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Review

Spectral-sensitive Pulsed Photometry to predict the fat content of commercialized milk

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ABSTRACT

Dairy industry has a strong interest in quick and inexpensive techniques able to estimate milk fat content both in off and on-line processes. In the present study, a simple and inexpensive optical technique is preliminary described, discussed, and then tested for the prediction of this parameter. It's essentially based on the different intensity and spectral emission of a tungsten lamp during its progressive lighting combined with the photodiode wavelength sensitivity. Measurements were carried out at 22 °C on thirteen samples of commercialized milk (and mixture of them) (fat content ranging from 0.05% to 3.8%). The influence of the milk temperature on the optical behaviour was also investigated. The detected voltage waveform was strictly and not-linearly correlated with the fat content (R^2 up to 0.985) and predictions with R^2 up to 0.997 were obtained by using artificial neural networks (ANN). Milk temperature differently influenced the measurements for milks with various fat content.

Contents

1. Introduction

Milk is a popular nutritional food with a world consumption exceeding 700 million tons per year (Bogomolov and Melenteva, 2013). The composition of milk is an important factor in dairy industry, for quality control, consumer information and for determining the economic value of the product (Iñon et al., 2004). Among milk components (fat, protein, lactose, calcium), the fat content is recognised as a main direct indicator of milk quality. So, fast, at a reasonable price, and accurate methods for the quantification of this component are needed (Lin et al., 2014).

The traditional chemical milk fat detection methods (Gerber,

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Rose-Gottlied, Babcock and Tesa methods) achieve higher accuracy but are labour and time consuming. Particularly, the official Gerber method, as a consequence of the complicated handling and inevitable usage of toxic corrosive chemicals, such as the sulphuric acid, is a dangerous technique not suitable for non-trained hands.

Optical spectroscopy is the most common technique for the industrial quality control (Bogomolov et al., 2012). Middle (MIR) and near (NIR) infrared spectroscopy were intensively investigated to monitoring the milk composition (Aernouts et al., 2011; Soyeurt et al., 2010; Kawasaki et al., 2008; Kalinin et al., 2008; Iñon et al., 2004; Luginbühl, 2002; Chen, 2002; Tsenkova et al., 2000, 1999). MIR spectroscopy evolved into a standard AOAC method (Biggs, 1972) but, in order to obtain a good accuracy of the analysis, it requires an intensive sample preparation, a periodic instrument calibration, and a sophisticated automation. As a consequence, this technique is hardly adaptable for in-line process evaluation. NIR spectroscopy is considered a feasible alternative to MIR for the in-line monitoring, but the price to paid is a lower accuracy, sensitivity and selectivity (Bogomolov and Melenteva, 2013).

In the visible light (Vis) region, where the light scatter phenomenon is dominant, turbidimetric methods based on the correlation between fat content and light dispersed from homogenised milk samples, were assessed at individual wavelengths (Walstra et al., 2006; Nakai and Le, 1970; Ashworth, 1969). The light scattering is influenced by the number and size distribution of fat milk particles; as a consequence, the turbidimetric analysis in the Vis region is not suitable for the evaluation of non-homogenised sample milk (raw milk). Nevertheless, Bogomolov and Melenteva (2012) recently suggested a quantitative determination of homogenized milk fat based on visible light scattering. Particularly, a method combining the Vis-NIR spectroscopy (up to 1000 nm) with the multivariate statistical analysis was developed with good determination coefficients between fat content and spectral signal (R^2 up to 0.973).

Kucheryavskiy et al. (2014) proposed an interesting method to determine the fat and total protein contents in raw milk using conventional digital imaging. However, as it could be expected, the fat prediction accuracy in raw milk was influenced by a significant variability of composition and particle sizes. The reported results are worse than those obtained by a physically similar scatter-based method using ViS-NIR spectroscopy.

Other alternative methods have been proposed to measure fat in milk. Phillips et al. (1995) investigated a possible correlation between milk fat content and colour, showing that the Hunter colour depends on fat content. By using a method based on thermal conductivity measurements and a specific probe, it was possible to assess the milk fat content with a sensitivity better than 0.1%, in about 1 s (Gustavsoon and Gustafsson, 2006). Moreover, Mabrook and Petty, 2003 reported that the milk electrical conductance decreases with increasing the fat content. The milk complex permittivity has not proven to be reliable and useful to determine the milk fat content by Nunes et al. (2006). A first fluorescent sensor based on the BODIPY compound showed a remarkable linear signal increment with increasing concentration of fat in commercial milks (Xu et al., 2014). The disadvantage of this method lies in the fact that it is not a chemical-free determination and it cannot be applied on-line. The fat content of raw and pasteurized milk samples was estimated (R^2 up to 0.99 with six samples) by using microwave and convective drying methods by Lakatos et al. (2010).

The present study refers about the setting up of a simple and inexpensive prototype able to assess the fat content of commercialized milks by exploiting the optical properties of milk, such as light scattering according to the Mie theory (only the big molecules as fat can cause light scattering while the other particles can be substantially ignored) (Xin et al., 2006). The proposed technique deeply differs from the traditional optical spectroscopy, although it maintains a sensitivity to the wavelength of the radiation. The obtained data were analysed by using classical statistics and artificial neural networks (ANN).

2. Materials and methods

2.1. The technique

The technique is based on the optical response of a material under test to a source of radiation (variable during time in intensity and spectral composition) such as a miniature tungsten filament bulb lamp. The light emerging from the material can be detected by a photodiode characterized by a sensitivity depending on wavelengths. The current coming from the photodiode depends on the response to the incident spectral components of the light. When the lamp is progressively lighted, in a short time (e.g 1 s), the output current pulse takes a characteristic shape (peak, amplitude, curvature) as function of interposed medium. By using photodiodes with different spectral sensitivities, it is possible to evaluate various optical phenomena such as molecular vibrational absorbance or, more simply, the light scattering.

The technique could be named *Spectral-Sensitive Pulsed Photometry*, and differs much from the conventional spectroscopy, in which the absorption is measured at different, known and selected wavelengths.

2.2. The prototype and acquisition setting up

The prototype was characterised by a miniature bulb lamp with a lent to focalize the radiation (rating voltage of 3.8 V and power of 0.3 W), a sample holder (a glass cuvette with a path of 10 mm) and a photodiode (Fig. 1). The distance between lamp and photodiode was 13.5 mm. The photodiode (OPTEK Technology Inc., model OP993) mainly operates in the NIR-B range with a peak sensitivity at around 890 nm. Its spectral sensitivity is reported in Fig. 2.

The progressive lighting of the lamp was controlled by Arduino® Uno Rev. 3 board, programmed by the Arduino integrated development environment software, and by using one of its pulsed with modulation (PWM) output pins (high frequency square pulses, e.g. 490 Hz, constant voltage, increasing pulse duration). The voltage applied to it increases during lighting (up to 1.2 V), as a consequence of the load resistance variation of the current amplifier, due to the incandescence of the filament. The duty cycle is the time during which the voltage is high compared to the time when it is low and increased from 0% to 69.0% in 4.63 s. The maximum light of the lamp (duty cycle = 69.0%) was chosen in order to obtain a filament weakly radiating with an relative abundant emission in

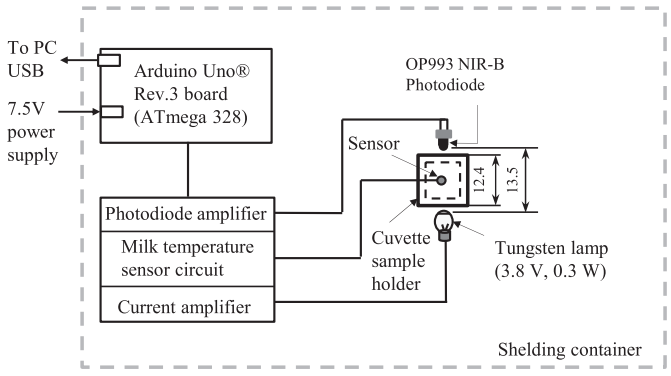

Fig. 1. Layout of the photometer prototype.