Single Cell Performances Based Glass Composite Membrane for Low Temperature H_2/O_2 Fuel Cells

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Abstract: Membrane electrode assemblies (MEAs) for a low temperature H_2/O_2 fuel cell were fabricated using glass composite membrane and Pt/C electrode were evaluated by various operating condition. The stability and durability of the cell and polarization characteristics of membrane electrode assembles (MEAs) were reported. Electrochemical performances on MEAs consist of PWA {(12-tungsto(VI) phosphoric acid, *n*-hydrate)}/P_2O_5(phosphoric acid)/SiO_2 (TEOS, tetraethoxysilane) glass composite membrane electrolyte and Pt/C electrode have been demonstrated representing a major milestone towards developing a viable atmospheric low temperature H_2/O_2 fuel cell system. MEAs were showed good performances under various functions of the temperature and relative humidity. A maximum current density of 141 mA/cm^2 was obtained at 35 °C with 30% relative humidity by using a PWA/P_2O_5/SiO_2 (5/5/90 mol%) glass composite membrane and Pt/C (0.1 mg/cm^2) electrode. Polarization curves were recorded and their results support the conclusions obtained from the electrochemical impedance spectroscopy (EIS).

Keywords: Glass, Composite, MEA, Pt/C electrode, Fuel cell, Low temperature.

1. INTRODUCTION

Fuel cells have been considered as promising alternative power sources due to the high conversion efficiency of the chemical energy of fuel to electrical energy with low emission of pollutants [1-3]. Fuel cells present a highly efficient and environmentally friendly alternative technology for decentralized energy production. Fuel cells offer the tantalizing promise of cleaner electricity with less impact on the environment than traditional energy conversion technologies. This is because fuel cells are direct electrochemical energy conversion devices. Proton exchange membrane fuel cells (PEMs) in general have a high power density, its ability to provide an ionic path for protons to travel from the anode to the cathode, moderately efficient in their conversion of chemical energy to electrical energy, low operating temperatures and low greenhouse gas emissions [4, 5]. Proton exchange membrane (PEM) fuel cells are one of two types of fuel cells being intensively investigated today because they have the potential to be versatile. Applications of PEM fuel cells are seen in electric utility, portable power, and transportation [6, 7]. The low operating temperature of a H_2/O_2 PEMFC has both advantages and disadvantages.

Low temperature operation is advantageous because the cell can start from ambient conditions,

quickly, especially when pure hydrogen fuel is available. It is a disadvantage in carbon monoxide containing fuel streams, because carbon will attack the platinum catalyst sites masking the catalyst activity and reducing cell performance [8]. The effect is reversible by temperatures must be greater than 120 °C, at which point there is a reduction in chemisorptions and electro-oxidation. The heart of fuel cell is a device involving multi-physics coupling phenomena: mass transport (in the electrodes and electrolyte), charge transport (in the electrolyte), and electrochemical kinetics (at reactive sites). Currently there are many types of electrolyte materials for application as PEMs. However, each material has specific limitations restricting its usefulness for application to fuel cells. Current densities should be higher than 100 mA/cm^2 electrode area to make the fuel cell economically attractive [9-11]. Current density is limited by both the electrolyte resistance and the catalytic efficiency of the electrode materials.

Proton conductors have a promising future due to unique advantage such as flexibility, high mechanical strength, thermal stability, excellent processibility and high proton conductivity. For instance, perfluoro-sulfonated ionomer (Nafion®) membranes have been used for this purpose, due to their efficient proton conduction (10^{-1} S/cm in their fully hydrated protonic form) and long lifetime [12-15]. Lower cost polymers with similar properties are strongly desired as alternative materials [16-20]. The proton conducting glass membrane was yielding high proton conductivities of 10^{-2}-10^{-1} S/cm at room temperature [21, 22]. A proton-conducting membrane is an integral

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part of a fuel cell system. Fuel cells offer the best alternative to conventional power generation technologies. They are inherently very efficient and clean. In our lab, electrochemical activity tests have been performed on a H_2/O_2 fuel cell fed with H_2 and O_2 at room temperature utilizing silica phosphate and heteropolyacid glass membranes [23]. Based on previous work, our interest is to fabricate the MEAs on cell performances operating by various operating conditions for H_2/O_2 fuel cell.

HPAs with the Keggin structure, and in particular the compounds $H_3PMo_{12}O_{40}$ and $H_3PMo_{12}O_{40}$ have received particular attention due to their acidic and redox properties, stability at elevated temperatures, commercial availability and relative ease of synthesis [24, 25]. HPAs are known proton conductors, which may be exploited in the design of the membrane electrode assembly (MEA). Depending on their chemical environment not all HPA loose all their bound water molecules below 100 °C, allowing the real possibility of proton conduction at elevated temperatures. These materials have even been investigated as possible electrocatalysts for fuel cell applications with limited success; they have been shown to increase the tolerance of a platinum containing anode to deactivation by carbon monoxide [26-28]. There are many HPAs that are easily synthesized that have not been investigated as PEM fuel cell electrocatalysis. This paper presents a more exhaust report on the glass composite membrane for low temperature fuel cell in order to define the MEA optimum operation and the effects of MEA preparation on the performance of a PEMFC. The objective of our research aim is to improve the cell performances by different operating conditions with glass composite membrane. The present study demonstrates the development of a H_2/O_2 fuel cell with a $PWA/P_2O_5/SiO_2$ glass composite electrolyte and a Pt/C electrode as well as its performance at low temperature.

2. EXPERIMENTAL SECTIONS

The $PWA/P_2O_5/SiO_2$ ((5/5/90 mol%) glass composite membrane preparation procedures were adapted from our previous work [23,29] and the preparation procedure of the catalyst layer was similar to our previous study [30]. Furthermore, PWA glasses [31] for H_2/O_2 fuel cell electrolytes were tested at room temperature and the proton conductivity of 10^{-2} magnitude order was obtained at 90 °C and 30% RH. Catalyst slurry ink composed of Pt/C (0.1 mg/cm²) powder, Nafion ionomer and polytetraflouroethylene

(PTFE) was sprayed onto carbon sheet as a catalyst layer. Here, used the glass composite and catalyst layer thicknesses are 0.7 and 0.20 mm. The MEA compartments were separated by the glass ceramic electrolyte, each face of which was covered with porous Pt/C electrode. The total thickness of MEA is 0.9 mm. The MEA were those of a typical fuel cell with a 0.75 cm² surface area (projected area). Each MEA compartment was sealed by a carbon cloth. The electrode was prepared with metal Pt/C (0.1 mg/cm²) loading at 100 °C. The cell temperatures were 35, 45, 55 °C, and humiliation level, 35, 45, 55%, respectively, controlled by NF−Electrochemical fuel cell evaluation system, Japan. The gas flow rates of 50 mL/min for oxygen to anode sides and 100 mL/min for hydrogen to cathode sides, respectively, 1 atmospheric pressure. It was controlled by special A520 software. After the cell was conditioned, it was put on open circuit voltage (OCV) with hydrogen and oxygen as feed gases on the anode and cathode sides, respectively. The OCV was monitored over the life of the cell during throughout the electrochemical measurement.

The catalyst layer consists of Pt/C phase, ionomer phase and gas pore phase. During fuel cell operation, O_2 reduction reaction occurs at the three-phase boundaries: where O_2 from gas pores, protons from ionomer and electron from Pt/C. The total voltage losses in the catalyst layer include kinetic loss, proton/electron transport loss and O_2 transport loss. All these lose are more or less dependent on the electrode composition and structure, such as Pt loading, Pt/C ratio, ionomer loading and electrode porosity.

The AC impedance spectra were monitored using a Solarton 1260 frequency-response analyzer, controlled by a Solarton 1287 electrochemical interface analyzer. The impedance spectra were measured in the constant voltage mode by sweeping frequencies over the 0.1 Hz to 1 MHz range, and recording 10 points/decade with voltage amplitude of 10 mV. The impedance data was carried out using the Solartron software, Z view. Prior to impedance measurements to determine the current corresponding to cell voltage, typically polarization curves were recorded. Each polarization curves were displayed for 1 hr time set after impedance spectra, the constancy of the I−V measured before and after each impedance measurements were taken as a criterion for the stability of the cell during the measurement. Two sets of polarization curves were recorded, one set is displayed as a function of temperature with constant relative humidity and another one was recorded as a function of humidity with constant temperature is 30 °C.

3. RESULTS AND DISCUSSION

Figure **1** (**a**, **b** and **c**) shows the result of the open circuit voltage test on PWA/P_2O_5/SiO_2 glass composite membrane operating at 30% relative humidity for various temperatures (35, 45 and 55 °C). Figure **1a**, the cell started the OCV at about 0.75 V and then reached to 1 V with 1 h time. The cell was operated in 6 h. After 6 h operation, the OCV of the cell was 0.95 V. By testing, all the temperatures, OCVs, above 0.9 V were obtained. Figure **1** (**b** and **c**) shows the similar trend, and they were confirmed the cell was constantly stable after 1 hr and shows good OCV for fuel cell test. The strength of MEA was very important to show the good performances in the H_2/O_2 fuel cell. Here we have run the OCV for 10 h at 55 °C, without any degradation, and recorded the high performances. Figure **2** (**a**, **b** and **c**) shows the OCV at 0.5 V for PWA/P_2O_5/SiO_2 glass membrane were operated at 30 °C with three humidity conditions (30, 40, 50%). For these three tests, the observation shows the OCV was stable and the trend was similar as Figure **1**. The operating time was 2, 3 and 5 h (Figure **2** (**a**, **b** and **c**)), the cell voltage were decreased after 1 h, so the experiments were stopped. However, the variation of cell voltage loss due to the ohmic resistance, that was recorded. From those observation on OCV, further can improved the cell voltage can be increased by changes of gas flow rates to the anode and cathode sections, respectively.
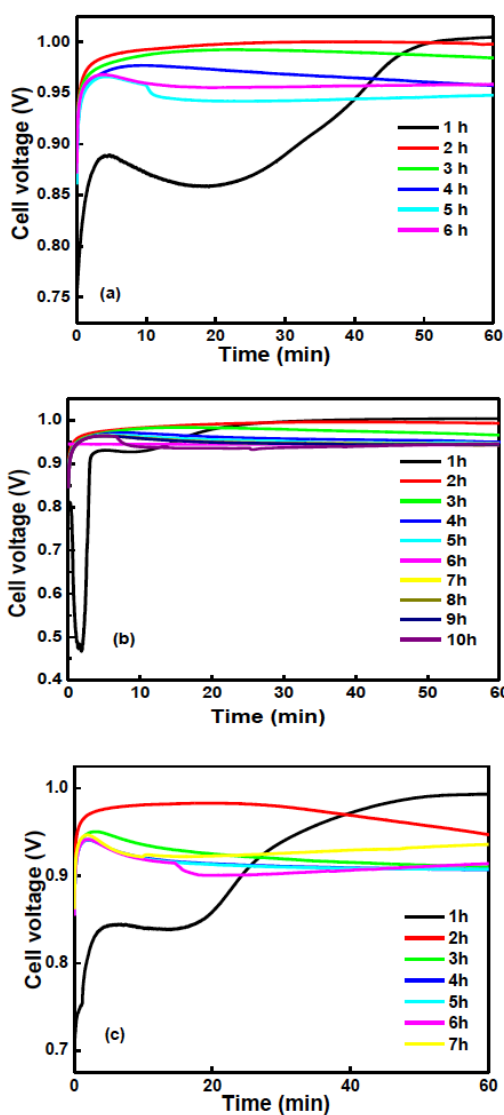

Figure 1: Time vs cell voltage curves for PWA/P_2O_5/SiO_2 glass composite membrane measured at various temperatures condition with 30% relative humidity: (**a**) 35, (**b**) 45 and (**c**) 55 °C.
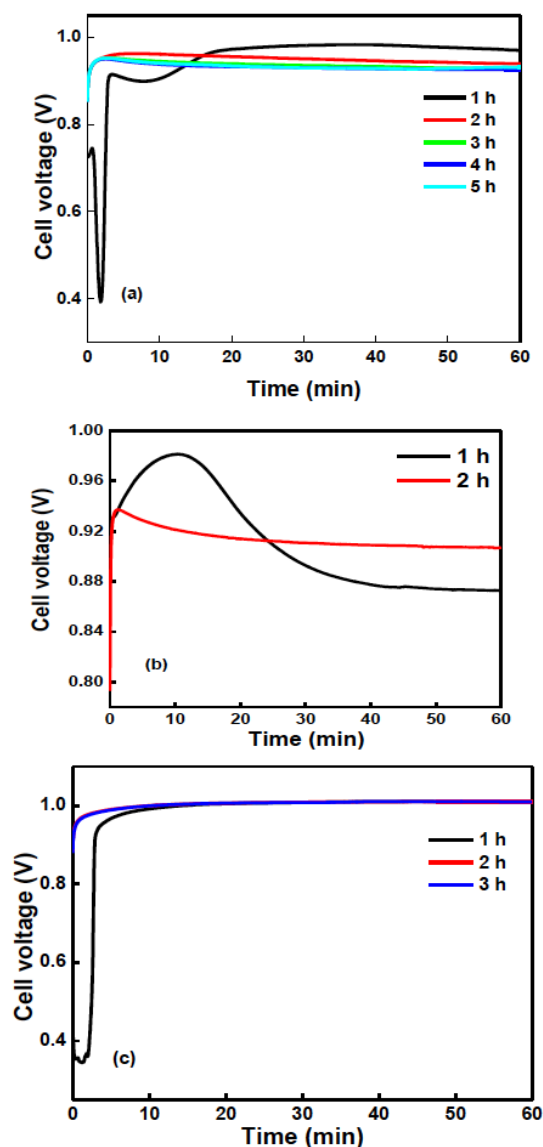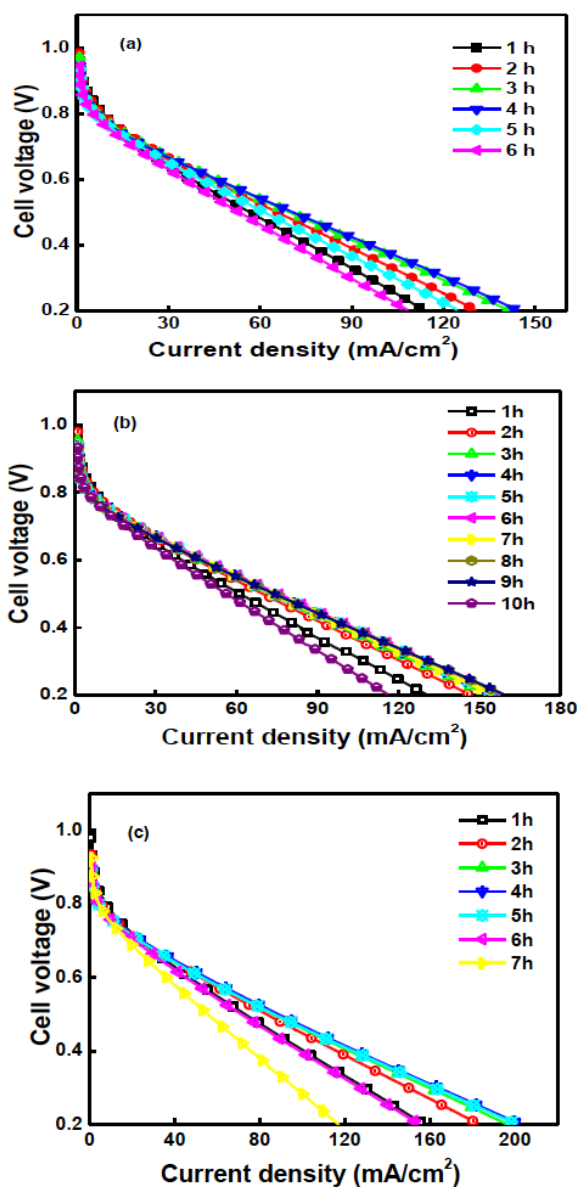
Figure 2: Time vs cell voltage curves for PWA/P_2O_5/SiO_2 glass composite membrane measured at various humidity condition with 30 °C. (**a**) 35, (**b**) 45 and (**c**) 55%.

Fuel cell tests were carried using the 0.9 mm thick MEA between 35 and 55 °C under constant humidified (30%) condition. The current-voltage (I-V) curves of the fuel cell are shown in Figure **3**. All the current-voltage slopes became lower as the operating time increased. The power density value of 38.5 mW/cm^2 and current density value of 141 mA/cm^2 at 35 °C with 30% relative humidity were obtained for 4 h (Figure **3a**). The cell performance was increased with increase the cell temperature at 45 °C, the power density was 41.5 mW/cm^2 at 108 mA/cm^2 (Figure **3b**). Further, the maximum power density was reached 51 mWcm^2 at 55 °C and current density was 198 mA/cm^2 for 4 h measurement, after that both values were decreased

Figure 3: I−V curves for PWA/P$_2$O$_5$/SiO$_2$ glass composite membrane measured at various temperature conditions with 30% relative humidity: (**a**) 35, (**b**) 45 and (**c**) 55 °C.

(Figure **3c**) and also observed the cell voltage decreased from 1 V to 0.9 V. However the performances are higher compared to previous work at low temperature using the same glass composite membrane [23].

Figure **4** and **5** show the polarization curves for PWA/P$_2$O$_5$/SiO$_2$ glass composite membrane in H$_2$/O$_2$ fuel cell at various humidity conditions. Hydrogen and oxygen was flow to the anode and cathode compartment at 50−100 mL/min at 1 atm pressure. At 30 °C and 30% RH, the cell resistance was 3.9 ohm cm^2 and the maximum power and current density values of 30.5 mW/cm^2 and 120 mA/cm^2, respectively, for 4 h measurement, after that those values were decreased including cell voltage from 0.78 V to 0.98 V (Figure **4a** and **5a**). The humidity conditions were increased from 30 to 40 and 50% and observed the cell performances, at similar operating conditions; the power density values were increased 33 and 38 mW/cm^2 at 0.5 V and also current density values were increased [Figure **4** (**b**, **c**) and **5**(**b**, **c**)]. Fuel cell polarization curves increase with increasing operating time. Conversely, the polarization curves decrease with decreasing operating time. The current-voltage curves are linear, typical of a cell with high internal resistance. The increase in temperature and humidity level, results the cell performances were improved, but the values are not high like Nafion membrane was showed higher performances at low and high temperature fuel cells [32, 33]. This power density was considered to be the highest output presently known when using the PWA/P$_2$O$_5$/SiO$_2$ glass composite membrane with a 0.1 mg/cm^2 Pt/C electrode in H$_2$/O$_2$ fuel cells at low temperatures.

During the same experimental conditions (electrical potential 0.5 V), the distance between the end of the platinum wires and the edge of active electrodes of the fuel cell was always much larger than three times the thickness of the glass membrane to avoid potential gradients. The chemical reaction of PWA containing glass membrane with hydrogen is due to the reduction of PWA species to a heteropolyblue compound that is a mixed-valence intervalence transition. The heteropolyblue species have the same structure as the parent heteropolyacid but they are characterized by a tungsten atom in the Keggin structure having 5$^+$ oxidation state. The reduced PWA species migrate to the cathode in the PWA cell where they are reoxidized by dissolved oxygen. The evidence of a chemical reaction of H$_2$ with phosphotungstic acid is strongly supported by the observed color change of the
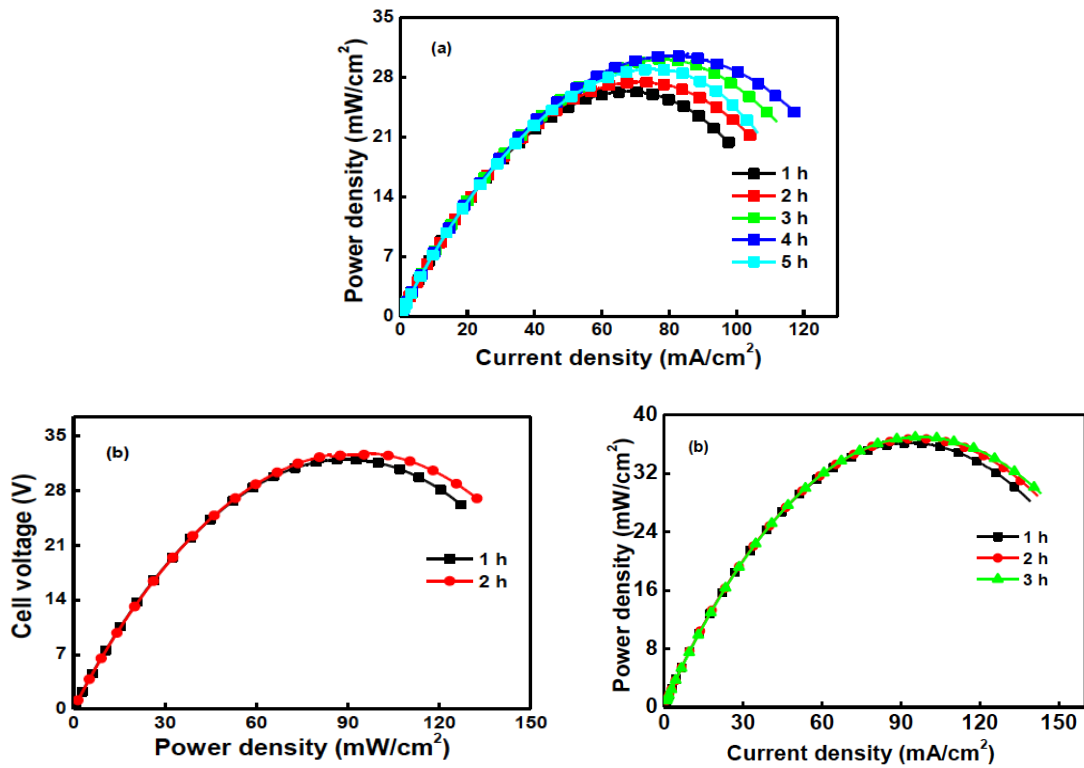
Figure 4: Polarization curves for PWA/P$_2$O$_5$/SiO$_2$ glass composite membrane measured at various humidity conditions with 30 °C. (**a**) 35, (**b**) 45 and (**c**) 55%.
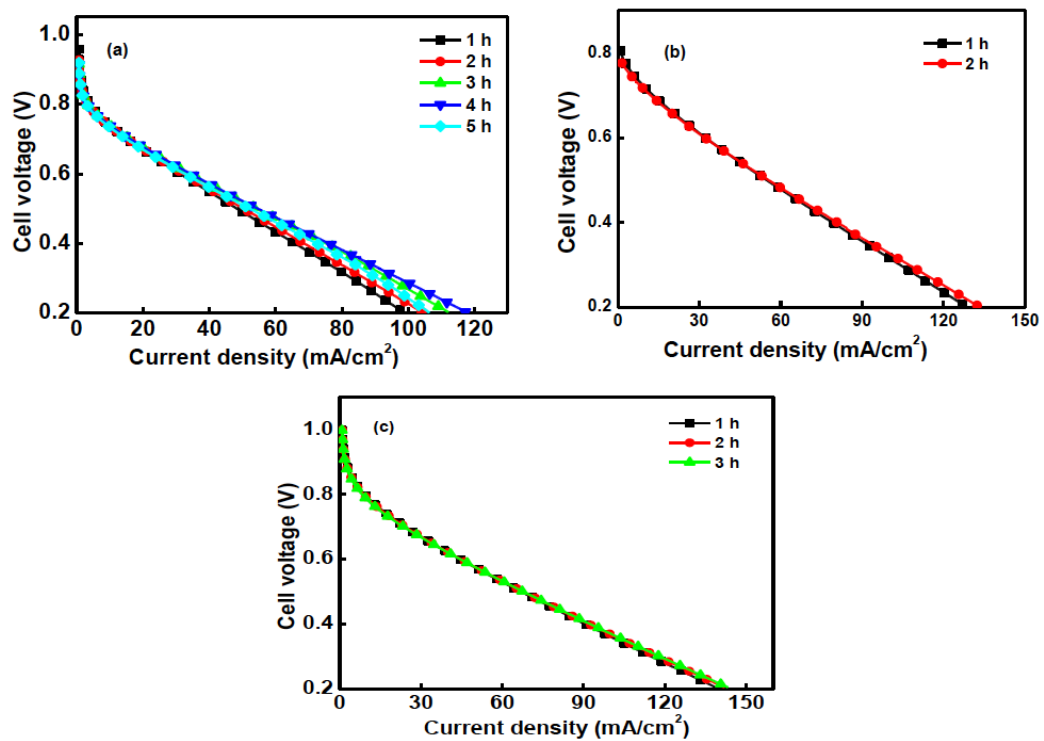

Figure 5: I−V curves for PWA/P$_2$O$_5$/SiO$_2$ glass composite membrane measured at various humidity conditions with 30 °C. (**a**) 35, (**b**) 45 and (**c**) 55%.

electrolyte coming out from the anodic side of the cell, from colorless to a deep blue color, which can be explained by the formation of reduced PWA species [20].

Figure **6** (**a**-**c**) present typical complex impedances obtained at cell voltage 0.5 V for electrode with a content of Pt/C (0.1 mg/cm^2). The effect of temperature on the EIS system was measured with constant humidity condition at 30%. The strong dependence of temperature provides the reports on EIS behavior of H₂/O₂ fuel cell reveal the presence of two loops. The higher frequency intercepts of the kinetic loop and the real impedance axis is a measure of the total ohmic resistance of the cell. The total resistance can be expressed as a sum of the contributions from contact resistances and ohmic resistances of the cell components including membrane, catalyst layer and gas diffusion layer [34], among which, the biggest contributor is the membrane resistance. Another series of experiments was performed by changing the humidification and keeping fixed cell temperature is 30 °C during electrochemical measurements. The observed changes results of ohmic resistance values were noted from Figure **7**. The shape of loops is similar as Figure **6**, one is small loop and second one is big loop. In our experiment, both effects were small, since the operating temperature was low and the humidification also moderate. The semicircle of the high frequency region, as seen in Figure **7**, was not included because this semicircle at the high frequency region is not associated with the electrode process [35]. The loop at the lower frequency region corresponds to the electrode process and the loop at the higher frequency region is not associated with electrode process. Freire and Gonzalez [36] measured the Ohmic resistances at different currents by Ac impedance through the high frequency intercepts values of the kinetic loop. Their results indicated that the Ohmic resistances, mainly membrane resistances change with current. On the contrary, comparison of both effects on EIS recorded in similar condition at different temperature (35–55 °C) and humidity (35–55%) revealed little different shapes of loops at low and high frequency region. The ohmic resistance values were obtained for both experiments in the range from 2 to 4 ohm/cm^2. However, we emphasize that the PWA/P₂O₅/SiO₂ glass ceramic composite membrane sample showed a low resistance of 1.38 ohm/cm^2, while maintaining a high OCV of 0.9 V.

Figure 6: A.C impedance spectrum measured at various temperature conditions: (**a**) 35, (**b**) 45 and (**c**) 55 °C with relative humidity for PWA/P₂O₅/SiO₂ glass composite membrane.

Performing AC-impedance measurements on fuel cells can assist in identifying problems within the fuel-cell components as well as track deviations in the fuel cell assembly process. More specifically, such measurements can be helpful in identifying the kinetic resistances in the fuel cell system and the ohmic resistances in the system and in the electrolytic. Fuel cell components that can affect the impedance include current collectors, porous electrodes, the catalytic layer and the membrane. When the temperature was increased from 35 to 55 °C, there is no further decrease in ohmic resistance, suggesting that the membrane resistance is no longer the main contributor to the ohmic resistance. However, the charge transfer resistance decreases consistently with the increase of temperature and humidity.
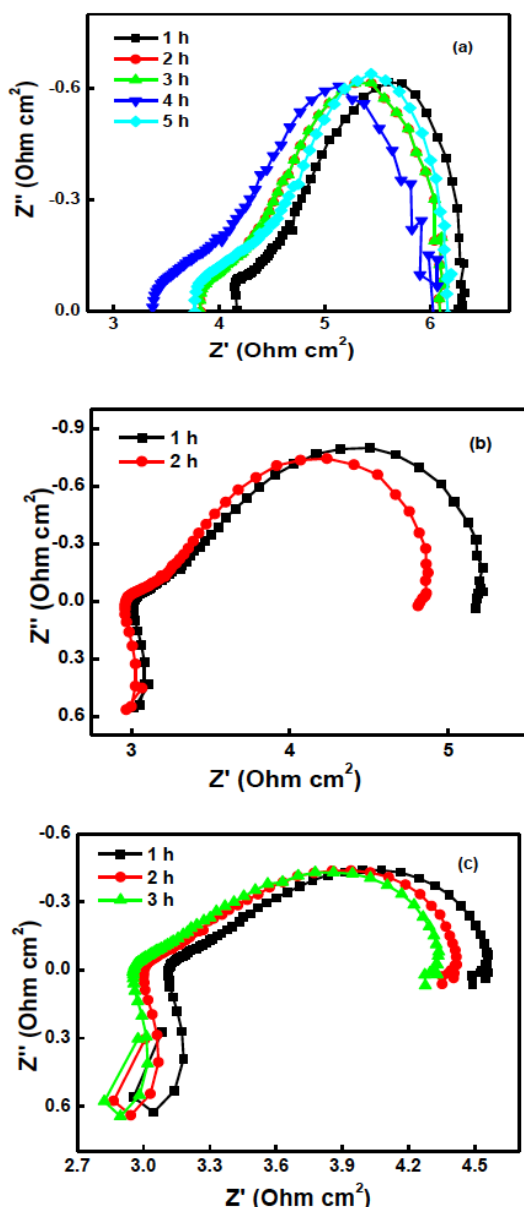
Figure 7: A.C impedance spectrum measured at various humidity conditions: (**a**) 35, (**b**) 45 and (**c**) 55% for PWA/P_2O_5/SiO_2 glass composite membrane at 30 °C.

4. SUMMARY

Because of our interest we measured the cell resistance value at various potential using a good MEA, its only showed high performance, because of heteropolyacid in the phosphosilicate glass. Further in future, require to study about the effect of heteropolyacids in glass composite how could be affected at similar operating condition for H_2/O_2 fuel cell. Our EIS results showed the ohmic resistance of the electrolyte and electrode which can be estimated from the high and low frequency limit of the spectrum. Operating at low temperature under constant atmospheric pressure, the cell delivers a maximum power density of about 51 mW/cm^2 and a current of 198 mA/cm^{-2} at 55 °C and 30% RH. These values are high and promising, and suggest that the PWA/P_2O_5/SiO_2 glass composite membranes are suitable for use in low temperature H_2/O_2 fuel cells. This work thus imparts a distinctive opportunity to improve fuel cell performances by tuning the glass composite membrane properties through optimization and using non-noble metals as electrode.

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