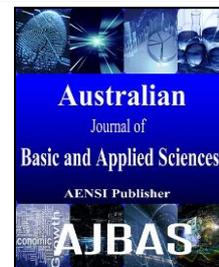




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Bucky Paper as an Electrode Material for Fuel Cell Applications

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ABSTRACT

The development of fuel cell electrodes technique was demonstrated. The replacement of the carbon fiber paper (CFP) by a Bucky Paper (BP) give an advantage in the fuel cell technique. The two major advantages in the replacement of carbon fiber paper (CFP) by the Bucky Paper (BP) are; the carbon fiber paper was brittle whereas the BP is flexible. The other major advantage point is that the Platinum (Pt) film can be deposited direct on the Bucky Paper through the deposition of Copper without needing to the deposition of the Gold (Au) on the substrate. The result out of this work shows that the x-ray photoelectron spectroscopy (XPS) spectrum of 1, 3, and 5 monolayer of Pt deposited on Bucky paper with no need to the Au as a substrate. The two peaks of Pt at 122ev and 160ev are clear, and shows that the Pt is stable at 3 and 5 monolayer. The Cyclic Voltammetry (CV) test shows that at 5 monolayer the Pt surface is completed through the reduction noticed at 0.15V which is due to hydrogen adsorption/desorption.

INTRODUCTION

The effect of the Au-carbon fiber paper (CFP) morphology was explored on the structural and chemical properties of Pt catalyst films and determining how changes in these properties influence the electrochemical performance and durability of the catalyst architecture as a whole, the ultrathin platinum over layers were prepared on two types of Au structures grown on CFP (Shuang Cheng, *et al.*, 2011). This work is considered as a future work for developing the Fuel cell electrodes by replacing the Carbon Fiber Paper (CFP) with Bucky Paper (BP). This replacement gives an advantage in the deposition of Pt on the electrode of the fuel cell. However, the replacement of the CFP by the BP is considered to be a great improvement, since the carbon fiber paper, as compared to the flexible Bucky paper, is brittle. Another very important reason is that the platinum (Pt) film can be directly deposited on the Bucky paper through the deposition of copper (Cu) with no need to the deposition of gold (Au).

We are going to demonstrate the deposition of Platinum (Pt) on Bucky Paper, and we studying for different monolayers (1, 3 and 5) (Mauricio J. Prieto and Germano Tremiliosi-Filho, 2013; Juan A. Santana, Sven Krüger, and Notker Rösch, 2014). The Bucky paper is a thin sheet made from an aggregate of carbon nanotubes or carbon nanotube grid paper (Phil coale, 2008). Among the possible uses for the Bucky paper are that; Fire protection: covering material with a thin layer of Bucky paper significantly improves its fire resistance due to the efficient reflection of heat by the dense, compact layer of carbon nanotubes or carbon fibers (Zhao, Zhongfu and Gou, Jan, 2009). If exposed to an electric charge, Bucky paper could be used to illuminate computer and television screens (James B. Lewis, 2008). It could be more energy-efficient, lighter, and could allow for a more uniform level of brightness than current cathode ray tube (CRT) (Peters-Michaud *et al.*, 2011) and liquid crystal display (LCD) technology (Jonathan W. Steed and Jerry L. Atwood, 2009).

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Bucky papers also operate as electrodes in an electrochemical cell, and because of the difference in the injected charge between two sheets of Bucky paper, electrolyte ions form a double layer. This is demonstrated by a bimorph cantilever actuator, in which a Bucky paper was applied to either side of an adhesive tape and immersed in a 1M NaCl solution (Karthik Mayilvahanan, 2013).

Another factor to consider is the alignment of the CNTs in the Bucky paper. To properly mimic muscles, Bucky papers must be aligned to maximize the conversion of electrical energy to mechanical energy. Despite the fact that there are still production limitations, the future is bright for artificial muscles made of Bucky paper (Fryasse, J., Minett, *et al.*, 2002). In this study we demonstrate the deposition of Pt on the BP at different monolayer 1,3 and 5 monolayers. The Cyclic Voltammetry (CV) and the X-ray Photoelectron Spectroscopy (XPS) test of these samples is studied as well.

Experimental Work:

To synthesize the Bucky paper from CNTs, a popular method involves dispersing the CNTs in a surfactant solution and then filtering the suspended solution. One of the more commonly used solution is Dimethylformamide (DMF) which mixed with MWCNTs and pouring in glass container on ultra-sonication for 24 hour. After that the mixing were casting in Buchner funnel which contain the Teflon filter paper dispersed effectively in the surfactant solution. They are filtered using Buchner Filtration, as shown in Figure (1.1) (Rafique, M. M. A. and Iqbal J., 2011; Byrne, M. T. and Gun'ko Y. K., 2010).

This method, published in 2008, optimizes the alluring properties of Bucky paper, using multi-walled carbon nanotubes (MWNTs) ((Rafique, M. M. A. and Iqbal J., 2011; Byrne, M. T. and Gun'ko Y. K., 2010). In this process, chemical vapor deposition (CVD) is used to form a forest of vertical CNTs. Then, a microporous membrane is placed on top of the array of CNTs as a cylinder, pushed at a constant pressure, flattens the array of CNTs. With the help of Van der Waals forces, the CNTs align to form a Bucky paper. After the array is flattened with the microporous membrane, the Si substrate is removed first, and the microporous membrane is removed next by applying ethanol, which permeates the membrane and makes the membrane easily removable (Wang, D., *et al.*, 2008).

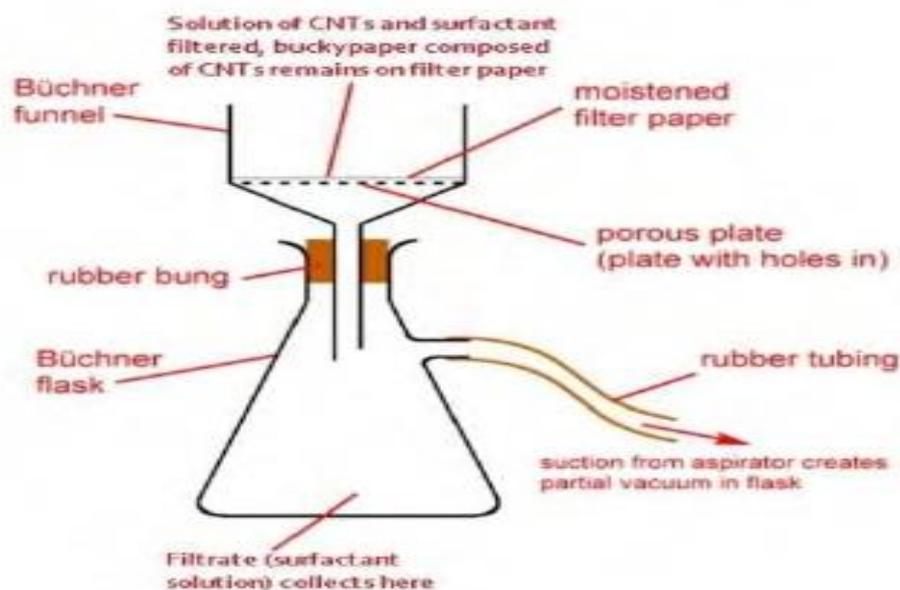


Fig. 1.1: A set up of Buchner filtration to filter suspended solution of dispersed CNTs in surfactant solution (Byrne, M. T *et al.*, 2010).

The Bucky paper was first cut into small pieces and then stuck to small pieces of glass by conductive tape to deposit the Pt on it by Under Potential Deposited (UPD) technique and Cu replacement processes. Figure (1.2) shows the Bucky paper sheet. The small pieces of Bucky paper were washed by DI water as a first step and followed by the Cu UPD step performed in 50 mM H_2SO_4 mixed with 10 mM CuSO_4 with the three electrodes connected to the sample.

The external cell shown in figure (1.3) was checked and the Cu UPD was run to start the sweep for one cycle to deposit one layer of Cu on a Bucky paper piece. This sample was insert in 1 mM H_2PtCl_6 solution for replacement the Cu layer with the Pt when the three electrodes were connected and the external cell was checked to run the Pt deposition. This procedure was taken about 10 minutes. The deposition produced 0.3 monolayer of Pt on Bucky paper sample, and 1 monolayer of Pt was deposited on Bucky paper when reearete this process (Cu UPD and Pt deposition) for 3 times. So for 1, 3 and 5 monolayer of Pt deposited on Bucky paper samples we repeat this process 3, 9 and 15 times respectively.

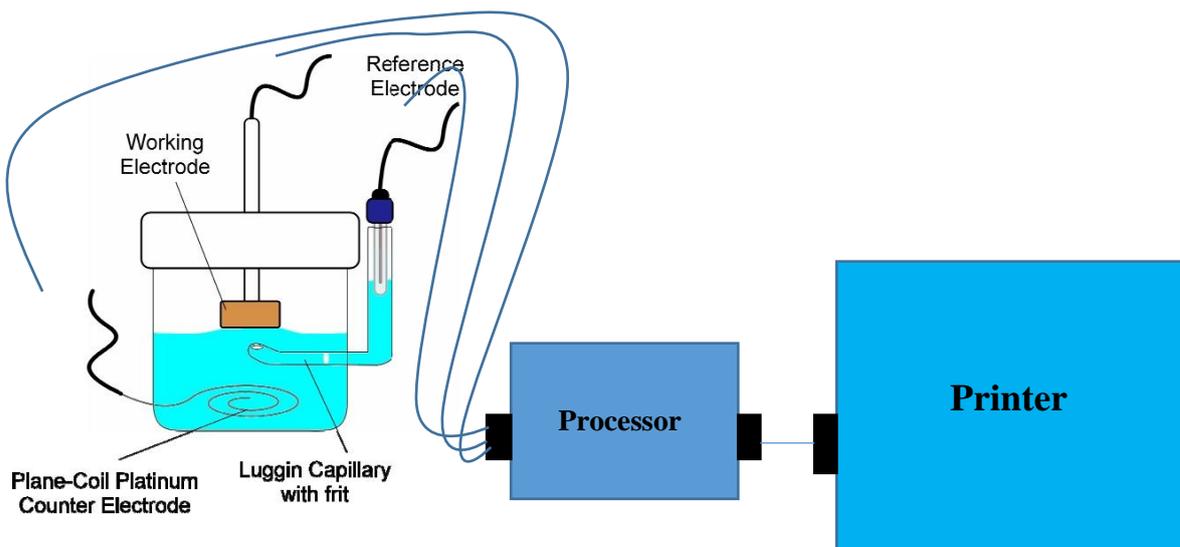


Fig. 1.3: Illustration of electrochemical cell geometry employed to Cu UPD step and Pt deposition.



Fig. 1.2: Bucky Paper Sheet.

The Pt-BP samples were rinsed in DI water for washing and inserted in Sulfuric acid solution (H_2SO_4) to clean off the sample from the chemical residual when the three electrodes were connected and the external cell was checked to begin the sweep for one cycle. After 1, 3 and 5 monolayers of Pt deposited on BP samples; these samples were cleaned by H_2SO_4 solution. Finally, the samples were rinsed in DI water, dried by nitrogen gas, and kept in small containers.

X-ray Photoelectron Spectroscopy (XPS) for the Pt-BP samples were explored and the Cyclic Voltammetry (CV) data were collected using a Solartron 1287 potentiostat and a pine Wave Nano potentiostat.

RESULTS AND DISCUSSIONS

The Bucky Paper (BP) was used as a substrate instead of Carbon Fiber Paper (CFP) because the Pt can be deposited on the BP directly through the Cu nano film without deposition of the Au. So, BP was given more interest especially at high temperatures in the (100-400) °C range.

At high temperatures, the problem which has been persisted with the Au diffusion in CFP has now been eliminated in the BP. This advantage is currently being used in fuel cell operation. The disadvantages of the BP reside in its mechanical properties which can be improved through the research.

X-ray Photoelectron Spectroscopy (XPS):

The over layer Pt atoms are first examined via x-ray photoelectron spectroscopy (XPS) using a tunable light source, effectively allowing a depth profile study to be performed on catalyst samples. Using a tunable XPS system with low energy incident photons, the chemical states of the Pt can be examined at varying 'information depths' on BP structure. This method enables a case study sensitive enough to examine the surface of Pt-BP samples and allows for the analysis of the thickness-dependent structural features of Pt over layers. XPS 'information depth' values were obtained based on inelastic mean free path modeling functions by Cumpson and Seah (Cumpson, P. J. and Seah, M. P., 1997), Utilizing low energy photons available from synchrotron radiation allows precise examination of the over layer that can't be achieved with laboratory XPS. The resultant Pt sublayer from one iteration corresponds to roughly 1/3 of a complete monolayer (Cheng, S.; Rettew, *et al.* 2011).

Figure (1.4) shows the XPS spectrum for 1, 3 and 5 ML of Pt samples deposited on the Bucky paper (BP) prior to being heated with incident photon energy of 400 eV. It can be noticed from the figure that the Pt is characterized as a 4f electron doublet ($4f_{7/2, 5/2}$) at ~ 122 and 160eV. This means that at 1 ML selected incident photon energy of 400 eV is still able to penetrate past the deposited Pt over layer deposited on BP, these results agree with the reference (Shuang Cheng, *et al.*, 2011).

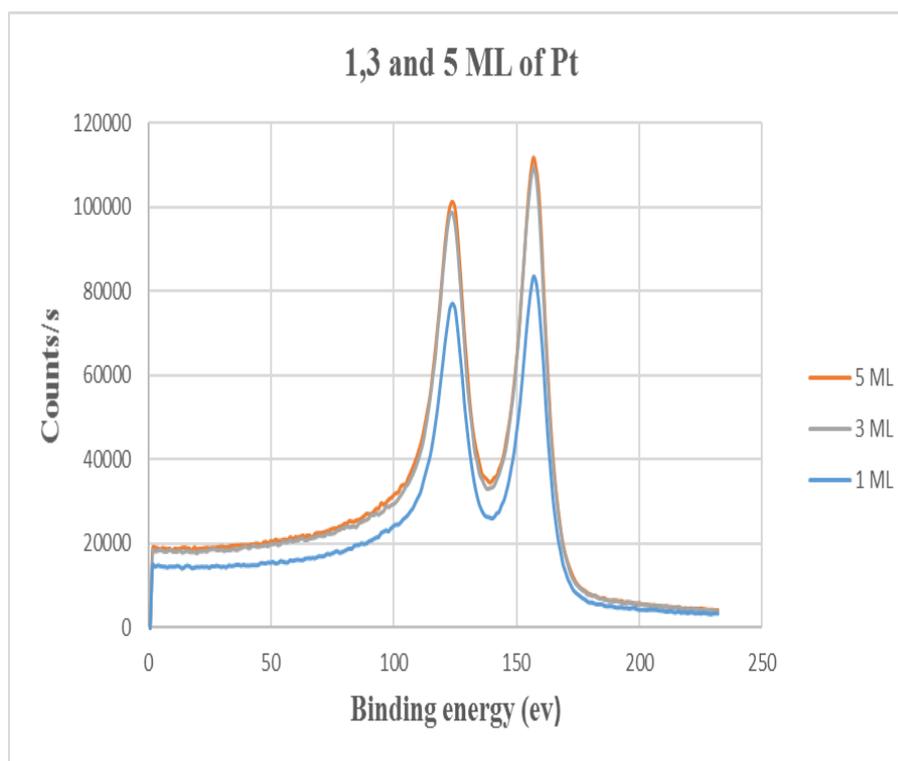


Fig. 1.4: XPS spectrum for 1, 3, and 5 ML samples of Pt deposited on Bucky paper (BP).

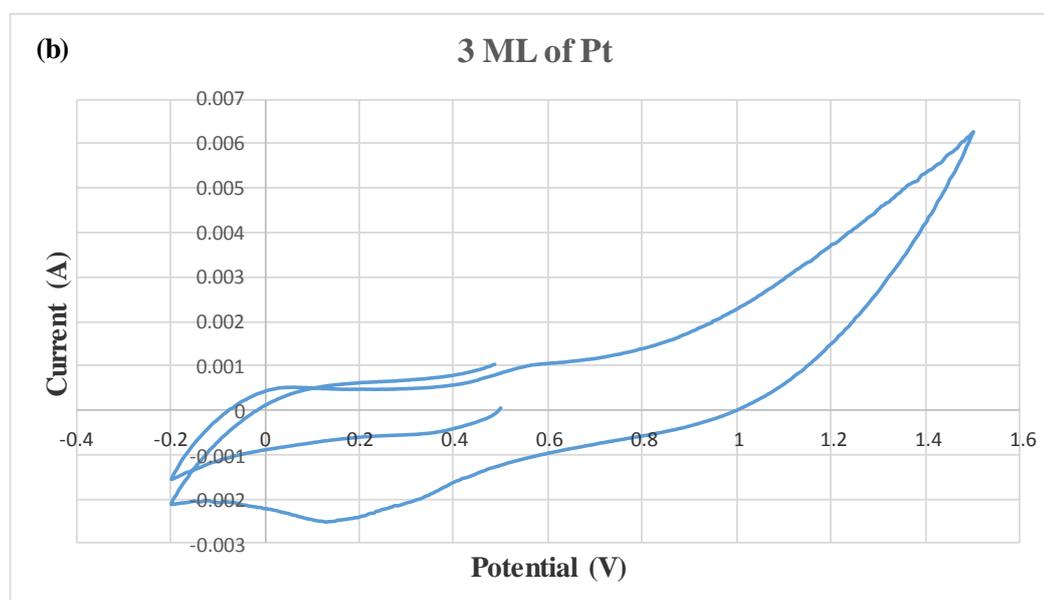
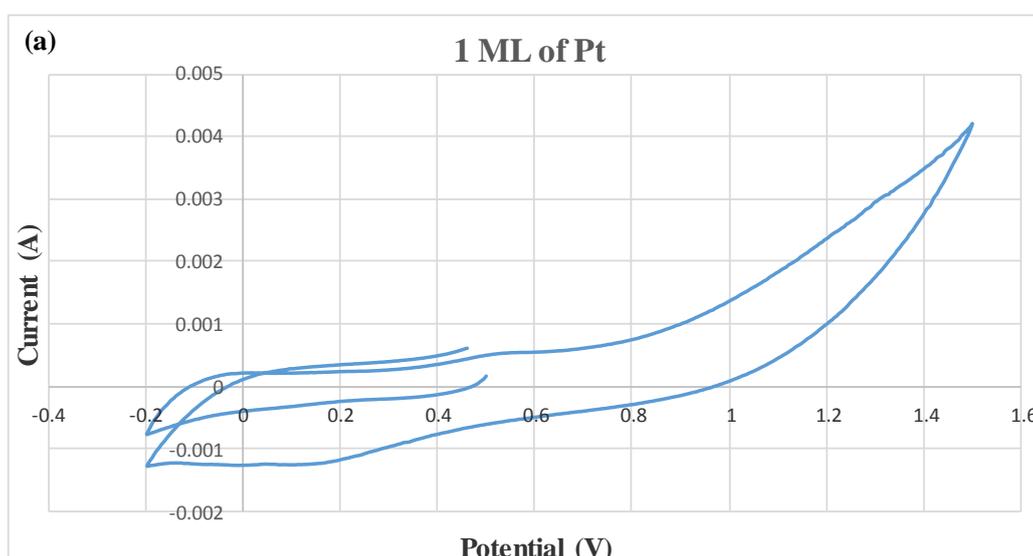
However, for 3 and 5 ML samples, photon energies around 400 eV can only provide information of the Pt over layer; electrons from the underlying BP don't have enough energy to eject from the sample.

Cyclic voltammetry (CV):

To further investigate the catalyst surface, the Pt coverage of samples was also examined via cyclic voltammetry (CV). Voltammograms were conducted in N_2 -saturated 0.1 M H_2SO_4 by sweeping from -0.2 to 1.5 V at a scan rate of 20 mV/s. The electrolyte was chosen to match the $CuSO_4$ solution used during electrochemical layer-by-layer synthesis. CV is a surface-sensitive technique where the observed currents are generated from reactions at the metal–electrolyte interface. Changes in Pt surface area can be tracked by either the Pt oxide reduction feature near 0.45 V or the hydrogen adsorption/desorption curve from -0.2 to 0.0 V against an Ag/AgCl reference electrode. These methods of tracking Pt coverage on another materials have often been employed (Cheng, S.; Rettew, *et al.* 2011; Abdelhafiz, A., *et al.*, 2015; Zhai, J.; Huang *et al.*, 2007; Tang, H.; Chen, 2004).

The results of 1, 3 and 5 ML of Pt can be seen in figure (1.5) a-c. CV is a surface sensitive technique where the current seen are generated from reactions at the metal – electrolyte interface. Changes in Pt surface area can be tracked by either the Pt oxide reduction feature near 0.15 V or the hydrogen adsorption / desorption curve from - 0.2 to 0.0 V against an Ag/AgCl reference electrode. These results agree with other researcher reports (Robert E. Rettew, *et al.*, 2009).

Finally, it is possible to replace the brittle film of CFP by the flexible BP film. This can be consider as a good advantage in the fuel cell industry. As a manuscript for future work we try to heat the Pt-BP samples at different temperatures from (100-400) C with two types of furnaces (under air and under vacuum) to see the effect of the environment on these samples which effects on the fuel cell application.



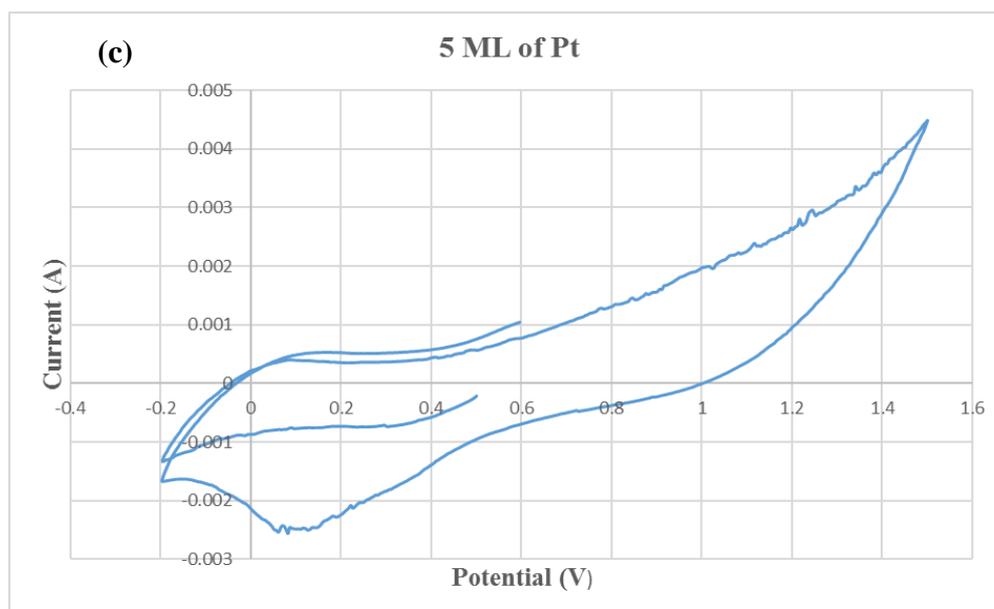


Fig. 1.4: CV spectrum for 1,3 and 5 ML samples of Pt deposited on Bucky paper (BP);(a) 1 ML of Pt-BP. (b) 3 ML of Pt-BP. (c) 5 ML of Pt-BP.

Conclusions:

The development of fuel cell electrodes technique was studied. The replacement of Carbon Fiber Paper (CFP) by Bucky Paper (BP) give rise to more advantage in the fuel cell industry. The brittle CFP is replaced by flexible BP can be solve an industrial problem in the fuel cell technology. The deposition of Pt on the BP at different layers (1, 3 and 5) layers are studied. The deposition was easier than the deposition of Pt on CFP. These two points can be considered as a developing stage in the fuel cell technology. The result of this work shows that the x-ray photoelectron spectroscopy (XPS) spectrum of 1, 3, and 5 monolayer of Pt deposited on Bucky paper by Cu under Potential Deposited Cu (UPD) which appear two clear peaks of Pt at 122ev and 160ev. The Cyclic Voltammery (CV) test shows that at 5 monolayer the Pt surface is completed through the reduction noticed at 0.15V which is due to hydrogen adsorption/desorption. The deposition of Pt on Bucky paper can be reduced effects of metal catalyst constituents and is worth further exploration.

Future Work:

The replacement of carbon fiber paper (CFP) by Bucky paper (BP) give rise to more advantage in the fuel cell industry. The brittle CFP is replaced by flexible BP can be solve an industrial problem in the fuel cell technology. The Cyclic Voltammery (CV) test shows that at 5 monolayer the Pt surface is completed through the reduction noticed at 0.15V which is due to hydrogen adsorption/desorption. The Bucky paper samples required to be heated to different temperatures (100-400) °C by using two types of furnaces (tube and muffle) with and without Oxygen, and study the oxygen reduction reaction (ORR) step before and after heating to check the effect of environment on Bucky paper samples.

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