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A low cost instrumentation system to analyze different types of milk adulteration

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ABSTRACT

In this paper, the design of a complete instrumentation system to detect different types of milk adulteration has been reported. A simple to use indicator type readout device is reported which can be used by milk community people. A low cost microcontroller based automatic sensing system is also reported to detect 'synthetic milk', which has been reconstructed after adulterating the milk with 'liquid-whey'.

1. Introduction

Food quality monitoring is a serious issue and addressed by different researchers [1–4]. Among these milk is one of the most vulnerable food which is adulterated very easily. Adulteration achieved with low value ingredients like water and whey into natural milk is known as 'economic adulteration'. It is a very common practice by the milk community people to add water or 'liquid-whey' [5,6] to increase the volume, this in turn reduces the milk quality. Additionally whey retains many natural properties of milk, so preparation of synthetic milk from liquid-whey is simple and can camouflage the natural milk easily. Hence, this becomes a serious concern to the dairy firms who buy milk from thousands of different milk suppliers and need a simple, robust and bio-compatible automated sensing system for quality control.

There are several methods [7–9] available in the literature to detect different types of adulterants in milk. Methods to determine additional water present in the milk are explained in [10–12]. The volume of water mixed in milk is detected by electrical conductivity (EC) method. In this method [11], quantity of ions decreases so the resistance is higher and the EC is changed. To detect fat and water content present in milk, electrical admittance spectroscopy [7,10] method has been used. Another method used to determine the additional water content in milk is freezing point osmometry [12]. In [13–16], different methods are proposed for determination of whey as an adulterant. Low priced whey [14,16] and rennet whey solid [13,15] are often mixed with liquid milk and milk powder. To detect the fraudulent addition of rennet (enzymes for curdling of milk) whey solid in ultra high temperature (UHT) milk [13], capillary electrophoresis is used. Sometimes urea [17–21] is added to milk to increase the shelf life. In [17] a potentiometric biosensor method is reported to detect urea adulteration in milk. Measure of change of pH [19,21] is another method of urea detection in milk. Enzyme based piezoelectric sensor [18] is also used to detect urea in milk by measuring the pressure of the gas evolved in the milk sample. Addition of urea in milk is also detected by near infrared spectroscopy method [20]. But most of these methods are expensive and time consuming and need skilled manpower. It is, therefore, necessary to develop a new and easy to use instrumentation system for the rapid and reliable detection of this kind of fraud.

So the aim of this paper is to develop a low cost, user friendly instrumentation system for measurement of these kinds of adulteration and can be easily installed in dairy industry.

In this paper a constant phase element (CPE) sensor is used to detect the adulteration in milk. The sensor is a stick type two terminal device and when dipped inside a medium the phase

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angle between the two terminals remains constant (hence, the name constant phase element (CPE)). But if the property of the measuring medium is changed the phase angle also changes [22]. This means that the phase angle of the sensor will be different for plain milk and the milk with some impurity [9]. The change of phase angle is then detected with the help of a phase detector circuit [23]. Indicator LEDs are used to display the type of adulteration. An automatic detection system for synthetic milk is also developed and studied. The advantages of using such sensor are that the electrodes are coated with the poly-methylmetahacrylate(PMMA), which makes it bio-compatible and the milk property does not get changed when the sensor is dipped in. Moreover, it is a stick type rigid probe and can be easily dipped inside the measuring medium, an essential requirement for automatic detection.

The paper is organized as follows: Section 2 describes the building blocks of the instrumentation system which includes sensor design, signal processing block with the detector circuit and display unit. Section 3 presents the automatic detection system and conclusion is given in Section 4.

2. Design of the instrumentation system

Fig. 1 shows the block diagram of the developed instrumentation system to detect the adulteration of pure milk with different types of adulterants. It includes sensor, signal conditioning circuit and display circuit.

2.1. Development of the sensor

The sensor, shown in Fig. 1 [9], is a copper cladded printed circuit board, cut into a suitable dimensions and coated with a thin film of Polymethyl-methacrylate (PMMA) by using spin coating technique [24]. For determination of different adulteration of milk the sensor has to be dipped inside the test medium as it has been observed that its impedance changes with the interaction of the sensor with the test medium [9]. In particular, the phase angle changes when milk sample is adulterated with different materials [9,24,25]. Hence in this measurement "change of phase angle" is considered as the sensor output. Moreover, this sensor has the property that for a particular measurement the phase angle remains constant over almost one decade of frequency which is an essential requirement for sensing system where change of signal frequency for interference or other effect will not hamper the measurement.

The sensor with coating thickness 13 μm, named as FOE13, is selected for designing the instrumentation system for detection of tap water and urea as an adulterant in pure milk in the frequency range 5–12 kHz. It will be worth to mention that the phase angle of the FOE13 sensor remains constant in this frequency range at a particular test medium. Other sensors with different film coating can be fabricated which gives constant phase angle in different frequency zones [9].

FOE13 sensor is considered for development of the instrumentation system because the circuit elements used are suitable for the frequency range 5–12 kHz. So, here for development of the instrumentation system, measurements are performed at 7 kHz which is almost at the middle of the bandwidth of the sensor. Hence the choice of frequency depends on the bandwidth of the circuit element and the range of frequency where phase angle remains constant. This value is chosen because due to environmental or other effect, if the frequency of the detector circuit shifts, the output of the sensor will remain the same.

2.1.1. Calibration

Before calibrating the instrumentation system, each block has to be characterized. For that purpose, the resolution, sensitivity, repeatability and reproducibility of the sensor block are checked with 7 kHz signal frequency.

The phase angle of FOE13 sensor is measured with LCR meter (Agilent Precision Impedance Analyser 4294A), after dipping 2 cm of the sensor separately in different test mediums. Four different buffer pH solutions (pH 2.0, pH 4.0, pH 7.0 and pH 9.2), pure milk, milk adulterated with different % of tap water and milk adulterated with different concentration of urea are the testing mediums. For measurement purpose, the frequency of the sinusoidal signal is varied from 100 Hz to 4 MHz with a peak to peak voltage of 1 V. The readings are repeated 5 times to check the repeatability and the average values are considered for analysis. It is observed that the deviation from its average values are negligibly small.

FOE13 sensor is characterized by standard buffer solutions of pH 2.0, pH 4.0, pH 7.0 and pH 9.2. The graph of phase angle with respect to pH value is shown in Fig. 2 and the average values are tabulated in Table 1. A line fit, $y = mx + c$ yields 'm', 'c' and fit factor

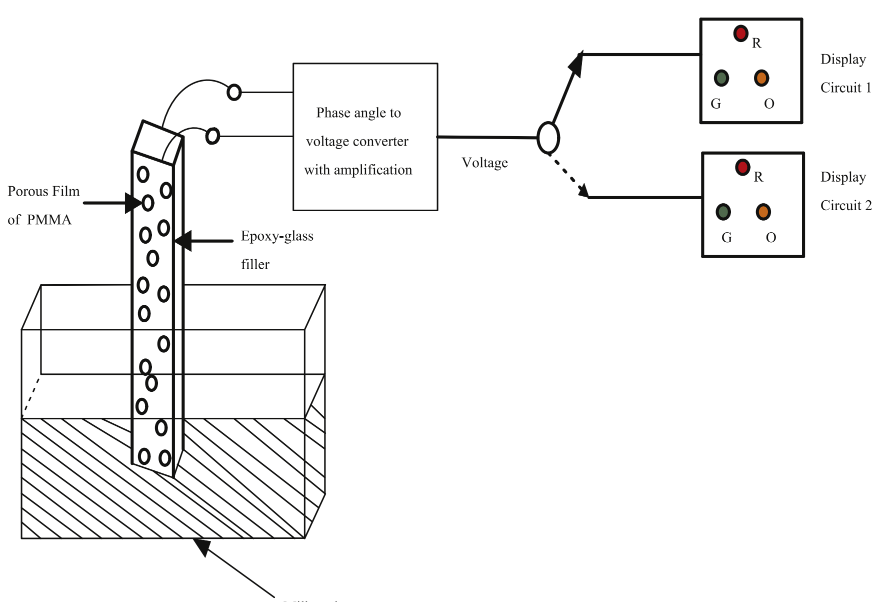

Fig. 1. Block diagram of the instrumentation system.