

Design of a Low-Cost ‘Constant Phase Angle’ Based Sensing System to Detect Natural Milk and ‘Synthetic-Milk’ Reconstructed from ‘Liquid-Whey’

Siuli Das, M. Sivaramakrishna, Bhaswati Goswami
Department of Instrumentation & Electronics Engineering,
Jadavpur University, Kolkata-700098, India
e-mail: bg@iee.jusl.ac.in

Karabi Biswas
Department of Electrical Engineering,
I.I.T Kharagpur, Kharagpur-721302, India
e-mail: karabi@ee.iitkgp.ernet.in

Abstract—In this work, a low cost automatic sensing system is proposed to detect ‘synthetic milk’, which has been reconstructed after adulterating the milk with ‘liquid-whey’. The constant-phase angle (CPA) based sensor detects the synthetic milk and a micro-controller based circuit drives a stepper motor to close the valve installed at the outlet of the milk supply line to prevent mixing. The sensor is stick type, rigid and hence it is easy to mount. The electrodes of the sensor are coated with polymethyl methacrylate (PMMA) film, which makes it bio-compatible and suitable for the application.

Keywords—constant-phase-angle; milk adulteration; liquid-whey; automatic sensing system; phase detector circuit.

I. INTRODUCTION

Milk adulteration is a century old problem [1, 2]. The oldest and simplest method of adulterating milk is by dilution with water to increase volume [3] and then to compensate specific gravity, different types of salt or sugar [4] are used. Sometimes the color change due to adulterants are corrected by the addition of a small amount of coloring matter [5] which may cause serious health problem [6].

A similar type of milk adulteration problem is reported in this work, where the volume is increased by addition of ‘liquid-whey’ (liquid by-product of cottage cheese) to increase the volume. The liquid-whey addition makes the natural milk a little acidic and lowers its pH value which is compensated by adding NaOH.

It is a well known fact that NaOH may cause health hazards to the patients suffering from heart disease and hypertension. It also deprives the body from utilizing lysine which is required for growing babies. Moreover, some greedy cheese maker uses cheap muriatic acid (a chemical composition of hydrochloric acid) to make cheese from milk, which causes more health problems.

The profit of using liquid-whey, as milk adulterants is double folded. First of all it is in abundance to the cheese maker, so cheap and easily available to the milk supplier. Secondly whey retains many natural properties of milk, so preparation of synthetic milk from liquid-whey is simple and can camouflage the natural milk easily. As a result adulteration of natural milk with liquid-whey became a wide spread fraudulent activity, where huge amount of cottage cheese is used everyday to make different varieties of sweets. 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 to their milk incoming line for quality control.

In this work a stick type probe, whose electrodes are coated with porous film of PMMA [7], is used as a sensor for developing the sensing system. The advantage is that it can be easily dipped inside the medium and at the same time

biocompatible due to the PMMA coating on the electrodes. More over, the detection is based on the principle of change of phase angle with the ionic property of the medium. The phase angle remains constant over a band of frequency, so measurement can be frequency independent, which is an important requirement for automated sensing system. The other advantages are - it is simple, easy to fabricate, cheap and can be replaced easily.

The paper is organized as follows: Section II describes the principle of operation of constant phase angle based sensor to detect milk adulteration. Section III provides fabrication and characterization of the CPA-based sensor. Section IV details the integrated sensing system with the detector circuit. Section V discusses the experimental observations and results. Section VI presents a conclusion.

II. PRINCIPLES OF CONSTANT PHASE ANGLE –BASED MEASUREMENT

In earlier works, the construction and working principle of the constant phase angle based sensor has been reported [8]. The impedance of the sensor (Fig. 2) dipped inside the medium can be represented as

$$Z(s) = Qs^{-\alpha} \quad (1)$$

where Z is the impedance, Q is a constant, α is a real number and s is the Laplace operator. So, magnitude $|Z| = Q\omega^{-\alpha}$ and phase angle $\theta = -\alpha\pi/2$, where θ is expressed in radians and independent of frequency. When $\alpha = 1$, $Z = Qs^{-1}$ and Z represents a capacitor. Similarly for $\alpha = 0$ and -1 , Z represents a resistance and inductance respectively.

For the above sensing system, it has been observed experimentally that the constant phase angle θ is a function of three parameters of the measurement arrangement and expressed as

$$\theta = f(A, t, \sigma) \quad (2)$$

where ‘ σ ’ is the property (e.g., ionic, dielectric) of the medium under test, ‘ A ’ is the area of contact of the probe with the test medium and ‘ t ’ is the thickness of PMMA-film coated on the electrodes [7, 9].

In this work, the change in constant phase angle θ for pure milk, adulterated milk and synthetic milk (reconstructed after adding liquid-whey) has been observed to identify synthetic milk. For a particular measurement, area of contact of the probe with liquid medium and the thickness of film coated on the electrodes are kept constant. However the effect of these two parameters needs to be studied for standardizing the sensor and the optimum area and thickness is to be chosen for measurement [10]. It has also been observed that this

constant phase angle changes with the change of physical property of the polarizing medium (e.g., ionic concentration). This property of the CPE can be used for sensing purpose- this means the phase angle of the CPE will be different for plain milk and the milk with some impurity.

It has been already mentioned that the phase angle remains constant over frequency which makes the measurement independent of the frequency. However the bandwidth is limited to 10 kHz to 20 kHz. In this report all the measurement is performed at 15 kHz, so that in automatic sensing, if due to environmental or other effect the frequency of the detector circuit shifts, it will not effect the measurement.

III. FABRICATION AND CHARACTERIZATION OF THE SENSOR

The principle of development of thin PMMA-film coating on the probe electrodes are similar as reported by the authors in earlier report [11]. The sensor used in present application is 6 cm long, 6 mm wide, cut from double sided copper clad PCB, generally used in electrical circuit fabrication. A thin film of PMMA is coated on the electrode surface using spin coating technique. The different film thickness can be achieved by changing concentration of the PMMA chloroform solution and the speed of the rotation of the spin coating machine. In the present study sensor with coating thickness of 18 μm is used.

The fabricated sensor is characterized by measuring impedance (Z) and phase angle θ with a LCR meter (Agilent Precision Impedance Analyser 4294A). The sinusoidal signal frequency is varied from 100 Hz to 4 MHz and peak to peak voltage is 1 V. The phase angle change is noted with standard buffer pH 4.0, pH 9.2 solutions, and also in pure milk. Each measurement is repeated five times and the average value of phase angle is plotted. It can be observed in Fig. 1, that the sensor gives a constant phase angle in the frequency range 10 kHz-20 kHz. The entire experiment was performed in controlled room temperature of 20°C, since temperature is one of the dependent parameters of pH value.

IV. DESIGN OF THE SENSING SYSTEM

Fig. 2a shows the proposed sensing system to detect the synthetic milk. The physical dimension of the probe is shown in Fig. 2b. The first block consists of the CPA-based sensor dipped inside the test sample. The output of the sensor is a phase angle (which remains constant over a frequency band). A phase detector circuit [12] measures the phase angle and gives voltage output. The display will glow indicator circuit whenever adulteration is detected. The microcontroller based stepper motor drives the control valve so that whenever adulteration is detected the control valve at the outlet of the milk line shuts off to prevent mixing.

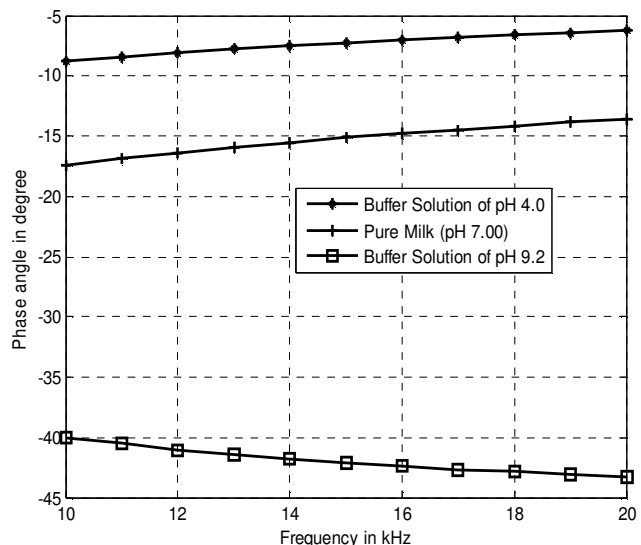


Fig. 1: Constant Phase behaviour of the sensor in three standard test medium.

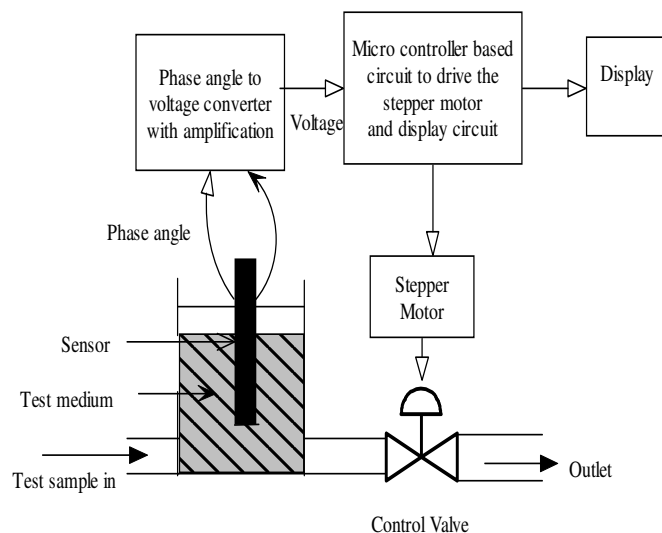


Fig. 2a: Block diagram of the proposed sensing system.

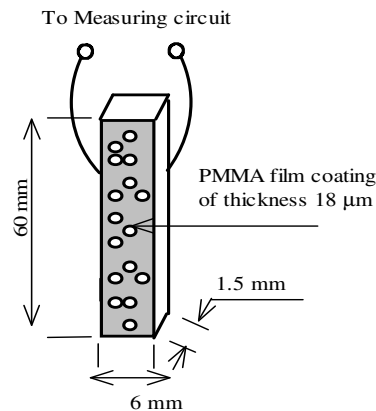


Fig. 2b: Schematic diagram of the probe (Dimensions are not in scale)

A. Sensor block

The sensor block consists of the CPA sensor dipped inside the test medium. The interaction of the sensor with the test medium changes its impedance. In this measurement the “change of phase angle” in different test medium is considered as the sensor output. In Eqn. 2, it has been mentioned that the change of phase angle is dependent upon the ionic property of the test medium, hence the milk sample consisting of different ions will result different values of the phase angle. The phase angle mode is advantageous as the subsequent signal transduction becomes easy [8]. More over, for a particular measurement the phase angle remains constant over a frequency band, which is an essential requirement for automated sensing system [13, 14] where change of signal frequency for interference or other effect will not hamper the measurement.

B. Phase angle detector

The phase detector circuit [11] is shown in the Fig. 3. In this circuit four op-amps and one XOR gate are used. XOR gate mainly perform the phase detection. First two Op-Amps (Amplifier 1, Amplifier 2) act as amplifier circuit and second two (Comparator 1, Comparator 2) are comparators. The lower part of the circuit i.e., Amplifier 2 and Comparator 2 are used for reference signal. In the inverting terminal of Amplifier 1, a resistor of 1 kΩ is connected whereas in the Amplifier 2, a resistor with different phase angle is connected. So the outputs from the Amplifier 1 and 2 have some phase difference when a sinusoidal excitation is applied. Then the output signals are fed to the comparator. The comparator converts the sinusoidal signal to square wave.

From the truth table of the XOR gate it can be seen that when d , the phase difference between the two inputs of the XOR gate is zero the output voltage $v_o = 0$, and when the phase difference is 90° the output will be maximum (half of the V_{DD} applied to the XOR gate chip). The output of the XOR gate is fed to a low pass filter containing a resistance and capacitance. So, it gives dc output voltage proportional to the phase difference between the two inputs.

Fig. 3 shows the phase detector circuit used for the sensing system. Though the circuit is a standard one, but as the constant phase angle is to be measured over a frequency range, the phase characteristics for individual component is to be studied. To do that, frequency response of the individual op-amp has been studied before fabricating the complete circuit.

C. Actuator

The micro-controller based stepper motor driver closes the control valve to perform the actuation. The output voltage corresponding to the pure milk is stored in the micro-controller and whenever a deviation occurs the driver circuit of the stepper motor is activated. This closes the control valve at the outlet and cuts off the milk supply line.

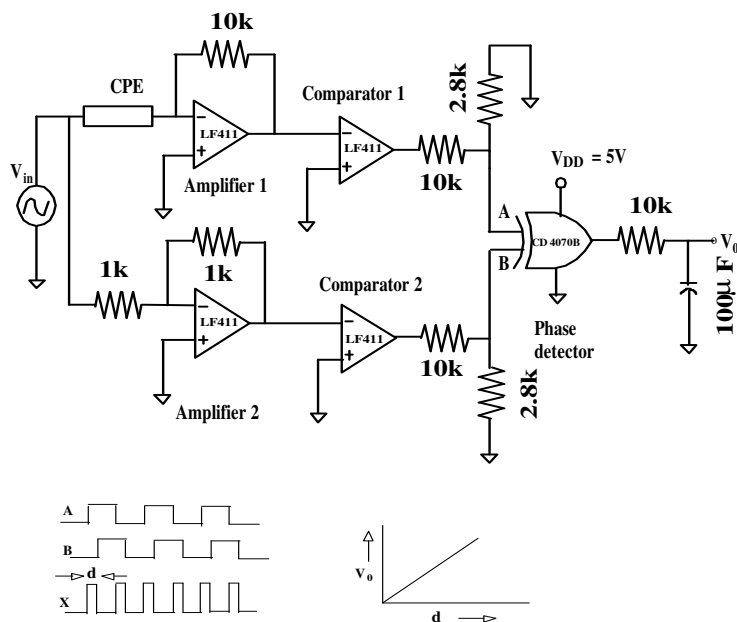


Fig. 3: Phase detector circuit

D. Display unit

The display unit consists of LCD display and a LED array system. The output voltage can be read from the LCD and a comparator based LED driver system is used to glow the ‘RED’ or ‘GREEN’ LED to indicate the status of the milk.

V. EXPERIMENTS, RESULTS AND DISCUSSION

A. Sample preparation

250 ml of pure-milk with pH value 7.00 (complete specification is given in Appendix-I) is taken in a 500 ml beaker and boiled for 15 minutes. Then 1.5 ml of “muriatic acid (a chemical composition of HCl) is added to it. In few minutes light green color liquid (120 ml) is formed in beaker while white colored solid part gets separated. This light green colored liquid is filtered by filter paper into the conical flask and this liquid is called as ‘liquid-whey’. The white solid part is termed as cottage cheese. The chemical composition of these cottage cheese and whey is provided below.

TABLE 1
COMPOSITION OF COTTAGE CHEESE AND WHEY

	Cottage cheese	Whey
Fat (%)	25-35	0.30
Protein (%)	15-25	0.70
Lactose (%)	2.0-2.5	4.40
Minerals (%)	0.3-0.4	0.60

The above mentioned ‘liquid-whey’ (pH value 4.33-4.98) is added to the pure milk which makes the mixture acidic (lowers the pH value). To compensate that small amount of NaOH is added till the pH value matches the original pH value of the pure milk. This reconstructed ‘synthetic milk’ is the test sample.

B. Experiment

The experiments are carried out as discussed in Section III. The sensor is dipped inside the tests samples (first column of TABLE-2) and then the value of pH is measured by precision pH meter (Systronics Digital pH Meter 335). The magnitude and phase angle values of impedance of the sensor are measured by using precision LCR meter (Agilent Precision Impedance Analyzer 4294A) varying input frequency. And then output voltage is noted from the LCD by varying the frequency of the input signal. The results are tabulated in TABLE-2.

C. Results and Discussion

From Fig. 1, it is apparent that the sensor shows almost constant phase angle in the frequency zone 10 kHz–20 kHz and can be used for the proposed constant phase angle based sensing system.

The second and third columns of TABLE-2 show the pH value of the test samples and corresponding phase angle obtained by the sensor at 15 kHz. Point to be noted the phase angle is almost constant in the frequency range (10 kHz-20 kHz) which changes with the different ionic property of the test medium.

From Fig. 4, it can be observed that the output voltage of the sensing system also remains almost constant in the prescribed frequency zone (15 kHz-20 kHz). Though output voltage versus phase angle (Fig. 5) does not show a linear curve but exhibits a definite relationship, i.e., with the increase of phase angle (i.e., decrease of ionic concentration), output voltage decreases.

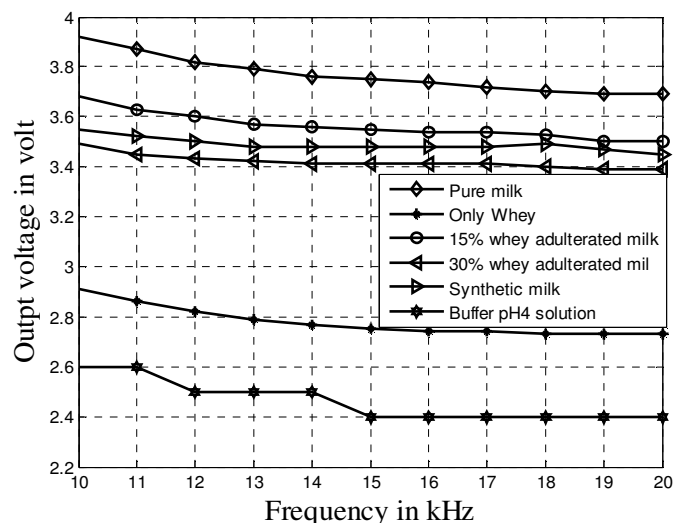


Fig. 4: The output of the sensing system for different test samples.

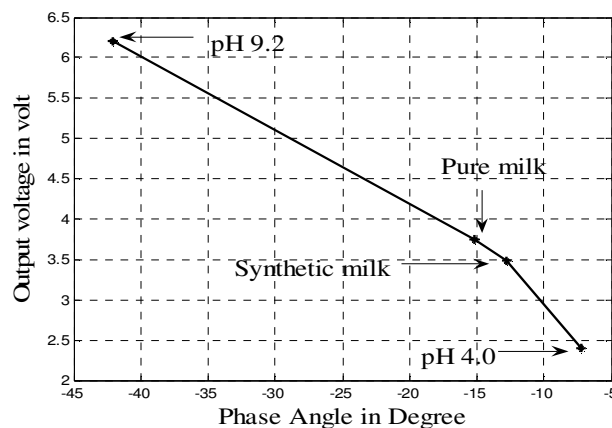


Fig. 5: The output voltage of the sensing system versus the phase angle of the sensor.

TABLE 2
PERFORMANCE OF THE SENSING SYSTEM IN THE TEST MEDIUM
AT 15 KHZ FREQUENCY

Test samples	pH value	Phase angle	Voltage (V)
pH4.0	4.00	-7.24	2.4
Pure milk	7.00	-15.15	3.75
Pure milk+15% whey adulteration	6.95	-13.02	3.55
Pure milk+30% whey adulteration	6.87	-11.81	3.41
Synthetic milk (reconstructed after adding 30% whey and NaOH)	7.00	-12.81	3.48
pH9.2	9.20	-42.11	6.2

It will be worth mentioning here, that after adding NaOH to the whey adulterated milk though the pH value is brought to the same value of the pure-milk, the sensor could identify between these two test samples and shows different phase angles. This is also reflected in the voltage output (3.75 volt for pure milk and 3.48 volt for Synthetic milk).

VI. CONCLUSION AND FUTURE WORKS

In this work, a low cost automatic sensing system is designed to detect synthetic milk. The synthetic milk is reconstructed after adulterating pure milk with ‘liquid-whey’. The main difference between the pure milk and synthetic milk is the presence of different ions which the sensor is capable to detect. Moreover, the measurement is based on the change of the value of the phase angle due to the presence of ions with different concentration in the test medium. The phase angle is again constant over a frequency range, which is an added advantage for such automatic sensing systems. The instrumentation system is such designed, that whenever

adulteration is detected the micro-controller based stepper motor shuts off the control valve in the milk supply line and prevents mixing pure milk with adulterated milk.

The stick type PMMA-film coated probe is rigid and hence, easy to install in the system and also replace whenever necessary. The PMMA coating makes it biocompatible, an essential requirement for the system.

The proposed system can detect synthetic milk when 30% whey is added, but need to increase the resolution of the system. Further research work is on in this direction.

Modification of the electronic transduction circuit to improve sensitivity and linearity remain as one of the future scopes of work. Field testing of the sensing system is to be carried out and the life time of the sensor as well as other components of the system is to be investigated in future.

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APPENDIX-I

Specification of the milk used for experimentation
 Mother Dairy Cow Milk

Vitamin A Enriched
 Homogenised & Pasteurised
 Net content 500ml
 Enriched with Vit A 2000 IU per 1000ml
 Milk Fat 3.5% Minimum
 Milk SNF 8.5% Minimum

Nutritional information per 100 ml.

Protein(g)	: 3.20	Fatty Acids(g)	: 0.008
Carbohydrate	: 4.5	Mineral(g)	: 0.70
Added vit(IU)	: 200	Vitamin(mg)	: 4.9
Energy value (K cal)	: 62	Fat(g)	: 3.5
Cholesterol(g)	: 0.01		

Marketed by : Mother Dairy Calcutta
 P.O. Dankuni Coal Complex,
 Dist. Hooghly(WB), Pin- 712310
 Customer Care No. 033-2659-2342

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