Abstract

As the global human population increases, so does our per capita energy demands. Yet, our society still relies heavily on petroleum-based chemical energy. One focus of Chemical Engineering is to design catalysts, which are materials that increase the rate of a reaction without being appreciably consumed. However, a limiting factor of identifying ideal catalysts is the ability to be highly active, stable, and selective towards their reaction. In the following study, electrocatalysts were synthesized to improve the hydrogen evolution reaction and oxygen evolution reaction, shown in figure 1.

Figure 1: Hydrogen Evolution Reaction & Oxygen Evolution Reaction

Introduction

When comparing electrocatalysts, the quality of the material needs to be referred to a defined quantity of the catalyst. Electrocatalysts can be referenced to the mass of the electrocatalysts, geometric surface area of an electrode, geometric surface area of the electrocatalyst, or to the electrochemical active surface area of the material. As a researcher approaches the end of the previous list, the quality of comparing electrocatalysts increases as well as the difficulty of obtaining the value.

The electrochemical active surface area (EC/SA) is determined utilizing two different techniques. This first technique is an integration under the hydrogen desorption region correcting for the double layer. The values calculated are then referenced towards 210μC/cm², the charge of stripping a monolayer of hydrogen on platinum. Carbon Monoxide (CO) stripping is then used as the second technique to corroborate the surface area determined by the previous integration. The integration in the CO oxidation region is compared to a value of 420μC/cm², the charge of stripping a monolayer of CO on platinum.

The final objective of this project is to create active, stable electrocatalysts for the hydrogen evolution reaction and the oxygen evolution reaction. Platinum surface atomic concentrations for a series of 5, 10, 20wt% Platinum/Carbon (Pt/C) electrocatalysts were explored using cyclic voltammetry. However, only the 20wt% Pt/C sample is shown. Figure 2 shows a representative SEM image of Pt supported on high temperature annealed carbon nanotubes (here 5wt% Pt/C).

Figure 2

SEM of 5wt % Pt/C

Methods

Synthesis

1. Incipient wetness impregnation with Pt salt/Water/Ethanol solution and a chosen carbon nanotube followed by air drying
2. Reduction of the catalysts in a tube furnace under hydrogen at 200°C for 30min shown in figure 3
3. Catalyst dispersion in 0.5wt% nafion solution by sonication
4. Placement 100μL of catalyst on glassy carbon electrode followed by air drying as shown in figure 4

Testing

1. A standard three electrode set up was constructed with a Ag/AgCl reference electrode, Pt wire counter electrode, and glassy carbon working electrode shown in figure 5
2. All experiments were conducted in 0.5M H₂SO₄
3. Pre-treatment run using voltages as shown in figure 6
4. CO stripping technique performed
5. Surface area integration determined

Figure 3: Tube furnace

Figure 4: Glassy Carbon electrode with catalyst

Figure 5: Electrochemical setup

Figure 6: Pre-Treatment and CO stripping parameters

Results

Stripping of the under potential deposition region of hydrogen (UPD-H) occurs around 0.0V as shown in figures 7 and 8. This is indicative of how much hydrogen was adsorbed. By comparing this value to the charge of the formed monolayer, 210 μC/cm², the surface area can be calculated. This change is affected by the surface characteristics and is necessary to ensure an effective monolayer.

CO is easily adsorbed to the electrode surface and displaces pre-adsorbed hydrogen. During CO stripping, CO is desorbed from the electrocatalyst surface. The value is then compared to desorption value of a monolayer of CO, which is 420 μC/cm². It is important to ensure oxygen has been removed during these measurements.

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Future Research

- Determination of the electrochemical surface area on other high temperature annealed carbon supports

Works Cited


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