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Honors Research Project

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Platinum-Rhodium Alloy Electrocatalyst Activities in the Methanol Oxidation Reaction

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Honors Project 4200:497

The University of Akron

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Abstract

This study evaluates the electrochemical activity of several compositions of platinum-rhodium alloys on carbon support to identify potential replacements for the expensive platinum electrocatalysts currently used in direct methanol fuel cells (DMFCs). The electrochemical active surface areas (ECSAs) of each sample were determined using cyclic voltammetry (CV) in a 0.5 M H_2SO_4 solution to normalize CV currents generated in the methanol oxidation reaction (MOR). The activity of five compositions was tested; Pt_3Rh, Pt_2Rh, PtRh, Pt_2Rh, and Pt_3Rh. 100 mg of each catalyst was synthesized at 10% by weight platinum loading using a surfactant-free impregnation technique. TEM images were taken of one sample to examine morphology and confirm alloying of the two metals. A volcano-like relationship was found between decreasing platinum loading and electrochemical activity, with a peak seen with the Pt_2Rh sample at 0.66 mA/cm^2. This value is higher than a similarly prepared commercial platinum catalyst which had an activity of 0.6 mA/cm^2. These findings indicate that Pt_2Rh has the potential to be better suited for use in DMFCs than commercial platinum, and could be used to bring down the cost of DMFCs and make green energy one step closer to reality.

Executive Summary

Direct Methanol Fuel Cells (DMFCs) are devices that are able to utilize the methanol oxidation reaction (MOR) to generate electricity with high fuel efficiency and almost no environmentally harmful emissions. This technology has the potential to replace more widely used, inefficient, and environmentally damaging internal combustion power sources currently used in automobiles and portable/backup generators. However, MOR proceeds too slowly to generate useful levels of power, so catalysts are used to improve the reaction kinetics. Currently, platinum on carbon support is the most widely used electrocatalyst. Unfortunately, the high cost of platinum per activity increase is a large barrier to the more widespread implementation of DMFC technologies, causing the development of highly effective, inexpensive electrocatalysts to be an area of great interest. In this project, several compositions of platinum-rhodium alloys on carbon support were prepared to evaluate the materials' effectiveness as an electrocatalyst.

To evaluate each sample, as shown in figures 8 through 10 and table 3, the electrochemical activities, peak potentials, and onset potentials at 0.05 mA/cm2, respectively, of the prepared samples are as follows: 0.48 mA/cm^2, 0.45 V, and 0.38 V for Pt_3Rh, 0.66 mA/cm^2, 0.56 V, and 0.36 V for Pt_2Rh, 0.20 mA/cm^2, 0.54 V, and 0.39 V for PtRh, 0.08 mA/cm^2, 0.54 V, and 0.40 V for $PtRh_2$, and 0.08 mA/cm^2 , 0.45 V, and 0.38 V for $PtRh_3$.

According to Formo et al., the activity of a similarly prepared and tested commercial platinum catalyst on a carbon support is 0.6 mA/cm2. PtRh, $PtRh_2$ and $PtRh_3$ all exhibited low activities relative to the commercial platinum sample, but Pt_3Rh had a very similar activity to platinum and the activity of Pt_2Rh exceeded that of platinum. Of the prepared samples, Pt_2Rh also had the lowest onset potential, which is preferable. Pt_2Rh appears to be the "best" electrocatalyst for MOR of the prepared samples, and should be further evaluated for use in DMFCs.

In undertaking this project, many technical skills relevant to the field of chemical engineering were required. First, laboratory skills were utilized. Sample preparation required the meticulous calculation of reagent amounts, weighing out materials, dissolving materials in the proper solvents, pipetting, drying, ensuring the proper conditions were set in the furnace for all samples, tracking results in real time to see if they follow expectations, and taking good notes in the lab notebook. Second, data analysis skills were utilized. This included running cyclic voltammogram (CV) tests, exporting the data, learning how to use new software such as Origin Labview, researching various sources to understand what the output data means and how to analyze it, integrating over a certain portion of the data and multiplying by conversion factors to calculate the electrochemical active surface area (ECSA), normalizing MOR CV data to the ECSA, looking up literature values to compare results to, generating graphs to effectively display the results of the study, and writing up a report about the data to convey this information to other individuals. Additionally, this is the first study looking into the electrochemical activity of PtRh alloys. The electrochemical activity of Pt_2Rh is very promising, and if other properties such as durability are suitable, it may be a potential low-cost alternative to the expensive platinum electrocatalysts currently in use, which could aid in bringing environmentally clean and highly fuel efficient power to a state of widespread use.

Going forward, additional studies should be performed on Pt_3Rh and even more so on Pt_2Rh due to their promisingly high activity. Chronoamperometry curves should be generated under MOR conditions to determine the durability of these catalysts, as durability is often times a challenge that plagues platinum alloy electrocatalysts. Additionally, more in depth studies should be performed on the morphology of these electrocatalysts, such as TEM or X-ray diffraction (XRD) to determine a more exact morphology of Pt_2Rh and Pt_3Rh to better understand the structure of the formed alloys and what effect it may have on the elevated catalytic activity.

Introduction

Direct Methanol Fuel Cells (DMFCs) are devices that produce power by utilizing the chemical conversion of methanol to water and carbon dioxide in the redox reaction shown in the background section [1]. Methanol is much easier and safer to transport than hydrogen gas, making DMFC's more desirable for portable usage than the more commonly used hydrogen-based Proton Exchange Membrane Fuel Cells (PEMFCs) [1]. One major drawback to both DMFCs and hydrogen PEMFC's is that expensive platinum catalysts are required at the anode and cathode to allow the Methanol Oxidation Reaction (MOR) to occur with the kinetics needed to generate useful power. This high cost makes the common usage of DMFCs for power generation uneconomical, despite both their energy efficiency and low environmental impact [1]. To make DMFCs commercially relevant, new catalysts must be developed to reduce this cost [2]. The United States Department of Energy has set an MOR anode active area target of 150 mA/cm^2 at 0.6 V and a loading of less than 2.7 mg Pt/cm^2 to promote research in this field [3].

Recently, there have been several attempts to alloy platinum with other transition metals to create catalysts that maintain the excellent performance levels of platinum at a reduced cost, with varying degrees of success being shown with Pt_y-Ni_x [4] and Pt_2CuNi [5] alloys. To continue research into possible replacement catalysts, this project aims to synthesize and test the MOR activity of five compositions of platinum-rhodium alloy catalysts; Pt_3Rh, Pt_2Rh, PtRh, $PtRh_2$, and $PtRh_3$. Rhodium is a transition metal, making it a potential candidate for creating a platinum alloy with acceptable catalytic properties. This paper aims to provide background, an overview of the experimental methods employed, the data and results of the study, and a discussion/analysis of the results.

Background

For several decades, fuel cells have been seen as a possible alternative to the combustion engine used to provide power in generator systems as well as automobiles due to their properties of relatively high efficiency and low emissions [1]. Hogarth defines a fuel cell as "an electrochemical engine which can convert the free energy change of a chemical reaction directly into electrical energy." [1] In Direct Methanol Fuel Cells, the following reactions:

Anode (oxidation): $CH_3OH + H_2O \rightarrow CO_2 + 6\ H^+ + 6\ e^-$

Cathode (reduction): $3/2\ O_2 + 6\ H^+ + 6\ e^- \rightarrow 3\ H_2O$

Overall (redox): $CH_3OH + 3/2\ O_2 \rightarrow 2\ H_2O + CO_2$

are used to generate electricity [1]. This reaction occurs at much lower temperatures than the combustion reaction used to power current technologies, causing fuel efficiency to be higher and environmentally harmful emissions to be lower [1]. Other types of fuel cells use the reaction of hydrogen and oxygen to form water as the source of electricity. While having the advantage of plentiful and inexpensive fuels, having an energy density seven time higher than methanol and the only byproduct being water, Hydrogen is notoriously difficult to contain and transport due to its small size. An entirely new and expensive fuel infrastructure would need to be created for distribution if widespread use is ever to be attained, while methanol is compatible with current distribution sources and is no more dangerous than gasoline.

As seen in **Figure 1**, in DMFCs, a mixture of water and methanol permeates into the porous carbon electrode and reacts on the catalyst surface to form positive hydrogen ions, free electrons, and carbon dioxide. The carbon dioxide exits the system as a gas, while the hydrogen ions travel through the polymer electrolyte to the cathode and the free electrons travel up the anode, through a load to perform work, and finally to the cathode. The cathode is exposed to open air and oxygen permeates the porous carbon electrode. At the catalyst surface, the oxygen molecules react with the electrons and briefly form negative oxygen ions which then rapidly react with the positive hydrogen ions to form water molecules.

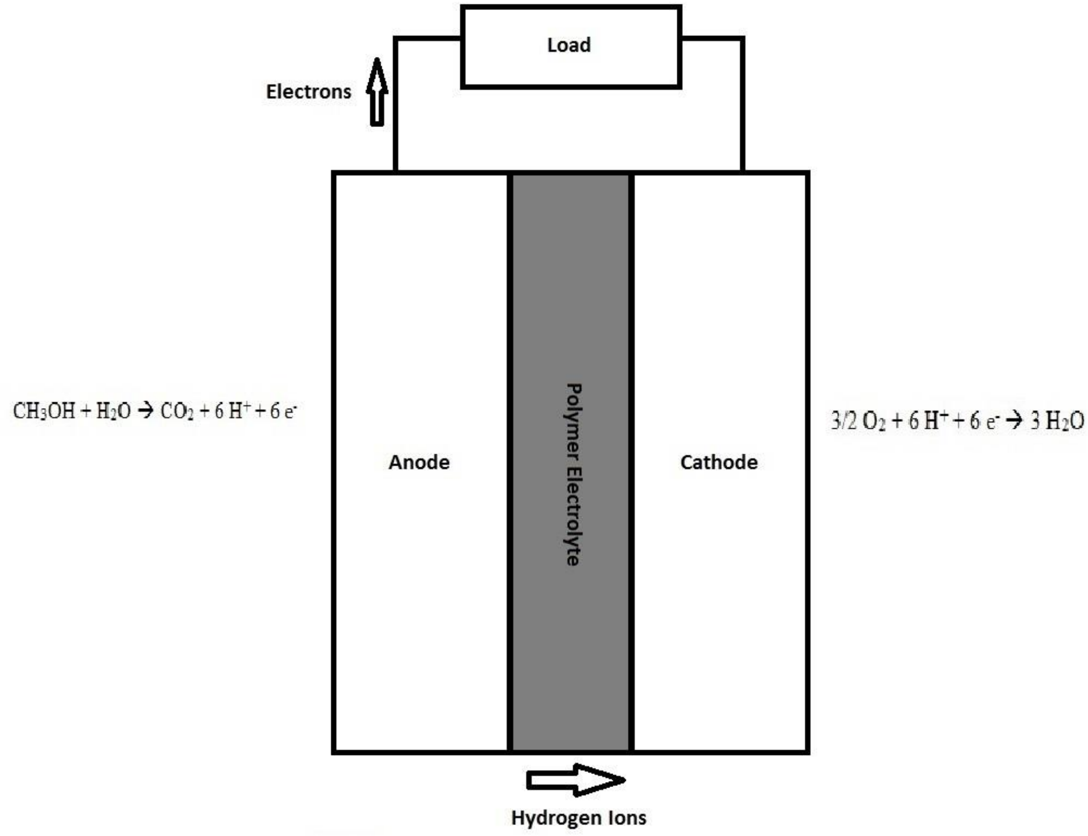

Figure 1 shows a diagram of a Direct Methanol Fuel Cell in operation.

Under normal conditions, the Methanol Oxidation Reaction (MOR) occurs very slowly and is not a feasible source of power [4]. For this reason, a catalyst is needed at the surface of the anode and cathode. Catalysts are substances that improve reaction kinetics by providing a surface for the reaction to take place upon in such a way that the energy of activation for that reaction is lowered. Currently, platinum is the most widely used catalyst on both the anode and cathode of DMFC's due to its exceptional catalytic properties caused by the unique D-orbital electron structure on the surface of its nanocrystals that promotes the adsorption of some molecules [1]. Unfortunately, platinum is an expensive metal which drives up the price of DMFCs. Because of this, there is much interest in developing alternative electrocatalysts which have a higher activity per cost [6]. One avenue of research is the alloying of platinum and other transition metals to create reaction surfaces similar to platinum, with several successes demonstrated with platinum-nickel [4] and platinum-nickel-copper [5] alloys due to the formation of surfaces with electron configurations which promote molecule adsorption very well.

Several standard methods are used to characterize the electrochemical activity of catalysts in MOR. The main tool used in this process is cyclic voltammetry. Cyclic voltammetry setups consist of an electrolytic solution, a reference electrode, a working electrode coated with the catalyst, and a counter electrode [7]. The potential of the working electrode is varied linearly with time at a constant scan rate, first decreasing to a minimum, then increasing to a maximum, while the potential of the reference electrode remains constant [7]. The current generated at the working electrode is measured as a function of its potential vs. the reference electrode, and the resulting curve is known as a cyclic voltammogram (CV) [7]. To normalize the activity of a catalyst, the electrochemical active surface area (ECSA) is calculated. The ECSA is the total surface area of active catalyst on the electrode. Due to roughness at the molecular level of the electrode, the ECSA is often larger than the area of the electrode. To measure the ECSA of the catalyst, a CV is generated with an electrolytic solution consisting of 0.5 M H_2SO_4 [6]. This CV is known as a hydrogen adsorption-desorption cyclic voltammogram [6]. The region of hydrogen adsorption, which occurs after the last peak on the backwards potential scan, is integrated over and divided by the scan rate and a conversion factor for platinum nanocrystal catalysts from literature to calculate the ECSA of the material, as shown in equation 1 [6]. Next, a CV is generated with the electrodes submerged in a solution of 0.5 M H_2SO_4 and 0.5 M CH_3OH to evaluate the catalysts activity in MOR [6]. The peak of the forward sweep on this CV normalized by ECSA is a good measure of the anodic activity of the catalyst. Using these methods, commercial platinum catalysts have been shown to have an anodic activity in MOR of 0.6 mA/cm^2 [6]. Additionally, the Department of Energy (DOE) has set goals of developing anode electrocatalysts with an “activity of 150 mA/cm^2 at 0.6 V and a loading of less than 2.7 mg Pt/cm^2” by the fourth quarter of 2015 [3]. Although this deadline has passed, these can be considered as guidelines for any new MOR electrocatalysts.

$$\boldsymbol{ECSA = \frac{Area\ of\ Hydrogen\ Adsorption\ Region}{scan\ rate * 0.000210\ C/cm^2 Pt}}$$

Equation 1 shows the equation used to calculate the ECSA from the hydrogen adsorption desorption CVs.

Experimental Methods

The methodology for electrocatalyst synthesis in this experiment is scalable and surfactant free. 100 mg of each sample was created, each having a 10% by weight loading of platinum. First, precursor rhodium(III) acetyleacetonate (97%) and platinum(II) acetylacetonate (97%), both from Sigma Aldrich, are added to a small vial in varying amounts outlined in table

1. Next, 3.2 mL of acetone is added to the vial and the contents of the vial are vigorously mixed in a vortex laboratory mixer until all solid is dissolved. A varying amount of charcoal outlined in table 1, which has been left overnight in a small furnace at 300 °C to remove moisture, is then added to a ceramic dish which has been stored in a drying chamber for several hours to remove all water. The metal precursor solution is then pipetted dropwise directly onto the charcoal in 130 µL increments in a spiral pattern to maximize surface area exposed. After each increment is delivered, the sample is allowed to dry for several seconds, and then stirred for 30 seconds to promote more complete drying and even catalyst distribution.

The prepared catalyst precursor is then treated in a furnace under a CO_2/H_2 flow of approximately 2 mL/s. The sample is transferred to a ceramic boat container and placed inside of the glass tube of the furnace. The apparatus is closed off and Nitrogen is run through the tube for several minutes to remove oxygen from the environment. CO_2/H_2 gas flow is then activated and the furnace begins heating at a ramping rate of 5 °C per minute for 36 minutes, heating from room temperature to 200 °C. After the furnace has maintained a temperature of 200 °C for one hour, the gas flow is cut and nitrogen flow is introduced to the furnace for at least 20 minutes until the sample is cool enough to handle.

The electrochemical activity of the samples in the methanol oxidation reaction is then tested using cyclic voltammetry. First, 12.0 mL of DI water, 8.0 mL of isopropyl alcohol, and 80 µL of Nafion are added to a large vial and mixed thoroughly. 5.0 mg of the catalyst is placed in a small vial and dissolved in 5.0 mL of the Nafion solution. The vial is then wrapped in a polymer film to prevent leaking and sonicated for 10 minutes. If the solid does not appear to be fully dissolved in the liquid, the vial is sonicated for an additional 10 minutes repeatedly until sufficient mixing is achieved. At this point, the electrode is soaked in a beaker with ethanol and sonicated for several seconds. The electrode is then polished on a microfabric pad with DI water to remove any additional contaminants. The remaining liquid is blown away and the electrode is attached to a rotating stand. 10 µL of the catalyst solution is pipetted onto the metal surface of the electrode, the rotating stand is set to 200 rotations per minute, and a hair dryer is placed a foot away from the stand blowing at a low speed. When the catalyst ink has completely dried, the surface is examined to confirm that the ink has been evenly distributed on the surface of the electrode.

To begin the electrochemical testing, 60 mL of 0.5 M H_2SO_4 solution is added to a beaker. The beaker is covered with a polymer film and several punctures are made to make room for the working electrode, the reference electrode, the platinum counter electrode, and a gas flow line. Nitrogen is bubbled through the solution for approximately 15 minutes to remove oxygen. The nitrogen flow is then adjusted so that it is flowing over the top of the solution instead of bubbling through it. The working electrode coated with the sample is attached to the CHI760D Electrochemical Workstation, lowered into the beaker, and hydrogen adsorption-desorption CV's are generated using a previously defined sweep profile. Next, 2.45 mL of methanol are added to the beaker and two MOR CV's are generated. The electrode is then cleaned using ethanol and DI water to remove the catalyst and the acid solution is discarded as waste.

Sample	**Pt(ac) (mg)**	**Rh(ac) (mg)**	**Carbon (mg)**
Pt_3Rh	20.00	6.79	88.26
Pt_2Rh	20.00	10.18	87.39
PtRh	20.00	20.36	84.77
$PtRh_2$	20.00	40.70	79.50
$PtRh_3$	20.00	61.08	74.31

Table 1 shows the amounts of precursor materials used in the preparation of each catalyst.

Data and Results

For each of the five prepared platinum-rhodium alloy electrocatalysts, hydrogen adsorption-desorption cyclic voltammograms (CV) are shown which were generated using an electrolytic solution of 0.5 M H_2SO_4. The highlighted area on each graph represents the hydrogen adsorption region, which is used to calculate the electrochemical active surface area (ECSA) of each catalyst using equation 1. Figure 7 shows the ECSA normalized cyclic voltammograms for each catalyst in the methanol oxidation reaction (MOR) which were generated using an electrolytic solution of 0.5 M H_2SO_4 and 0.5 M CH_3OH. All CV's were generated using a scan rate of 10 mV/s. Figures 8 through 10 compare the peak currents , the peak potential, and the onset potential at 0.05 mA of each sample as well as the reported values of a standard commercial 20% by weight platinum sample on carbon support prepared in a similar fashion.

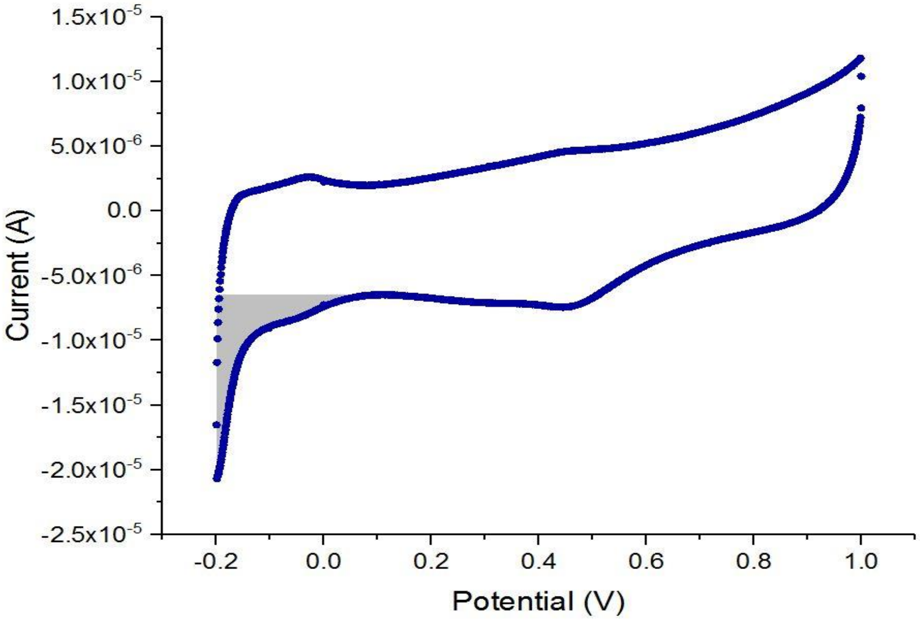

Figure 2 shows the hydrogen adsorption-desorption cyclic voltammogram for Pt_3Rh vs. Reference Hydrogen Electrode (RHE) at a scan rate of 10 mV/s. The hydrogen adsorption region used to calculate the ECSA is shaded in gray.

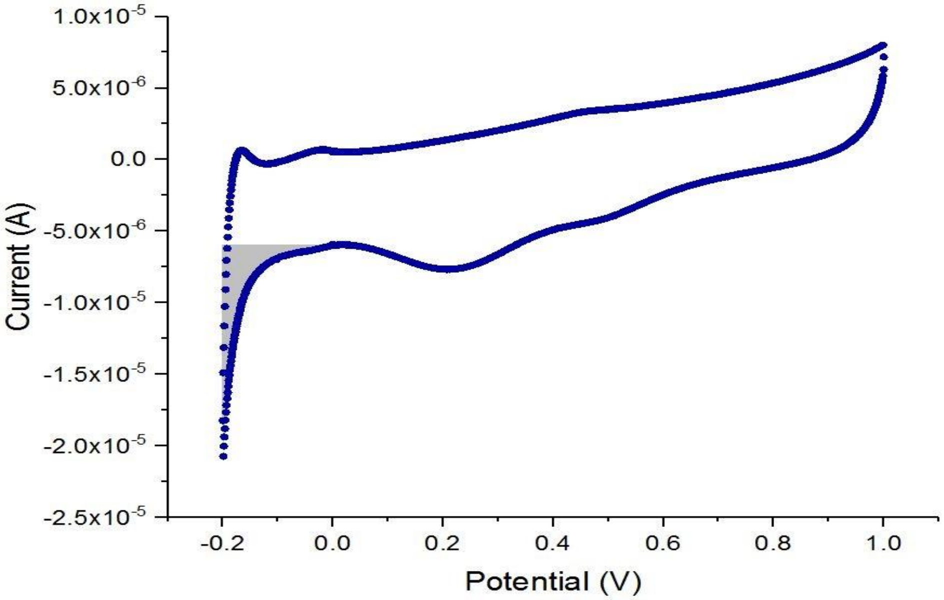

Figure 3 shows the hydrogen adsorption-desorption cyclic voltammogram for Pt_2Rh vs. RHE at a scan rate of 10 mV/s. The hydrogen adsorption region used to calculate the ECSA is shaded in gray.

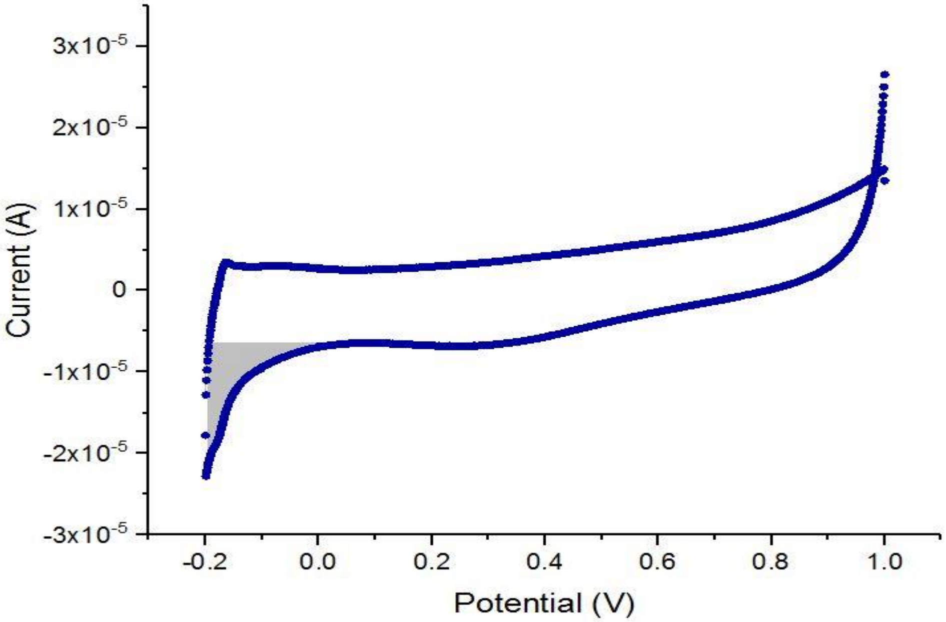

Figure 4 shows the hydrogen adsorption-desorption cyclic voltammogram for PtRh vs. RHE at a scan rate of 10 mV/s. The hydrogen adsorption region used to calculate the ECSA is shaded in gray.

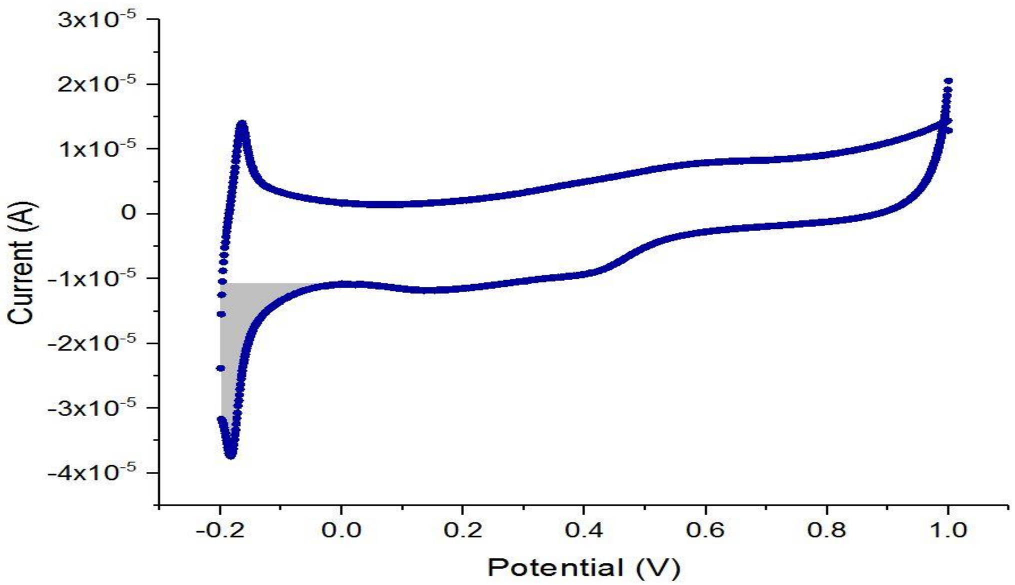

Figure 5 shows the hydrogen adsorption-desorption cyclic voltammogram for $PtRh_2$ vs. RHE at a scan rate of 10 mV/s. The hydrogen adsorption region used to calculate the ECSA is shaded in gray.

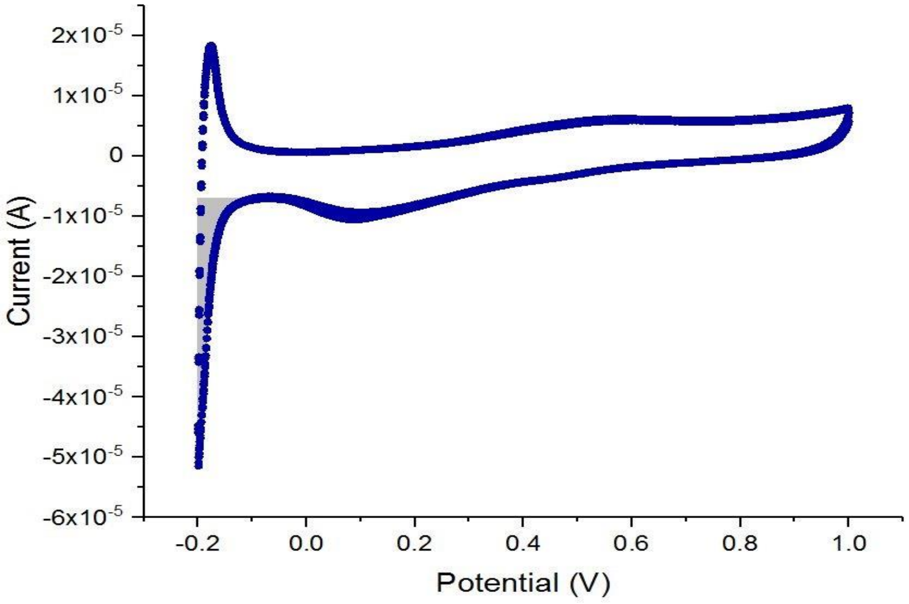

Figure 6 shows the hydrogen adsorption-desorption cyclic voltammogram for $PtRh_3$ vs. RHE at a scan rate of 10 mV/s. The hydrogen adsorption region used to calculate the ECSA is shaded in gray.

Sample	**ECSA (cm^2)**
PtRh	0.426
$PtRh_2$	0.607
$PtRh_3$	0.436
Pt_2Rh	0.218
Pt_3Rh	0.380

Table 2 shows the calculated ECSA for each preparation of electrocatalysts.

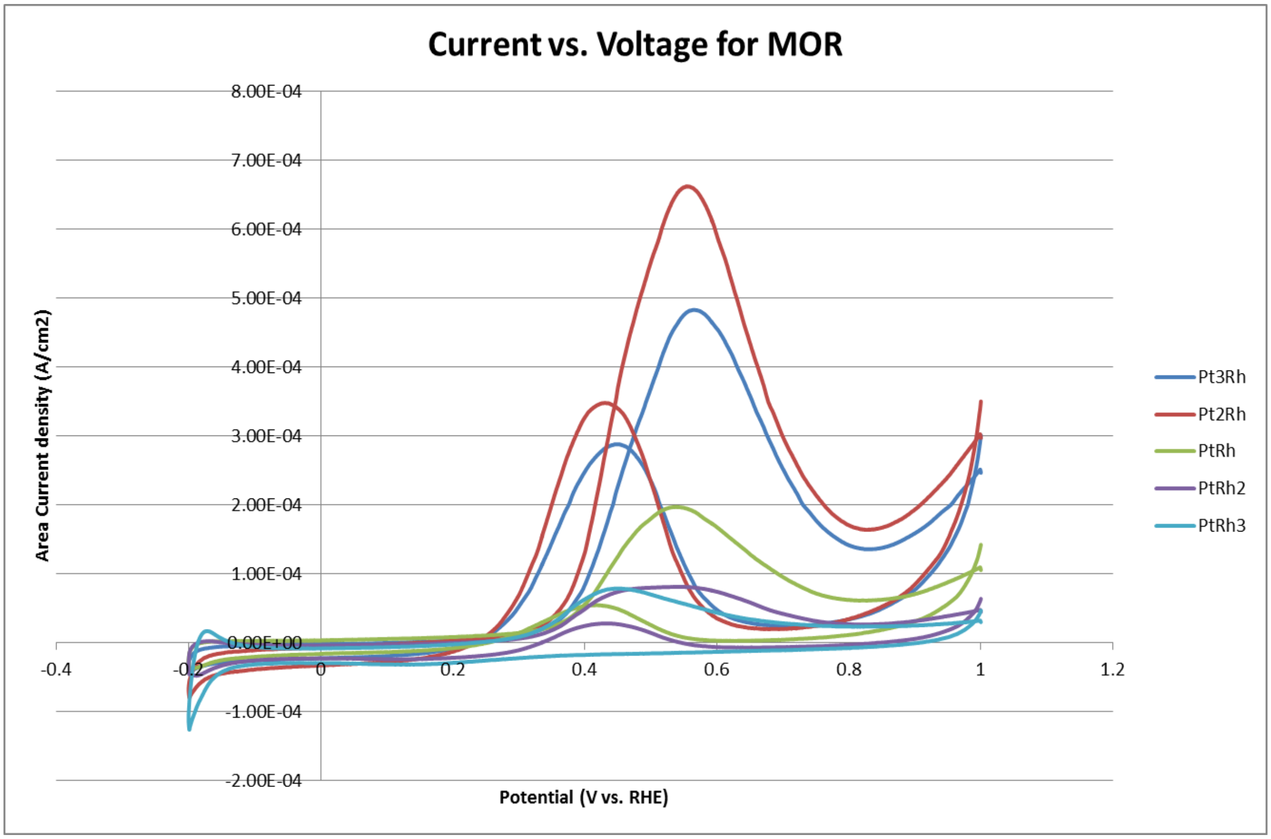

Figure 7 shows the recorded CVs in MOR for each prepared catalyst vs. RHE at a scan rate of 10 mV/s. The metrics of interested are detailed in table 3.

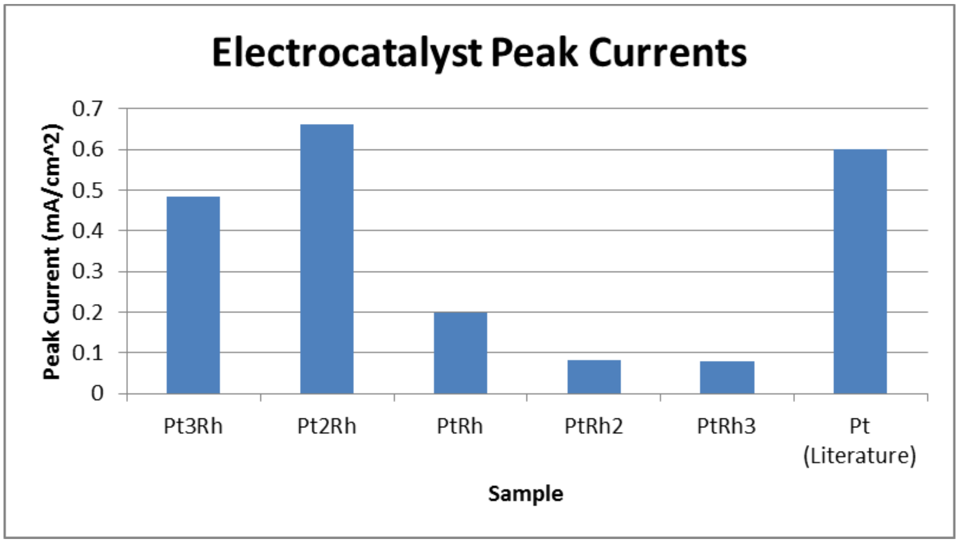

Figure 8 shows the peak activity of each prepared electrocatalyst normalized to ECSA as well as a literature value for commercial platinum.

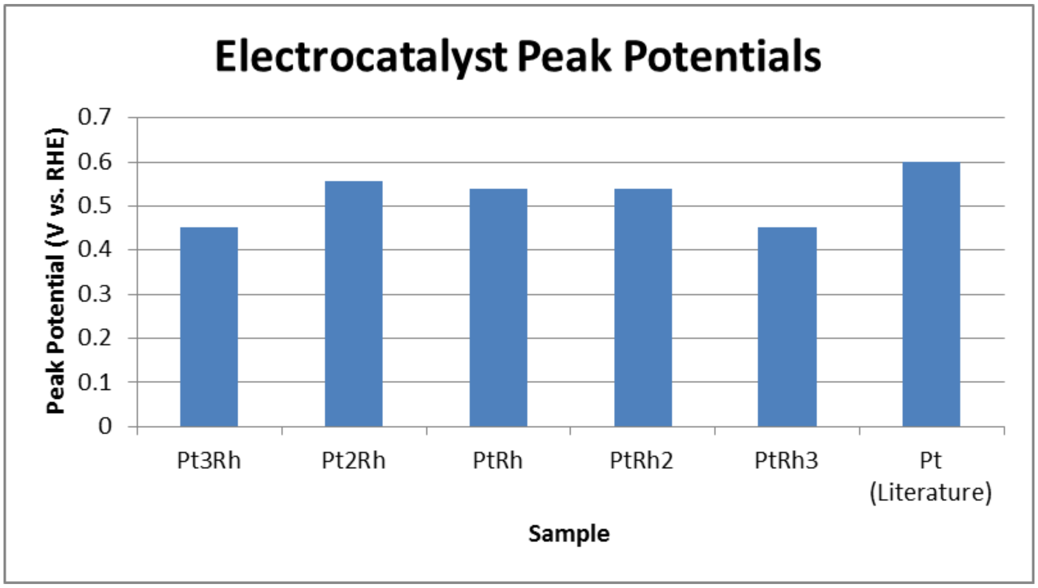

Figure 9 shows the potential of the peak activity of each prepared electrocatalyst as well as a literature value for commercial platinum.

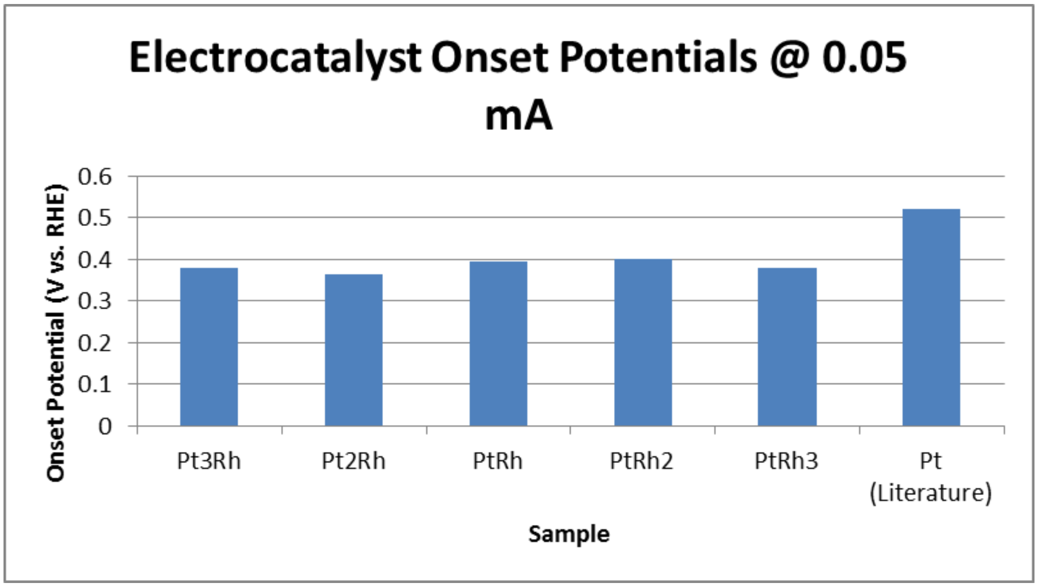

Figure 10 shows the onset potential measured at 0.05 mA of each prepared electrocatalyst as well as a literature value for commercial platinum.

Sample	**Peak Current (mA)**	**Peak Potential (V)**	**Onset Potential (V) @ 0.05 mA**
Pt3Rh	0.48	0.45	0.38
Pt2Rh	0.66	0.56	0.36
PtRh	0.20	0.54	0.39
PtRh2	0.08	0.54	0.40
PtRh3	0.08	0.45	0.38
Pt (Literature)	0.60	0.60	0.52

Table 3 shows the values for the peak current, peak potential, and onset potential of each prepared electrocatalyst as well as a literature value for commercial platinum.

Several transmission electron microscope (TEM) images were taken of the prepared PtRh sample to examine particle morphology. The images below show dark cubic particles on a lighter support structure, which is in line with what is expected to be seen if the alloying of platinum and rhodium was successful. The image was processed using ImageJ software to examine the particle size distribution, which is shown in table 4 and figure 12.

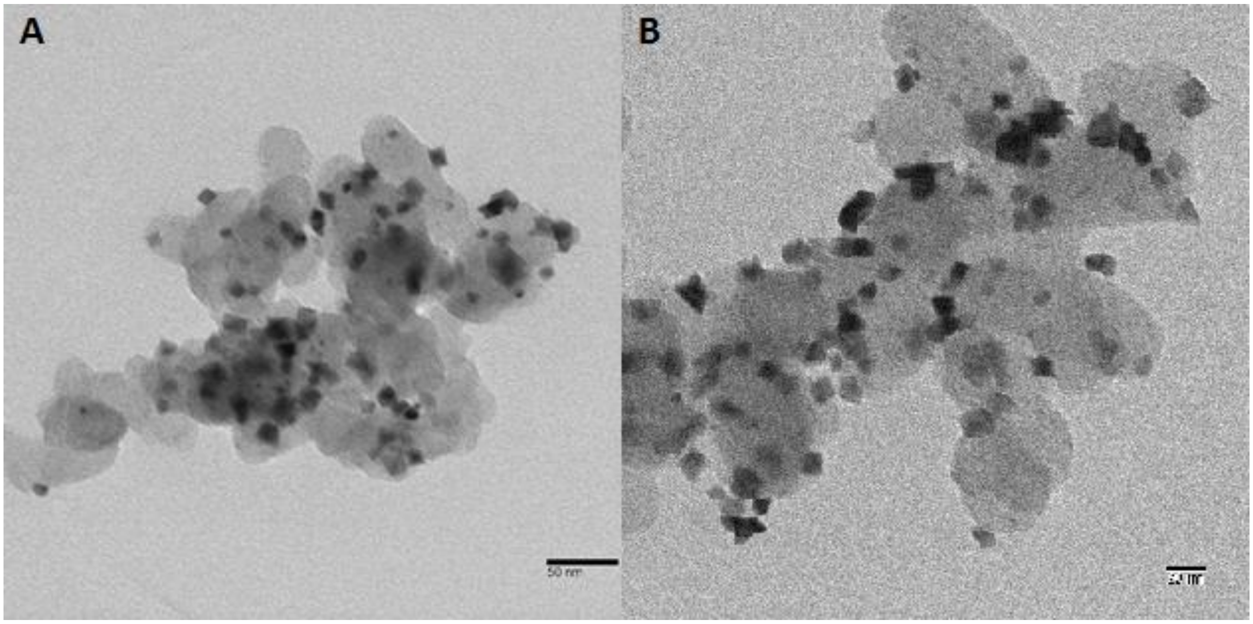

Figure 11 shows a TEM image of A) the prepared PtRh catalyst with a 50 nm scale shown and B) a TEM image of the prepared PtRh catalyst with a 20 nm scale shown. The dark cubic particles are the platinum-rhodium alloy, while the lighter amorphous structure is the carbon support.

Metric	Particle Size (nm^2)
Average	109
Min	2
Max	386
Std Dev	83

Table 4 shows the average, minimum, and maximum particle sizes from figure 11B as well as the standard deviation for the data set.

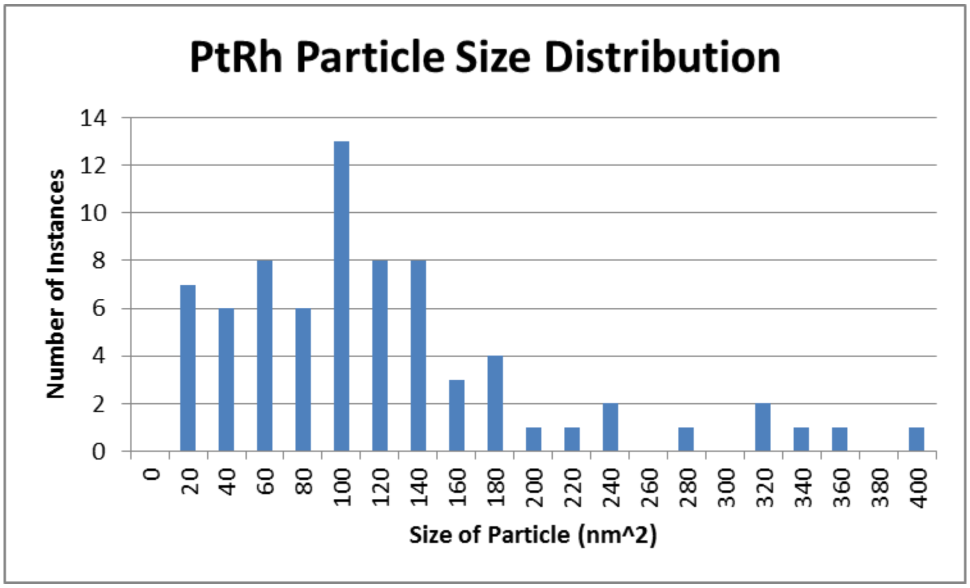

Figure 12 shows a histogram containing the size distribution of the PtRh particles seen in figure 11B.

Discussion and Analysis

In this study, five alloys of varying composition of platinum and rhodium were synthesized and characterized for use as electrocatalysts in the MOR reaction. Figures 2 through 6 are hydrogen adsorption-desorption CVs that were generated to calculate the ECSA of each coated electrode. CVs were then generated using these same electrodes in a methanol solution to calculate the peak currents generated during MOR. These currents were then normalized by ECSA to calculate the area specific activities of each electrocatalyst, an intensive property that is useful for evaluating the effectiveness of a catalyst.

The PtRh, $PtRh_2$, and $PtRh_3$ samples all exhibited low activities compared to commercial platinum catalysts on carbon support. However, Pt_3Rh exhibited a very similar activity to commercial platinum, and Pt_2Rh exceeded the activity of commercial platinum. Platinum-rhodium alloy catalysts appear to exhibit a volcano-like relationship between platinum loading and area specific activity in MOR, with a peak near the Pt_2Rh composition, as seen in figure 8. This indicates the formation of a surface that is highly effective at promoting the adsorption of methanol and hydrogen. Figure 11 shows TEM images of the PtRh sample, which confirmed that a cubic structure was formed, suggesting the formation of an alloy of platinum and rhodium using these procedures.

Going forward, additional studies should be performed on Pt_3Rh and even more so on Pt_2Rh due to their promisingly high activity. Chronoamperometry curves should be generated under MOR conditions to determine the durability of these catalysts, as durability is often times a challenge that plagues platinum alloy electrocatalysts. Fuel cell testing should also be performed to determine the resistance of these electrocatalysts to carbon dioxide poisoning. Additionally, more in depth studies should be performed on the morphology of these electrocatalysts, such as TEM or X-ray diffraction (XRD) to determine a more exact morphology of Pt_2Rh and Pt_3Rh to better understand the structure of the formed alloys and what effect it may have on the elevated catalytic activity.

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