Determination of solids and fat contents in bovine milk using a phase-locked resonant cavity sensor

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Abstract—**Monitoring the milk quality is of fundamental importance in the food industry since milk is one of the most common product in a daily diet. The control of milk composition and detection of adulteration in raw materials are crucial in order to assure the quality of dairy based products. Different technologies for the monitoring of milk constituents in substitution of chemical processes have been reported. Recent studies showed that microwave sensors have a good potential for measuring the milk moisture content. Here, we proposed a measurement system using a resonant cavity sensor for the determination of total solids and fat contents. Its main advantages comprises non-invasive and non-destructive characteristics and fast response. Additionally, the geometry of the cavity is adapted for easy deployment in industrial facilities. We used nine characterized milk samples in order to demonstrate the feasibility of the system. The results showed a good correlation between the system response and the volume fractions of total solids and fat constituents, proving that the system is a useful platform for the analysis of milk quality.**

I. INTRODUCTION

Milk is one of the most common food present in daily diet of many cultures, being recommended in most dietary guidelines around the world. Furthermore, it is considered as a rich source of essential amino acids for human nutrition, and its protein may have good effects on growth and recovery from undernutrition [1], [2]. Bovine milk contributes with 85% of global production [3]. According to the most recent report of the Food and Agriculture Organization of the United Nations [4], world milk production in 2013 was 769 million tonnes. Within this scenario, Brazil is the major milk producer in Latin America and the fourth in the world, with 34.3 million tonnes annually [5], [6]. Because of the dairy sector importance in the economy as a whole, the constant monitoring of the milk quality becomes imperative in industrial manufacturing facilities for characterizing the different types of milk, and also to identify possible adulteration. Additionally, it turns necessary to keep an effective control in the selection of raw materials for the production of certain dairy products, as for example, specific types of cheeses, offering authentic products for the consumers [7].

One of the biggest obstacles in proper determination of milk quality arises from a wide diversity among various milk samples. Bovine milk is a complex multi-component liquid whose total solids, fat droplets and other components are immersed or dissolved in an aqueous background to form an intricate substance. The constituents of milk exhibit widely differing characteristic lengths, leading to wide spatial scaling [8]. The determination of milk constituents are generally made through laboratory analysis by means of different chemical processes. As reported in [9], the routine chemical approaches used are costly and time consuming, require skilled labor, suffer from large amounts of chemical reagent, demand high water consumption, and generates high waste liquid emissions.

Recently, some alternative technologies have been applied for monitoring the milk quality and detecting adulteration. In [10], a methodology based on electrical impedance measurement is presented for the detection of bovine milk adulteration, specially due to the addition of water. The developed dedicated system allows to perform in-situ measurements. Milk adulteration is also investigated using a near-infrared diffuse reflection based methodology [11], where the system response is monitored while gradually adding water to the samples, validating the feasibility of using this process in substitution of cryoscope-based laboratory tests. In [12], an optofluidic microviscometer is implemented in order to investigate its potential employment in milk adulteration analysis by means of the addition of water, flour, starch, and urea. A sensor based on microwave absorption is explored in [13] for determination of moisture content in milk. The results showed a good correlation between the total solids content of various milk samples with the normalized signal strength. The main disadvantage of this technique is the invasive way of the measurements. In [14] the feasibility of using a resonant cavity sensor (RCS) for detection of different milk compositions is studied. They used S-parameters information in order to estimate the fat and protein content of three different kinds of milk (whole, semi-skimmed, and skimmed). It is also observed the sensor capability for detecting spoiled milk samples. The results proved to be useful for the implementation of real-time measurement systems based on resonant cavity sensors.

All the previous sensors are well adapted for in-situ measurements, but in a laboratory scale. A complete real-time system for online milk quality assessment in an industrial facility

is described in [15]. The system is based on near-infrared spectroscopic sensing and it is used for monitoring various milk constituents, such as, fat, protein, and lactose contents during milking by a milking robot with good precision and accuracy. Despite the fact that this technology presents good results it can be very expensive, as stated in [14].

In this work, the feasibility of using a resonant cavity sensor for online monitoring of moisture, total solids and fat contents is investigated. A complete measurement system is proposed, which comprises a controller, an RCS operating around 200 MHz, and a signal acquisition and processing circuit. This circuit consists of off-the-shelf radio frequency (RF) components and is based on a phase-locked loop (PLL). Nine milk samples were used in order to validate the measurements, whose physicochemical characteristics were previously analyzed following a rigorous methodology. Preliminary results show a linear stable response of the sensor with a good sensitivity, proving that the system is very promising for real-time monitoring of milk quality at industrial scale.

II. MATERIALS AND METHODS

A. Physicochemical analysis and sample preparation

Physicochemical analyses on three different milk samples (whole - W, semi-skimmed - SS, and skimmed - S) were carried out, in triplicate, according to the methodologies described by Association of Analytical Chemistry (AOAC), using analytical grade chemicals. In order to ensure the quality of the samples, their density, acidity and pH values were determined. The milk density ($g\,cm^{-3}$) was measured with a Thermo Lacto-Densimeter (Quevenne Tip), while the titratable acidity (lactic acid g/100g), with 0.1 mol equival/L NaOH, and the pH values were measured using a pH meter (MP220, Metler-Toledo) by the potentiometric method. Additionally, the total solids and fat contents were determined. Total solids content, (g/100g), was determined from 5 g samples by drying to constant weight at $105\,°C$, while fat content, (g/100g), was analyzed by Gerber method [16].

From the three main characterized samples (W, SS, and S) we prepared six additional (A1, A2, A3, B1, B2, and B3) by successive mixing. A test tube was used for measuring the contents of each sample in order to fill the total available volume inside the RCS (2000 mL). The temperature of each sample was measured with a mercury thermometer before each measurement. Table I lists the monitored temperatures and resultant compositions of each sample. The physicochemical composition of the last six samples were calculated by mass balance.

The obtained physicochemical data was expressed in terms of mean and standard deviation. It was possible to note that for all milk samples probed the density, titratable acidity and pH values (Table II) are in accordance with the official regulation established in Brazil [17]. Likewise, the total solids and fat contents are in consonance with the Brazilian Normative Instruction No. 62 [17], that establishes minimal values of 8.40 g/100g and 0.00 g/100g, respectively.

TABLE I
CHARACTERISTICS OF THE SAMPLES

Samples	Temperature [°C]	Composition [mL]
W	25	2000 W
SS	24.8	2000 SS
S	25	2000 S
A1	24.7	1000 W + 1000 SS
A2	24.5	1000 W + 1000 S
A3	24.5	1000 SS + 1000 S
B1	24.5	1000 A1 + 1000 A2
B2	24.5	1000 A1 + 1000 A3
B3	24.5	1000 A2 + 1000 A3

B. Measurement system setup

The RCS is a cylindrical cavity designed to resonate between 150 MHz and 250 MHz. Its geometry allows the easy deployment in an industrial facility. It is built of a 7.62 cm diameter PVC pipe contained inside a 12.7 cm diameter metallic pipe, both of 15 cm length. The resonance frequency of the sensor for the TE111 propagation mode is given by

$$f_o = \frac{k\ c}{\sqrt{\varepsilon_m}} \tag{1}$$

where k is a constant dependent on the geometry of the cavity, c is the speed of the light in vacuum, and ε_m is the effective relative permittivity of the milk flowing inside the RCS's PVC pipe [18].

According to (1) the resonance frequency of the RCS is inversely proportional to the square root of the effective relative permittivity of the milk flowing inside the sensor. Therefore, f_o will change as ε_m varies according to the moisture content of the milk [19].

The block diagram of the measurement system is shown in Fig. 1. It consists of a Phase Comparator, a Controller, a Voltage-Controlled Oscillator (VCO), and the RCS [20]. The VCO output signal crosses the RCS and the phase difference between its phase-shift φ_{sen} and the VCO signal phase φ_v is compared with the reference phase ϕ_{ref} in the Phase Comparator. The Phase Comparator output signal u_{er} is proportional to the phase error between ϕ_{ref} and $\varphi_{sen} - \varphi_v$ and it is applied to the input of the Controller. The controller adjusts the VCO frequency by the control signal u_c, which is proportional to the VCO oscillating frequency, in order to minimize the phase error over time. The VCO will then oscillate at the frequency where $\varphi_{sen} - \varphi_v = \phi_{ref}$. Hereby, the phase-locked system tracks the resonance frequency of the RCS as ε_m changes. The voltage level of the VCO control signal u_c is used as the measurement parameter and from now on we will mention it as the system response.

All measurements were done in a single day at the laboratory with a room temperature fixed at $25\,°C$. Fig. 2(a) shows the whole measurement setup consisting of the RCS attached to the RF circuit (detailed in Fig. 2(b)) and the computer with the software control. The test tube and the three main samples

| Samples	Density [g cm^{-3}]	Titratable acidity [g lactic acid/ 100g]	pH	Total solids [g/100g]	Fat [g/100g]
W	1.031 ± 0.000	0.131 ± 0.003	6.87 ± 0.02	11.50 ± 0.25	3.20 ± 0.00
SS	1.034 ± 0.000	0.128 ± 0.003	6.86 ± 0.01	9.95 ± 0.09	1.20 ± 0.00
S	1.035 ± 0.000	0.131 ± 0.003	6.83 ± 0.02	9.04 ± 0.13	0.10 ± 0.00
A1	1.033 ± 0.000	0.131 ± 0.003	6.81 ± 0.07	10.73 ± 0.15	2.20 ± 0.00
A2	1.033 ± 0.000	0.127 ± 0.003	6.78 ± 0.01	10.27 ± 0.18	1.65 ± 0.00
A3	1.035 ± 0.000	0.126 ± 0.005	6.79 ± 0.03	9.50 ± 0.08	0.65 ± 0.00
B1	1.033 ± 0.000	0.131 ± 0.003	6.80 ± 0.00	10.50 ± 0.16	1.93 ± 0.00
B2	1.034 ± 0.000	0.127 ± 0.003	6.84 ± 0.00	10.11 ± 0.11	1.43 ± 0.00
B3	1.034 ± 0.000	0.128 ± 0.002	6.90 ± 0.01	9.88 ± 0.13	1.15 ± 0.00
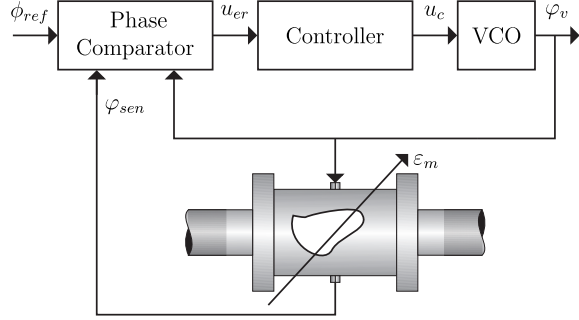
Fig. 1. The block diagram of the measurement system based on a phase-locked resonant cavity sensor.
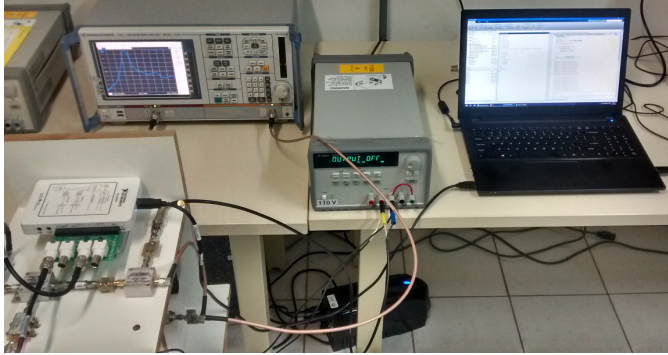
(a)

(b)

(c)

Fig. 2. (a) The whole measurement setup. (b) Detail of the RCS attached to the RF circuit. (c) The main samples (W, SS, and S) and the test tube used for accomplishing the additional samples.

used for preparing the additional samples are shown in Fig. 2(c).

III. RESULTS AND DISCUSSION

The correlations between the system response (u_c) and the total solids and fat contents are shown in Figs. 3 and 4, respectively. As we can deduce from Fig. 4, u_c increases as the fat content increases. This can be explained because the real part of the permittivity decreases as the milk moisture decreases [19], consequently raising the resonance frequency of the RCS, in agreement with (1).

We can also observe from Fig. 4 that the three main samples (W, SS, and S) are disposed near to the straight line, with W and S samples in the opposite extremes of the interval, and SS near to the middle. The rest of obtained samples are all within this interval. This proves that the measurement system may be roughly linear even for a small range for both total solids ([9.95 to 11.5] g/100g) and fat ([0.1 to 3.2] g/100g) contents. The sensitivity for the total solids and fat contents are approximately [0.075 and 0.095] g/100g mV, respectively. The discrepancies from the linear response may be explained from the standard deviation of the physicochemical composition analysis indicated in Table II. An additional source of error can be related to the preparation of the six mixed samples (A1 to B3) due to the test tube scale, which was not taken into account for mass balance calculus.

Based on Pearson's correlation coefficient and the p-values with 95% confidence intervals for the coefficient estimates, the system response u_c is highly correlated (P < 0.05) with total solids and fat contents. These results allow the prediction of the concentration of total solids and fat contents by monitoring u_c.

All the information about the correlation analysis are summarized in Table III.

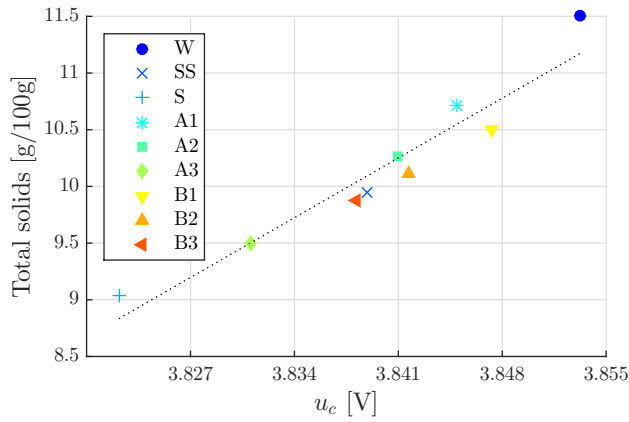| Constituent	Pearson's coefficient	p-value	Sensitivity [g/100g mV]	y-intercept [V]	R^2
Total solids	0.961	<0.001	0.0753	3.705	0.9227
Fat	0.959	<0.001	0.0950	3.824	0.9188

Fig. 3. The correlation between the total solids content and the system response signal u_c.
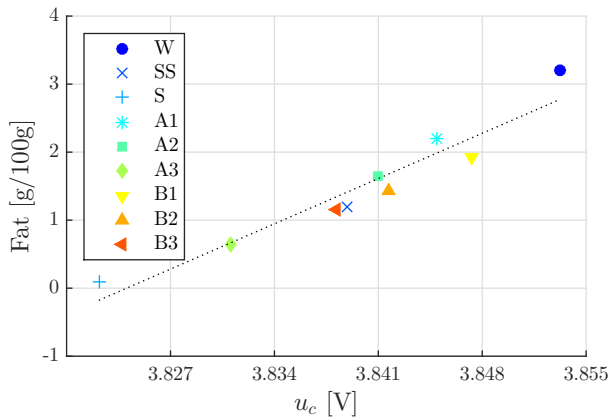

Fig. 4. The correlation between the fat content and the system response signal u_c.

IV. CONCLUSIONS

The measurement system using a phase-locked resonant cavity sensor presented in this work may be effectively used for determining the concentration of total solids and/or fat constituents in milk. Measurements were performed using several samples from mixtures of different brands and types (whole, semi-skimmed and skimmed) of commercially available milks. The samples physicochemical constituents were obtained following AOAC methodology, verifying that they were in consonance with official regulation in Brazil. A high correlation and a linear dependence between the system response and the volume fraction of total solids and fat were found. Furthermore, the sensor's geometry was designed for compatibility with milking pipeline at industrial facilities. As a result, the complete system proves very attractive for online non-invasive and non-destructive monitoring of milk composition, providing a faster response and simpler installation compared to other reported techniques. Moreover, these encouraging results should allow us to explore other similar applications such as detection of milk adulteration used as raw material in derived dairy products.

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