

Characterization of Interdigitated Electrode Structures for Water Contaminant Detection Using a Hybrid Voltage Divider and a Vector Network Analyzer

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Abstract— Interdigitated capacitive electrode structures have been used to monitor or actuate over organic and electrochemical media in efforts to characterize biochemical properties. This article describes a method to perform a pre-characterization of interdigitated electrode structures using two methods: a hybrid voltage divider (HVD) and a vector network analyzer (VNA). Both methodologies develop some tests under two different conditions: free air and bi-distilled water media. Also, the HVD methodology is used for other two conditions: phosphate buffer with laccase (polyphenoloxidase; EC 1.10.3.2) and contaminated media composed by a mix of phosphate buffer and 3-ethylbenzothiazoline-6-sulfonic acid (ABTS). The purpose of this study is to develop and validate a characterization methodology using both, a hybrid voltage divider and VNA T-II network impedance models of the interdigitated capacitive electrode structure that will provide a shunt RC network of particular interest in detecting the amount of contamination existing in the water solution for the media conditions. This methodology should provide us with the best possible sensitivity in monitoring water contaminant media characteristics. The results show that both methods, the hybrid voltage divider and the VNA methodology, are feasible in determining impedance modeling parameters. These parameters can be used to develop electric interrogation procedures and devices such as dielectric characteristics to identify contaminant substances in water solutions.

I. INTRODUCTION

Electrode structures at low frequencies with small dimensions relative to the wavelength of the stimulation signals can be modeled as an interconnection of lumped passive or active electrical components having unique voltages and currents defined at inputs and outputs. However, when the signal are either RF or beyond ($>1\text{MHz}$) this model could be enhanced by using two port network analysis as suggested by microwave engineers [1]. The precise characterization and modeling of electrode structures have important implications in the continuous monitoring of different bioprocesses such as cell differentiation and microfluidic separation using dielectrophoresis principles. Microfluidic biosensors and electrodes have been successfully tested in experimental set ups with promising applications such as: the development of a micro-flow

injection amperometric biosensor system to determine the glucose content of pharmaceutical injections [2]; dielectric spectroscopy and dielectrophoresis to sense changes in dielectric properties of cells [3]; different dielectric techniques used to measure cell viability and their utility [4]; characterization of dielectric properties of oil palm biocomposites [5]; and analyses of the frequency domain characteristics of a single-cell covered microelectrode impedance for cellular biosensing applications [6].

In addition, impedance spectrometry has been used to determine electric characteristics of biological substance experimental set ups and Radio Frequency (RF) or Microwave (MW) stimulation have been applied to determine dielectric characteristics of biological substances. Impedance spectrometry has brought important developments with the following important contributions: impedance characterization of cell-electrode interface using an equivalent circuit approach [7]; development of capacitive impedance spectroscopy (CIS) to investigate the electric properties of electrochemical materials [8]; demonstration of cell manipulation and analysis using dielectrophoresis and micro-electrical impedance spectroscopy in chromaffin cells and red blood cells [9]; measurement of impedance in biofluids to examine the impact of the length and concentration of free-floating double-stranded DNA molecules [10]; and using a resonant sensing electrode structure having an external inductor to sense human blood cells [11]. RF and MW signal stimulation have been used in bioprocessing to characterize dielectric properties of materials such as: use of measurement of moisture content in grain and in considering dielectric heating applications of agricultural products [12]; evaluation of the RF safety of mobile phones in terms of specific absorption rate [13]; development of a microwave nondestructive evaluation to measure dielectric properties of liquids [14]; measurement of the scattering transmission parameters to determine the dielectric properties of wheat, corn and soybeans [15]; and measurement of dielectric properties of homogeneous isotropic medium using microwave frequency signal stimulation [16].

The present work proposes a characterization of interdigitated capacitive electrode structures which will be used to monitor contaminants in water solutions. An advantage of using a capacitance sensor for the measurement is the quick results obtained using electrical measurements and the sensitivity of the capacitor to changes on the dielectric medium, in this case the measurement of a change

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in a water buffer due to an external contaminant. The characterization of the capacitive sensor is based upon two methods, a Hybrid Voltage Divider methodology (HVD) and measurements from a Vector Network Analyzer (VNA). This paper is divided in eight sections: introduction, HVD methodology, VNA methodology, COMSOL capacitance modeling, validation of the proposed model, results and discussion, conclusions, and references

II. HYBRID VOLTAGE DIVIDER METHODOLOGY

The HVD consists of an RC network where the capacitive reactance is connected to ground and it is calibrated at the half power point. This is, at operating frequencies, the resistance value equals the capacitive reactance such that the detection and measurement of the impedance are performed roughly at the point where the impedance magnitude equals the value of R. Fig. 1 shows the typical hybrid voltage divider.

The hybrid voltage divider detection shows some important characteristics:

1. The device behaves as a low pass filter of the input signal VS with a phase going from 0 to -90°.
2. According to this behavior, the magnitude of the output voltage reduces to 70% of the DC value when $\omega_0=1/RC$.
3. When the capacitive reactance $X_C=1/(\omega_0C)$ equals the resistance of the divider, R, the magnitude of the output voltage also reduces to 70% of the input signal magnitude (half power point).

III. VECTOR NETWORK ANALYZER METHODOLOGY

When considering high-frequency networks, equivalent voltages and currents, and their related impedance and admittance matrices, become an artificial abstraction tool for characterization and modeling purposes. An electrode structure where high-frequency stimulation is performed to describe the behavior of biological media can be modeled as a two port network as shown in the Fig. 2. A good representation of this network which is appropriate for direct measurements having incident, reflected and transmitted waves can be obtained by the so called scattering matrix or S-matrix. The scattering matrix provides a complete description of the two port network and their parameters can be calculated using network analysis techniques. Moreover, those scattering parameters can be measured by a Vector Network Analyzer (VNA) and then converted to impedance form as illustrated later in this section. The scattering matrix

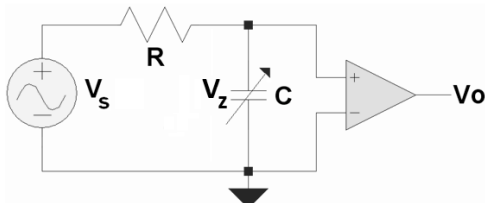


Figure 1. Hybrid voltage divider to detect and measure variations of the interdigitated capacitive electrode at C

[S] is defined in terms of incident and reflected voltage signals as follows:

$$\begin{bmatrix} V_1^- \\ V_2^- \end{bmatrix} = \begin{bmatrix} S_{11} & S_{12} \\ S_{21} & S_{22} \end{bmatrix} \begin{bmatrix} V_1^+ \\ V_2^+ \end{bmatrix} \quad (1)$$

Where V_i^- represents the amplitude of the voltage wave reflected from port i, V_i^+ represents the amplitude of the voltage wave incident to port i, and the specific elements of the S-Matrix are defined as follows.

$$S_{ij} = \left. \frac{V_i^-}{V_j^+} \right|_{V_k^+ = 0 \text{ for } k \neq j} \quad (2)$$

Which means that S_{ij} is determined by driving port j using incident signal wave V_j^+ and measuring the reflected wave amplitude V_i^- at output of port i. Thus, S_{ii} describes the reflection S-parameter seen looking into port i when the other port is terminated in matching load (usually 50 Ω) and S_{ij} describes the reflection S-parameter from port j to port i when the other port is terminated in matching load (usually 50 Ω)

A transformation of the S-Matrix model is convenient to illustrate an impedance model. The impedance model is expressed from the transformation from currents to voltages as follows:

$$\begin{bmatrix} V_1 \\ V_2 \end{bmatrix} = \begin{bmatrix} Z_{11} & Z_{12} \\ Z_{21} & Z_{22} \end{bmatrix} \begin{bmatrix} I_1 \\ I_2 \end{bmatrix} \quad (3)$$

Where Z_{11} is the input impedance, defined as the ratio of voltage V_1 to current I_1 measured at port 1 with open output port 2 or $I_2=0$ (forward measurement), Z_{21} is the forward transfer impedance, defined as the ratio of voltage V_2 to current I_1 measured with open output port 2 or $I_2=0$ (forward measurement), Z_{12} is the reverse transfer impedance, defined as the ratio of voltage V_1 to current I_2 measured with open input port 1 or $I_1=0$ (reverse measurement), and Z_{22} is the output impedance, defined as the ratio of voltage V_2 to current I_2 measured at port 2 with open input port 1 or $I_1=0$ (reverse measurement).

IV. COMSOL CAPACITANCE MODELING

Using the simulation software Comsol Multiphysics, the proposed interdigitated capacitor is modeled and simulated to obtain the capacitance. The proposed model contains 50 electrodes equally spaced between them with a length, width and height of 50.292mm, 0.508mm and 0.035mm respectively. The electrodes are separated in two groups of 25 that are connected through the same node with

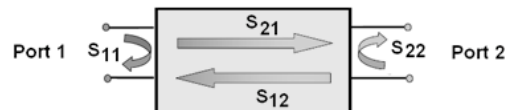


Figure 2. Two port network for a simple electrode structure showing S-Matrix elements

dimensions of 50.292mm by 2.54mm, forming an array where each electrode is equally spaced a distance of 0.508mm with respect to the neighboring electrode and a separation of 1.016mm between electrodes arrays (see Fig. 3).

After the design of the electrode we consider the materials that will be used for the prototype and testing purposes. The prototype will be done on a printed circuit board (PCB) with FR4 epoxy ($\epsilon_r=4.5$) as the dielectric. For the simulations we consider two active capacitances taking place over the prototype, the capacitance of the FR4 and the capacitance of the surface where the biological samples will be measured. For the surface capacitance, air and bi-distilled water will be used to compare the change in capacitance. Results from the simulations are showed in Table I.

With these results we see that the total capacitance in air for the prototype would be 64.42 pF while the same capacitor with bi distilled water over the surface would be of 959 pF.

V. VALIDATION OF PROPOSED MODEL

Fabrication of the prototype is done using standard PCB and a 50 Ω track and SMA connector is added for connection to the VNA. Results obtained from the VNA are in the form of Z-parameters that we use to convert them to a T- Π circuit like is represented in Fig. 4. Results from the VNA are presented in Table II with the equivalent impedances for the Π circuit at 500 kHz.

If we use the Π circuit, we can relate the impedance ZC to the capacitance of the electrodes. Calculating the capacitance from the imaginary part of ZC we obtain results for bi-distilled water of 803.81 pF and for air is 36.17 pF. We see that the results obtained from tests with the HVD are comparable to the results obtained from the Π circuit obtained through the VNA. The methodology for the hybrid voltage divider follows the test of the components of the circuit; from the probes of the oscilloscope to the complete circuit as is showed in Fig. 1. Calibration of a function generator to a sine wave of 500 kHz and amplitude of 5 Volts peak to peak is done for comparison purposes with results from the VNA.

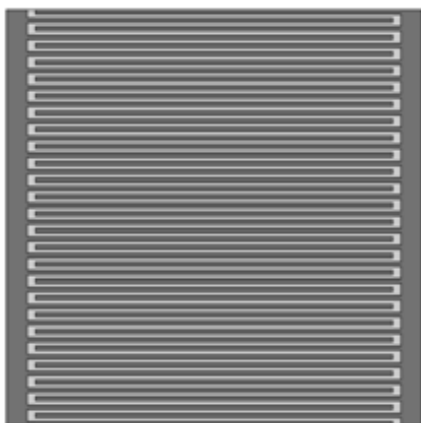


Figure 3. Interdigitated capacitor model

Table I. Capacitances by material

MATERIAL	CAPACITANCE
FR4	53.09 pF
AIR	11.38 pF
BI-DISTILLED WATER	905.5 pF

Table II. Parameters for Π circuit by VNA

Parameter	Air	Bi-distilled Water
ZA	2.50 -j6.25 k Ω	1.97 -j7.2 k Ω
ZB	15.5 -j17.6 k Ω	8.3 -j35.26 k Ω
ZC	0.364 -j8.88 k Ω	0.0698 -j0.396 k Ω

A first measurement is done over the oscilloscope probes to see the capacitance contribution from them into the measurement of capacitance from the electrodes. After the measurement of the oscilloscope probes, the electrodes are connected to the circuit and new measurements are done with Air and Bi-Distilled water.

From the measurements, if we remove the capacitance of the oscilloscope probes we would see that the capacitance of the electrodes in air correspond to 58 pF and in bi-distilled water goes from 840 pF to 866 pF depending of the resistor used. We see that results from the VNA and hybrid voltage divider are comparable between them as we see in Table III.

VI. RESULTS AND DISCUSSION

From using the HVD methodology with the electrodes, a test using 0.5 ml of laccase enzyme from *Pycnoporus sanguineus* fungus on a concentration of 8 U/ml is diluted on a buffer of 7 ml of bi-distilled water and the change in capacitance measured, after that a 0.5 ml solution of ABTS with a concentration of 5 mMol is mixed with the bi-distilled water. During the next 10 minutes an oxidation reaction occurs where the ABTS loses an electron forming the radical ABTS+ which are observed in the solution as a change in color of the buffer from colorless to an intense green (darker electrode) as seen on Fig. 5. After 10 minutes measurements in the electrodes detects any change in capacitance product of the oxidation reaction. Results from these experiments are showed on Table IV.

A second test with 7 ml of phosphorus buffer at 50 mMol replacing the bi-distilled water is proposed to compare the results from the measurements. This second test uses the

Table III. Comparison of sensor capacitance using both the VNA and the HVD methods

	VNA S1 (PF)	VNA S2 (PF)	VNA ZC (PF)	HVD (PF)
AIR	46.343	83.255	35.809	58
BI-DISTILLED WATER	876.92	917.41	803.71	853

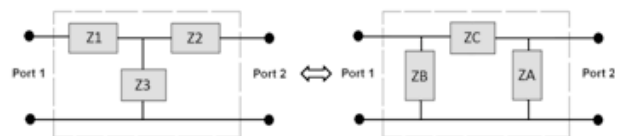


Figure 4. Model transformation from T to Π using the VNA methodology

Table IV. Capacitive sensor characterization using the HVD method

	RESISTANCE (OHMS)	FREQUENCY (KHERTZ)	VA (VOLTS)	Vz (VOLTS)	PHASE (NSECONDS)	CALCULATED CAPACITANCE (NFARADS)
LACCASE (BI-DISTILLED WATER)	330	500	4.56	1.1	92	8.36
LACCASE + ABTS (BI-DISTILLED WATER)	330	500	4.48	0.538	44	45.5
PHOSPHORUS BUFFER	330	500	4.48	0.504	44	49.1
LACCASE (PHOSPHORUS BUFFER)	330	500	4.4	0.432	36	70.9
LACCASE + ABTS (PHOSPHORUS BUFFER)	330	500	4.4	0.328	20	177

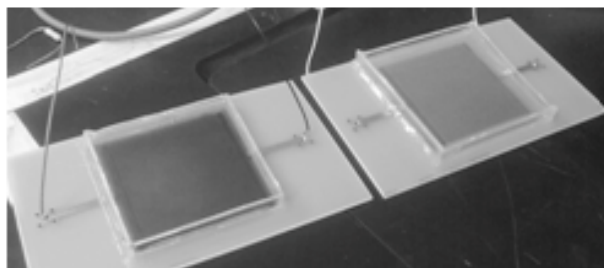


Figure 5. Experimental set ups for the interdigitated capacitive sensor devices (right electrode is before the oxidation reaction and left one is after the oxidation reaction)

same amount of laccase and ABTS and same 10 minutes to take measurements after the ABTS is mixed in the buffer. The results obtained using Phosphorous buffers are also included on Table IV. From the results obtained we see that an increase on capacitance can be measured in each step of the tests and this difference is big enough to be detected using the HVD method. The reaction between laccase and ABTS is not measured directly in these experiments due to the lack of immobilization of the laccase over the electrodes. This research aims to measure this reaction on future experiments with new and smaller electrodes and new methodologies like the immobilization techniques for laccase for more precise results in water monitoring.

VII. CONCLUSIONS

Changes in capacitance due to physical properties of materials and reactions are measurable using a HVD scheme or a VNA in conjunct with interdigitated electrodes. Differences in the capacitance between simulations in COMSOL and results from VNA and HVD are due to non ideal conditions in the fabrication of the electrodes and losses not considered on the simulation but results are close to a 10% of difference. Also results between VNA and HDV differ due the use of an external circuit for the use of a HDV methodology and use of commercial resistors and protoboard which add some interference. Measurements in the presence of laccase and ABTS indicate that the system is sensitive enough to detect the introduction of these substances and their reaction indicating that the HVD methodology works in these conditions.

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