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by

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**A Study of Optimized Carbon-based Ultracapitors**

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**A Study of Optimized Carbon-based Ultracapitors**

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**Report**

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## **Dedication**

This is dedicated to my parents Herman and Marilyn Roessler who taught me life's lessons, how to live with God in my heart, and the lesson of tenacity even when others do not believe that you are capable of it. Also thanks to my family and friends who have always been there for me. Finally to three teachers who made a difference in my life by vastly distinct ways, the late Ms. Sandra Blair and her guidance on how to live life and how to teach with a full heart, Mr. Mark Vossemer who taught me the value of believing in everyone no matter their ability, and Mrs. Elaine Bigham who endlessly helped with all my career transitions.

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## **Abstract**

### **A Study of Optimized Carbon-based Ultracapacitors**

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The University of Texas at Austin, 2010

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Research was compiled in regards to the effect of various changes in the development and fabrication of the ultracapacitor. The binder was changed from Teflon to a mix of Teflon and Nafion. Results demonstrated that an increase in capacitance was seen in the ultracapacitor and is may be attributed to the structure of Nafion. Furthermore water soluble graphene, graphene oxide, and ionic liquid graphene was tested in order to determine the effects of those alterations on the overall capacitance.

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## INTRODUCTION

Society has been using batteries since before 250 B.C., when rudimentary systems were built using metals pots and rods as the electrodes, and common acids, such as wine or vinegar, as the electrolyte. In the 1800's, scientists began to develop capacitors in order to improve the energy storage capabilities of batteries. Today capacitors are significantly improved and often termed super- or ultracapacitors. Modern ultracapacitors, in particular, are composed of double layers of charge and use various ionic solutions that are better for the environment and more sustainable. One goal is to find an ionic solution that will maximize the performance of the ultracapacitor and enable cost-effective mass-production (Bielawski 2008). Currently, scientists have been exploring systems, which utilize one-atom thick layers of  $sp^2$ - hybridized carbon sheets, known as graphene (Si et al 2008). Evidence currently indicates that graphene may double the energy capacity of current ultracapacitors (Agnihotri, 2008). In fact, research has demonstrated that the energy stored in the capacitor is inversely proportional to the thickness of the double layer and therefore the capacitors have an extremely high energy density compared to conventional dielectric capacitors (Agnihotri, 2008). The overall aim of the project in which I am involved is to develop a feasible design for an ultracapacitor with large energy density capacity and one that can be mass produced for low cost so that the technology can be widely implemented in areas ranging from household products up to transmission and energy storage.

Ultracapacitors have properties that make them ideal for our increasing energy demands as well as our desire to improve and protect the environment. These advantages

include higher power capacity, longer life, wide temperature ranges, lightweight and more flexible packaging, lower maintenance, and environmentally friendly preparations (Agnihotri, 2008). Batteries currently have a higher energy density in comparison to ultracapacitors. However, ultracapacitors release and store their energy at a faster rate than batteries (Agnihotri, 2008). Unlike batteries (e.g. lead acid), it is possible to use non-toxic chemicals or more environmentally friendly chemicals in ultracapacitors, such as KOH and other common salts. Furthermore, they are ideal for applications having a short load cycle and high reliability requirements such as recapturing energy, like those used hybrid cars or trains in Japan (Agnihotri, 2008). Ultimately, ultracapacitors may deliver higher energy densities ratings than rechargeable batteries, but this is the current outstanding challenge.

Energy density refers to the amount of energy stored in a system over a given amount of space. Electrodes used in contemporary ultracapacitors are being constructed of a variety of materials including high surface area conductive carbon materials such as chemically modified graphene sheets (Agnihotri 2008). The conductivity of the materials is approximately  $2 \times 10^2$  S/m which closely approaches that of pure graphite (Agnihotri 2008). Recently discovered the graphene sheets are not pristine (i.e., pure carbon), there are topographical defects, which range in size from 10 Å high and 10-200 Å wide (Ruoff, 2009). Some of the causes may include absorption of hydroxyl radicals or an-harmonic coupling occurring due to thermal fluctuation (Ruoff, 2009). If these ripples could be removed then the surface area could be increased and the energy density would increase. Activated carbons are believed to store charge in tunnel-like pores, so with the reduction of ripples would increase these tunnels and increase the energy density (Agnihotri 2008).

The benefits of the use of chemically modified graphene in comparison to that of activated carbon include high surface area which leads to gains in energy density, increased compatibility of high voltage electrolytes, and they do not need additional additives in order to show increased conductivities (Agnihotri, 2008). Furthermore, the chemically modified graphene sheets have a low equivalent series resistance, which increases performance, decreases the temperature fluctuations during loads, and prolongs the life of the capacitor as well as the unit in which the capacitor is being utilized (Agnihotri, 2008). Another benefit of chemically modified graphene sheets is that they reduce the adverse effects of water that is present in activated carbon (Agnihotri, 2008).

Another area of development is in ionic liquids for use as the electrolyte layer. Ionic liquids are defined as salts that melt below 100°C and are composed of specific ions. These salts have unique properties, which make them ideal for battery development and also for ultracapacitors, including non-volatility, non-flammability, high thermal stability, good solvating ability, and large liquid ranges (Ruoff, 2009). Some of these show promising properties including 1-ethyl-3-methylimidazolium tetrafluoroborate and 1-butyl-3-methylimidazolium bis(trifluoromethanesulfonyl)amide that are first dissolved in alkyl carbonates (Ruoff, 2009). So far, researchers have found that not only will the type of ionic liquids aid in further improvement of the ultracapcitor, but the ratio of these liquids to water need to be optimized in order to influence the properties of the nanosheets (Agnihotri 2008). There are properties that need to be met by the ionic liquids in order for them to be viable in the use in ultracapacitors. These properties include the following: increased electrochemical window, decreased melting point, lower viscosity, increased thermal and environmental stability, reduced cost and toxicity, and

able to be readily scaled and purified (Agnihotri 2008). There are several specific goals for the research and development of ionic liquids for the development of ultracapacitors, including reducing the melting point of ionic liquids by decreasing the interactions between the cation and anions so that there is an increase in various structures (Agnihotri 2008). The viscosity of the ionic liquids also needs to be reduced, which will improve the thermal stability of the ultracapacitor. Fluorinated anions will be explored and used to broaden the electrochemical window of ionic liquids because fluorinated anions also can increase the thermal as well as chemical stability. Finally, the ionic liquid must be developed so that it is low in cost and toxicity (Agnihotri, 2008).

Researchers are currently focused on improving the properties of ionic liquids used in ultracapacitors. Currently they have discovered that ionic liquids that contain non-coordinating counter anions will be liquids at temperatures that are close in a specific range and will demonstrate high thermal and chemical stability. The halides that are attached in the process can be removed through filtrations. Another area of interesting development is ionic liquids with bisimidazolium salts. These salts are composed of two linearly opposed imidazolium salts which are situated opposite of each other. This arrangement in the molecule yields them with large aromatic surface area which may have increased interaction with the graphene sheets. This unique property may also lead to increased electrochemistry properties (Bielawski, 2008).

Ultracapacitors have specific needs and qualities that must be met in order for them to be a feasible solution. They must be capable of facilitating cycling, efficient at charge storage, scalable, have high energy, large power density, be environmentally friendly, and cost effective (Agnihotri, 2008). If these needs are met then

ultracapacitors are a feasible solution for our demanding need for fast and effective energy storage.

## BACKGROUND

### Ultracapacitors

In the development of the ultracapacitors, the concept of the energy stored being inversely proportional to the thickness of the double layer provides parameters for the construction of the ultracapacitor. Also there are some limitations of the lifetime of the ultracapacitor which is due to faradic redox reactions occurring within the electrode (Stoller 2008). Chemically modified graphene has similar conductivity to that of pristine graphite which enables the electrodes to be constructed of greater thicknesses. This allows more flexibility in the construction, thus more versatile for various purposes. The two electrode test cell used to characterize the ultracapcitor is composed of a stainless steel base followed by a sheet of Mylar used as insulation, then a current collector, the electrode, and charge separator (see Illustration 1 and Fig 2). Then the inverse of this pattern is repeated (Stoller 2008).

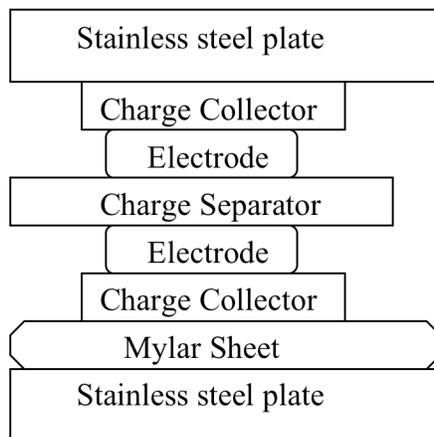


Illustration 1: Schematic of an Ultracapcitor



Figure 1: Photo of an assembled Ultracapcitor

There are two types of test used to evaluate the capability of ultracapacitors. These two tests include cyclic voltammetry and chronopotentiometry. Cyclic voltammetry the electrode potential is tested linearly in comparison to the time. It shows the charge and discharge potential in the electrode over time and with varying potential. In an ideal situation the charge and discharge should occur quickly and therefore the graph should appear almost rectangular. In chronopotentiometry the current is held constant and analysis of the electrode is based on the rate of change in the potential versus the time. Two equations are used to provide a uniform analysis of the material. The first one is used with the cyclic voltammetry and takes in account the mass of electrodes (see Figure 2).

$$C_{sp} \text{ (F/g)} = 4 * C/m$$

Figure 2: Equation used in analysis of cyclic voltammetry

Using the relationship between the slope of the discharge and the current that was utilized in the experiment uses the second equation with the chronopotentiometry in order to determine the capacitance (see Figure 3).

$$C = I / (dV/dt)$$

Figure 3: Equation used in analysis of Chronopotentiometry

### Water Soluble Graphene/ Covalent Functionalization

Researchers are also evaluating the use of surface functionalized graphene for the use in ultracapacitors. When examined at one atom thickness, the graphene appears as a thick planar sheet constructed of  $sp^2$  bonded carbon; a honeycomb matrix (see Illustration 2).

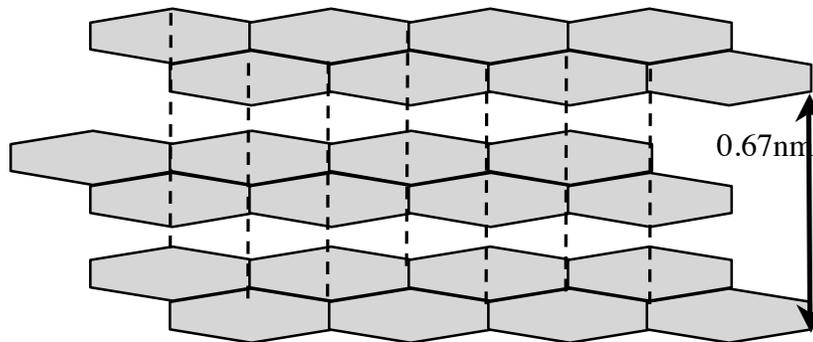


Illustration 2: Diagram of  $sp^2$  bonded carbon

Again it is these surfaces that enable charges a place to reside and enables the material to be utilized in energy capacitance. One area of research examines this as well as the possible improvement of electrical conductivity by restoring the  $sp^2$  bonds conjugation system. Exfoliation is used to produce graphene sheets by utilizing a strong acid to entice rapid thermal expansion. The problem is that there are defect sites created thus creating electronic perturbations in the material. In order to achieve the desirable properties of the graphene, vigorous reduction must take place. This reduction causes the graphene to become graphene oxide. There is a two-step process currently being studied in order to produce graphene sheets with sulfonated functional sheets as well as improved water solubility (Si & Samanski 2008). The first part of the process is to remove the remaining oxygen functional groups then introduce the sulfonate functional groups. Current findings indicate that this method had a decrease in lateral sheets in the graphene; however, that may be attributed to too much sonication. Furthermore they discovered that the electrical conductivity test was restored and it is believed that the honeycomb structure of the  $sp^2$  was restored even though this graphene is water-soluble (Si & Samanski 2008).

Another area of interest in the development of the chemically modified graphene electrodes includes investigation of the covalent functionalization. The original method for exfoliation of graphene was completed by use of scotch tape (Hummers & Offman 1958). The problems of this method includes failure to produce stable sheets capable of long term use as well as the graphene's problematic use in variety of solvents as well as the length of time needed in order to produce the graphene. Thus this amount of time also leads into scalability issues. Ionic liquids have advantages for the use with chemically

modified graphene sheets due to their wide solubility, non-volatility, non-flammability, and thermal stability (Bielawski 2008). With the aid of an atomic force microscope, evidence is indicating that graphene that has been treated with an ionic liquid have interlayer spacing in the sheets of greater approximately 1.49 nm which is greater than that of unexfoliated graphene oxide (Yang, et al 2009). Evidence in the research indicates although some of the solvent was trapped between the sheets, excess and unwanted molecules were lacking. This was accomplished by synthesizing the graphene sheets with an amine terminated ionic liquid through covalent functionalization (Yang, et al 2009). Lastly this technique provided graphene that was considered stable in that the material remained in tack for three months.

## Nafion

Teflon proves to be an adept binder for the graphene in that it has a high melting point, binds the carbon, and does not stick to other surfaces (see Illustration 3). It provides the structure for the electrodes to be formed whether if graphene or other carbon related materials are used. The material by itself can not form a useable electrode, so a binder is needed in order to cause the carbon based material to adhere together and fabricate a functional electrode. Nafion is similar to Teflon, but it differs in that it incorporates more ionic components along its polyfluoroethylene backbone (see Illustration 4).

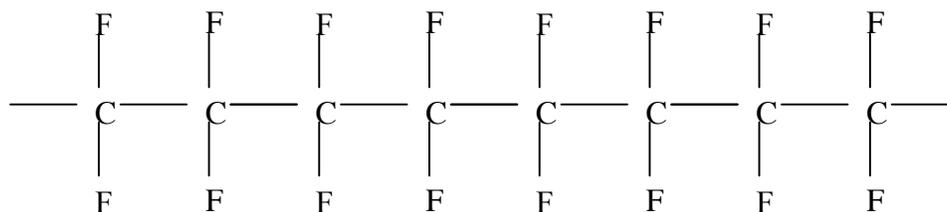


Illustration 3: Diagram of the Teflon Structure

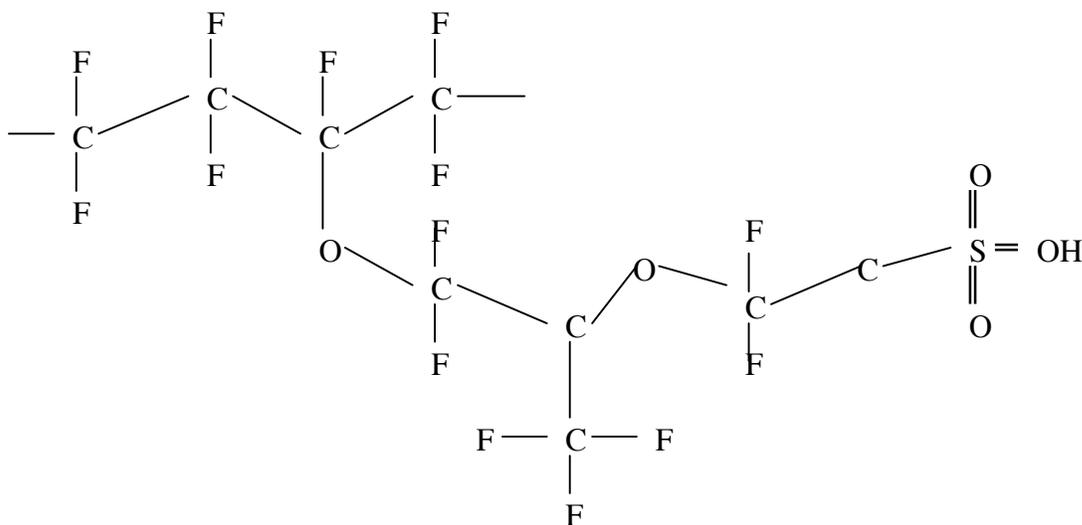


Illustration 4: Diagram of the Nafion Structure

It is thought that this ionic component would increase the electrical capacity of the ultracapcitor. Nafion has additional ionic properties associated with it due to the ionomers that are attached to it. It is believed that the additional sulfonic groups along with the increase amount of fluorines attributes to the increase electronegativity, thus making it conducive to increasing capacitance in an ultracapcitor. A problem lies in that Nafion is extremely hard to dissolve into a liquid.

## Graphene Oxide

One of the original methods for producing graphene oxide is referred to as the Hummers method. This method utilized potassium permanganate and sulfuric acid in order to produce the graphite oxide. In order to control the exothermic reaction an ice bath was utilized in order to control the reaction (Hummers & Offman 1958). The

overall process is highly exothermic and has to be controlled at all steps of the process. Recent studies have demonstrated that in the final phase of the production of the graphene oxide hydrazine or sodium borohydride can be used to complete the final reduction process of the graphene oxide. This process produces graphene that is very similar in physical and chemical attributes to that of pristine graphene. Graphene oxide is favored due to its flexibility is attachment of various functional groups. However some functional groups are easier than others to attach onto them and also others are easier than others to remove. Further understanding of the functional groups on the graphene oxide produces various properties that could be utilized in fabrication process for various electrical engineering projects (Dreyer et. al 2009).

## METHODS

The first phase of the research involved understanding how to make the electrodes. Currently activated carbon is being used for the electrodes because it has tunnel like structures that yield a place for the charges from the liquid to be housed. The carbon compound that is used must be a fine ground compound. This enables the binding process to be smooth and the area for charge becomes uniform. To begin the process of making an electrode, 0.020-0.025 grams of Norit Super 30 Carbon is measured out on the balance. The mass is recorded in this step so that that information can be used later to determine the ratio of binder to carbon. Then an automatic pipet that is set for a standard of 2.5  $\mu\text{L}$  of polytetrafluorethylene (PTFE; 60 wt% in water), also known as Teflon, is added above the graphene in the mortar and not directly. The pestle is used to mix carbon and PTFE until they are homogenized.

The next part of the process of making the electrodes was the rolling of the electrode. The bound carbon compound was placed on a double folded sheet of aluminum foil. A stainless steel or aluminum rolling pin of approximately one-inch thickness was used to begin rolling the electrode. Then the thickness was checked periodically to ensure a specific thickness of 0.08-0.06 mm thick. One centimeter diameter electrodes were cut using a small punch. The electrodes were then dried *en vacuo* to remove residual water.

Once the electrodes have been allowed to have adequate time to remove the water from them, then the electrodes are massed again. The electrodes were then soaked for at

least 24 hours in either 6 M KOH (aq) or 1 M tetraethylammonium tetrafluoroborate (TEA BF<sub>4</sub>) in either propylene carbonate (PC) or acetonitrile (AN). The 2-electrode test cell was then assembled according to Illustration 1, where conductive vinyl (aqueous KOH) or carbon-coated aluminum (TEA BF<sub>4</sub> in PC or AN) was used as the current collectors, the electrolyte-soaked carbons as the electrodes, and a porous polypropylene (Celgard 3501) as the separator.



Figure 4: Preassembled Ultracapacitor

The parameters for cyclic voltammetry depend on the type of solution that was used for the soaking. For potassium hydroxide the parameters were based on water. So the low voltage was set at zero and the high was set at 0.8. The scan rate began at 0.05 Volts and was increased to 0.015 and 0.025 V. There were at least ten sweeps done on the sample at each of the voltages. Since the sweeps were paired for a charge and a discharge cycle, then technically there were five sweeps total.

The parameters for chromopotentiometry included the cathode current being set for 0.005 A and the anode current set for the same value. The high voltage for the test was set to 0.8 V and the low was set to zero. Just as in the cyclic voltammetry the ultracapacitor charges and discharges so the number of sweeps was five sets. Thus the parameter for segment sweeps was ten. On this graph the very top of the peak on the discharge side has an irregularity that occurs regularly on all peaks. This is due to internal resistance of the ultracapacitor and is inevitable (Stoller & Ruoff). During the test one must make sure that the cell is not being overdriven or that the rates of discharge are not too low. Both of these methods leads to overstating of the ultracapacitors capability and are unreliable for actual applications (Stoller and Ruoff).

## **Other Binders**

The next phase of the research was to determine what other binders could be used as well as what results electrically they would yield. There were nine polymers which included: Nafion, polyethylene glycol monoethyl ether, polyvinyl alcohol, polyacrylic acid, polystyrenesulfonic acid, Poly (2-vinyl pryridine)-b-Poly (ethylene oxide), Poly (4-vinylpyridine) [linear polymer], polyethyleneimines, and vinyl acetate. Currently Nafion is of high interest due to its high thermal and mechanical stability. The same procedures that were described above were used to attempt the assembly of an electrode. None of the polymers bound the Norit Super Carbon with only 2.5  $\mu$ L of the chosen polymer. It was hypothesized that it would take more of these polymers to bind the carbon as compared to that of the polytetrafluoroethylene. All of the polymers that were used did require more

than  $2.5\mu\text{L}$  of solution to attempt the binding process of the carbon. Not all polymers produced the binding results needed to roll for electrode production. So this process was repeated again to see if results could be produced. Each time that an attempt was made to fabricate functional electrodes, the material would not bind on itself and was brittle.

So the next phase of testing the nine potential polymers included some minor changes. One alteration occurred with the replacement of the foil with Teflon boards. This time when the mixture was rolled out, the substance was placed in between two Teflon boards and pressure was applied to the boards with the pin rolling above the sandwich. Unfortunately, when the metal spatula was brought near the carbon sheet the potential carbon bounded sheet separated by induced charges. So the spatula was coated with Teflon tape, and the effect was minimized but still displayed in each of the nine polymers material.

### **Teflon/ Nafion Mixtures**

Due to the nature of Nafion, it was decided to attempt to mix Teflon with Nafion in order to produce electrodes so that data could be derived. The procedure that had been used to produce electrodes as before was utilized; the only change was that there would be two substances used as the binder. The first ratio that was used was a fifty-fifty mix. Two micro liters of sixty percent Teflon and two micro liters of ten percent of Nafion were used. The next ratio was one micro liter of Teflon with two micro liters of Nafion followed by one to five micro liters of Teflon to Nafion. The final ratio was two micro liters of Teflon to five micro liters of Nafion. All of the ratios yielded electrodes that

could be analyzed in order to determine if the Nafion did increase the capacitance of the ultracapcitor.

## **Nafion Electrodes**

The problem with the Nafion was that when it was used by itself it seemed to be absorbed into the carbon and appeared not to bind. However, if too much was added at one time, the mixture became too wet and unusable. The overall desire was to produce a set of testable electrodes that consisted of one hundred percent Nafion as the binder. So the process was changed as an attempt of achieving this goal. Previously the process of weighing out twenty five milligrams of carbon with the addition of Nafion yielded too small of an area for punching out electrodes. So when the carbon was weighed for this new attempt, the carbon was doubled to account for any absorbing of the Nafion in the carbon. Approximately 60 milligram of activated Norit Carbon was weighed out in the mortar on the scale. Then thirty three microliters of Nafion was added to the carbon. This was ground together until no liquid could be seen. Then another thirty three microliters of Nafion was added to the material, mixed, and then placed on weighing paper for transfer. The mix was then transferred to a vial and vacuumed for at least three hours. Then the process was repeated until the desired micro liter concentration was achieved. Each time the desired concentration was achieved, the material was vacuumed off.

Then molds were made out of foil for use with the heat press that was available at the Mechanical Engineering building. A sheet of foil was removed from the aluminum

foil container. It was approximately five by six inches. Then two small blue vial lids were placed in the center and a ruler was used to box this area off. This would be the area that the carbon would sit in for the press. Then the parallel ends were carefully folded onto itself six times until a thickness of 0.10 millimeters was achieved. The foil itself had a thickness of 0.02 millimeters, so the difference between the two levels would yield 0.08mm thickness. Next the other parallel sides were folded. Any place of potential overlapping area between all four areas was cut off to ensure the desired thickness all around the area. Another sheet of foil was cut up into rectangles and was used as the cover for the mold.



Figure 5: Heat Press Machine at the Mechanical Engineering Building

Then the vials and molds were then taken to the heat press at the Mechanical Engineering building. The temperature of the top and the bottom plate was 250 Celsius. Once the temperature was achieved, then the carbon was dumped out onto the center of the molds. The mix was spread throughout the center of the mold and then the foil was

placed on top. It was then placed on the bottom press plate. The two plates were pressed together to a desired pressure of five thousand psi. This was left in the press for about two minutes. It was then checked and repressed. After the second press the samples were removed and checked again. They were left in the molds until they were returned to the lab.

Then at the lab the punch was used to punch out the electrodes from the Nafion-Carbon sheets. These electrodes were weighed, placed in a vial, and soaked in acetonitrile solution. The Nafion electrode with only 164  $\mu\text{L}$  broke once the solution was pipetted onto the electrode. This set of electrodes had the thinnest thickness being at 0.24 mm. The second set that consisted of 264  $\mu\text{L}$  and had a thickness of 0.35 mm survived the transfer of the liquid in the vial.

### **Nafion/Teflon Electrode Vacuum Preparation**

Two techniques were combined in order to attempt to fabricate an electrode with a binder that consisted of almost entirely Nafion. Just like in the fabrication of the Nafion electrodes the amount of activated charcoal that was acquired was twice that of the original electrode technique. Then sixty micro liters of Nafion was pipetted into the mortar with the activated carbon in two stages. The material was mixed with the Nafion until no Nafion residue was seen in the mortar. Afterwards the carbon mixture was placed on weighing paper for transfer into a marked vial. It was vacuumed for ninety minutes in order to remove the excess water from the material.

The vial was removed and the contents were dumped into the mortar. Only one micro liter of Teflon was added to the mixture and it was kneaded with the pestle until it adhered onto itself. The material slowly bound and formed the roll-able sheet. Then it was rolled out to the desired thickness and electrodes were punched out of it. Just as with the other procedure, the electrode filled vials were vacuumed once more, weighed, and then solution was added for soaking.

## **Graphene Oxide**

The next phase of the research was to produce the graphite oxide by beginning with flaking graphite and using sulfuric acid and potassium permanganate. The reaction is an exothermic reaction so control of the reaction was vital. Two grams of flaking graphite was weighed out and placed in a clean two hundred fifty milliliter round bottom flask. Then the catalyst sulfuric acid was measured out to a quantity of one hundred milliliters. This was added to the round bottom flask and carefully swirled to ensure that there were no chunks in the solution. Then the flask was placed in an ice bath. Ice was moved around and up to the top of the curve of the flask but not too the neck of the flask. A stirring rod was placed in the flask to ensure constant stirring since the solution over time becomes more viscous due to the hydrophobic properties being reduced and changed to hydrophilic properties due to the reaction that is being done. Once the solution has the same temperature as the ice, then six grams of potassium permanganate is weighed out. Then the potassium is added slowly in a controlled manner over a three hour time frame.

This keeps the reaction under control and prevents the exothermic reaction from getting out of control.

After three to four hours in the ice bath all of the potassium permanganate was added to the solution and the mixture had been thoroughly mixed. The flask was then removed from the ice bath and placed on a heating/stirring plate. No heat was needed to complete the reaction; rather the stirring mechanism is the only vital part of the phase. The solution was continuously stirred until the solution reaches room temperature. Once the solution reaches this point, then it is placed in a mineral oil bath. It remains in the oil bath for a minimum of twelve hours or more.

Once the graphite solution has remained in the mineral oil bath for the desired time frame, then the color is checked. It should appear to be a grayish color and should appear to be very viscous. The solution is then placed in a 1000mL Erlenmeyer flask with a stirring bar in it. Slowly the graphite solution is poured into the Erlenmeyer flask and the stirring knob is turned on so that it is stirring constantly. Then the solution is diluted up to the one thousand milliliters line with deionized water and is placed on the heating/stirring plate. The solution turns to a dark maroon color. The solution is allowed to continuously stir while the temperature of the solution equalizes to the room temperature.

When the solution in the flask has equalized to the room temperature then the color appears muddy brown. Slowly thirty percent hydrogen peroxide is pipetted into the solution. About thirty milliliters or more is pipetted into the solution. As it is pipetted into the solution bubbles appear throughout the solution. The purpose of the hydrogen

peroxide is to quench the excess potassium permanganate. Otherwise if the potassium permanganate remains in the solution it will form manganese oxide which is insoluble in water and therefore provides poor filtration later in the experiment. The hydrogen peroxide oxidizes it and forces the magnesium to form manganese sulfate which can be removed through filtration. About fifty milliliters of the hydrogen peroxide was added to the solution. The color changed to a golden rod color which was used as an indicator that quenching was successful.

Then a Frit Filter apparatus was assembled for filtration. The purpose for the filtration is to remove the excess liquid from the graphite oxide even though it is hygroscopic. If the substance was heated the oxides on the graphite would be removed. The oxides provide the functional groups of interest in the research. The golden brown solution was slowly poured through the filter and the excess liquid was filtered out. This liquid was the sulfuric acid from the previous steps. The substance in the filter had to be stirred occasionally since the graphite oxide builds up along the filter. After it was filtered, then the graphene oxide in the frit filter was gently lifted from the apparatus so that the liquid could be disposed of properly.

Once the liquid was disposed of properly, then apparatus was assembled again. This time six molar hydrochloric acid is used to flush the graphene oxide. One liter of hydrochloric acid is slowly added to the frit filter. The purpose of the hydrochloric acid is to remove the remaining sulfuric acid. Hydrochloric acid is easier to remove from the graphene than the sulfuric acid. However it will not neutralize the solution nor does it readjust the pH of the graphene oxide toward the neutral range.

After the flushing with the hydrochloric acid, then the graphene oxide is flushed with deionized water. Again one liter is the quantity of choice. When the water is added, the pH is adjusted back to near neutral range and there is an observable color change in the graphene oxide. It turns a murky dark brown in color. Just as before a stirring rod is used to keep the bottom of the filter clear for filtration. This filtration takes a longer period of time due to the hydroscopic nature of graphite oxide and therefore this step takes several days to filter one liter of water through the graphite oxide.

Once the graphene oxide has been filtered with the one liter deionized water, then the material is gathered and placed in glass vials with screwable lids. These vials are taken to the vacuum hut with the lids slightly loosened. The graphene oxide is allowed to remain in the hut for at least two days or until the remaining water is removed. Then the graphene oxide is placed all in one vial for further treatment.

The graphene oxide is weighed out on the scale in order to determine the ratio of hydrazine to graphene oxide. After it is massed, then the material is placed in a round bottom flask. About one hundred milliliters of deionized water is added to the flask. Then it is allowed to sonicate for at least two hours. Once this is complete the solution should appear to be clear. Three microliters of hydrazine is plunged and released into the bottom of the flask. The use of three microliters is based on the relationship that one micro liter of hydrazine is needed for each three milligrams of graphene oxide. Once this is complete then the solution is placed on a hot plate for six to eight hours. After this phase the solution has separated and the graphene oxide has precipitated out of the

solution. To collect the graphene oxide, the solution is then filtered with a filter. The material is collected and placed in a marked vial.

### **Other Carbon Electrodes**

There were two vials of carbon that had been prepared with a few changes on them. The first vial had an Ionic Liquid combined with the carbon. Since this carbon was chunkier, the first step was to grind it to a fine powder. Once the powder texture was achieved the carbon was weighed out and the Teflon was used as a binder. The carbon soaked up the Teflon as soon as the Teflon approached the carbon. The carbon mixed properly, but on the first and second roll out, the surface was enumerated with holes throughout. So the material was packed again with the mortar and pestle until it formed a small and compact ball structure. Then it was rolled out again. This time only the outside had holes in it and the middle was devoid of patches. Two electrodes were punched out. The material was rolled out again. One last set of electrodes was punched out before the material became too tough to manage. The thickness range of 0.06-0.08 milliliter was attained.

The second vial contained water soluble graphene. This graphene was squishy and very hard to grind to a fine powder. Tweezers were used to shred the graphene into smaller bits. Once small enough bits were obtained then 2.5  $\mu\text{L}$  of Teflon were used to attempt the binding process. However the graphene would not roll together, so another dose of Teflon was added. The same results were seen in which the binding of the graphene did not occur even after lots of grinding in the mortar and pestle. A final dose

of Teflon was added and the graphene rolled up into one full sheet. The graphene was then moved to the foil sheet and several rolls were attempted. The graphene had the texture of crushed vermiculite that was gray. There were lots of holes throughout the graphene even after several folding and rolling. So the sheet was placed in a vial and allowed to sit in the drawer over night.

The next day the graphene was placed in the mortar and pestle and the graphene was rolled over and over again. The color changed from a white gray to a dark gray and when it was rolled out it began to have the consistency of the Norit Carbon that had been used on the first day. Six electrodes were able to be punched from the water soluble graphene oxide. These electrodes were treated in the same manner as previously stated in the first method of the production of electrodes.

## RESULTS

### **Teflon/Nafion Electrodes and Potassium Hydroxides**

The control in the experiment was the Teflon electrode, which had a capacitance of 0.06 Farads. The mass of the electrode was 0.0055 grams and when this is normalized the value of capacitance per gram is 10.90. Each time that the ratio between the Teflon and Nafion was altered so that the Nafion was higher in relationship to the Teflon, the capacitance increased. The greatest capacitance seen in the trials was that of the one to five ratio of the Teflon to the Nafion. In this trial the capacitance rose to 0.18 Farads. When using the mass of the electrode normalizes the results the capacitance does demonstrate an increase in capacitance with the increase of the Nafion, but the increase is much less than the original growth from the full Teflon bounded electrode and the 50:50 ratio electrodes. The 2:5 ratio has a capacitance of 19.63 F/g and the 1:5 ratio has a capacitance of 19.79 F/g. So once again capacitance increased but a limit of increase of capacitance may be close to being achieved in the material.

Table 1: Values for Chronopotentiometry of KOH electrodes with Nafion/ Teflon Mixtures

Teflon		KOH
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First Peak	0.06147037	0.08134
Last Peak	0.06192717	0.08074
Teflon/ Nafion KOH		50/50 Ratio
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 v_{max}) / (t_2 - t_1)$
First Peak	0.109452736	0.045681818
Last Peak	0.110358666	0.045306818
Teflon/ Nafion KOH		1:2 Ratio
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First Peak	0.118276892	0.042273684
Last Peak	0.118631369	0.042147368
Teflon/ Nafion KOH		2:5 Part Mix
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First Peak	0.117853561	0.042425532
Last Peak	0.118424333	0.042221053
Teflon/Nafion KOH		1 to 5 ratio
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 v_{max}) / (t_2 - t_1)$
First Peak	0.180135101	0.027756944
Last Peak	0.179775281	0.0278125
Teflon/ Nafion KOH		1:60 Ratio
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First Peak	0.107151757	0.046662791
Last Peak	0.10823588	0.046195402

The chronopotentiometry for the 1:5 ratio of Teflon to Nafion electrode soaked in potassium hydroxide produced a graph with rigid charge and discharge lines. The slopes in the graph were mirror like images of each other, which demonstrates a smooth charge

and discharge (see Fig. 6). This is also amplified in the cyclic voltammetry graph seen in Figure 7.

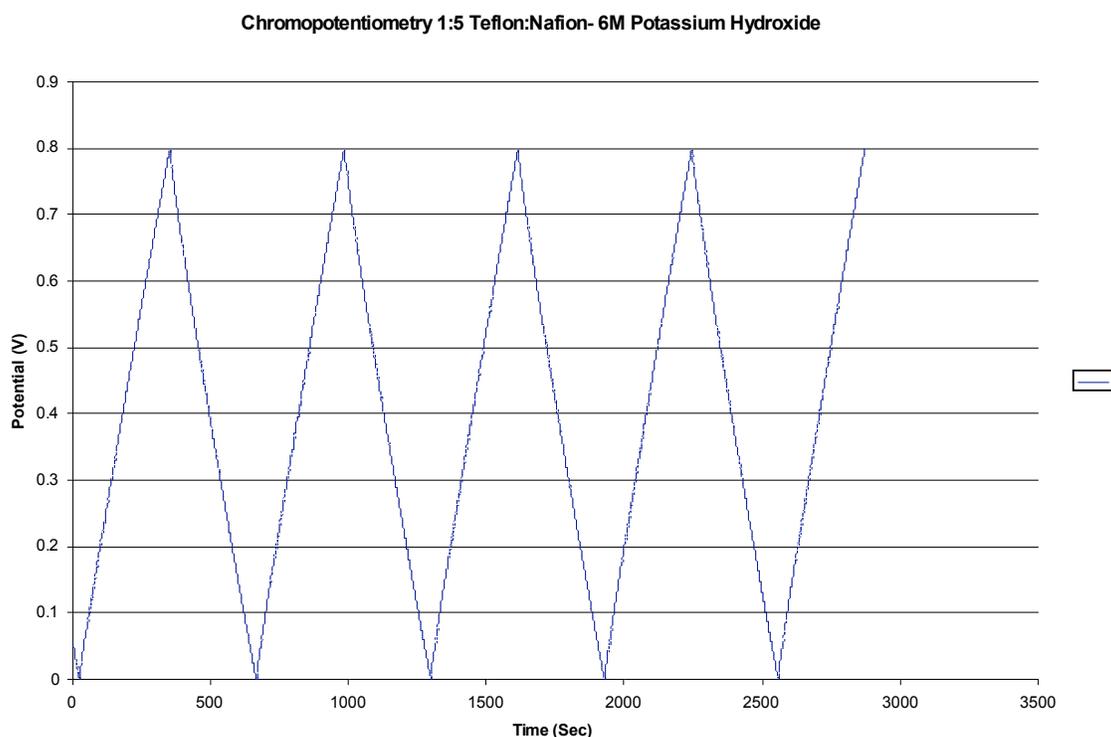


Figure 6: Chromopotentiometry for 1:5 Teflon to Nafion ratio soaked in 6 M potassium hydroxide

The cyclic voltammetry for each of the scan rates at this ratio were closer to the ideal rectangular shape that demonstrates rapid charge and discharge in the cell (see Fig 6). The trial where the activated carbon was saturated with the Nafion, then vacuumed, and then Teflon was added to it did not produce results that were above the results seen in the fifty-fifty ratio.

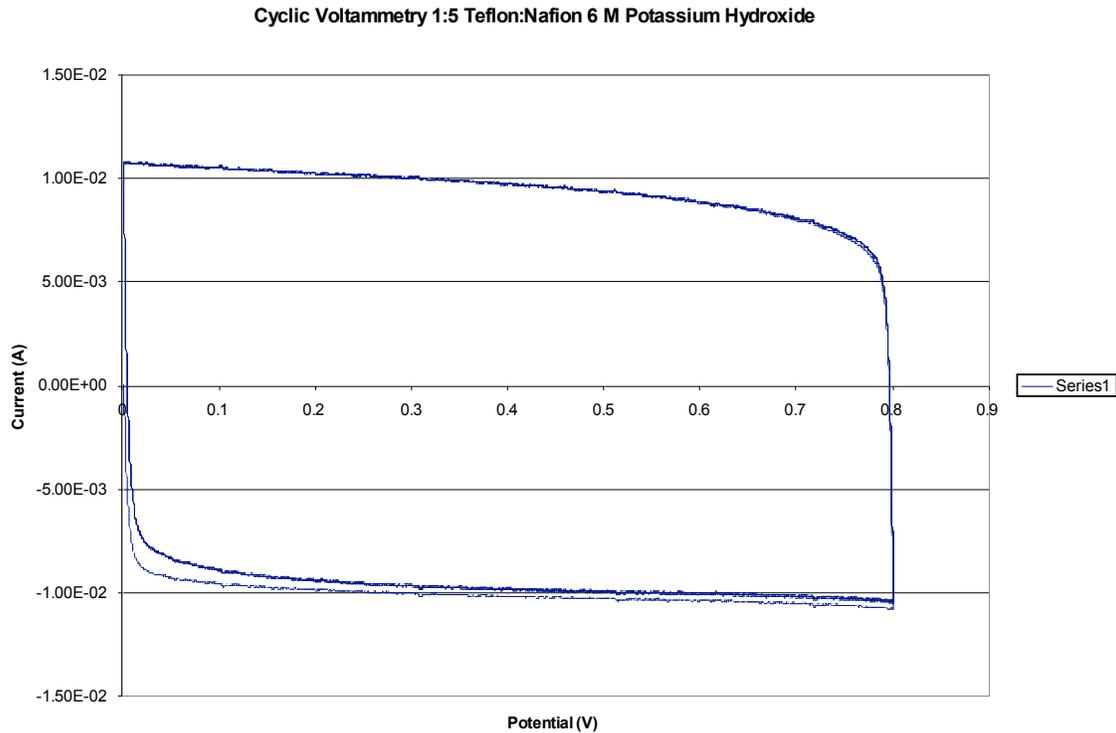


Figure 7: Chromopotentiometry for 1:5 Teflon to Nafion ratio soaked in Potassium hydroxide scan rate of 0.05.

### **Teflon/Nafion Electrodes and Acetonitriles**

In regards to the tetrabutylammonium tetrafluoroborate dissolved in acetonitrile there is an increase of capacitance in the electrode with the change in ratio between the Nafion and the Teflon. When compared to the control each step increases. At the 2: 5 ratio of the Teflon to Nafion the capacitance increases from 0.04 Farads to 0.08 Farads. When this is also equalized the capacitance is then 8.40 F/g and 13.84 F/g respectively. Then when the ratio is altered to a 1: 5 ratio then the capacitance increases to 0.12 Farads and when the electrodes mass is taken into account the capacitance per gram is 16.77.

However just as before with the electrode that was constructed of sixty microliters of Nafion and only 1 microliter of Teflon the capacitance did not increase significantly rather it was equal to that of the two to five ratio (see Table 2).

There may be a limit in capacitance that was reached or the process of vacuuming out the excess liquid and only placing liquid Teflon in it as a final binder may be the cause of the limit seen in this trial. Nafion may need to be in a liquid form in order to be equally displaced in the electrode so that the charge caused by the sulfonic group can be dispersed throughout and enable the solution's charges to also be dispensed equally.

Table 2: Values of capacitance of electrodes soaked in Acetonitrile with various ratios of Nafion and Teflon.

Teflon CH3CN		100%
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First peak	0.042016807	0.119
Last peak	0.043634908	0.114587156
Teflon/Nafion CH3CN		50/50 Ratio
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
first peak	0.058446757	0.085547945
last peak	0.06362545	0.078584906
Teflon/Nafion CH3CN		1:2 Ratio
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
first peak	0.066453163	0.075240964
last peak	0.0672	0.074404762
Teflon/Nafion CH3CN		2:5 Part Mix
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First Peak	0.087234894	0.057316514
Last Peak	0.087234894	0.057316514
Teflon/Nafion CH3CN		1:5 Ratio
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 v_{max}) / (t_2 - t_1)$
First Peak	0.120848339	0.041374172
Last Peak	0.1192	0.041946309
Teflon/Nafion CH3CN		1 to 60 Ratio
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 v_{max}) / (t_2 - t_1)$
First Peak	0.085794094	0.05827907
Last Peak	0.087130296	0.057385321

The chromopotentiometry unlike the previous one seen in with the ionic salt (see Fig. 6) does not have the rigid slopes. Here the charge and discharge slopes have a curve

surface on the charge side of the slope (see Fig. 8). The discharge side of the chart appears to look like the slopes from the previous chromopotentiometry (see Fig. 6).

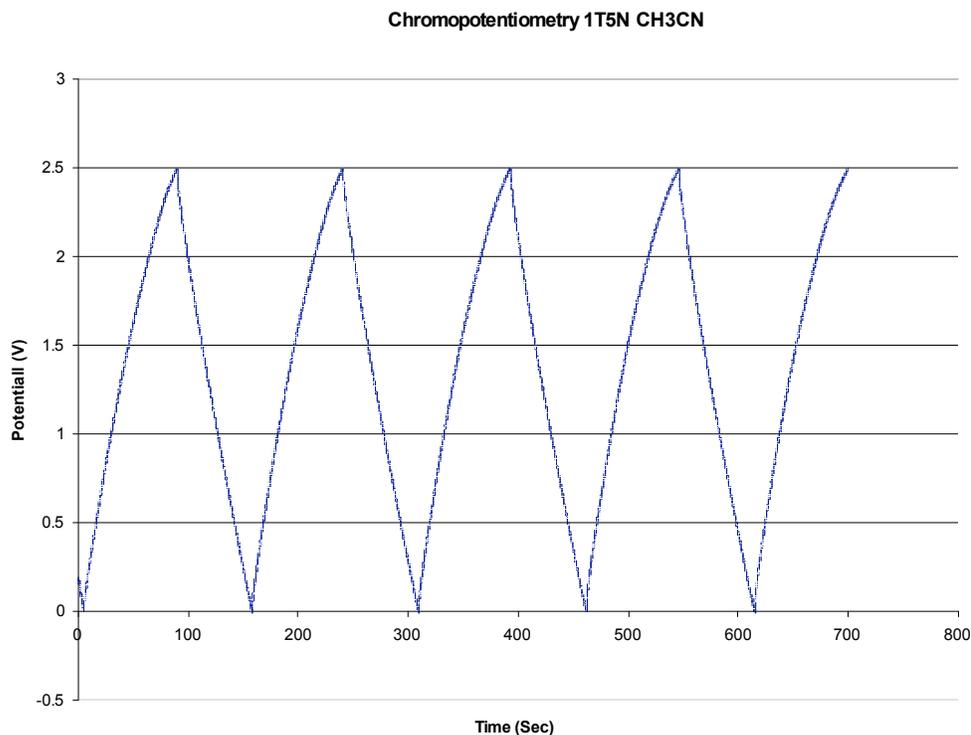


Figure 8: Chromopotentiometry for 1:5 Teflon to Nafion ratio soaked in Acetonitrile

The cyclic voltammetry for these electrodes were more rectangular in comparison of previous test with this solution (see Fig. 9), but the potassium hydroxide produced results that were more ideal (see Fig. 7). Here at the edge of the cycle where the ultracapcitor is going from charge to discharge states there appears to be a slight hook seen in the graph. This could be due to irregularities in the electrode.

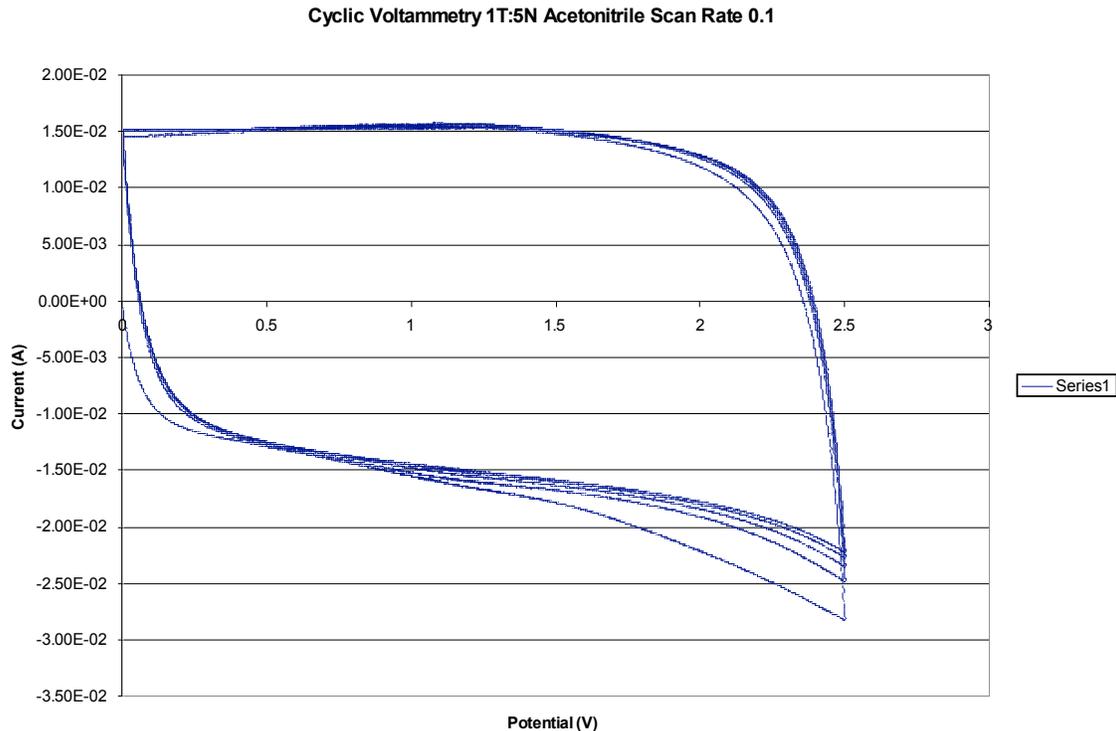


Figure 9: Cyclic Voltammetry for 1:5 Teflon to Nafion ratio soaked in Acetonitrile Scan Rate of 0.1

### **Teflon/Nafion Electrodes and Propyl Carbonate**

The propyl carbonate electrodes also showed an increase in capacitance with the change of ratio between the Nafion and the Teflon as the binder. However the greatest capacitance was demonstrated at the one to two ratio. The ratio of one to five came close to the highest ratio. When these values are normalized with the electrode mass the values become 17.25 F/g for the 1:2 ratio and 13.44 F/g for the 1:5 ratio of Teflon to Nafion. There may have been inconsistencies with the electrodes or with the solution; however when compared to the control there is still an increase when Nafion is used as a binder. Likewise as before with the previous organic salt solution, there appears to be a limitation

on the capacitance increase with Nafion as a part binder. Although just as before with the saturated Nafion electrode, the capacitance was almost equivalent to that of another previous ratio.

Table 3: Values of capacitance of electrodes in Propyl carbonate solution

Teflon PC		
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First Peak	0.024009604	0.20825
Last Peak	0.023609444	0.211779661
Teflon/ Nafion PC		50/50 Ratio
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First Peak	0.024264121	0.206065574
Last Peak	0.024028835	0.208083333
Teflon/Nafion PC		1:2 Ratio
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First Peak	0.106971154	0.046741573
Last Peak	0.1056	0.047348485
Teflon/Nafion PC		2:5 Part Mix
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First Peak	0.082065653	0.060926829
Last Peak	0.0824	0.060679612
Teflon/ Nafion PC		1:5 Part Mix
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First Peak	0.0968	0.051652893
Last Peak	0.095846	0.052166667
Teflon/ Nafion PC		1 to 60 Ratio
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First Peak	0.055422648	0.090215827
Last Peak	0.055555556	0.09

Here the chronopotentiometry shows less curved structure on the top part of the charge slope, but it is still present (see Fig. 10). It does not have the mirror image in slopes as seen in the potassium hydroxide chronopotentiometry.

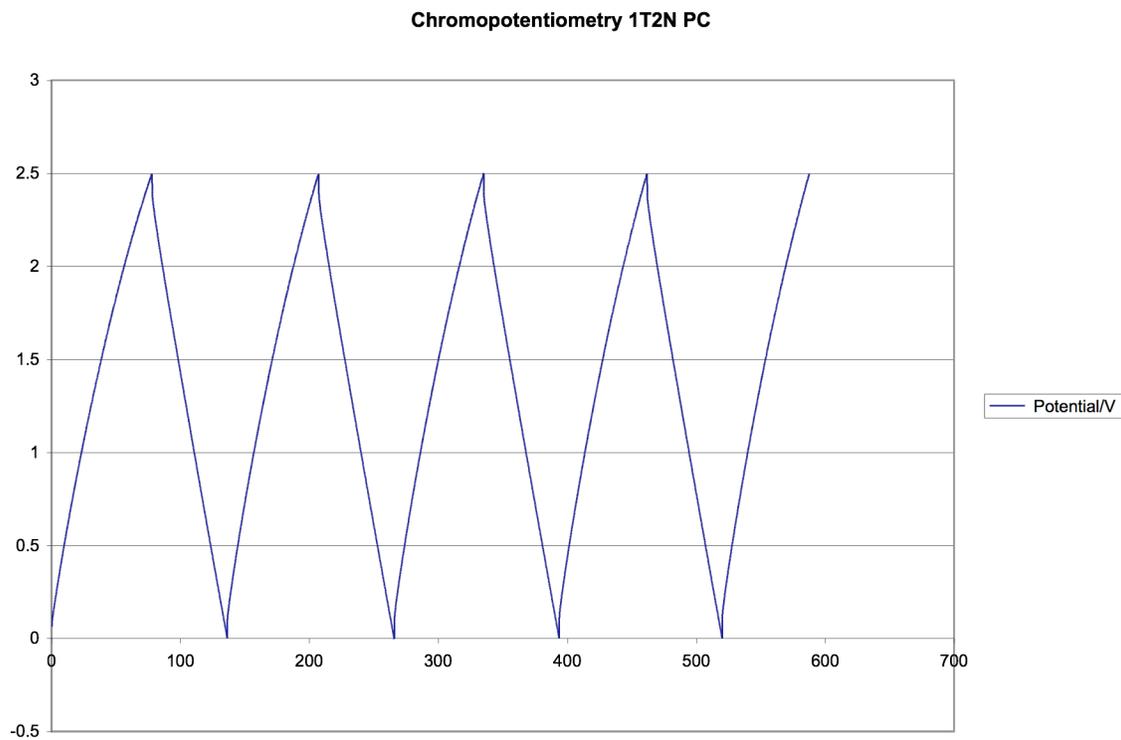


Figure 10: Chronopotentiometry for 1:2 Teflon to Nafion ratio soaked in Propyl Carbonate

The cyclic voltammetry when compared to the potassium hydroxide cyclic voltammetry is not the perfect box shape. Here again there appears to be a slight issue with the charge and discharge of the ultracapacitor. The charge and discharge is not occurring very fast and there appears to be irregularities near the turnover areas.

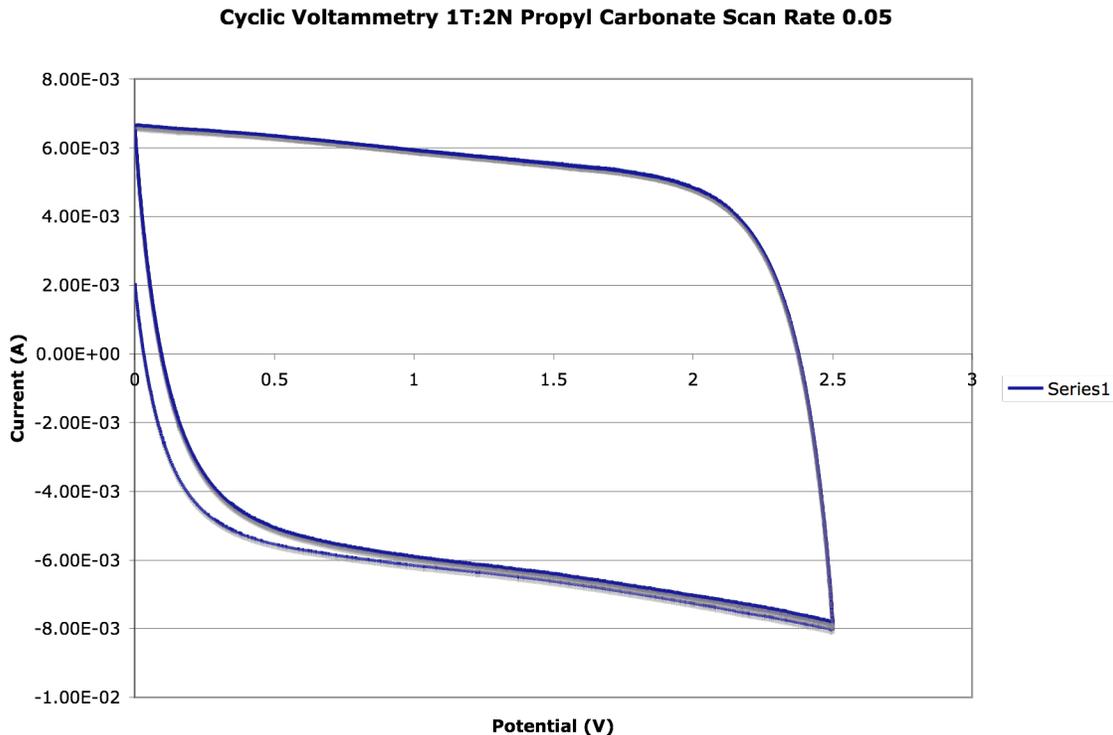


Figure 11: Cyclic Voltammetry for 1:2 Teflon to Nafion ratio soaked in Propyl Carbonate with a Scan Rate of 0.05

### WATER SOLUBLE GRAPHENE

In comparison to the Teflon electrode, the water soluble graphene did demonstrate increased capacitance. However, in comparison to the electrodes with the Nafion-Teflon mix, the water soluble graphene electrodes were not as high. In fact when compared with the Teflon electrode soaked in potassium hydroxide the value for the control was 10.9 F/g whereas the water soluble electrode had a value of 5.319 F/g. For the electrode soaked in acetonitrile was 8.40 F/g whereas the water soluble electrode had a

capacitance per gram value of 1.17. The propyl carbonate had a value of 5.21 F/g and the water soluble electrode was 3.46 F/g. The water-soluble has a higher capacitance with the potassium hydroxide in comparison to the organic salt solutions, but these values are still lower than with the Norit Carbon 30 electrodes with Teflon. No results were derived successfully from the self-produced graphene oxide nor of the ionic liquid carbon. The tests were flat lined on the capacitance.

Table 4: Values of capacitance of water soluble graphene oxide in various solutions

Water Soluble Graphene Oxide KOH		
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First Peak	0.071276729	0.070149123
Last Peak	0.072527198	0.068939655
Water Soluble Graphene oxide CH <sub>3</sub> CN		
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First peak	0.014411529	3.47 E -01
Last peak	0.014417301	3.47 E -01
Water Soluble Graphene Oxide-PC		
	$c = I/(dv/dt)$	$dv/dt = (v_{max} - 1/2 V_{max}) / (T_2 - T_1)$
First peak	0.046018407	0.108652174
Last peak	0.045618247	0.109605263

## GRAPHENE OXIDE

Electrodes were made with the graphene oxide and with graphene treated with ionic liquids. However no results could be derived from those electrodes. The testing of

the electrodes produced almost a complete flat line on the cyclic voltammetry test on each of the electrodes tested.

### **OTHER POLYMERS**

None of the nine polymers by themselves were able to produce the same consistency in an electrode like that of the Teflon. Most of them were very dry and unrollable. Even after the material had been placed in the vacuum chamber and the polymer was added to the material multiple times, there were no useable electrodes. Furthermore with the addition of the use of the Teflon boards to counter the problem of the electrodes sticking to the board, electrodes were unable to be produced. This included the Nafion polymer ,which was a focus in this project.

Table 5: Results of test with various polymers

Polymer Name	Amount Used	Results/ Observations
Polytetrafluoroethylene (Teflon)- 60% in solution	2.5 $\mu\text{L}$	Binds into a smooth malleable texture- Easy to roll and punch out electrodes
Nafion- 10% in solution	35 $\mu\text{L}$	never bound together Appears dry and powdery
polyethylene glycol monoethyl ether	5 $\mu\text{L}$	Did not bind in the mortar Attempted rolling and it stuck to the pin
Polyvinyl alcohol	9.9 $\mu\text{L}$	Did not bind in the mortar thoroughly Began rolling the graphene out Was roll able- thickness remained at 0.27mm and began to dry out at the edges
Polyacrylic acid	2.6 $\mu\text{L}$	Began to bind, on the first roll the sheet of graphene broke apart and would not bind to itself extremely brittle
Polystyrenesulfonic acid	10 $\mu\text{L}$	Would not bind together at all
Poly (2-vinyl pyridine)-b-Poly (ethylene oxide)	10 $\mu\text{L}$	It appears dry throughout Began to bind mildly Roll able- brittle Crumbled with more rolls
Poly (4-vinylpyridine) [linear polymer]	67.5 $\mu\text{L}$	Appeared to bind together, but after a few minutes it dried out again. Was roll able-brittle
Polyethyleneimines	5.5 $\mu\text{L}$	Non roll able- fell apart once it was rolled
Vinyl acetate	30 $\mu\text{L}$	Dry- extremely powdery No binding exhibited at all

## CONCLUSION

The results demonstrate a positive effect by the Nafion on the overall capacitance of the ultracapacitor. Almost each set of electrodes in which the Nafion was added in addition to the Teflon, the capacitance increased. However it appears that there may be a limitation on how much the capacitance can increase and this may be due to the solution or the structure of Nafion. When compared to the water soluble electrodes, the standard Teflon electrode had a higher capacitance per gram. However in comparison to that with the Nafion an increase of almost double was seen in the electrodes. If this is coupled with further positive alterations of the carbon backbone, then the overall capacitance of the ultracapacitor could increase even more.

Some avenues of future research could include evaluating where the saturation point occurs with the Nafion. It appears that the structure of Nafion does increase the capacitance in the electrodes and knowing where the maximum capacitance could be reached would be helpful in achieving the goal of increasing the ultracapacitors. Also it one could evaluate the effects of Nafion/Teflon mixed binder on the water soluble graphene. The water-soluble graphene structure did increase the overall capacitance and if the trend seen in the other activated carbon electrodes holds true to the water-soluble graphene then that capacitance will either be comparable or possibly greater than that of the Nafion/Teflon electrode.

Fabrication of one hundred percent electrode binded with the use of Nafion only was consistently an issue throughout the issue. The process of vacuuming the electrode with the addition of heat and pressure did not produce an electrode that was useable. Although the material appeared to bond together when the punch was applied, the material shattered. A suggestion was offered to try to make the Nafion rich carbon

material a paste and then add the heat and pressure to it so that the molecules could more easily rearrange so that the electrode could be produced.

The second technique which was a combination the two processes did produce a punch able electrode. The electrode was vacuumed and then the Teflon was added afterwards. This process did produce an electrode with similar consistency to that of the previous electrodes; however, once the electrode was soaked in solution, the electrode did lose its shape in that a spatula was used to remove the electrode from the bottle. This flexing of the electrode occurred worse in the organic salt solutions. This technique of loading the carbon up with Nafion, vacuuming the excess water, and then adding the Teflon did not significantly increase the capacitance of the material. One change that could be attempted could be to repeat the process but then add a small amount of Nafion instead of just Teflon. This would produce an electrode almost completely of Nafion and it would be rollable due to the Teflon, which would therefore be testable in order to determine if there is a limit in capacitance by the Nafion.

## **APPLICATIONS TO PRACTICE**

When it comes to lab procedures there is a time and a place for step by step procedures. These labs introduce ideas and techniques and procedures to students who are unfamiliar with them, but after this phase is completed there needs to be a time for controlled exploratory learning. Students need to be given time to evaluate the previous procedures and then be given a deviation from the previous lab. Research is a lot like playing on a team. Everyone has their specialty that they bring to the team. However just like a team sport, these individuals have to come together to develop the end-desired product. This research opportunity presented me with first hand knowledge that I can use to entice my students.

Open-ended labs give the students these opportunities to develop these skills. Even if their attempts to solve the problem do not produce an actual product, the student needs to take that moment and reflect on it. They need to ask why it did not work and then try to determine what changes they would need to do in order to make it work. Also students need to learn to be adaptable in various situations. They need those times in lab in which not all the equipment is there or the procedure did not produce the results desired. The students need to be able to look and evaluate a solution and find a feasible solution. Lastly they need to learn how to work together. Research has individual components in it. Each member brings their strength to the research but it is a team effort to produce the end results. Sometimes what was thought to work fails to work, and

talking to another person enables another idea to be born from the failure of the experiment.

Finally chemistry teachers can adjust the curriculum to incorporate the concepts of ultracapacitors into the lessons. Usually in the electrochemistry unit the focus is on the different types of batteries like dry cells, alkali batteries, and lithium batteries. This would be a great opportunity to discuss other applications of electrochemistry as well as how research is improving this area. Teachers could utilize the students own technology like the various smart phones and discuss Students receive limited exposure to electricity throughout their school years, yet they work and play with electricity everyday.

Furthermore students have marginal understanding of what electricity is and the parts of common electrical equipment. Likewise electrochemistry is a great opportunity to discuss green technology. This would be a great lesson in which students research the different types of green technologies and investigate the pros and cons of each. To further incorporate this with what has occurred here at the University of Texas, students should also research what the academic field is pursuing in regards to the problems that are yet to be solved.

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## **Vita**

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