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Preparation and Characterization of Platinum on Activated Carbon Support as the Electrode in Proton Exchange Membrane Fuel Cell

Tey Chen Han[a] and Hadi Nur[a]*

[a]*Department of Chemistry, Faculty of Science, Universiti Teknologi Malaysia, 81310 Johor Bahru, Malaysia*
**Corresponding Author: hadinur@utm.my*

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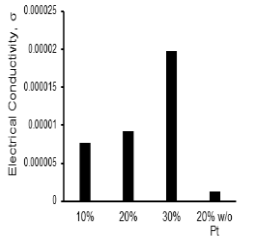
GRAPHICAL ABSTRACT



ABSTRACT

Activated carbon (AC) is an inexpensive and environment friendly material which is widely used as the electrode in proton exchange membrane fuel cell (PEMFC). However, it suffers from a few drawbacks including low electrical conductivity and mechanical strength. To overcome this problem, Polytetrafluoroethylene was used as a binder to improve the mechanical strength of the samples together with the hydraulic press and platinum was used to enhance the conductivity of the sample. In this work, the electrode was successfully prepared using different concentration of PTFE solution (10%, 20% and 30%). From Electrochemical Impedance Spectroscopy analysis, the conductivity of the sample has increased significantly from 10%PTFE to 30%PTFE, while it also confirmed that there was a significant effect of adding platinum which helps to improve the electrical conductivity of the samples. Scanning Electron Microscopy analysis has illustrated that as the concentration of PTFE is increases, the activated carbon tends to bind closely and strongly with each other to give a more compact electrode. Energy Dispersive X-ray analysis clearly shown that the distribution of PTFE and platinum are very uniform on the surface of the samples. FTIR analysis showed the presence of materials used in the electrode. The results suggested that Pt/AC with 30% PTFE was the best electrode which gave the highest conductivity of 1.98×10^{-5} $\Omega^{-1}cm^{-1}$.

Keywords: Activated carbon (AC), polytetrafluoroethylene (PTFE), platinum, electrical conductivity

1. INTRODUCTION

As the number of populations in the world is gradually increasing every year from 7.3 billion in 2015 to 7.7 billion in 2019, the global energy demand is also keep increasing by 2.3% in 2018 which is twice than in 2016 of only 1.1%. The major factor that contribute in an increased of global energy demand is due to the usage of fossil fuels. Because of the use of fossil fuels can cause significant effects to the environment, researchers were trying to find an alternative way of producing energy and fuel cell has been the most popular topic being discussed for producing a clean energy. There are several different types of fuel cell and Proton Exchange Membrane Fuel Cell (PEMFC) has received attention because of its low operating temperature [1]. PEMFC involved an electrochemical process that produce electrical energy from chemical energy which involve the process of oxidation and reduction reaction also known as redox reaction. PEMFC most often required the supplied of external sources which is usually the hydrogen from the fuel and oxygen coming from the air to generate the electricity.

One of the components in the fuel cell is the electrode which is mostly made from carbon that produced from the coal which is non-environment friendly. Therefore, an alternative way of sources of carbon as electrode material has been discovered to replace carbon produced from coal. Activated carbon draws the attention from the researchers because of the wide range of applications including water treatment, purification, fuel cell, electrodes for electric double layer capacitors and others. Activated carbon has been used as the electrode material which is low cost. The sources of carbon to produce activated carbon can be obtained from the agricultural waste or lignocellulosic materials [2]. The agricultural waste and lignocellulosic materials which are inexpensive and effective such as coconut shell, risk husk, bamboo, wood, kernaf fiber, corn cob, hazelnut shell and many others [3]. Activated carbon is obtained from the organic materials that undergoes pyrolysis at a very high temperature. The carbon materials produced can then be activated through two ways which is physical activation and chemical activation. Physical activation, it involves two process which is carbonization and activation. During carbonization, the materials with carbon content will be pyrolyzed under inert gases like nitrogen gas at a range of temperature from 600 to 900°C [4]. After that, the carbonized product will be activated through the exposure to the oxidising atmosphere usually carbon dioxide at above 600°C. On top of that, chemical activation is also used to produce activated carbon and this process is also known as wet oxidation which require activating agent to be impregnated into the precursor. Activating agent also has the function known to be dehydrating agents and oxidants. The reagent usually used for the chemical activation is usually acid or strong base such as phosphoric acid, H_3PO_4, sodium hydroxide, NaOH, potassium hydroxide, KOH, calcium chloride, $CaCl_2$, zinc chloride, $ZnCl_2$ [5]. Strong bases such as sodium hydroxide and potassium hydroxide will produce higher surface area and microporous carbon structure [6]. Some surface oxygen

functional groups can be introduced into the precursor carbon when react with the activating agent. The most important step in the chemical activation is the washing step as it will determine the porosity of activated carbon. Hence, the porosity of the activated carbon is significantly determined by the washing step [7]. Chemical activation is preferred over the physical activation as it requires relatively low temperature and more effective of developing the porosity of activated carbon. The porous size of the activated carbon can be varied with a very particular diameter size from micropores, macropores to macropores and this can be achieved during the activation process.

A binder is needed for the preparation of electrodes as it helps in binding of the discrete porous particles to form an electrode which facilitates the electrochemical activities in the system. Polytetrafluoroethylene (PTFE) provide enough strengthen during the formation of electrodes. In addition, PTFE has often been used as a binder because it is excellent in chemical and thermal resistance because of the fluorine atoms in PTFE that make it to be hydrophobicity and the electronegativity of fluorine atoms make it insoluble to most of the solvents. The cost of PTFE is relatively lower as compared to Nafion [8]. PTFE is usually used in the form of aqueous dispersion rather than powder form as to obtain a uniform distribution. However, the use of PTFE binder will affect the electrochemical performance of the activated carbon as some of pore of the material will be blocked.

Both electrodes which is anode and cathode will undergo oxidation and reduction process at the respective electrode and at the same time diffusion of liquid and gaseous will also take place on the surface of electrode. Despite of the activated carbon electrode itself without the catalyst will not give a good performance as an electrode. Therefore, platinum is used and supported on the activated carbon. The loading of platinum on the activated carbon will affect the surface properties of the carbons. One of the problems that being found out when using activated carbon as catalyst support is that the large ash content left after the activation process that usually will be concentrated on the surface. Some inorganic constituents on the surface of activated carbon may interact with the metal which directly affecting the catalytic activity.

2. EXPERIMENTAL

2.1. Pt/AC electrodes with PTFE Preparation

3g of activated carbon powder was weighed on the electronic balance and mixed with 1mg of platinum powder. After that, different concentration of PTFE suspension solution was prepared by diluting the 60 wt% PTFE to 10, 20, and 30% of PTFE solution. Next, different concentration of diluted PTFE solution was added into the mixture to prepare 10, 20, and 30% PTFE activated carbon electrode. Approximately 2mL of 1,3-propanediol solution was added into the mixture to create the paste. The paste was then kneaded and compress with a round shape mould using hydraulic press at 5tons of pressure. Lastly, the paste was then dried in the oven at 70 °C for 1 hour

2.2. Samples Characterization

All the synthesized samples were characterized by FTIR-ATR, EIS and SEM-EDX. Perkin Elmer instrument model was used in Fourier Transform Infrared – Attenuated Total Reflection (FTIR-ATR) analysis. The electrical conductivity of the samples was conducted by Electrochemical Impedance Spectroscopy (EIS). The surface morphology of the samples was studied using Scanning Electron Microscopy (SEM) and Energy-dispersive X-ray spectroscopy (EDX) analysis. The samples were scanned with resolution of 10 µm (10.0 kV).

2.3. Electrical conductivity

The electrical conductivity of the electrode was calculated using equation 1, where σ is the electrical conductivity, ι is the conduction length, *R* is the ohmic resistance of the sample and *A* is the area of the pellet face. On top of that, the conduction length and area were measured from the dimensions of the sample.

Electrical conductivity,

$$\sigma = \frac{\iota}{RA} \tag{1}$$

3. RESULTS AND DISCUSSION

3.1. Fourier Transform Infrared- Attenuated Total Reflection (FTIR-ATR)

The functional group of the electrodes were determined using (Fourier-Transform Infrared – Attenuated Total Reflection). Figure 1 shows that a wide band was observed at around 3213.37 – 3224.24 cm^{-1} corresponding to the O-H stretching in the all four samples; (i), (ii), (iii) and (iv) due to the chemical used of 1,3-propanediol and 60 wt% dispersion in H_2O solution for the preparation of different concentration of PTFE solution. The peak at 1052.34 – 1054.50 cm^{-1} is due to the vibration absorption of C – O in all four samples. Two peaks are clearly observed at 1151.41 cm^{-1} & 1204.88 cm^{-1} and 1150.08 cm^{-1} & 1204.95 cm^{-1} in (i) and (iv) respectively due to the presence of C-F bonding.

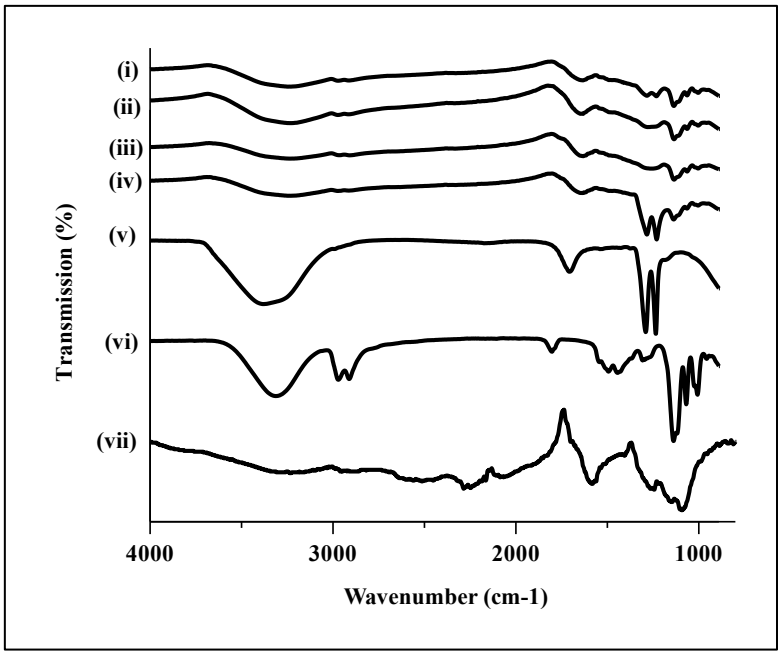


Figure 1. FTIR-ATR spectrum of AC (i) Pt/AC with 30% PTFE; (ii) Pt/AC with 20% PTFE; (iii) AC with 20% PTFE; (iv) Pt/AC with 10% PTFE; (v) PTFE; (vi) 1,3-propanediol and (vii) AC

3.2. Electrochemical Impedance Spectroscopy (EIS)

Electrical Impedance Spectroscopy was employed to determine electrical conductivity of the samples. Based on Figure 4.2, it can be seen that the ohmic resistance, |Z| was decreased gradually from 10% to 30% PTFE of Pt/AC, while AC with 20% PTFE without the platinum has an increased in the ohmic resistance, |Z|. In other words, the conductivity of the samples were increased from 10% to 30% PTFE of Pt/Ac. Pt/AC with 30%PTFE has the highest electrical conductivity of 1.98×10^{-5} $\Omega^{-1}cm^{-1}$among the others while 20%PTFE of AC without platinum has shown that the conductivity is the lowest at 1.3×10^{-6} $\Omega^{-1}cm^{-1}$. The average conductivity of the samples was calculated, and it is also confirmed that the average conductivity of the samples increased from 10% PTFE to 30% PTFE as shown Figure 2. The increased in the conductivity of the samples are due to an increased in the amount of PTFE used. As the concentration of PTFE is higher, it makes the activated carbon bind strongly with PTFE to become more compact. Thus, the electrons are more easily to be conducted from place to place and leading to higher conductivity. On the other hand, the conductivity of the sample also influenced by having the platinum incorporated into the samples which help the samples to be more conductive. This statement is shown in the table 1 where there are two electrodes with the same concentration of PTFE at 20%, but one of it is without putting platinum in the sample. From the calculation, it is found out that the conductivity of the electrode with platinum has higher than the one that do not have platinum with. The conductivity of Pt/AC electrode is 9.18×10^{-6} $\Omega^{-1}cm^{-1}$ while AC electrode without platinum is 1.30×10^{-6} $\Omega^{-1}cm^{-1}$.

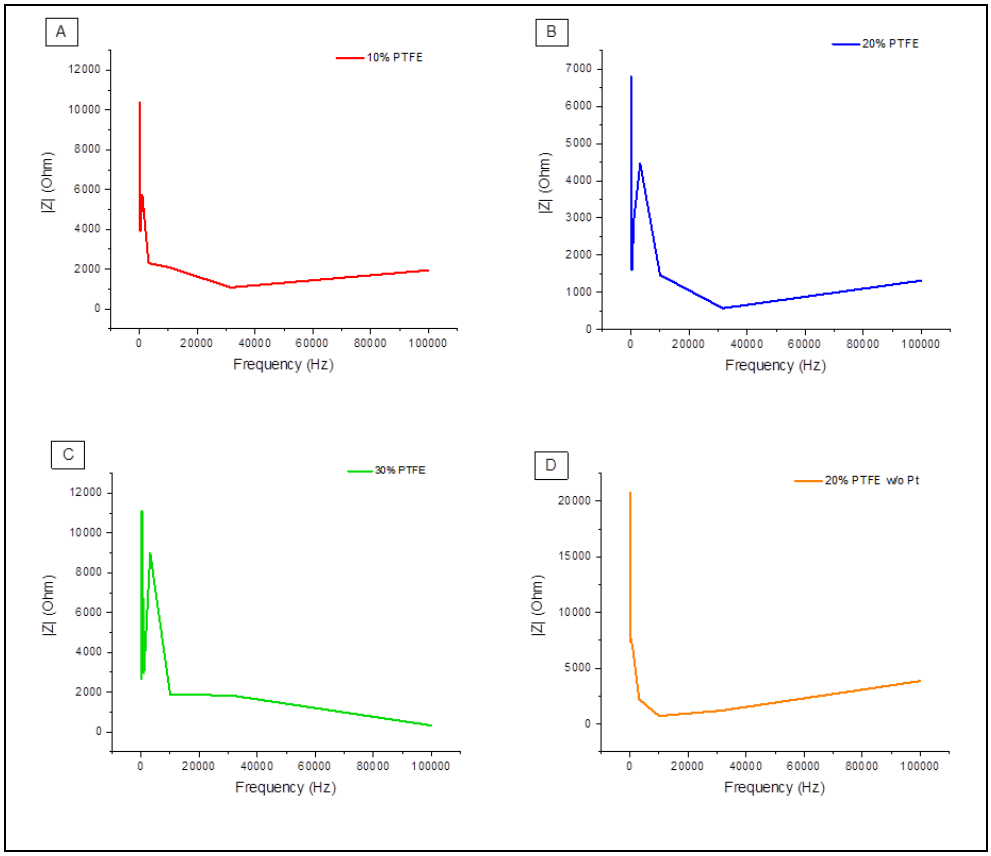


Figure 2. The absolute impedance plots of (A) 10%PTFE; (B) 20%PTFE; (C) 30%PTFE and (D) 20%PTFE w/o Pt

3.3. Scanning Electron Microscopy-Energy-dispersive X-ray (SEM-EDX)

Figure 3 illustrates the EDX elemental mapping for the electrodes with a different concentration of PTFE. EDX is used together with SEM to detect the emission of five major elements on the surface of the electrode which are carbon, fluorine, oxygen, and small percentage of silica and platinum. The compositions shown in Figure 3 are taken from the same mapping area. There is a small percentage of platinum in the electrodes to act as an electrocatalyst to enhance for the dehydrogenation process. Besides, the fluorine element comes from the PTFE which used as a binder to improve the mechanical strength of the electrodes. From the EDX mapping, it can be clearly seen that the fluorine dots green colour) in 10% PTFE are the least concentrated as compared to the other electrodes. This can be proved by the atomic percentage of fluorine which increases as the concentration of PTFE increases from 10% to 30% PTFE. On the contrary, the atomic percentage of carbon is inversely proportional to the atomic percentage of fluorine. The electrodes are successfully incorporated with the platinum and it is uniform distributed on the surface of the electrodes. The SEM image at resolution of 1000 displayed that as the concentration of PTFE increases, the activated carbon become more closely packed and compact with each other.

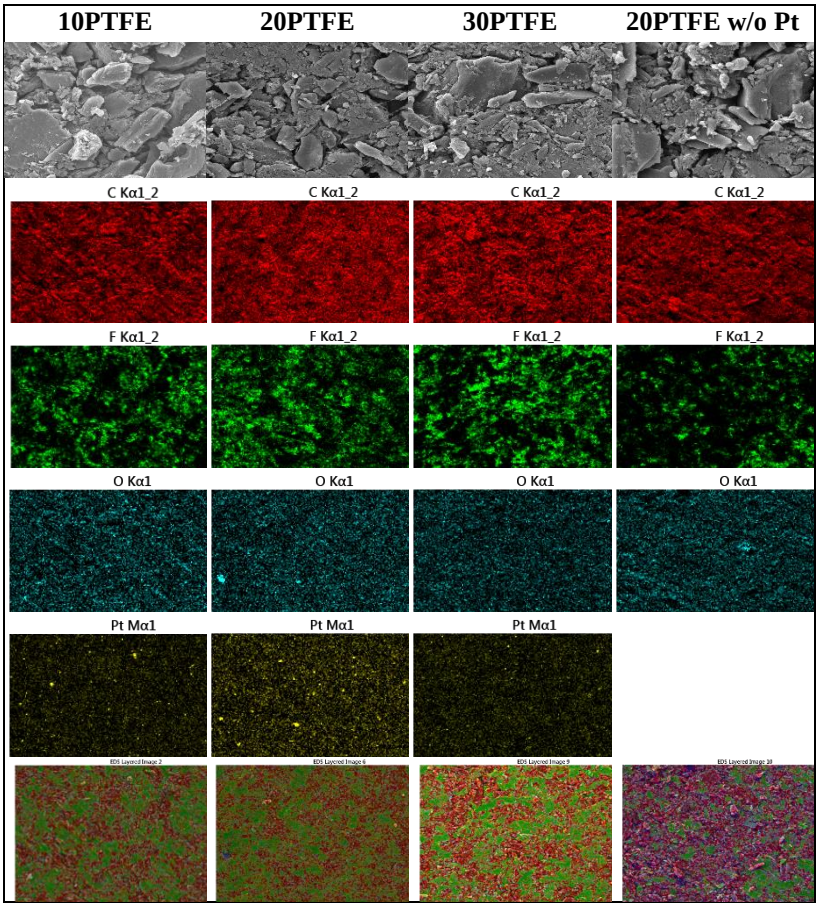


Figure 3. EDX mapping of prepared electrodes using different amount of PTFE

4. CONCLUSION

The electrode was successfully synthesized by mixing the activated carbon, platinum, and PTFE solution and pressed using the hydraulic press. The synthesized samples were characterized using FTIR-ATR, EIS and SEM-EDX. In FTIR analysis, all the significant peaks of the functional groups of the samples has been detected in all samples and also confirmed the presence of fluorine in samples. For EIS analysis, it is concluded that the higher the concentration, the higher the conductivity and 30%PTFE has the highest conductivity of 1.98×10^{-5} $\Omega^{-1}cm^{-1}$. With SEM-EDX analysis, it is clearly seen that all the PTFE and platinum are uniform distributed on the surface of the electrode. SEM analysis also displayed that with higher concentration of PTFE, the activated carbon powder can bind strongly with higher mechanical strength and this helps in the conduction of electron becomes more easily.

REFERENCES

[1] Rajashekara, K., & Rathore, A. K. (2015). Power conversion and control for fuel cell systems in transportation and stationary power generation. *Electric Power Components and Systems*, 43(12), 1376-1387.

[2] Alternor, S., Carebe, B., Emmanuel, E., Lambert, J., Ehrhardt, J.J., & Gaspard, S. (2009). Adsorption studies of methylene blue and phenol onto vetiver roots activated carbon prepared by chemical activation. *Journal of Hazardous Materials*, 1651(1-3), 1029-1039.

[3] Georgin, J., Dotto, G. L., Mazutti, M. A., & Foletto, E. L. (2016). Preparation of activated carbon from peanut shell by conventional pyrolysis and microwave irradiation-pyrolysis to remove organic dyes from aqueous solutions. *Journal of Environmental Chemical Engineering*, *4*(1), 266-275.

[4] Ioannidou, O., & Zabaniotou, A. (2007). Agricultural residues as precursors for activated carbon production—a review. *Renewable and sustainable energy reviews*, *11*(9), 1966-2005.

[5] Zhengrong, G., & Xiaomin, W. (2013). Carbon materials from high ash bio-char: a nanostructure similar to activated graphene. *American Transaction On Engineering Applied Sciences*, 2(1), 15.

[6] Fernandez-Ruiz, C., Bedia, J., Bonal, P., Rodriguez, J. J., & Gómez-Sainero, L. M. (2018). Chloroform conversion into ethane and propane by catalytic hydrodechlorination with Pd supported on activated carbons from lignin. *Catalysis Science & Technology*, *8*(15), 3926-3935.

[7] Ahmadpour, A., & Do, D. D. (1997). The preparation of activated carbon from macadamia nutshell by chemical activation. *Carbon*, *35*(12), 1723-1732.

[8] Dong, H., Yu, H., Wang, X., Zhou, Q., & Feng, J. (2012). A novel structure of scalable air-cathode without Nafion and Pt by rolling activated carbon and PTFE as catalyst layer in microbial fuel cells. *Water research*, *46*(17), 5777-5787.