A Phase Angle Measurement Based Conductivity Sensor for Low Conductance Solution

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ABSTRACT

This work presents a simple, small size, low cost, stable conductivity sensor which is able to measure a solution conductivity of the range 10 μS/cm to 1 mS/cm with ±5% full scale accuracy. The sensor is simple to fabricate, light weight and small in size (3 cm × 0.6 cm × 0.16 cm). It is fabricated by coating a thin film of polymer, DQN-70, on a double-sided copper-clad epoxy strip. Here, the sensing principle adopts a phase measurement technique, and thus, avoids bulky and complex 4-electrode measurement set up. The sensor requires very small amount of solution (0.5 ml) making it suitable for the cases where sample amount is not abundant (e.g. in diagnostic and pharmaceutical applications). The sensor is tested over a year, in different ionic solution and is found to retain its sensor characteristics. A proper signal conditioning scheme has also been proposed to develop a multichannel conductivity meter with the developed conductivity sensor.

Key Words: *Conductivity Sensor, Phase Detector, Low Conductance Solution.*

1. INTRODUCTION

In recent years, measurement of conductivity of a solution is gaining paramount importance in different fields of applications. The conductivity of a solution is now, not a mere interest of material scientists, rather a tool in oceanography, water treatment, pollution control, study of corrosion and medical diagnostics. In oceanography, the conductivity measurement is a primary tool in characterizing sea water [1]. However, one of the primary concerns for such oceanographic sensor is that, the sensor should sustain its property in high percentage saline water as well as it should not pose any threat to marine life while doing in-situ measurement [2], especially when marine animal itself is the platform of such sensor [3]. That also requires the sensor to be of small size and light weight. So, miniaturization of conductivity meter is one of the main research interests in this subject [4].

Beside sea water, significant attention is also being grown in quality assessment of fresh water. And change in water conductivity relative to nominal values, is being used to detect environmental changes and pollution events [5]. On the other hand, water coming from a lake, river, or tap usually contains large amount of ionic contaminants which causes scaling and corrosion in various process plant equipment like cooling towers, boilers and heat exchangers [6]. So, quality assessment of water is essential in different industries also. Though conductivity measurement is a non-selective technique (as it does not distinguish individual concentrations of different ions), still, serves the gross purpose of water quality assessment more simply than any other ion-selective processes; hence, more popular in different industrial applications.

Leak detection and study of corrosion, mainly in different types of reservoirs, and piping due to potentially harmful contamination employs conductivity sensing technique [6]. One main aspect of such applications is that, the sensor must be stable and retain its characteristic even in foul environment [7]. To develop a high calibration-stability sensor is another prime research interest and some works has already reported in this line [8]. Again precise measurement will be of most importance when interface of two liquid of different conductivity is being detected by conductivity sensor. Different food and beverages and chemical processing plants utilizes such type of applications [6]. De-ionized water plants and distilled water plants, both thermal (evaporative) and membrane (reverse osmosis) type, make extensive use of conductivity sensing technique to monitor their processes. Their household version needs small size of sensor. Small size sensor is required in pharmaceutical and diagnostic applications also. MEMS based sensor is another solution to meet this challenge of miniaturization [5, 9-10]. Besides, conductivity sensing for diagnostic applications needs to be functional with only very few volume of liquid.

As a conductivity meter can sense the presence of ionic component in water, it is very useful in pollution control as well as in aqua agriculture [11]. Conductivity sensors are reported to be useful in different type adulteration detection like presence of whey, urea etc. in milk [12]. However, all these area needs a simple, cheap and rugged sensor so that common people can use them.

The rest of the paper is organized as follows: the Section 2 describes the motivation and objective of the work. In Section 3, the fabrication of conductivity sensor is detailed and in Section 4, the sensing principle is explained. Section 5 gives the experimental results; Section 6 describes the signal conditioning circuit for proposed prototype of a conductivity meter. The work is concluded in Section 7, defining the future path of work.

2. OBJECTIVE AND MOTIVATION

The main objective of the work is to develop a simple, small size, low cost, stable conductivity sensor which would be able to measure conductivity effectively in the range- 10 μS/cm to 1 mS/cm. Most of the commercially available conductivity sensors employ four electrode techniques for precise measurement of conductivity. Here, two of the electrodes are used to establish the sensor drive potential, while the other two senses the flow of current between the drive electrodes and maintain proper drive potential. 4-electrode system gives more accurate result with respect to 2 electrode system. Many research works are also devoted to such 4-electrode type conductivity sensors [13]. However, such systems are usually bulky. The electrodes are also made of costly metals like, titanium [14] or platinum [15] coating, making the sensor costly. Such sensors are cell type, hence; require extra care in handling. So, though very accurate, such sensors lack their utilization in many fields, especially, where size or cost or ruggedness of the sensor is also a pressing issue.

In this work, we develop a sensor which is actually a polymer coated copper clad epoxy strip of very small size (3 cm × 0.6 cm × 0.16 cm). The fabrication procedure is simple and cheap and the sensor is rugged and easy to handle. The sensor is experimentally validated to measure the conductivity in the range 10 μS/cm to 1 mS/cm. The sensor is found to retain its characteristics over a year. Besides it is quick to response and takes very small volume of sample to measure conductivity.

3. FABRICATION OF SENSOR

It is a polymer coated Copper clad 2-electrode type sensor, fabricated by following method-

3.1. Electrodes

The electrodes are thin Cu layer on either side of a rectangular epoxy-glass (relative permittivity 3.6) block (viz. fig. 1a) of dimension 3 cm × 0.6 cm × 0.16 cm. This is actually prepared by cutting a double sided-Cu-clad printed circuit board (PCB) in given dimensions. Cu layer are on the largest faces of the blocks and lead wires are soldered to them.

3.2. Polymer

The coating polymer is a proton exchange type polymer-Naphthalene dianhydride (NTDA) based semifluorinated sulfonated copoly(ether imide)s. It is synthesized by one-pot high temperature polycondensation reaction using sulfonated diamine, 4,4’-diaminostilbene-2,2’-disulfonic acid

(DSDSA) and fluorinated quadridiamine (QA). The synthesized polymer is termed as DQN-xx, xx indicating mol % of DSDSA in the polymer [16]. For this work, we used polymer with 70 mol% of DSDSA, to be termed as DQN-70 (number average mol wt: 158,700 gm/mol) for rest of the paper.

3.3. Coating of Polymer DQN-70 on Electrodes

First, a measured quantity of the synthesized DQN-70 polymer is dissolved using dimethyl sulfoxide (DMSO) (15 wt%) as solvent. Next, DQN-70 in DMSO solution is coated on the surface of Cu electrodes by dip coating technique and is dried under thermo controlled oven at 100 °C for 12 hour for slow removal of the solvent, then the temperature is slowly raised to 120 °C, 140 °C and 160 °C each for 2 hour. Finally, all the samples were further dried in vacuum oven at 180 °C for overnight (8 hour) to remove any trace amount of solvent.

These coated probes (viz. fig. 1b) can now be used as conductivity sensor. It is fitted vertically in a 2ml plastic container (made by TARSONS) (viz. fig. 1c), within which only 0.5 ml of the sample solution needs to be put for sensing the conductivity (viz. fig. 1d).

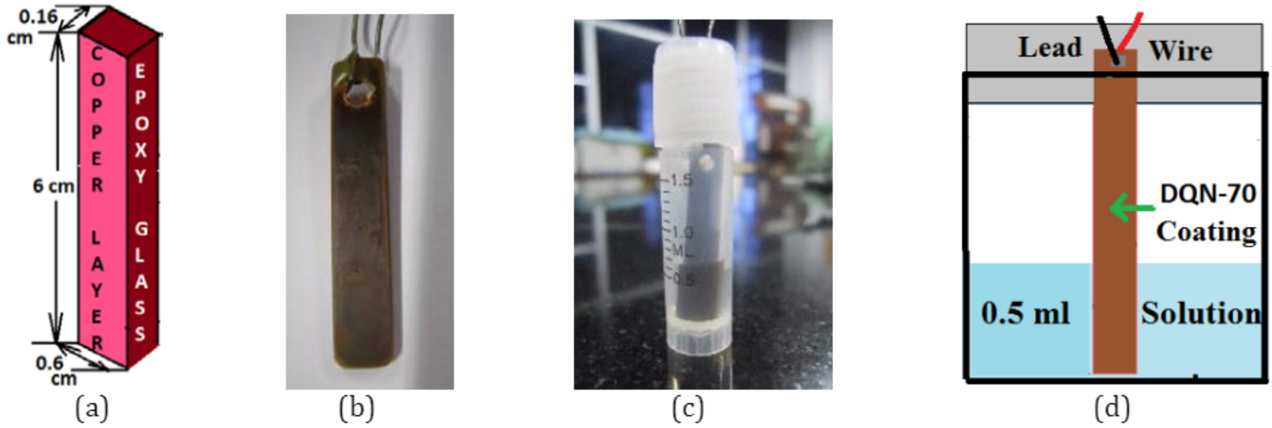


Figure 1: (a) Schematic of Cu-clad Epoxy Block, (b) DQN-70 Polymer Coated Probe, (c) Conductivity Sensor with Sample and (d) its Schematic Diagram (Front View)

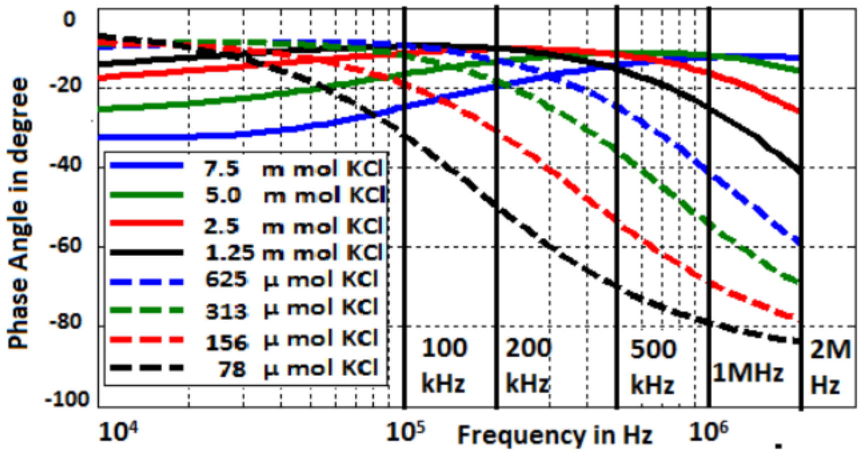
4. THE SENSING PRINCIPLE

The principle for conductivity measurement is based on the impedance behaviour of the above fabricated probes in ionic samples. To develop a sensing principle, we first study the impedance between two lead wires (viz. fig. 1d), putting the coated probe in 0.5 ml of KCl solutions of different concentration. The concentrations of KCl solutions are measured by a standard conductivity meter (SYSTRONICS 304) [15]; and impedance between the lead wires is measured by Agilent 4980A LCR meter. The impedance is measured using frequency sweep option for the frequency range 20 Hz to 2 MHz in Z-θ mode. The variation of phase angle (of impedance) with respect to frequencies is shown in fig. 2a for varying solution concentration.

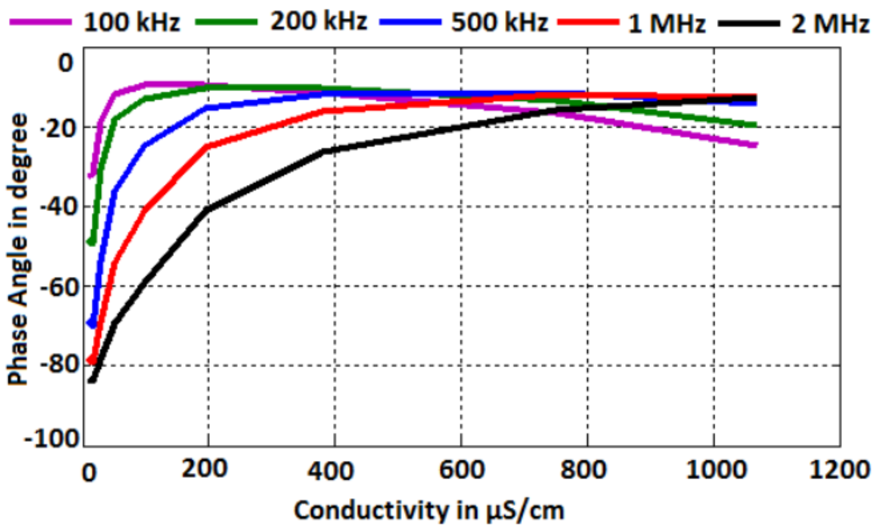
Table 1.KCl Solution Concentration versus Conductivity (measured by Conductivity Meter, SYSTRONICS)

Concentration of KCL Solution (in μmol/lit)	7500	5000	2500	1250	625	313	156	78	39
Conductivity (in μS/cm)	1070	750	380	194	98	51	28	15	8

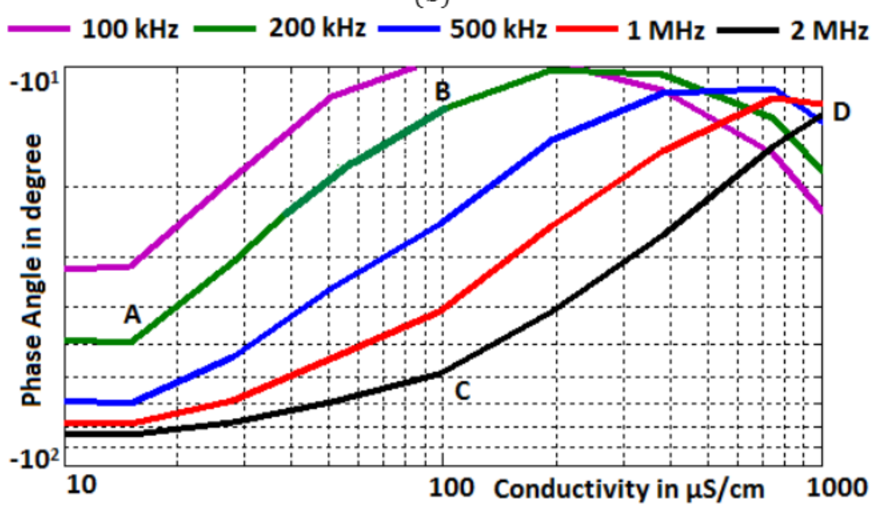
As we see, at high frequency zone (beyond 100 kHz), the phase angle for different concentration is quite different to each other. Now different concentration of KCl has different conductivity as shown in table 1. So, there is a relation between solution conductivity and corresponding impedance phase as shown in fig. 2b. Now, the relationship is non-linear and different for different excitation frequency. However, if the phase versus conductivity is plotted in log-log scale as shown in fig. 2c; two linear zones can be found:



(a)



(b)



(c)

Figure 2: (a) Phase versus Frequency Plot at different Concentration of KCl, (b) Phase versus Conductivity Plot at Different Excitation Frequency, (c) Phase versus Conductivity Plot at Different Excitation Frequency in log-log Scale

1) zone AB (viz. fig. 2c)- on the plot for excitation frequency- 200 kHz; for the conductivity range 10 μS/cm to 100 μS/cm and,
2) zone CD(viz. fig. 2c)- on the plot for excitation frequency- 2 MHz; for the conductivity range 100 μS/cm to 1000 μS/cm

[There are five curves shown in fig. 2c. Each curve has different linear zones. Out of them, the zones AB and CD are mutually exclusive zone and of almost equal range in log-frequency scale, hence these two particular zones are selected.]

Now, the best fitted linear equations for these two zones are-

1) For zone AB: 200 kHz excitation frequency

$$-\log|\theta| = 0.727\log|s| - 2.54 \qquad 10 \le s \le 100 \qquad (1)$$

(*Goodness of fit: Root Mean Square Error, RMSE = 0.03*)

2) For zone CD: 2 MHz excitation frequency

$$-\log|\theta| = 0.655\log|s| - 3.10 \qquad 100 \le s \le 1000 \qquad (2)$$

(*Goodness of fit: RMSE = 0.02*)

Where, θ:phase angle in degree, s: conductivity of solution in μS/cm.

With the equation (1) and (2), we, here, propose the following algorithm for a 2-channel conductivity sensing-

Channel 1: For conductivity 10 to 100 μS/cm, operating frequency of the sensor is 200 kHz; and equation (1) is the governing equation to find out the conductivity from measured phase angle. [The range of measured phase angle for this measurable zone of conductivity is -65^0 (10 μS/cm) to -12^0 (100 μS/cm); if the measured angle is less than -65^0 the conductivity is beyond the measuring range of the sensor, if the phase angle is more than -12^0 one should move to the channel 2]

Channel 2: For conductivity 100 to 1000 μS/cm, operating frequency of the sensor is 2 MHz; and equation (2) is the governing equation to find out the conductivity from measured phase angle. [The range of measured phase angle for this measurable zone of conductivity is -62^0 (100μS/cm) to -13.5^0 (1000μS/cm); if measured angle is less than -62^0 one should move to the channel 1, if the phase angle is more than -13.5^0 the conductivity is beyond the measuring range of sensor at this operating frequency]

5. EXPERIMENTAL RESULTS

Table 2: Measurement of Conductivity of NaCl Solution of Different Concentration (Oct, 2014)

Concentration of NaCl Sol. (Approx. Normality)	N/128	N/256	N/512	N/1024	N/2048	N/4096	N/8192	N/16384
Conductivity (μS/cm) measured by standard commercial meter	1010	510	260	130	71	34.4	20.1	11.7
Operating Frequency	2 MHz (Full Scale: 1000 μS/cm)				200 kHz (Full Scale: 100 μS/cm)			
Measured Phase Angle (in degree)	-14.0	-21.8	-36.3	-49.28	-15.18	-25.4	-37.79	-51.74
Conductivity (μS/cm) by developed sensor	961.6	490.5	224.5	140.8	73.95	36.43	21.09	13.69
% Full scale error in	-4.84	-1.95	-3.55	+1.08	+2.95	+2.03	+0.99	+1.99

measurement								

We employed the developed sensor and applied the above explained algorithm to determine the unknown conductivity of other type of solutions and compared the result with the reading of a standard, commercial conductivity meter (Conductivity Meter, SYSTRONICS 304). We found the proposed sensor is measuring conductivity with ±5% full scale accuracy. In table 2 and table 3, we have shown the experimental results in measuring conductivity of different concentration NaCl solution with developed sensor and validated it with the described standard meter reading. The reported sensor was developed and calibrated in August, 2013. With the same calibration, the sensor had been used over a year for testing in different solution. The data presented in table 2 and table 3 have been collected in October, 2014 and January, 2014 respectively. In both the cases, full scale error is less than equal to ±5%. The good acceptability in sensor response, depicted in table 2 and table 3, also establishes the sensor stability and reliability over a year.

Table 3: Measurement of Conductivity of NaCl Solution of Different Concentration(Jan, 2014)

Conductivity (μS/cm) measured by standard commercial meter	1020	520	270	132	75	37.8	19.8	10.9
Operating Frequency	2 MHz (Full Scale: 1000 μS/cm)				200 kHz (Full Scale: 100 μS/cm)			
Measured Phase Angle (in degree)	-13.5	-20.7	-36.0	-47.1	-14.33	-23.83	-39.05	-53.77
Conductivity (μS/cm) by developed sensor	1017	529.3	227.4	150.9	80.05	39.77	20.16	12.98
% Full scale error in measurement	-0.30	+0.93	-4.26	+1.89	+5.05	+1.97	+0.36	+2.08

In fig. 3a and 3b, time response of the developed sensor has been depicted. The phase angle of the developed sensor in different media is measured at four different instants of time- 30 s, 2 min, 5 min, and 15 min after dipping. We see the response is almost steady within 2 min from dipping. This shows the sensor is quickly responsive and to measure solution conductivity, it needs to put the sensor in the medium only for 2 min.

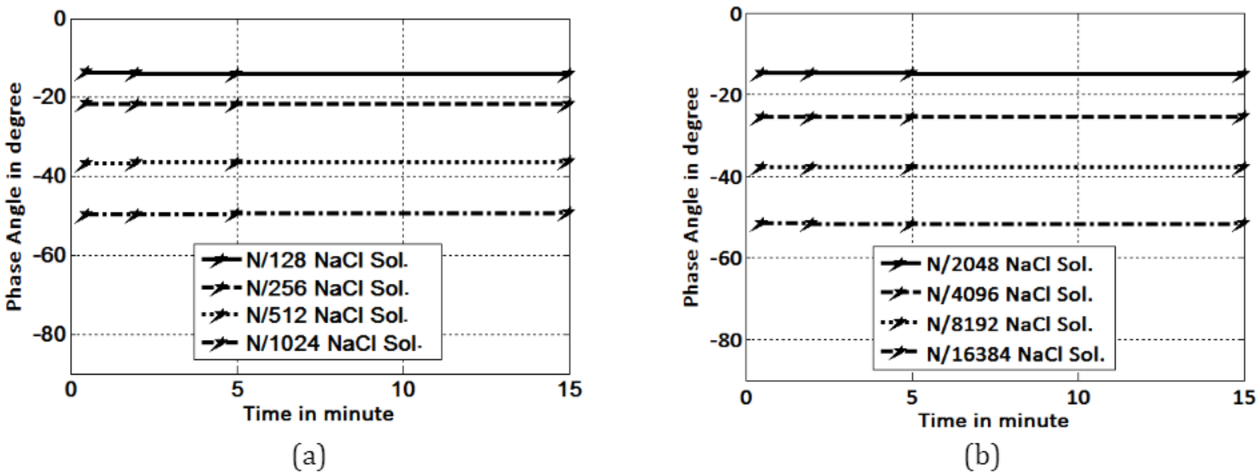
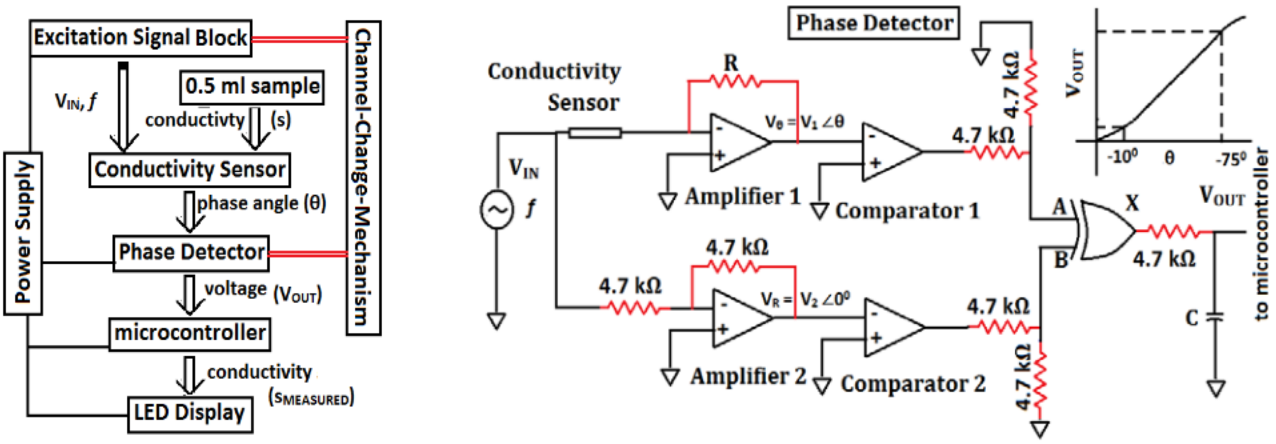


Figure 3: Change of Phase Angle in Different Ionic Solution with respect to Time (a) At 2 MHz Excitation Frequency, and (b)) At 200 kHz Excitation Frequency.

6. PROTOTYPE OF A CONDUCTIVITY METER

Prototype of a conductivity meter, employing the above developed sensor, shall include following parts as depicted in fig. 4a.

First is the excitation part which will include a high frequency sine wave generator capable of generating signal of 200 kHz and 2 MHz; and a channel-change-mechanism which will control the frequency of the generated sine wave. Next is the sensor which will sense the sample conductivity in terms of impedance phase angle. Third part is the analogue signal conditioning circuit- a phase detector. We adopt the phase detector circuit as shown in fig. 4b. The circuit has already performed successfully in some previous works [17]. The output of the circuit is a DC voltage signal (V_{OUT}) which is proportional to the phase difference (θ) between reference signal (V_R) and sensor signal (V_θ). The V_{OUT} versus θ plot is found linear in the range 10^0-75^0 which includes the measurement range of sensor in each channel (viz. Section 4).



(a) (b)

Figure 4: (a) Block Diagram for Prototype of a Two-Channel Conductivity Meter, and (b) Phase Detector Circuit.

However, necessary modification needs to be done to incorporate the channel-change-mechanism which will change the excitation frequency as well as the value of R and C in the phase detector circuit. The output of the phase detector circuit is voltage which will be interpreted in terms of conductivity by the equation (1) or (2) (depending up on corresponding channels) and the transfer function of the phase detector (V_{OUT} versus θ plot). This part of signal conditioning will be done digitally in microcontroller. The output of the microcontroller will be fed to a LED display to show conductivity in μS/cm. However, this part of the work has not been implemented yet, and defines the future path of this work.

7. CONCLUSION

In this work, a new type of conductivity sensor has been developed. It is a two electrode type sensor and easy to fabricate. The sensor is light weight, small in size and of low cost. It is quickly responsive and needs very small amount (0.5 ml only) of sample solution to sense the conductivity. All these properties make the sensor compatible in many applications (described detail in Section 1) where the common 4-electrode cell type sensors cannot be used due to bulkiness and cost.

The novelty in the sensing principle is that, it senses conductivity in terms of phase of the impedance unlike common magnitude based sensors. The sensor is showing good result while compared to a standard commercially available conductivity meter. It can sense 10-1000 μS/cm with ±5% full scale accuracy. The sensor is tested in different solution over a year and found to retain its characteristic over the year. No special care is needed to preserve its characteristics. It is found to be stable in normal ambient condition. No special cleaning is needed before re-using the sensor. Once used, the sensor needs to be washed with distilled water only, and to be dried with a tissue paper before re-use.

The future scope of this work is to develop a prototype of the conductivity meter as described in Section 6 and to use the meter in measuring different test samples. Besides, to modify the sensor, such that, it can be operated at lower excitation frequencies and can measure wider range of conductivity is another scope of work.

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