A method for testing the quality of milk using optical capillaries

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Abstract—The milk quality is determined by its visual appearance, absence of adulterating substances and ability to meet specific quality standards for somatic cell count (SCC), and bacteria count. There exist several diagnostic tests of milk quality. Some of them are applicable on dairy farms, like ,for example, the California Mastitis Test (CMT) and the Milk Conductivity Test (MCT). Other tests, such as the bulk milk bacterial count, the bulk tank somatic cell count and tests for adulterants like water, sediments or antibiotics, are used in laboratories. The knowledge required to successfully apply the existing milk quality tests can be rather extensive and pertains both to the methodology and the diagnostic capabilities of a given test. Therefore, there is a need for new simple and low-cost methods of milk quality testing. This paper presents a new method of milk quality classification using low-cost optical capillaries. In this method, milk quality is determined by observation of milk behaviour under specific heating conditions using a simple low-cost photonic system with optical capillaries. We show that the optical capillary is a suitable tool for analysing liquids showing high scattering of light, such as milk.

This high quality milk should be white in appearance, have no objectionable odors and be free of abnormal substances such as pesticides, added water or antibiotic and antiseptic residues. Normal milk from high producing Holstein or Friesian dairy cows is composed of water (87%), fat (3.8%), proteins (3.4% - of which 3/4 is casein), sugars (i.e., lactose, 4.5%) and other solids such as minerals (1.3%). Milk also contains a quantity of minor components, including sloughed somatic cells. Somatic cells are composed of white blood cells (WBC) and occasional sloughed epithelial cells. Most somatic cells found in normal bovine milk are a type of macrophages that function as early warning signals when bacteria invade the udder. Therefore, the milk quality goes down when the somatic cells start appearing and the usability of milk deteriorates when the bacteria turn up as well. In most developed countries milk quality at the dairy is defined by the somatic cell count and the bacterial count by a method called standard plate count.

Throughout the world, official regulatory standards for milk quality are based on determination of bacterial numbers present in raw milk and milk products, for example pasteurized milk. The SPC is the official reference method to specify the grade of Pasteurized Milk Ordinance (PMO) [1]. The PMO requires the SPC to be less than 100,000 cfu/ml for Grade A farms; grade B milk regulations require the SPC to be less than 300,000 cfu/ml. The SPC is an overall measure of milk quality but a single SPC value is not very useful diagnostically [2]. A high SPC is an indication of the presence of milk quality problems, usually caused by unsatisfactory refrigeration of milk or by poor cleanliness of the milking equipment.

It is very interesting that milk contains less water than most fruits and vegetables. Milk can be described as: an oil-in-water emulsion with the globules of fat dispersed in a continuous serum phase, a colloid suspension of casein micelles, globular proteins and lipoprotein particles or as a solution of lactose, soluble proteins, minerals, vitamins and other components. Skim milk of natural pH behaves as a Newtonian fluid; the parameters influencing its viscosity are the concentration of solids, temperature and heat treatment [3]. Because of high concentration and frequent interactions the casein contributes largely to the viscosity of skim milk. Most, but not all, of the casein proteins exist in a colloidal particle known as the casein micelle. In normal conditions the casein micelles have spherical shape. The casein micelles have diameters from 40 to 300nm and harbor the main part of casein in milk. The number of such particles in a milliliter of milk is in the range from 10^{14} to 10^{16}. They form a total surface of 5×10^4 cm^2/ml [4]. They are considered colloids in aqueous solute and they are stabilized from aggregating by steric and electrostatic stabilization due to k-casein molecules situated on the surface of the micelles. Changes in the physicochemical properties of the casein micelles can be inducted by changes in pH, salt concentration, or temperature. What is interesting is the time effects on these factors. Unpasteurized and non-sterilized milk ferments at room temperature. Lactic acid bacteria such Lactobacillus, Lactococcus or Leuconostoc convert milk monosaccharides (simple sugars, such as fructose) to lactic acid and energy, according to the following formula:

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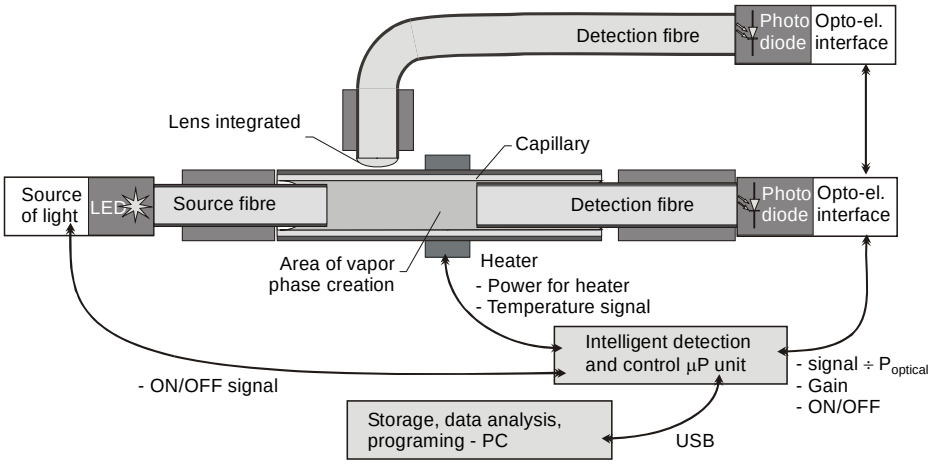

Fig. 1. Sensor configuration for the microlitre sample analysis of milk.

$C_6H_{12}O_6$ + lactic acid bacteria →

$$\rightarrow 2CH_3CHOHCOOH + 22.5 \text{ kcal}$$

The presence of the lactic acid lowers milk pH significantly. Low pH (pH of 4.6 at 20°C) leads to casein coagulation. Casein coagulation causes its aggregation and results in milk clot formation.

Optical properties provide the basis for many rapid, indirect methods of analysis such as proximate analysis by infrared absorbency or light scattering. Optical properties also determine the appearance of milk and milk products. Light scattering by fat globules and casein micelles causes milk to appear turbid and opaque. Light scattering occurs when the wavelength of light is near the same magnitude as the particle. Thus, smaller particles scatter light of shorter wavelengths. Skim milk appears slightly blue because casein micelles scatter the shorter wavelengths of visible light, more the blue ones than the red. The carotene precursor of vitamin A, ß-carotene, contained in milk fat, is responsible for the creamy color of milk. Riboflavin imparts a greenish color to whey. Refractive index (RI) is normally determined at 20° C with the D line of the sodium spectrum. The refractive index of milk is 1.3440 to 1.3485 and can be used to estimate total solids. Therefore, for spectrometric and light scattering setups milk is diluted. Moreover, fresh milk micelles have such sizes that scattering setups need sources and detectors that operate from $\lambda<300$ nm on, which is on the deep UV range boundary and covers the VIS range [5]. Such instrumentation is relatively costly. Classical test methods require a sample volume in the range of milliliters. When the sample volume is significantly lower, the opaque liquid sample can become transparent.

Our set-up is capable of analyzing a microlitre liquid sample examination. For the sampling purpose we used an optical capillary as a test tube [6]. Having in mind the influence of temperature on the structure of milk, we introduced local heating to the capillary [7]. Because milk shows significant light scattering we constructed our setup with a very short optical path length [8] and equipped it with nephelometric and turbidimetric arms (Fig. 1).

The opto-electronic subsystem of our set-up comprised a light source, fibre optical link, sensing head, and two photo-detection units controlled by an intelligent detection-and-control system built on the basis of the Atmel Co. AVR microprocessor. The set-up included a computer for data acquisition and storage. An in-house developed software package was used for internal data exchange and control. The measuring head comprised a few-centimeter-long section of a capillary with an inner diameter equal to 1 mm, and two pieces of HCS 400/430 hard clad silica fibre with removed coatings to function as the source and the turbidimetric arm. A piece of PFU-CD751-22-E PMMA fiber was used for the nephelometric arm with a local heater. The local heater was a wire coil 3 mm wide and of 1.3 mm inner diameter. The distance between the source and the transmission arm fibers is 7 mm. The nephelometric fibre position was self adjusting versus the capillary wall made on the head bed. This fibre was equipped with a thermally formed lens on its tip with a radius of 3 mm. The distance between the lens and the capillary was 1 mm. For the light source we used a super-bright white LED which we integrated with an opto-electronical optical power stabilization circuit. The photo-detection unit we constructed with an integrated photodiode-amplifier circuit from Burr-Brown with integrated band-pass filters. The light was modulated

with the frequency of 1 kHz.

We used a manual three-step procedure of milk sampling: drawing the milk sample into the capillary, placing the capillary in the measuring head and then inserting the source and transmission fibre. When the initial magnitude of signal was established, the time series were recorded for increased heater currents. We assume that for such small volume and current increases, the temperature inside the capillary has a uniform distribution which, due to the conduction, influences the region observed by the nephelometric arm of the head. We examined raw milk that was stored at two different temperatures 10° C (samples 1, 2 and 3) and 21° C (samples 4, 5 and 6). The recorded results for 10° C storage temperature and three different periods of storage equal 48, 49 and 50 hours respectively are presented in Fig. 2.

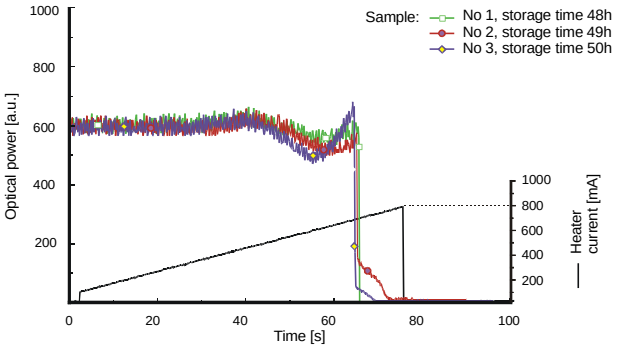

Fig. 2. Optical signal received during local heating from nephelometric arm for milk stored for 48, 49, 50 hrs at 10° C.

It can be seen that the optical signal is stable up to a certain temperature, when the amplitude forms a dimple. The dimple parameters can be correlated to the time of storage; its depth increases with the storage time. The signal for milk which underwent storage at conditions leading to its deterioration (21° C, 48 hrs) is presented in Fig. 3. The dimple is now significantly greater.

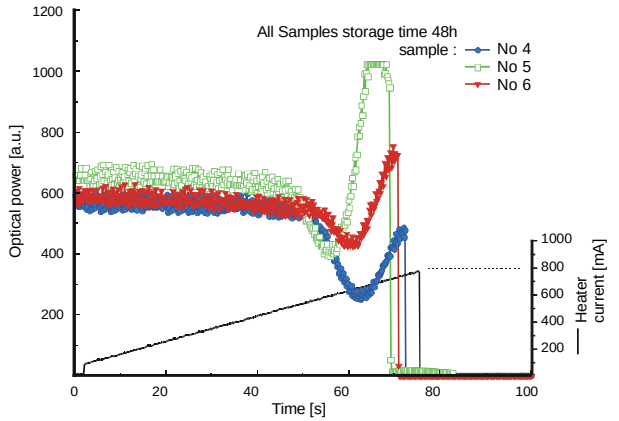

Fig. 3. Optical signal received during local heating from nephelometric arm for milk stored for 48 hrs, at 21° C.

What is very significant is that the dimple is not present for the same storage condition when pasteurized milk is under examination.

The proposed new method for testing the quality of milk using optical capillaries shows promising results by distinguishing between good and poor quality milk resulting from storage conditions. It is possible to show the dependencies of dimple parameters in the signal observed and bacteria presence in the milk. The method using optical capillaries and time series analysis can lead to construction of simple tools usable in real time milk quality classification.

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