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Co-Planar Sensor for Impedimetric Characterization of Aqueous Solutions

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Abstract—A minimal complexity co-planar screen-printed sensor has been developed to streamline impedimetric analysis and characterization of aqueous solutions, offering a less complex alternative to conventional liquid sample handling methods. The sensor design allows for repeatable measurement sensitivity and accuracy independent of analyte volume variations, within a range of $10\mu\text{L}$ to $18\mu\text{L}$. Comprehensive mathematical modeling, experimental evaluation, and statistical analysis were conducted to assess the sensor's performance. The results indicate significant potential for the sensor in practical applications, highlighting its efficacy and reliability in aqueous solution characterization.

Index Terms—co-planar sensor, impedance spectroscopy, screen-printed sensor, microfluidics

I. INTRODUCTION

Electrical impedance spectroscopy is a powerful analytical tool for analyzing the characteristics and electrical properties of different types of material by applying an alternating stimulus current and measuring the resulting voltage (or vice-versa). The stimulus can be at a fixed frequency or applied as a sweep of values across a certain frequency range [1].

Impedimetric analysis plays a crucial role in understanding the electrical behavior of liquids or aqueous solutions. It provides information on the ionic conductivity, dielectric properties, and molecular interactions within the solution [2]. Impedance spectroscopy is particularly useful in fields such as electrochemistry (studying redox reactions, ion transport, etc.) [3], material science (investigating polymers, electrolytes, adhesives, etc.), biotechnology/ biomedical diagnosis (characterizing blood and other body fluids) [4], industrial applications (where understanding the electrochemical behavior of solutions is important for applications such as battery development, petroleum refining, etc), biosensor design, and quality control in different manufacturing industries and environments [5].

For any impedimetric analysis, some key factors of the sample holder/sensor need to be determined for a stable and

reliable measurement. First, the configuration of the measurement (e.g. bipolar or tetrapolar), needs to be determined, followed by the electrode material and contact area, which affect the electrode-electrolyte interface. The latter is a crucial part of the measurement, and it mostly depends on the material of the electrode, which needs to provide a low polarization resistance to eliminate unwanted noise in the measurement [6]. Once these design parameters are finalized, the interrogated volume of the analyte also plays a vital role, especially in applications that involve microfluidics and ultra-low sample volumes (μL). In some cases, the portability of the sample holder and the time consumption (initial setup preparation, cleaning in case of repeated use) to prepare the setup before a measurement plays a vital role in the analysis as some analytes (e.g. biological or environmental samples) can degrade over time due to chemical instability that can alter the sample composition, leading to inaccurate results [5].

Whilst a range of both bipolar and tetrapolar microfluidic based devices for ultra-low sample volume (μL) interrogation have been previously proposed [7], they invariably require relatively elaborate specialized fabrication methods and dedicated instrumentation. Moreover, with such small samples, applying the sample volume with high precision is key to achieving repeatable outcomes. On the other hand, most generally used commercially available impedance analyzer instruments can be used to carry out impedance spectroscopy measurements in liquids through fluid cells or test fixtures which typically require sample volumes of several milliliters. Alternatively, metal plate capacitor configuration based instruments require at least 1mL [8] sample volumes for a single measurement. Moreover, commercially available liquid measuring setups are sometimes limited to bipolar configurations only (Keysight 16452A, Solarton 12962A, etc.). Given the increasing relevance of impedimetric measurements in biomedical and environmental applications, there is a need for a test fixture to handle low-volume liquid samples, without the need for

complex fabrication processes, ultra-precise sample handling, or dedicated electronics. If such a fixture could achieve repeatable measurements and be connected to any impedimetric instrument, it would open up such tests to a wide range of analytical laboratories and research facilities.

As a part of our domain of interest in the impedimetric analysis of liquid and aqueous media, we developed a simplified volume-independent co-planar sensor. The specified sensor minimizes the sample processing and handling complexities and provides consistent impedance data for analysis. Studies show that planar sensors provide better localised sensitivity in the desired region and can be implemented easily in a minimal design [9]. The proposed sensor can be easily configurable to be tetrapolar and can also be converted to other existing standard configurations if desired. The use of a widely available screen printing process employing non-polarisable (Ag/AgCl) electrodes lowers the sensor's electrode-electrolyte temporal variability and the relatively large contact area provides a lower noise profile, and ease of fabrication [10], [11]. The use of a fixed geometry electrode configuration, focusing on a well-defined measurement region ensures repeatability of measurement even if the applied sample volume is not exact. This is achieved in combination with a relatively large channel width combined with low channel height provided by an adhesive add-on layer, allowing for the interrogation of very low sample volumes. Thus the sensor can be effectively applied in experiments where the sample quantity is limited.

II. SIMULATION MODEL

A 3D computational model (Fig. 1a) was developed to simulate the sensor in COMSOL 5.5 using AC/DC module and electric current (ec). In this in-line tetrapolar configuration, the two outer electrodes function as CC (current-carrying) for current injection, while the inner pair of electrodes serve as the PU (pick-up) electrodes utilized for voltage measurement. The injection current was $100\mu A$ with an insulation boundary condition.

An 'extremely fine' mesh was employed for the simulation. In the Frequency Domain study, we utilized Sensitivity distribution, S , a commonly employed parameter for tetrapolar impedance measurement systems, which is

$$S = \frac{\bar{J}_{CC} \cdot \bar{J}_{PU}}{I^2} \quad (1)$$

where \bar{J}_{CC} represents the current density (A/m^2) resulting from the injected current through the CC electrodes, \bar{J}_{PU} represents the current density when the currents are injected through the PU electrodes, and I is the magnitude of the applied current. The sensitivity distribution along the sensor surface, as shown in Fig. 1b, indicates that the measured impedance should have higher contributions from the region between the two PU electrodes. Fig. 1c further illustrates that the sensitivity is relatively constant in the region between the PU electrodes, with the magnitude of sensitivity in all other regions being negligible. Therefore, the measured impedance should be affected by the material present in the middle region

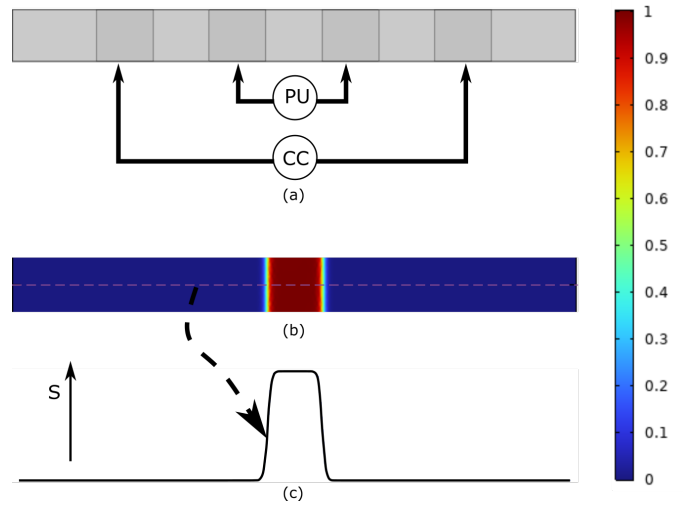


Fig. 1. Sensitivity distribution over the surface

of the sensor (the region between the PU electrodes). The sensor requires a sufficient volume of sample to cover all the electrodes, a critical factor in its operation. However, if there are liquids outside the CC electrodes, their effects on the measured impedance should be negligible. This reassures us of the robustness of the sensor, even in the presence of external factors. The transfer impedance was calculated by

$$Z_t = \frac{V_{PU}}{I} \Omega \quad (2)$$

Here, V_{PU} is the potential measured between the PU electrodes when an injection current of I is applied through the CC electrodes.

III. DESIGN AND DEVELOPMENT

A. Fabrication Process

Fabricating the Ag/AgCl test strip using screen printing involves several steps. A transparent polyester film ($90\mu m$ thick) with a hydrophilic coating is used as the base substrate. Ag/AgCl ink is screen-printed onto the hydrophilic surface to create electrodes, including extra features for registration points. The printed image consists of 4 identical sensor electrode sets with $1mm$ wide parallel lines separated by $1mm$ gaps and corner markers for sample label placement. The ink is dried at $50^\circ C$ in a dehumidifier box. The polyester substrate is then laminated to a $250\mu m$ thick polyester backing sheet using a free-film adhesive and cut into individual sensor cards. A sample label, made of a laminate of release liner, double-sided tape, hydrophobic coated mesh, and another release liner, is die-cut to form a flow channel and outer profile. The first release liner is removed, and the label is positioned over the sensor lines using corner markers. The hydrophobic mesh allows air to escape, ensuring no air pockets remain as the sample settles over the electrodes, resulting in a functional Ag/AgCl test strip. The CAD design of the sensor is provided in Fig. 2, where all dimensions are given in millimeters (mm).

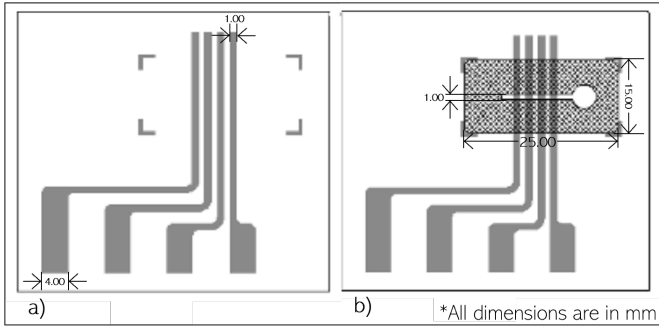


Fig. 2. CAD design with dimension

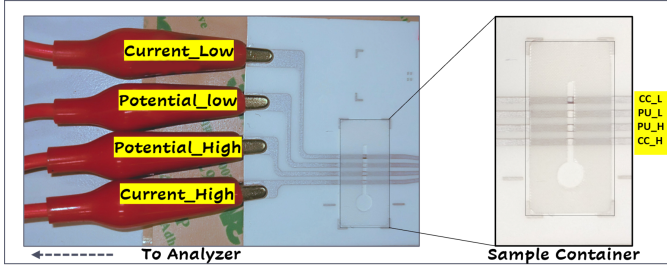


Fig. 3. Physical sensor with sample holding channel

B. Fabricated Sensor

Fig. 3 presents a fabricated sensor with individually indicated electrodes. The two outer electrodes are current injectors for a tetra-polar measurement, while the middle pairs, the potential pick-up electrodes, play a crucial role in the sensor's functionality. For a bipolar measurement, two adjacent electrodes need to be shorted. The zoomed area showcases the sample loading space, a circular area where the sample should be dropped. The capillary action of the sensor's surface aids in the even spread of the analytes over the electrode traces.

IV. RESULTS

A. Measurement and Analysis Setup

A fixed set of setups and configurations was strictly followed in all measurements. For taking the impedance measurements in tetrapolar configuration, a Keysight 4294a impedance analyzer was used, keeping the measuring parameter in R-X mode, and source oscillator frequency and levels were set to 110 MHz and 500 mV, respectively. A macro-enabled Excel worksheet (Excel VBA) from Keysight was used to acquire data from the impedance analyzer. All analyses are done in MATLAB R2023b. Every experiment was carried out in controlled environmental conditions (Temperature: 24°C, Humidity: 40±10%). Fig. 4 illustrates the overall measurement process in simplified steps.

B. Physical Evaluation of the Sensor

To effectively demonstrate the versatility and capability of the proposed sensor for impedimetric analysis and to validate the associated claims, a series of specific experimental

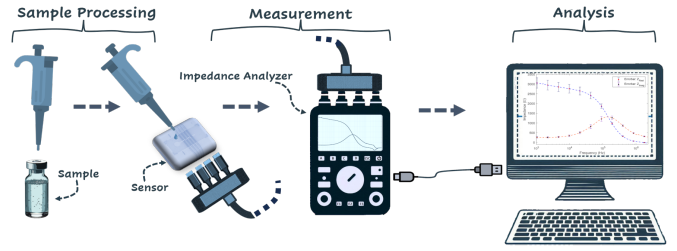


Fig. 4. Measurement process step by step

analyses are essential. These analyses are designed to thoroughly investigate and confirm the sensor's performance and accuracy in various conditions. Consequently, the following detailed experiments have been conducted to comprehensively characterize the sensor's properties, ensuring a robust evaluation of its functionality and reliability.

1) *Volume Independency*: The fabricated sensor has a sample capacity of around 18μL, where the minimum volume required for a measurement is 8μL (recommended 10μL to 18μL for standard measurement). Because of the co-planar design of the sensor, it enables the measurements to be volume-independent for the specific recommended volume range. Fig. 5 represents the bode plot from the experimental impedance data including the standard error limit from 10μL to 18μL 1.2% (w/v) NaCl solution with three repeats. The bode plot depicts the average experimental data of the real and imaginary parts. The error has been calculated by following the Standard Error(SE) formula,

$$SE = \frac{\sigma}{\sqrt{n}} \quad (3)$$

where, σ represents the standard deviation of the sample and 'n' is the number of items in the sample. The maximum error in amplitude found in the real part of the impedance is 19.4513Ω and in the imaginary part, it is 8.6835Ω.

Another measure to determine the consistency of the measurement is the deviation in the pole frequency of the imag-

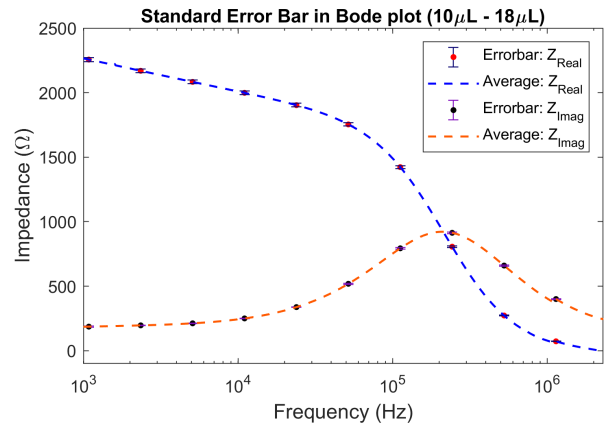


Fig. 5. Standard error limit in Bode plot of 1.2% NaCl solution (10μL - 18μL)

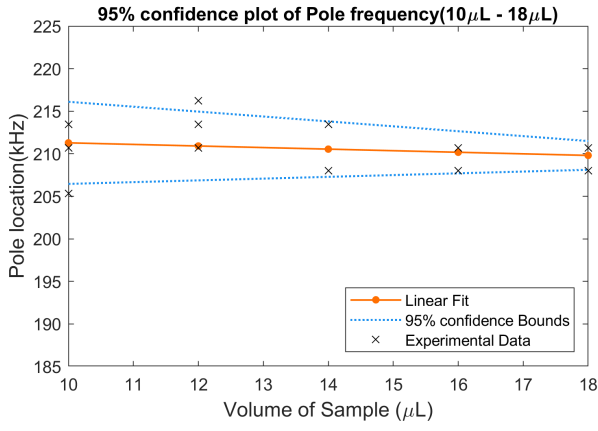


Fig. 6. Pole location of 1.2% NaCl solution(10 μ L - 18 μ L)

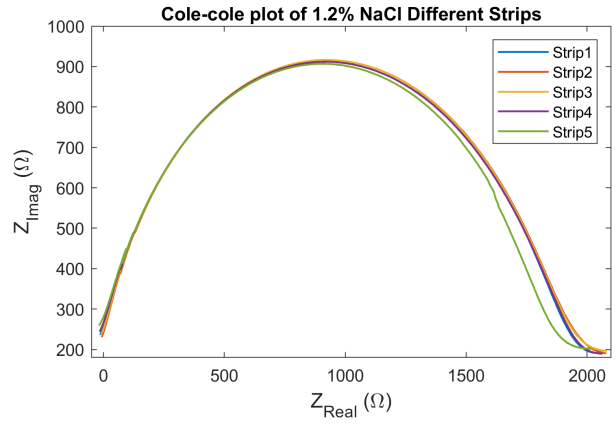


Fig. 8. Cole-Cole plot of 5 different sensors

inary part. Fig. 6 shows the pole location of the above-mentioned impedance data where analyte (1.2% (w/v) NaCl) volume ranges from 10 μ L to 18 μ L with 95% confidence interval limits. The confidence interval is calculated from Standard Error (SE) and the T-distribution data for the 95% confidence interval.

From Fig. 6 it can be seen that the confidence interval is between 5.1125 kHz and 1.9340 kHz for the 95% confidence limit.

2) *Sensor Consistency*: For analyzing different or the same type of samples in different strips or for any comparison study between different results obtained differently, the sensor properties must remain the same across different sensors. The same results for the same analytes between different sensors are required to validate this. The experimental data of 1.2% (w/v) NaCl shows a good consistency in inter-strip comparison. The comparison study results in Fig. 7, Fig. 8 reveal the consistency between strips from different production batches.

The bode plot with standard error (SE) following equation (3) gives a maximum 38.5987 Ω standard error limit in the real part of the impedance and a maximum 15.5565 Ω in the

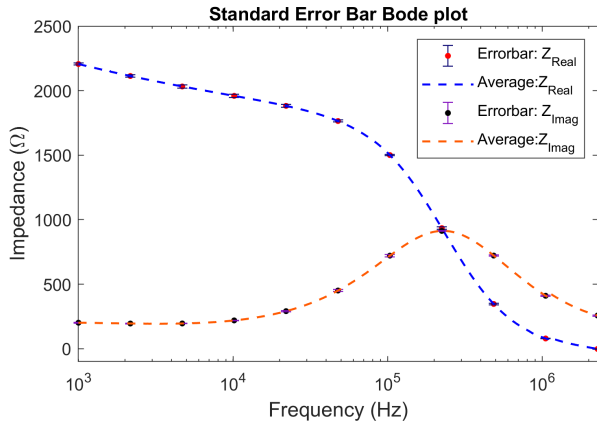


Fig. 7. Bode plot with standard error limit of 5 different strips

imaginary part of the impedance of the experimental data. The negligible amplitude variation can also be seen from the Cole-cole plot in Fig. 8. From these analyses, it can be stated that measurements between different sensors remain consistent throughout the experimental data.

V. DISCUSSION

It is often modeled as an equivalent electrical circuit to characterize material in the impedimetric domain, typically comprising a combination of resistors, capacitors, and constant phase elements (CPE). For instance, a homogeneous material like a NaCl solution can be represented by a parallel RC circuit, where the resistor (R) signifies the conductivity and the capacitor (C) signifies the permittivity of the material. The impedance's related properties (such as amplitude/phase or real/imaginary components) vary with frequency depending on the model used. At the resonance point, a pole is identified, and multiple poles may be observed in more complex models or when multiple RC combinations are present. The location of this pole in the frequency spectrum is directly related to the values of the associated components (R and C).

The most common form of a single pole RC (a parallel combination of R and C) model will have the impedance value according to the equation (4) and the pole location is defined by the equation (5).

$$Z = \frac{R}{1 + j\omega RC} \quad (4)$$

$$f_c = \frac{1}{2\pi RC} \quad (5)$$

Homogeneous salt solutions (a specific amount of standard NaCl mixed in de-ionized water to prepare salt solution where concentration ranging from 0.5% -1.5% (w/v)) were used as the sample to characterize an aqueous solution using the sensor mentioned above. The experimental data is portrayed in Fig. 9 and Fig. 10. It can be seen that the variation in the amplitude of the saline solution and the shift of pole location can be identified from the impedance data. The Bode plot (Fig.

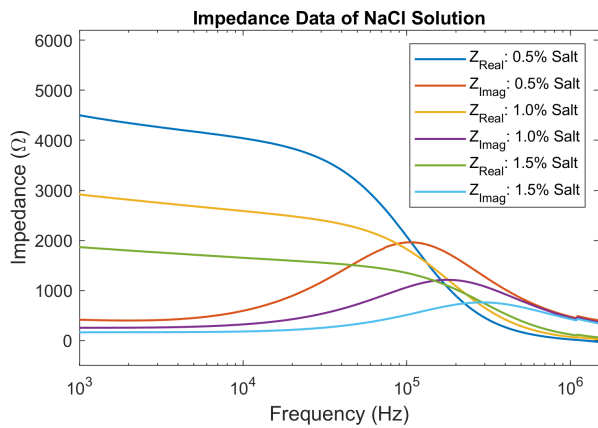


Fig. 9. Bode plot of different salt concentration solution

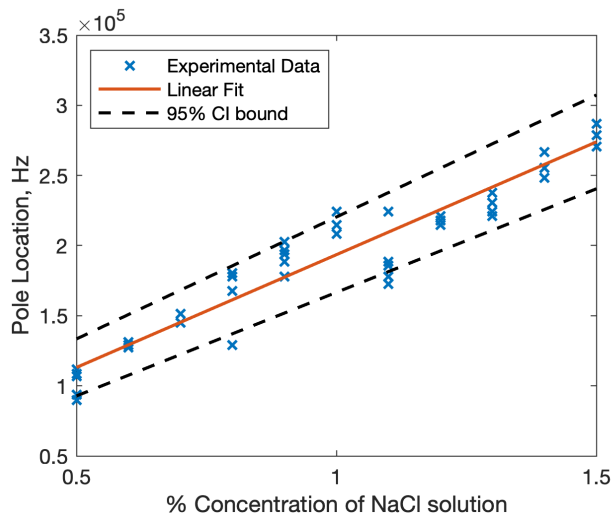


Fig. 10. Pole location with salt concentration level

9) of three different concentrations (0.5%, 1.0% & 1.5%) of NaCl solution shows that the overall impedance level decreases with the increase of salt concentration. On the other hand, the pole location shifts right (cut-off frequency increases) with salt concentration Fig. 10.

Fig. 10 represents the linear fit of the experimental data of different concentrations of Salt solution (NaCl) with 95% confidence interval bounds. Distinguishing between different salt concentrations can easily be identified from the impedance measurement. The location of the peak (frequency) increases with the increment of salt concentration, mathematically from (5) for any fixed value of capacitance (C); as resistance decreases, cut-off frequency increases and vice-versa. Increment salt concentrations with other parameters remain fixed, which means more conductive ions are available in the solution. Therefore, the amplitude level drops, and the location of the pole frequency increases, which agrees with the collected impedance data.

By mathematically correlating the results of the test with the above equations (4) and (5) and the prior stated performance

results of the sensor it can be considered that the sensor can be validated as a standard tool for aqueous impedimetric measurements.

VI. CONCLUSION

A new co-planar sensor has been presented for impedimetric analysis of aqueous solutions. A standard homogeneous NaCl salt solution was utilized as a reference sample for characterization and performance analysis. Measurements were carried out for different concentrations and different volumes of solution within the measurement capacity. The Ag/AgCl co-planar electrodes have excellent electrode-electrolyte interaction, which gives the sensor a high sensitivity and low temporal drift. As a result, the sensor can provide a good measurement of the analytes with a lower noise profile. Performance analysis of the sensor and the mathematical data exhibit the capability to characterize aqueous solutions. The exceptionally low requirement for accuracy in volumetric handling of the analyte within a range of values ($10\mu\text{L}$ to $18\mu\text{L}$) without affecting the results makes the sensor versatile and applicable where sample processing facilities and expertise are limited. Overall, this sensor offers measurement quality for ultra-low sample volumes combined with cost-effectiveness and reliability. The simplicity of the fabrication process of the sensor and the adaptability with the existing measurement techniques and apparatus make this sensor an enabling analysis tool for a range of non-highly specialized facilities for any aqueous impedimetric analysis.

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