Electrode Polarization Impedance in Weak NaCl Aqueous Solutions

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Abstract—In this paper, we characterize the polarization impedance behavior of several common metals in diluted NaCl solution operated at low current densities. The objective was to provide a useful reference for those wishing to calculate the electrode polarization impedance in diluted NaCl solutions. Serial equivalent resistance (R) and capacitance (C) for silver, aluminum, gold, platinum, and medical stainless-steel were measured as a function of frequency (10^{-2}–10^3 Hz) and NaCl concentration (2.4–77.0 mmol/L). The ratio of electrode polarization impedance with respect to the bulk resistance was calculated and plotted against concentration for each metal. Such a ratio shows the effect of the electrode polarization contribution as a function of electrolyte concentration when the bulk resistance of the solution changes. All metals showed a decrease of serial resistance R_p and capacitance C_p as a function of frequency. The medical stainless-steel electrode showed largest impedance values at lower frequencies compared to the other electrodes, and was concentration independent at all frequencies. Aluminum had smallest polarization impedance at low frequencies. Pure gold and platinum behaved similar with the exception that the serial resistance for gold showed a lower value at higher frequencies.

Index Terms—Chemical sensors, diluted solutions, electrode polarization, polarization resistance and capacitance.

I. INTRODUCTION

APPLICATION of metal electrodes in medicine varies from the recording bioelectric events and stimulating excitable tissues to electrochemical sensors. Despite the widespread use of electrodes, few have examined the electrical properties of the electrodes in diluted solutions with low voltage and frequencies. An example for such application is presented in a previous publication for measuring PCO_2 with conductivity measurement [1]. Since the CO_2 changes the conductivity of water by the dissociation of water into protons and bicarbonates, the conductance change can be measured by a voltameteric method as a measure of the PCO_2 changes in tissue. In spite of the simple measurements technique, one of the problems with the voltameteric method is the contribution of electrode polarization impedance, which is physically in series with the bulk resistance of the electrolyte. The problem would be eliminated when a four-electrode arrangement is applied for impedance measurements [2]. However, the disadvantage of the method for such an application would be an increased of sensor size and complicated design.

The behavior of electrode polarization for different electrode materials has been investigated earlier [3]–[5]. Specially, Gabriel [6] who reported impedance measurements on diluted electrolytes that were applied for correction of impedance of tissue. However, the experiment was focused on sputtered platinum electrodes, and the lowest selected frequency range was 10 Hz.

The contact area of the electrodes with electrolyte is playing a role on the electrode polarization impedance, as the electrode polarization impedance is inversely proportional with electrode surface area. Therefore, it is important to design the measurements cell with consideration of keeping the electrode area constant during the measurements. If the liquid penetrates the sidewall between the electrodes and the measurements chamber, this will result in an increasing electrode area that will change the impedance within time. We term this problem as "electrolyte seepage" in this paper. Another source of error is the sharp edges in the electrode geometry. Especially for wire geometries, the sharp edges of the electrode tip have a higher current density than the rest of the electrode area. An uneven current density distribution may affect the linearity behavior of the system [1], with earlier local onset of nonlinearity. The previous designs may have not considered the electrolyte seepage [7]–[9] or uneven current distribution problem [6].

By molding the electrodes in silicone, it is possible to have a constant geometry and avoid the electrolyte seepage because of the hydrophobic behavior of silicone. In addition, the silicone covers the edges of the electrode surface and eliminates uneven current density distribution. The disadvantage of such a method may be the error in geometry due to machining of the mold and shrinkage of the silicone material. If the procedures are done correctly, the error will be minimized to less than 2% of the original dimension [10].

The purpose of this study was to examine the behavior of the electrode polarization impedance of aluminum, gold, platinum, silver, and medical stainless-steel in diluted NaCl in the concentration range of 2.5–70.0 mmol/L, and frequency range 10^{-2}–10^3 Hz. By applying a new method to design the electrodes, we could keep the surface area between the electrode and electrolyte constant. The ratio of electrode polarization impedance and bulk resistance is presented to show the influence of electrode polarization impedance as a function of electrolyte concentration. The results of this study may provide quantitative data on the electrical properties of typical electrode materials used for recording, and chemical sensors in diluted solutions over a broad frequency range.

II. THEORY

When a metal is in contact with an electrolyte, a so-called dc boundary potential (V_0) will be generated in the electrode/electrolyte interface [11]. If another electrode is added to the system and an ac current (i) passes through the electrode/electrolyte interface, the boundary potential will be modulated by an alternating voltage (V).

The total measured impedance $(Z_{measured})$ which is the ratio of the alternating voltage and current may be

$$Z_{measured} = R_{bulk} + 2 \cdot Z_P \qquad (1)$$

where R_{bulk} is the bulk resistance and Z_p is the polarization impedance, respectively.

Further, the electrode polarization impedance can be presented by a series capacitance C_p and resistance R_p where the components are frequency and current density dependent [9]

$$Z_P = R_P + \frac{1}{j\omega \cdot C_P}. \qquad (2)$$

In addition to frequency and current density, both R_p and C_p are also dependent on the geometry of the electrode/electrolyte interface, electrode material, and electrolyte concentration [2].

III. MATERIALS AND METHODS

A. Electrode Material and Preparation

We selected the most common electrode materials used in biomedical application such as pure platinum, gold, silver, and two alloys: aluminum (AW 6060 T6), and medical stainless-steel (W.nr. 1.4044) for this investigation. The gold, silver, and platinum had impurity less than 1 ppm (Rasmussen AS, Norway). The electrodes had a shape of a disc with a diameter of 4 mm and a thickness of 1.5 mm before being processed.

Two grinding stages were applied on all electrodes: plane and fine grinding. At the plane-grinding step, SiC paper with grit 500 (Struers, Denmark) was applied to make sure that all electrodes were at the surface level with the brass electrode holder. During the grinding, de-ionized water was applied as a cooling agent. After a satisfactory result of the plane grinding, a fine grinding process was pursued. The fine grinding process was further divided in three stages with progressively finer SiC paper with grit 800, 1200, and 2400, respectively. When the fine grinding process was finished, the electrodes were slow-speed polished with a fine cloth. The electrodes were then cleansed with Triton X-100 (Wacker industries, Adrian, MI) and de-ionized water to remove any chemical rests or biological materials. Subsequently, the surface of each electrode was inspected under microscope (Olympus, SZx9) with a magnification of 100X for plainness and edge retention. The grinding processes were repeated when the grooves were larger than 10 μm in dimension.

B. Measuring Cell

To make the geometry of the electrodes in contact with electrolytes constant, the disk shaped electrodes were molded in silicone with a predefined circular open area in the mold. As the silicone was hydrophobic and could be attached to the electrode surface, the electrolyte would not penetrate to the nondefined area of the electrodes, and the electrolyte seepage would be eliminated.
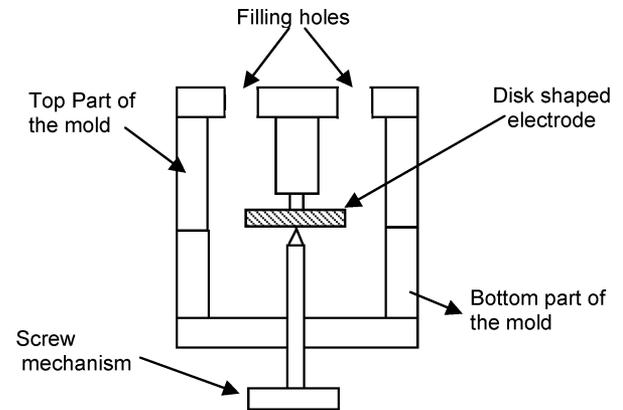

Fig. 1. The cylindrical shaped negative mold for the fabrication of the casting of each electrode in silicone.
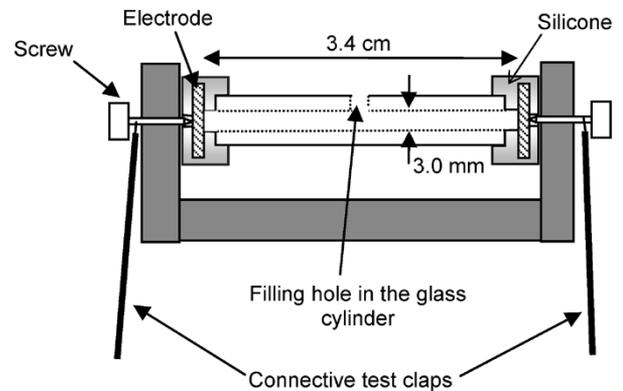

Fig. 2. The assembled measuring cell with connective test clamps for the instrumentation leads.

We fabricated a brass mold by a conventional milling process. The mold was made in two halves and screwed together. Inside the mold, a screw from the bottom part could hold the electrode horizontally during the molding process (Fig. 1). The pin inside the mold could keep the silicon away and make an exposed electrode area. The exposed electrode area was the same as the inner area of the glass tube, which was 0.07 cm^2.

Two component Elastosil RT601 (Wacker-Chemie GmbH, Münich, Germany) was mixed according to the instruction from the manufacturer. The mixed substance was centrifuged to remove any remaining bubbles produced during the mixing process. The bubble free silicone liquid was then carefully inserted into the mold with a small syringe. The casting process was carried out by placing the mold inside an air heater oven for about 1 hour at 70 °C. The demolded part was then kept for another 5 hours at 70 °C, to make sure that the silicone was vulcanized and maximum mechanical stability gained.

A hollow cylinder was made of glass with dimensions of 3-mm inner diameter, 8-mm outer diameter, and 3.4-cm length, corresponding to a cell constant of 48.6 cm^{-1}. In the middle of the glass cylinder, a 0.5-mm small hole was made for filling the measurement cell (Fig. 2). The doughnut shaped electrode parts could be placed on each side of the glass cylinder making the electrodes stand in parallel. A U-shape holder was constructed for supporting the glass cylinder and the molded parts. The U-shape holder had two screws on each end to hold the electrodes tight toward the end of the glass cylinder to avoid

any leakage, but they also made the electrical connection to the electrodes for further connecting to the measuring instruments (Fig. 2). Electronic test clamps were applied to connect the screws further to the instrument cables.

C. Preparation of the Electrolyte

Because the measurements were performed at low frequencies and the instrumentation had to be adjusted with large time constants, a NaCl solution was chosen. The NaCl solution is more stable than for example solution with dissolved CO_2 especially in such small volumes.

Saline 0.9% solution was diluted in de-ionized water to achieve lower concentrations, which was in the range of 2.4–77.0 mmol/L. Each of the concentrations were tested with a blood gas machine (ABL650, Radiometer AS, Denmark) to make sure that the pH value was within the range of 6.5 ± 0.5. The measuring cell was filled with each electrolyte through the insertion hole and made sure that no bubble was left in the measuring cell. A plastic foil was then applied to cover the measuring cell in order to slow down the evaporation of the electrolyte and keep the temperature constant. The lowest concentration was applied first and gradually changed to higher concentrations. Between each measurement, the measuring cell was cleaned with de-ionized water and dried carefully. After the completion of the filling procedure, measurements were postponed by 30 minutes allowing the electrode/electrolyte interface to reach equilibrium.

D. Instrumentation Setup

An impedance/gain-phase analyzer system (model 1260 + 1294 Solatron Instruments) was applied to perform the measurements. Open-ended coaxial cables (MIL-C-17-F) were used for all connections. Since the onset of nonlinearity appears virtually independent of frequency at an ac signal amplitude of about 100 mV [12], an amplitude of 50 mV was chosen for the impedance measurements.

From reported experiments and investigations [13], the electrode polarization impedance is minimized at higher frequencies. The stray capacitance of the empty cell connected to the instruments was about 10 pF, corresponding to a reactance of 1.6 MΩ at 10 kHz. The value of the bulk resistance for the 2.4 mmol/L concentrations was about 0.5 MΩ, which is 1/3 of the stray reactance value. The impedance for the medical grade stainless-steel showed high impedance values that made a noticeable influence on the stray capacitance over 1 kHz. Therefore, the upper limit for the analyzed frequency range was chosen to be 1 kHz.

Frequencies below 10 mHz were not favored since the measuring time constant had to be several hours to get stable measured values. Consequently, the vaporization of the solution would influence the measurements for such low-frequency measurements. The analyzed frequency was, therefore, selected between 10 mHz and 1 kHz.

A temperature sensor was glued to the side of the glass cylinder to continuously monitor the temperature. Silicone becomes soft at higher temperatures and the chance for electrolyte seepage may increase. Therefore, a controlled room temperature $(23 \pm 0.5\,^\circ\mathrm{C})$ was chosen.
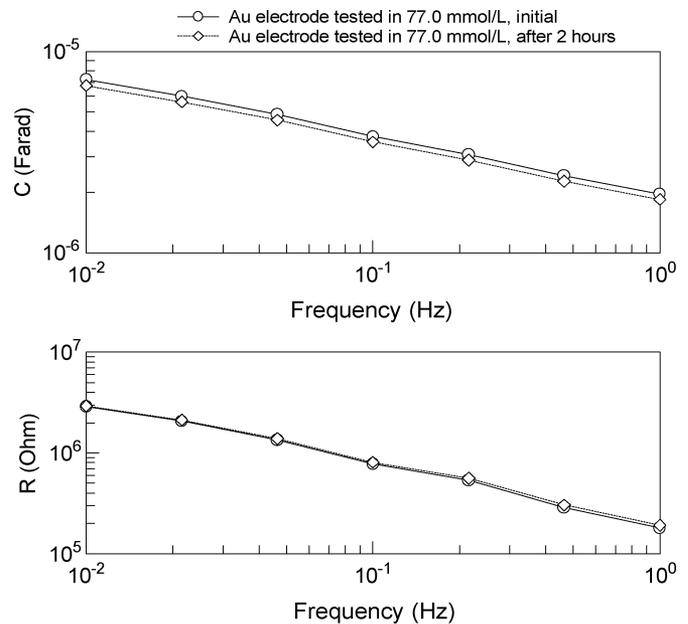

Fig. 3. Polarization impedance of Au electrode in 77.0 mmol/L for the control of electrolyte seepage within a two hours frame.

The impedance/gain-phase analyzer system was connected to a PC to collect the data by Solatron Impedance Measurements Software (Solatron Ltd) automatically. The data were later analyzed using the Zview software package (Scribner Associates Inc., Southern Pines, NC).

IV. RESULTS

To verify that the electrolyte seepage problem was eliminated, all electrodes were tested in 77.0 mmol/L measured at the frequency range of 0.01–1 Hz within a period of two hours. As an example, the result for gold electrode is shown in Fig. 3 as a parallel resistance and capacitances in order to show any changes due to the electrolyte seepage problem. All electrodes showed a decrease of the parallel capacitance within a range of 5%–10% of their initial values and the parallel resistance was either unchanged or increased within 10% of the initial value over the period of two hours. For longer periods than two hours, the electrolyte would evaporate out of the measurements chamber and may change the bulk resistance of the electrolyte. Thus, a measurement longer than 2 hour with our measurements cell was not feasible.

As explained in (1), the measured impedance results include both the impedance of the electrolyte and the electrode polarization impedance. At the higher frequencies, the bulk resistance of the electrolyte may dominate the total impedance measurements. Thus, one can obtain the electrode polarization impedance by measuring the impedance at a high frequency and subtracting it from the measured impedance at lower frequencies. The method is suggested by Schwan [7], and Ragheb and Geddes [5], however, they did not discuss the influence of stray capacitance with such a method. In order to minimize the error contribution from the stray capacitance, we calculated the mean value between the impedance at high frequency and the theoretical bulk resistance. As an example, for a concentration
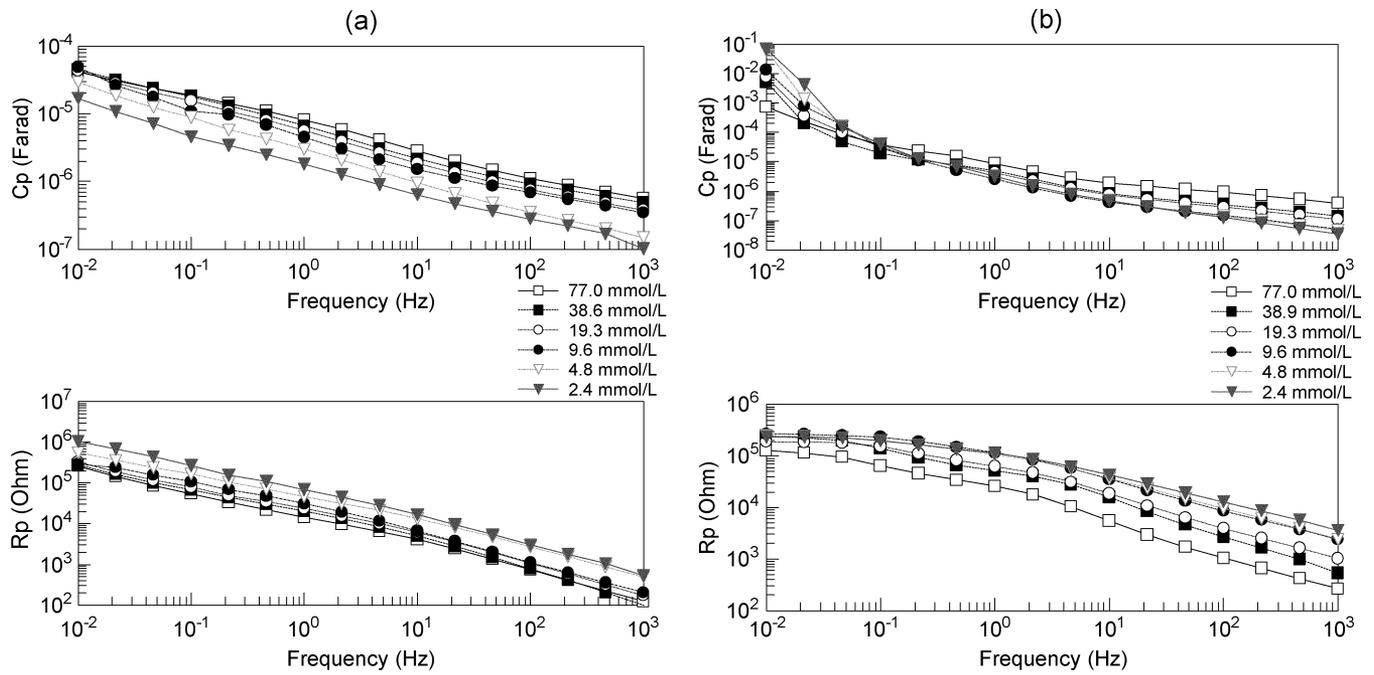
Fig. 4. Polarization impedance for (a) one silver and (b) aluminum electrode surface ($0.07 \ \mathrm{cm}^2$) in 2–77 mmol/L NaCl solutions.
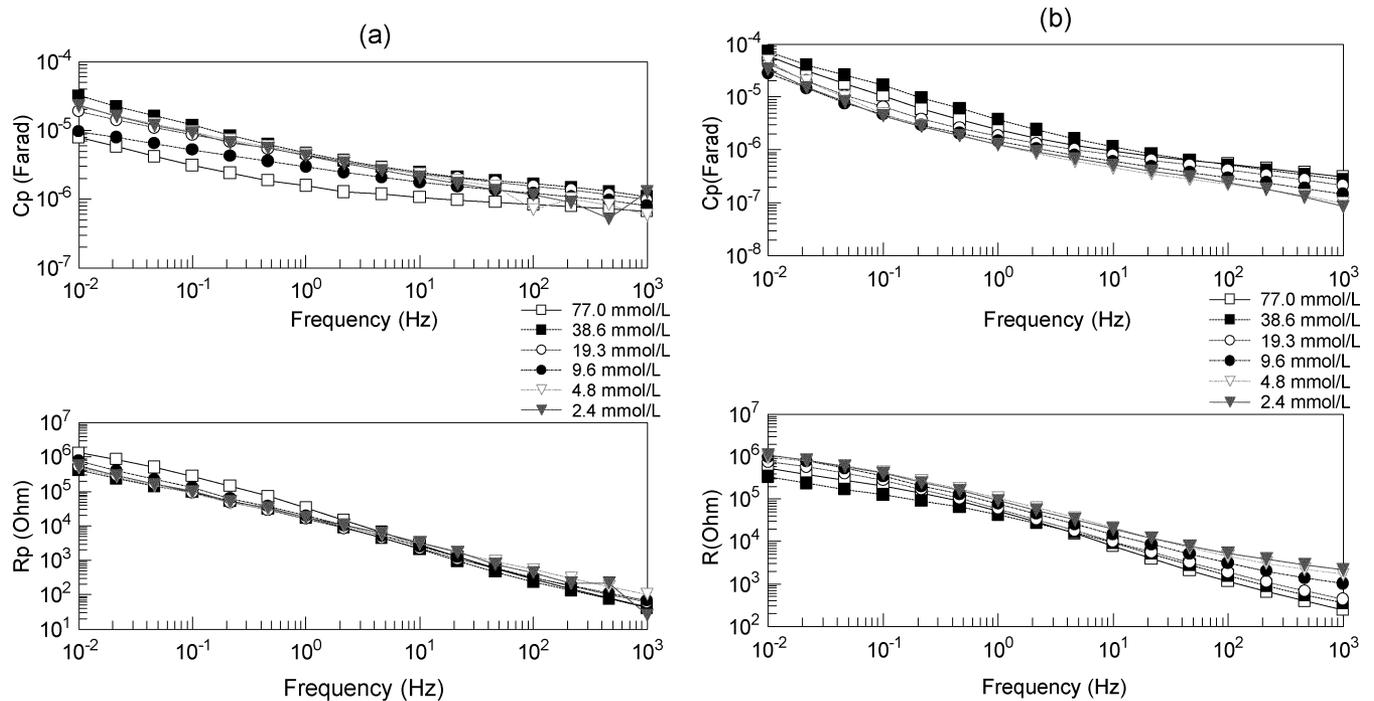

Fig. 5. Polarization impedance for one (a) gold, and (b) platinum electrode surface ($0.07 \ \mathrm{cm}^2$) in 2–77 mmol/L NaCl solutions.

of 77 mmol/L, a conductivity of 8.4 mS/cm was measured by a conventional conductance meter (WTW Cond 340i), and with a cell constant of $48.6 \ \mathrm{cm}^{-1}$, we calculated a bulk resistance of 5.8 kΩ. The mean value of the bulk resistance and the measured impedance at the highest frequency for platinum was about 5.7 kΩ. This value was then subtracted from the total measured impedance to achieve the electrode polarization impedance. The same procedure was applied for all metals and electrolyte concentrations.

To obtain the effect for one electrode surface according to (1), we divided the polarization impedance by the factor of two. The impedance values for all metals were then presented in series resistance R_p and capacitance C_p by (2). Fig. 4(a) shows the logarithmical value of R_p and C_p for silver (Ag) as the function of log frequency for the varied concentrations. Fig. 4(b) shows the results for aluminum (Al). The results for gold (Au), platinum (Pt) and medical stainless-steel are shown in Figs. 5(a), (b), and 6 in double logarithmical axis.
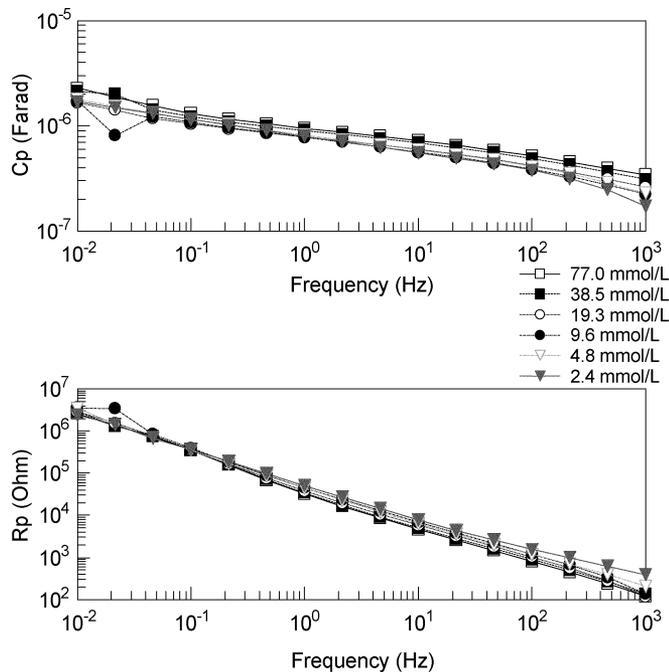
Fig. 6. Polarization impedance for one medical stainless-steel electrode surface (0.07 cm^2) in 2–77 mmol/L NaCl solutions.

Both R_p and C_p decreased with an increased frequency. The medical stainless-steel showed the largest serial resistance of about 2000 kΩ for almost all examined concentration at the frequency of 10^{-2} Hz, while aluminum showed a change between 150–300 kΩ with the same conditions. For the silver, gold and platinum the measurements at the same frequency showed a change in serial resistance between 200–1000 kΩ, 400–1500 kΩ, and 300–1000 kΩ, respectively.

At the higher frequencies as 1 kHz, the serial resistance was largest for aluminum, which changed from 200 Ω to 40 kΩ. Platinum showed the next largest changed of serial resistance with 200 Ω to 20 kΩ. Silver and stainless-steel showed changes of about 100–500 Ω, while gold had the minimal values and only varied between 20 and 70 Ω. The serial capacitance C_p for all metals decreased with the lower concentrations with the exception of the anomalous behavior of aluminum. The serial capacitance for aluminum was largest and changed between 900–10 000 μF at 10^{-2} Hz. At the same frequency silver, gold, and platinum had a serial capacitance change between 20–50 μF, 8–30 μF, and 20–80 μF, respectively. The value for serial capacitance for stainless-steel was around 2.0 μF showing concentration independency. At 1 kHz aluminum had the largest scattering of serial capacitance for different concentrations that was in the range of 0.04–0.6 μF, while silver had a change from 0.1–0.9 μF at the same frequency. Platinum had a serial capacitance of 0.1–0.4 μF, while gold showed values between 0.8–2 μF. Stainless-steel showed the lowest change of serial capacitance which varied between 0.2–0.7 μF. Both gold and stainless-steel showed less concentration variation at the higher frequency ranges from 100–1000 Hz.

The ratio of the impedance module of electrode polarization to bulk resistance for different frequencies was calculated and plotted as a function of the concentration. Figs. 7, 8, and 9 show the results for all investigated metals. The ratio for stainless-steel increased with increased concentration as a straight line, while for aluminum the ratio was increased first and from 10 mmol/L it changed less than 1% of its total change. The ratio for silver and platinum were similar for all concentrations. For gold, the ratio was changing as a liner line at concentration above 10 mmol/L while at lower concentrations it was less changes indicating larger influence from the polarization impedance.

V. DISCUSSIONS

A. Polarization Impedance Contribution

The Fig. 3 and the observation for the testing of other electrodes over time showed a decrease of parallel capacitance and an unchanged/or increased parallel resistance of electrode polarization in the frequency range of 0.02–1 Hz. If the electrolyte seepage problem were influencing the measurements, the capacitance would increase due to an increased area especially at such a low-frequency range. However, since the capacitance decreased due to instability of the system and the electrolyte seepage problem will not be influencing the measurements within 2 hours. Since our measurements are within the range of 1 hour, the polarization impedance measurements would be influenced by neither the electrolyte seepage nor the evaporation of the liquid.

The results showed for all metals that the electrode serial resistance increased in more diluted concentrations. The stainless-steel had the largest R_p in this study. Geddes *et al.* [7] has reported an R_p of about 42 Ω cm^2 at 100 Hz. The current density of their measurements was 0.025 mA (rms) cm^{-2} in physiological saline (154 mmol/L), while the electrode area was assumed to be 4.47 mm in diameter corresponding to an electrode area of 0.06. In our measurements, the highest current density at 100 Hz would be about 0.015 mA (rms) cm^{-2} for a concentration of 77.0 mmol/L with 50 mV rms and an electrode area of 0.07 cm^2. Our data has R_p for medical stainless-steel of about 56.7 Ω cm^2, for a concentration of 77.0 mmol/L at 100 Hz, and the extrapolated value for 154 mmol/L was found to be 45.5 Ω cm^2 at 100 Hz (see Fig. 6). The extrapolated value for R_p for 154 mmol/L (650 Ω) correlated well with Geddes *et al.* measurements (700 Ω).

Geddes *et al.* measurements showed a serial capacitance for saline solution of about 50 μF/cm^2 at 100 Hz. The C_p value based on data from Fig. 6, gave a C_p of 7 μF/cm^2 for the concentration of 77.0 mmol/L at 100 Hz. The extrapolated value for the C_p at 154 mmol/L would then be about 8.6 μF/cm^2. The electrodes in the Geddes *et al.* experiment were polished with emery papers, which may have affected the results for their serial capacitance values. Unfortunately the Geddes *et al.* investigation did not measure impedances under 30 Hz making any further comparison difficult.

The measurements of Onaral and Schwan [12], showed a R_p of 8.6 kΩ cm^2 and C_p 234 μF/cm^2 for platinum at 10 mHz for physiological saline with an electrode area of 0.0855 cm^2. The current density in that study was not mentioned but the ac potential was 65 mV. The R_p from our measurements was 28 kΩ cm^2, and C_p 614 μF/cm^2 at the concentration 77.0 mmol/L. The extrapolated value from our measurements for 154 mmol/L was 27 kΩ cm^2 and 571 μF/cm^2 for R_p and C_p at 10 mHz for an
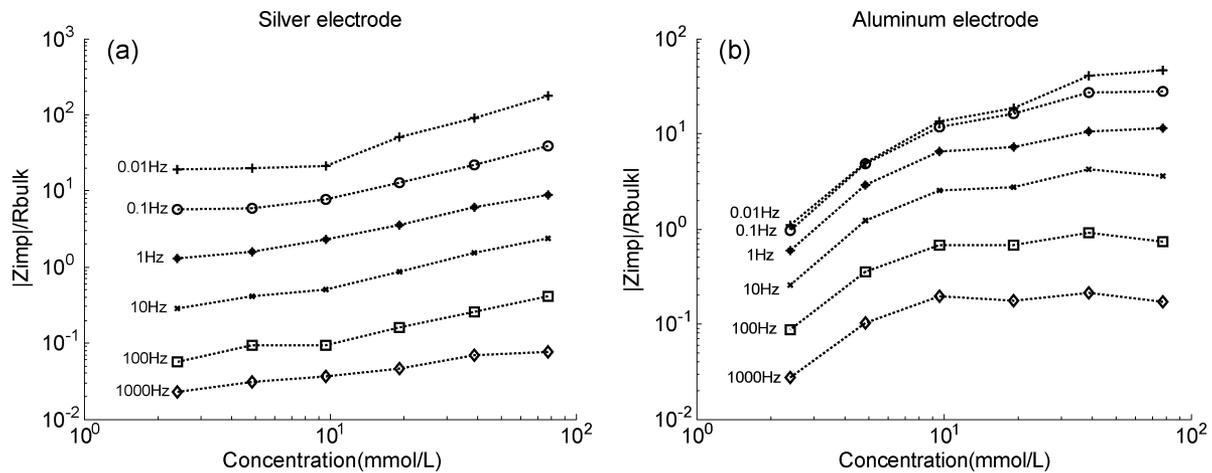
Fig. 7.　Ratio of impedance $|Z_{\mathrm{imp}}|$ and R_{bulk} as a function of concentration, for two (a) silver and (b) aluminum electrode surfaces.
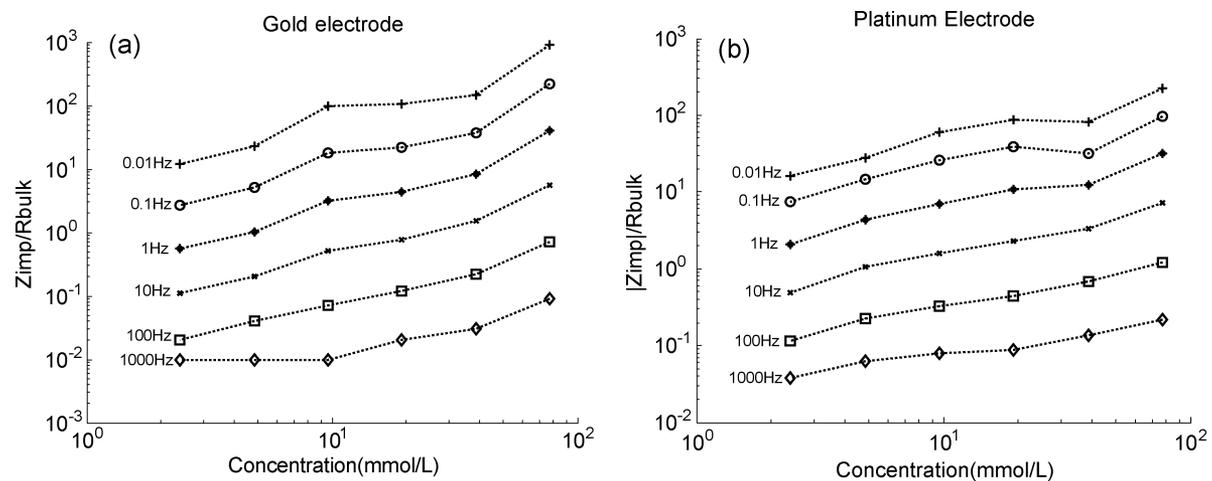

Fig. 8.　Ratio of impedance $|Z_{\mathrm{imp}}|$ and R_{bulk} as a function of concentration, for two (a) gold and (b) platinum electrode surfaces.
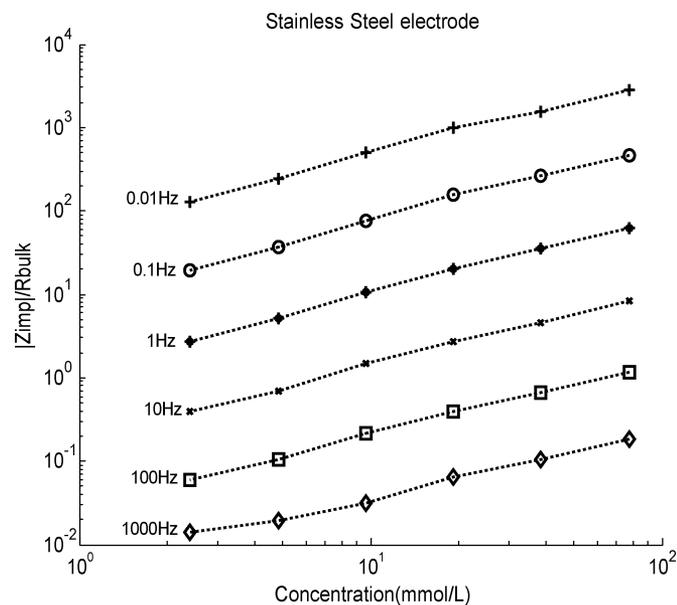

Fig. 9.　Ratio of impedance $|Z_{\mathrm{imp}}|$ and R_{bulk} as a function of concentration, two stainless-steel electrode surfaces.

electrode area of $0.07\ \mathrm{cm}^2$. In addition, the temperature of our experiment was two degrees lower than Onaral and Schwan's

reported measurements. By assuming that the temperature dependency was linear and about $2\%/^{\circ}\mathrm{C}$ for R_{p} and $-3\%/^{\circ}\mathrm{C}$ for C_{p} [14], we could expect that the values of Onaral and Schwan should be 8.6 kΩ, and $327\ \mu\mathrm{F}/\mathrm{cm}^2$ for R_{p} and C_{p} at the temperature of $23\ ^{\circ}\mathrm{C}$, respectively. Unfortunately, Onaral and Schwan have neither mentioned the purity of the platinum applied in their experiment, nor its surface roughness and how the surface of the electrode was treated making any further explanation of the difference between the measured values difficult.

The concentration independency of the electrode polarization impedance for stainless-steel makes the electrode as an ideal electrode for measurements in solution with different concentrations [15]. As a matter of fact, the material has been suggested for the application of dry electrode for biopontential recording [16]. Aluminum has been shown to have erratic measurements due to chemical response of its oxide to salt solutions and, therefore, may have low impedance at low frequencies [17], [18]. The ratio of polarization impedance and bulk resistance for aluminum showed a low value compared to other electrodes confirming a small influence of electrode polarization impedance. However, above 10 mmol/L the aluminum electrode showed small ratio changes indicating that the polarization impedance was more influent. The corrosive behavior of aluminum may change its impedance characteristic over time, which may not

preferred for a recording electrode or inside an electrochemical sensor such as the presented PCO_2 sensor in introduction. For silver, the effect of electrode polarization impedance showed its impact at concentrations lower than 10 mmol/L where the ratio plots were almost horizontal lines for all frequencies. Gold and platinum showed a similar ratio behavior. Nevertheless, the values for the gold at higher frequencies showed that gold had three times lower electrode polarization impedance than platinum at higher frequencies.

VI. Conclusion

The geometry of the electrode and the problem with respect to electrolyte seepage and homogeneous electrode geometry have to be considered when measuring impedance of a solution with voltameteric method with two electrodes. The polarization ratio was a ratio of polarization impedance and bulk resistance of the electrolyte. We found that the polarization ratio diminishes as a function of concentration in diluted NaCl solutions regardless of electrode material. The data for the medical stainless-steel behaved concentration independent and it had high electrode polarization impedance compared to other electrodes in our study. Aluminum showed small ratio changes for electrode polarization and bulk resistance at low concentrations; however, the influence of electrode polarization impedance was effective at higher concentrations. Gold, platinum, and silver showed a moderate concentration dependency for the electrode polarization contribution at low concentrations. For the application of a PCO_2 sensor, we could apply silver, gold, or platinum. The stainless-steel may not be applied due to its large electrode polarization contribution. The aluminum may not be preferred with respect to its time varied polarization impedance.

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