to 30 min).

2. Materials and methods

2.1. Milk samples

Fresh camel milk (2 L distributed into plastic bottles of 30 mL capacity) was obtained from an experimental station located in the center of Tunisia (ElJem region in Mahdia governorate). Camels of Maghrebine genotype, aged of 6 years were maintained on pasture feeding (yearly pasture of thorny plants and coquelicot) and supplemented with concentrate. The animals were inspected by a qualified shepherd on a daily basis, and routine animal care and vaccination procedures were conducted as prescribed by best practice protocols.

Fresh cow milk of Holstein Friesian genotype was collected from a regional farm of Lille (France). Cows were maintained on pasture feeding and supplemented with corn-based concentrate. Once arrival to the laboratory, milk samples were kept at −18 °C until analysis.

2.2. Samples preparation

Milk samples were thawed during 12 h at 4 °C and then kept at room temperature (~18 °C) for 10 min to avoid thermal shock. Series of milk samples were prepared by heating raw milk in a water-bath (Haake A25 AC 200 temperature controller, Thermo-Scientific. France) preset at 55, 60, 65, 70 and 75 °C for 0.5, 1, 5, 10 and 30 min at each temperature considered. Heated milk samples were cooled in ice water bath for 5 min and left at room temperature for 15 min. Raw milk was also studied.

2.3. Fluorescence spectroscopy

Fluorescence spectra were recorded using a Fluoromax-4 spectrofluorometer (Jobin Yvon, Horiba, NJ, USA). The incidence angle of the excitation radiation was set at 60° to ensure that reflected light, scattered radiation, and depolarization phenomena were minimized. The spectrofluorometer was equipped with a thermostated cell and the temperature was controlled by a Haake A25 AC 200 temperature controller (Thermo-Scientific. France). Milk samples were placed in 3 ml quartz cuvette and fluorescence spectra were recorded at 20 °C. The emission spectra of NADH (360–600 nm) and FMRP (380–680 nm) were recorded with the excitation wavelengths set at 340 and 360 nm, respectively. The excitation spectra of the vitamin A (252–390 nm) were scanned with the emission wavelength set at 410 nm. For each sample, 2 spectra were acquired.

2.4. Mathematical analysis of data

In order to reduce scattering effects and to compare samples, fluorescence spectra were normalized by reducing the area under each spectrum to a value of 1 according to others (Karoui, Bosset, Mazerolles, Kulmyrzaev, & Dufour, 2005c; Karoui et al., 2007a; Leriche et al., 2004). Specifically, the shift of the peak maximum and the peak width changes in the spectra were considered. PCA was applied to the normalized spectra to investigate differences between milk samples. The PCA transforms the original variables into new axes, or principal components (PCs). This statistical multivariate treatment was earlier used to observe similarities among different samples (Karoui & Dufour, 2003), reducing the dimension to two or three PCs, while keeping most of the original information found in the data sets.

In a second step, common components and specific weight analysis (CCSWA) was applied to the whole data sets. The objective of this technique is to describe several data sets observed for the same samples. The CCSWA takes into account the maximum inertia (total variance) of the data sets (NADH, FMRP and vitamin A spectra). It consists of determining a common space of representation for all the data sets. Each table (NADH, FMRP and vitamin A spectra) has a specific weight associated with each dimension for this common space. A large difference between the values of the specific weights for a given dimension would express the fact that this dimension reveals physical phenomena, which is visible by one method and not by the others. The CCSWA deals with analysis of co-inertia that is the total variance in data sets and enable the overall