Effect of Electrolyte Concentration on the Capacitance and Mobility of Graphene

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Effect of Electrolyte Concentration on the Capacitance and Mobility of Graphene

Flint Martino

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Abstract

The use of graphene field-effect transistors as a biosensor is increasingly being used to study biological phenomena, due to the sensitivity and low reactivity of graphene. To further improve sensitivity in biological environments, we examined how different salt concentrations affect the mobility and capacitance of the graphene. Samples were also measured after an annealing process. We report on the positive correlation between sensitivity and electrolyte concentration and speculate on methods to improve future detectors. Mobility of the device was found to change from $1.07 \times 10^3 \text{ cm}^2/(\text{V}\cdot\text{s})$ in de-ionized water to $2.78 \times 10^3 \text{ cm}^2/(\text{V}\cdot\text{s})$ in a 500 mM potassium phosphate buffer solution.
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Section 1

Introduction

1.1 Graphene

The investigation and study of graphene has become important in the creation of highly sensitive biosensors [1]. Most notably Konstantin Novoselov and Andre Geim are recognized for the initial study on this topic and received a Noble Prize for Physics in 2010. Their work showed that graphene has numerous properties including: strength, thermal conductivity, electrical conductivity, optical properties, and two-dimensionality [2]. The applications for graphene are immense and constantly growing as more study on the material is done. Our research considers how graphene can be applied to different biological environments as a biosensor, utilizing its high sensitivity, two-dimensionality and conductivity. Furthermore, the improvement of these properties and how they operate in different environments is investigated.

Graphite is an allotrope of carbon, in which the bulk structure is composed of 2-D sheets of a hexagonal structure that stack together as shown in Fig. 1.1 below. One of these 2-D sheets is called graphene; each layer is one atom thick with the height of a carbon atom. Due to the thinness of graphene its electrical conductivity is very sensitive to any change in the electric field.

![Graphite composed of layers of hexagonally structured graphene.](image)
1.2 Electronic Properties

Graphene’s conductive properties are unique in the field of 2-D materials. Novoselov and Geim’s investigation found that the resistance of graphene changes due to shifts in the electric field. To test this reaction, a gate voltage is applied to the device inducing an electric field perpendicular to the graphene sheet and if this applied voltage is swept, then resistance will change due to the voltage shift. This response is due to the vast amount of charge carriers within graphene [2].

Furthermore graphene is ambipolar; meaning that its charge carriers (electrons and holes) move around freely. In practice as shown below in Fig. 1.2 (a), when a positive bias is applied the majority carrier in graphene are electrons and when a negative bias is applied the majority carrier in graphene are holes. However, when there is no bias applied to the sheet of graphene it is near its Dirac point; a point where ideally there are no charge carriers. When at its Dirac point there are no available electronic states for graphene’s charge carriers to move into, therefore no current can flow. The further from the Dirac point graphene is pushed, the more states become available for charge carriers. For this reason it is expected that resistance becomes greater closer to the Dirac point as shown in Fig. 1.2 (b), and logically resistance is lower the further from the Dirac point. Thus, the further graphene is from its Dirac point the easier current can flow through it. Ideally the Dirac point is when, \( V_g = 0 \) V, (when the applied gate voltage is zero), because without an applied voltage to cause a charge carrier density shift the charge carriers do not move.

![Figure 1.2](image_url)

**Figure 1.2** (a) Density of States Diagram. Showing the difference from no applied voltage (the red dot and Dirac point) to where the voltage is applied showing slopes (the black lines). Also, the blue shaded region under the curve represents when holes are the majority carrier and the light red shaded region represents when the majority carrier are electrons. (b) Resistance vs. gate voltage curve showing that resistance increases as the Dirac point is neared.
1.3 Mobility
When resistance is decreased in graphene, as occurs with an applied gate voltage, the conductivity of the material also increases. This occurs because when resistance is lowered it makes it easier for the movement of charges through a material and the easier it is for a charge to move through a material the more conductive that material is. This action is due to an increase in the number of free charge carriers in the material, the fact these two actions are connected means that by changing the number of charge carriers one can change the conductivity of the material as shown below [2].

\[ \sigma = \mu n_e e \]  \hspace{1cm} (1)

Where \( \sigma \) is the conductivity of charge carriers in graphene, \( \mu \) is the mobility, \( n_e \) is the number of charge carriers, and \( e \) is the charge of an electron. Mobility describes how easily electrons move through a material. The easier electrons can move through a material, the more sensitive those electrons will be to a change in the environment. This reaction causes a greater shift in the conductivity of the material and this reaction can be detected if the voltage on the surface of the material is changed. For this reason, graphene is advantageous as a biosensor candidate [1]. A graphene biosensor has the potential to detect shifts in pH, electrolyte concentration, and bacteria [3, 4]. Increasing the sensitivity of these devices is of high importance and this can be done through increasing the mobility of the devices. Below shows how to characterize the increase of mobility to the slope of the conductivity curve, dependent on gate voltage.

\[ \mu = \frac{1}{C} \frac{\partial \sigma}{\partial V_G} \]  \hspace{1cm} (2)

Where \( C \) stands for capacitance, the capacitance in this equation is the total capacitance of the device, which will be explained further in section 2.1. Also, \( \frac{\partial \sigma}{\partial V_G} \) is the change in conductance over the change in gate voltage; this is represented by conductivity vs. gate voltage curve shown in Fig. 1.3. The steeper the curve is the more sensitive this device is, meaning the device can detect smaller shifts. [2]

![Figure 1.3](image-url) Conductivity versus gate voltage curves. The mobility of a device can be characterized by the slope of the curves shown. A higher mobility relates to a higher sensitivity, as \( \sigma \) will change more in (b) than (a).
1.4 Types of Devices

There are two different types of devices that are tested, one is a top gated device and the other is a bottom gated device. Both devices are tested with a voltage difference set across the graphene sheet creating a current through the material. A bottom gated device has an applied bias through the base (often SiO$_2$) till it affects the graphene on top of it. These devices are tested in vacuum and have mobility values of over 4,000 cm$^2$ / (V*s). Top gated devices have an applied bias into a liquid on top of the graphene; these devices cannot be tested in vacuum due to the liquid on top of them and have shown far lower mobility values than bottom gated devices such as 380 cm$^2$ / (V*s)[3]. Since an electrolyte solution on top of the graphene should not damage the device, it does not make sense for mobility to be significantly lower in top gated devices than in bottom gated devices. A hypothesis for this issue is that the liquid on top gated devices is scattering the charge applied to the device, this could explain this difference in mobility values. Therefore the mobility in top gated devices may be the same as in bottom gated devices; it may be that we are just not able to measure the mobility of top gated devices properly due to charge scattering [5].

![Diagram of top and bottom gated graphene devices](image)

**Figure 1.4** (A) Liquid top-gate graphene device. The purple represents graphene, the yellow represents metal pads, the blue represents the silicon base [5], and the light blue represents the top gated liquid. (B) Bottom gated graphene device. The purple represents graphene, the yellow represents metal pads, and the blue represents the silicon base.
Section 2

Theory

2.1 Capacitance

The capacitance of the graphene devices is derived from the changes in resistance due to the swept electric bias. Ohms law characterizes this interaction as the potential difference is equal to the current multiplied by the resistance as shown below. This method shows a path to derive the capacitance of graphene from the change in resistance due to a shifting electric field.

\[ V = IR \] (3)

Where \( V \) is the potential difference, \( I \) is the current across the graphene, and \( R \) is the resistance of the graphene device.

\[ R = \rho \frac{\ell}{\ell \cdot w} \approx \rho \frac{\ell}{w} \] (4)

Resistance is then converted into resistivity by multiplying resistance by the width of the graphene device. This can be done by separating the terms of area and cancel length out of the equation.

\[ \rho = \frac{1}{\sigma} \] (5)

The resistivity is then converted into conductivity and that conductivity can be utilized to solve for the mobility of the device. In the above equation the mobility of the device is shown equal to the conductivity over the number of charge carriers in the device and the charge of an electron. The number of charge carriers and the charge of an electron give the overall charge of the device as shown below.

\[ q = ne \] (6)

Using the definition of charge, Eq. 6 can be combined to solve for conductivity. Conductivity is shown in terms of mobility and charge using the definition of charge.

\[ \sigma = \mu q = \mu C \Delta V \] (7)

From Eq. 1, 2, and 6 the definition of charge is characterized as the change in voltage and the capacitance of the device.

\[ q = C \Delta V \] (8)

Where \( R \) is resistance, \( \rho \) is resistivity, \( \ell \) is the length of the graphene device, \( w \) is the width of the graphene device, \( \sigma \) is conductivity, \( \mu \) is mobility, \( n \) is the charge carrier density, \( e \) is the charge of an electron, \( q \) is the charge of the device, and \( \Delta V \) is the potential difference. Below shows the resistance of the device converted into conductivity.
Figure 2.1 (A) The resistance of the device plotted against the gate voltage. (B) The conductivity of the device plotted against the swept gate voltage. The conversion of resistivity to conductivity is shown.

2.1.1 Series Structure
Graphene devices have the ability to hold electric charge at a voltage, meaning the capacitance of a device can be measured, as discussed in the introduction. Earlier in section 1.4 the graphene devices were shown to be tested with an electrode in liquid as a top gate to apply voltage to the device. In this setup the gate voltage is applied with an electrode to the liquid which creates a separation of charge between the electrode and liquid and another separation of charge between the liquid and graphene. This creates a double layer capacitor between the electrode and graphene as shown in Fig. 1.4. It is also important to note that graphene has an intrinsic capacitance (quantum capacitance) as well. Due to Dirac point where there is an inability of electrons to move to another state, a separation of charge is created shown in Fig 2.2. The double layer capacitor and the quantum capacitor can be added in series as shown below mathematically and in Fig 2.2.

It is assumed that the interface between graphene and the electrolyte is ideally polarized. In this instance concentration polarization creates an uneven depletion of reagents creating concentration gradients. This effect allows for a redox reaction to occur, which transfers electrons through the liquid into the graphene without allowing electrolysis to occur [7]. Overall this process ideally makes the graphene liquid interface mostly capacitive. It is for this reason this interface is characterized as the equation shown below [8].

\[
\frac{1}{C_T} = \frac{1}{C_1} + \frac{1}{C_2} + \frac{1}{C_Q}
\]  

(9)

In these equations $C_T$ stands for total capacitance, $C_1$ and $C_2$ stand for the capacitors making up the double layer capacitance, and $C_Q$ stands for the quantum capacitance. However due to the setup of the device, the area of the electrode makes $C_1>>C_2$. This concept means that when these capacitors are added in series that the term with $C_1$ goes to zero, allowing one to characterize $C_2$ as the double layer capacitance as shown below.

\[
\frac{1}{C_T} = 0 + \frac{1}{C_2} + \frac{1}{C_Q}
\]  

(10)
Figure 2.2 The setup of a liquid top gated graphene device. An electrode applies a gate voltage to the liquid creating a double layer capacitor on the graphene made up of $C_1$ and $C_2$. The quantum capacitance intrinsically in the graphene is also shown.

2.1.2 Double Layer

A major portion of understanding the capacitance of a graphene device top layered with a liquid, is considering the double layer capacitor created by the setup shown above in Fig. 2.2. It is important to note that the double layer capacitor is not characterized by area the way a parallel plate capacitor would. The reason for this is that there are no plates in this model there is solely a separation of charge between the liquid and the graphene. The charge in this interface is dependent on the number of ions in the solution, which is dependent on the electrolyte concentration of the solution. All of this is taken into account when solving for the Debye length, therefore the equation below models the double layer capacitance well. This setup shows a double layer capacitor created by the electrodes, applied voltage, the salt solution, and the graphene itself. The double layer capacitor can be treated more simply by using the distance over which the potential drops completely to model it. Eq. 11 [8] models this capacitance as a single value,

$$\frac{1}{C_{dl}} = \frac{d}{\kappa \varepsilon_o}$$  \hspace{1cm} (11)

where $C_{dl}$ is the double layer capacitance, $d$ is the Debye length, $\kappa$ is the relative permittivity of the solution, and $\varepsilon_o$ is the permittivity constant in a vacuum.

2.1.2a Debye Length

The distance that the potential drops in a liquid on top of a graphene device can be modeled by the Debye length. The Debye length theoretically should increase in length with a lower concentration. The reason behind this is that this screening length needs a certain number of ions to fill it and a high electrolyte concentration has more ions than a low electrolyte concentration. This screening length creates a ‘bridge’ for charges to pass through and the shorter the bridge the easier it is for charges to pass through. This ease of movement allows the charges to travel a shorter distance through the solution thereby creating a shorter Debye length as shown in Fig. 2.3. This length gives us the size of the double layer capacitor, dependent on the solutions concentration. To calculate it for a solution due to a salt concentration it can be calculated as shown below [9].
\[ d = \frac{k \kappa \varepsilon_o RT}{2F^2C_o} \]  

(12)

Where \( \kappa \) is the relative permittivity of the solution, \( R \) is the gas constant, \( \varepsilon_o \) is the permittivity in vacuum, \( T \) is temperature, \( F \) is faraday’s constant and \( C_o \) is the concentration. For further explanation on the workings of the equation look at the text *Intermolecular and Surface Forces: with Applications to Colloidal and Biological Systems* by Jacob Israelachvili [10].

![Figure 2.3](image)

**Figure 2.3** Different electrolyte concentrations with different screening lengths. \( d \) is the Debye length, the blue dots are ions, and the dashed lines signify the screening length distance.

### 2.1.3 Quantum

As spoken about above, generally the quantum capacitance is the intrinsic capacitance of the graphene itself that occurs when testing it. It is important to note this capacitance as a major component to the overall capacitance of the device especially since the testing done with applying voltage through a liquid top gate uses far less potential than that of back gated devices. The quantum capacitance has a greater effect on a liquid top gated device because it is of a similar magnitude to the double layer capacitance of the device. However, in a bottom gated device the capacitance of the silicon is of a much larger magnitude than the quantum capacitance; meaning that when they are added in series the quantum capacitance would not affect the overall capacitance value. Since the quantum capacitance is intrinsic it should stay the same regardless of what solution is placed on the device. To solve for this value one look at the gate charge carriers and its impurities along with the fermi velocity of the graphene itself, below are the equations that have been found to solve for this intrinsic capacitance.

\[ C_Q = \frac{2e^2}{h V_{Fv}} \left( |n_G| + |n^*| \right)^{1/2} \]  

(13)

\[ n_G = \left( \frac{eV_{ch}}{hV_{Fv}} \right)^2 \]  

(14)

Where \( C_Q \) is the quantum capacitance, \( e \) is the charge of an electron, \( V_F \) is the fermi velocity, \( n_G \) is the gate charge carriers, \( n^* \) are the charge carrier impurities, and \( V_{ch} \) is the voltage being applied. For a more in depth look at this derivation look at the article *Measurement of the Quantum Capacitance of Graphene* by Jillian Xia, Fang Chen, Jinghong Li and Nongjian Tao [11].
2.2 Mobility

The mobility of graphene is in basic terms its sensitivity; this is expressed by how easily a charge moves through a material. This means the more conductive (faster a charge moves) the device is with less gate potential being applied gives a device a higher mobility. As shown in Figure 2.3 the graph on the left shows a graphene device with a less steep curve of conductivity vs. gate voltage than the graph on the right, therefore the graph on the right shows a device with higher mobility. The question is whether the concentration of the top gated solution of these devices can affect this mobility. Eq. 1 and 2 are necessary to calculate mobility and are provided by Giem and Noselov’s work on graphene devices [2].
Section 3

Experiment

3.1 Graphene Chip Safety and Preparation
The graphene chips used in this experiment were fabricated at Oregon State University using an advanced photolithography system. The fabrication of this chip allowed for graphene device testing with Linfield’s four-probe station. Since Linfield relied on Oregon State to fabricate these devices it was vital to have multiple safety procedures for the devices and a quick way to test if the devices would be viable for experimentation.

3.1.1 Cleaning/Precautions
To keep the devices on the Graphene chip usable for experimentation, the chip has to be clean. Any dust, hair, dried salt, or oil can damage the devices or leads. Latex gloves are always used when handling the chip and when moving the chip short distances the chip is held with tweezers while keeping one hand underneath the chip. When moving the chip any substantial distance, it is put into its holding container with raised ledges around it and a lid.

To clean the chip, it is sprayed gently on the edge with de-ionized water so that the water flows over the devices (you do not want to directly spray the devices) and leads. A sample chip is shown in Fig 3.1.1 (a) and (b) shows a sample device with pads, leads, and devices. The chip is dried with N$_2$ gas; there is a large Kimwipe underneath the chip to gather the liquid coming off the chip. The front and back of the chip are carefully dried till almost all the liquid is gone. Then the chip is set down, the tweezers are dried, and finally the chip is re-dried. Note: the N$_2$ gas pressure must be set at a very low setting (under 10 psi), if it is too high it could blow the chip out of the tweezers and break it. For this reason the pressure setting was checked every time the N$_2$ gas was used since others may use a higher setting. Furthermore if there are any significant amounts of salt or any other grime after testing, the chip was soaked in de-ionized water for several hours and then dried to try and dissolve any leftover particles that could be affecting the devices.

![Figure 3.1.1](image_url) (a) OSU Graphene Chip used in experimentation. There are 13 devices with pads at the top or bottom of the chip. (b) Model of a graphene device with its components. Inset: There is also a table of pad combinations that would be helpful to dry test.
3.1.2 Dry Test

Prior to testing the chip, it was studied under the microscope. The leads and pads of the devices were scanned at 5x magnification; to make sure none were broken, scratched, or dirty. At 10x and 50x magnification the devices were scanned and to make sure there were no tears or other issues. A few of the best devices were chosen and dry tested. This process will quickly let one know what devices to test without going through all of them during experimentation and whether the chip needs to be thoroughly cleaned. It is important to note that these devices were designed for four probe measurements, but our testing was done by just using two probes.

Next, to further test which devices on the chip are ideal one can perform a dry test. This test is done by using the probe station and two probes to touch down on different pads and check the resistance through the device (ideally there are 10 kΩ of resistance in the device but anywhere from 2-10 kΩ is usable). The probes are connected to an ohm meter and one just simply touches down on the different pad combinations of the devices to find the ideal ones.

When touching down on the pads be very careful, using the four probe stations x, y, and z capabilities to get precisely onto the pads. The point of the probe will often touch down and begin sliding a little once it hits the pad. On this chip each device has six pads and a few expected ways to touch down on them as shown in Fig. 3.1.1. On the devices it is good to test combination a, because that is how the current passes through the device and combinations b and c, because they provide a voltage difference. This dry test should give one the exact information they need to know which devices on the chip are best and which pad combinations to use to test them.
3.2 Solutions

Solutions are necessary to test a liquid top gated graphene device. For experimentation it is necessary to keep the pH of the solutions as close to 7.00 (neutral) as possible to make sure there are no extra charges from the solutions affecting the devices during testing. There was a motivation to test the devices with two different kinds of solutions at varying concentrations to see how these parameters would affect the capacitance, mobility, and overall sensitivity of these devices. Therefore, the goal was to make two stock solutions with a certain concentration of a specific salt and buffer solution (keeping the pH neutral) that could be diluted down into varying concentrations. Most of the equipment used for this portion of the experiment was originally from the Linfield Chemistry Department.

3.2.1 Molarity

The solubility and formula weight of the solutes have to be understood to make solutions with an ideal molarity. First the formula weight of the solute can be calculated by adding the weight in grams per mole of each chemical in the chemical formula, as shown below. (Also the chemical formula of a solute is often on its container.)

\[ F.W. = \frac{x_g}{1_{mol}} \]  

(15)

Where \( F.W. \) is formula weight, \( x \) is the amount of grams in a chemical formula in one mole. There are general tables with different solubility’s for different solutes that can be found. The solubility’s used for this experiment are in appendix A. With this knowledge the molarity can be found using the equation below.

\[ M = \frac{F.W.}{Total\ Volume} \]  

(16)

Where \( M \) is molarity in moles per liters, and total volume is the volume of the solution in liters.
3.2.2 Mixing
The stock solutions created were a 500 mM KH$_2$PO$_4$/HEPES Buffer and a 580 mM K$_2$HPO$_4$/KH$_2$PO$_4$ Buffer. To begin, stock solutions were made from all three given solutes (HEPES sodium salt buffer, potassium phosphate monobasic KH$_2$PO$_4$, and potassium phosphate dibasic K$_2$HPO$_4$) into solutions near their maximum solubility. The scale used to measure the solutes for these solutions had accuracy past μg, which was ideal. Also, the best way to measure mass amounts of solute is to set the container on the scale, zero the scale, and then add solute as needed. To get the precise amount of DI water to dissolve the solutes one can use a 100 mL graduated cylinder (make sure to get level with water level to make sure it is correct). After, creating the three basic stock solutions one must mathematically figure out what combinations of these stock solutions can create two different solutions with a neutral pH. This process is shown in section 3.2.4 and divulges that equal parts of KH$_2$PO$_4$ and HEPES Buffer will create a neutral solution as will 10 mL of K$_2$HPO$_4$ to 15.8mL of kH$_2$PO$_4$ buffer. The solutions are mixed carefully while using a pH meter and afterwards diluted down as needed for testing.

![Figure 3.2.2 Diluted solutions and a water dropper. All solutions are KH$_2$PO$_4$ with HEPES buffer at different concentrations. A.) 500 mM. B.) 50 mM. C.) 5 mM. D.) 0.5 mM. E.) 0.05 mM. F.) De-ionized water.](image-url)
3.2.3 pH Testing
To ensure that the solutions are neutral (have a pH of 7.00), for reasons discussed in sections 2.3 and 3.2.1, a pH sensor was used while mixing the final two stock solutions. To work the pH sensor one must turn the pH sensor on and calibrate it. To calibrate, the probe is placed in a standard pH 7.00 solution and set to 7.00 and this is also done with a standard pH 4.00 solution. This way the sensor is calibrated to a range of pH values and prepared for sensing actual solutions. Then the solution is tested as many times as are necessary while mixing the solution.

During the measuring process, add the solute to test the primary salt solution that you want a specific concentration for and test the pH as you add the buffer solution. This process should guarantee a nice neutral solution if done correctly. After the two stock solutions are made one can simply dilute them down to whatever concentration they want with de-ionized water, while keeping relatively the same pH.

Figure 3.2.3 pH sensor with probe. The probe is in a standard 7.00 pH solution container at the bottom to keep it protected.
3.2.4 pH Theory

For the solution to be usable as a gate it needs to be as neutral as possible so there are no extra charges affecting the graphene when it is tested. To do this one must first find the $K_a$ (acid dissociation constant) of the solutions being worked with and set up the acid base reaction that is taking place.

$$\text{Acid} + \text{Base} \rightarrow \text{Conjugate Acid} + \text{Conjugate Base}$$ \hspace{1cm} (17)

Where the acid and base solutions create a reaction and reform as a conjugate base and conjugate acid solution. Then this is used to figure out the $K_b$ (base dissociation constant) of the chemical equation as shown below.

$$K_b = \frac{\text{Base}}{K_a}$$ \hspace{1cm} (18)

Where the Base and $K_a$ (the acid dissociation constant) are in the form of ions and so is $K_b$ (the base dissociation constant). From here one uses an equation to solve for pH by using the known $pKa$, base, and acid as shown below in equation 18,

$$pH = pKa + \log \frac{\text{Base}}{\text{Acid}}$$ \hspace{1cm} (19)

which can be rearranged into,

$$10^{pH-pKa} = \frac{\text{Base}}{\text{Acid}}$$ \hspace{1cm} (20)

where pH is ideally neutral, $pKa$ is known, and the acid base ratio can be found to create this pH. With this setup the ratio of ions necessary is also the ratio of volume necessary to create a neutral pH system. This way the ratio of solutions needed to make a specific pH can be found.
3.3 Liquid Top Gate Testing

To test the liquid top gated graphene device as discussed in section 2.3 there are a few different pieces of equipment that need to work together. The probe station is used to contact the pads of the device, and create a gate voltage on the device, all while viewing the device through a binocular lens system. The pre amplifier (model # SR570) inputs a constant voltage difference between two of the probes and converts the subsequent current into a measurable voltage. The Lock-In amplifier collects the information from the pre amplifier and sends it to the computer, while applying a gate voltage from the computer program to the gate voltage probe. The reason behind using the lock-in amplifier is the capability of controlling it with the computer, the lock-in component of the amplifier is not actually used in this process. The LabView computer program takes sample measurements of the current going through the graphene device versus the gate voltage and saves the data to an outside folder. The voltage difference current measurement from the pre amplifier goes into the auxiliary input of the lock in amplifier and the auxiliary output of the lock in amplifier goes into the probe station to create a gate voltage on the graphene device.

Figure 3.3 The general experimental setup of testing top liquid gated graphene devices. A.) The probe station with four probes and a magnifying viewer (up to 2.0x) with a light. B.) The pre amplifier with a bias voltage, filter frequency to block out noise, and sensitivity settings. C.) The computer where the LabView program runs gathering the necessary data. D.) The Lock in amplifier which sends data from the pre amplifier to the computer and sends out gate voltage info from the labview program.
3.3.1 LabView Testing Program

The LabView testing program collects current amounts from the device while outputting a gate voltage to the device through the lock in amplifier. This process gives one the information of the current going through the device and the gate voltage creating that current. Therefore, the program takes this data down and graphs it as the gate voltage is being swept. The gate voltage can be swept from a set minimum to maximum voltage (to be safe always stay below +/- 1V, it is never necessary to surpass this), have its direction changed, and stop at end (one should always try to stop and start the voltage sweep from 0V as to not shock the device with voltage). One can control the steps at which the data is taken, 100 data points every 50 micro volts seemed to allow for good readings. The current sensor setting should match the pre amplifiers, which for our purposes was at 5μA/V.

It is necessary to set up what lock in amplifier you are using which was GPIB 4, set the Path to store your data, and create a file name for that path of data. File names cannot have dashes, periods or commas, so be careful when you are creating a file name or it may not save. At any rate this program allows one to see how their device is testing while manipulating the gate voltage, therefore if something is clearly off one can send their gate voltage back to zero and stop the process to check for issues. This is a vital part of the testing process and allows for a better use of time and less wear on the devices.

![LabView Testing Program](image)

**Figure 3.3.1** The front panel of the LabView computer program used to test the graphene devices. In the top left there is a resource setting for a lock in amplifier. Top middle provides one with the ability to characterize a file path and name and top left allows one to stop or control the direction of the gate voltage. The left allows one to control the data collection rate and the sensitivity on the current pre amplifier. The middle gives the user a real time graph of current vs gate voltage and the right allows one to set the maximum and minimum gate voltages used by the program.
3.3.2 Current Pre Amplifier

To create a voltage bias across the graphene device it is necessary to use the current pre amplifier (CPA). To check the bias voltage, connect the input of the CPA to a voltmeter and check your voltage, pick a voltage that is easy to use (for example we used 25mV of bias to create a current). The CPA is set to a low noise gain mode to clear out any extra noise and the sensitivity setting to record current is set to 5 μA / V. The output of the CPA goes into the Aux in of the lock in amplifier. The bias voltage and sensitivity can be shifted with the arrows in the sectioned off boxes. Remember to turn the bias voltage on with the on button in the bias voltage section and the power button in the power section as well. Forgetting to turn on both of these buttons is a common mistake.

3.3.3 Lock-In Amplifier

The lock-in amplifier allows the computer (LabView Program) to interact with the device reading the output voltage of the current pre amplifier and sending out voltage gate data from the computer. Often, the Lock In amplifier must be on for about an hour for optimal usage. For this experiment’s purposes LabView needs to know the address of the Lock in amplifier which is displayed on the right screen below in Fig. 3.3.3, to check this press the right interface button until address comes up (in this case it is 4). The most important part of handling the lock in is to make sure that it is connected correctly: this includes hooking it up to the computer, connecting the Aux in 1 to the output of the current pre amplifier, and connecting the auxiliary output to the gate voltage probe.

![Figure 3.3.3](image)

**Figure 3.3.3** the Lock-In Amplifier. One can see the general settings of the amplifier for the experiment and the amplifiers address in the far right screen (4).
3.3.4 Four Probe Station

The probe station was a vital resource for testing graphene devices. It allowed one to touch precisely onto a small pad a few hundred micrometers squared, which for graphene testing is necessary. The station had four probes with x, y, and z movement capabilities, a magnifying viewer, and lighting to help with viewing the devices. For this experiment three of the probes were used, two of the probes were set down on two pads to apply a voltage difference across the device and the third probe was lightly set into a solution droplet above the graphene device to create a gate voltage. When using the probe station one must be careful to make sure the correct probes are connected to the correct source (for example the third probe is connected to the Lock In amplifier to create a gate voltage). Also one must touch down onto the pads or water droplet carefully, it is not helpful to deeply scratch the pads or scratch the graphene device itself. Use the magnifier (Up to 2.0x magnification) to zoom out to touch carefully down on the water droplet and zoom back in for the pads. If one does not touch down correctly the data in lab will simply jump randomly above and below zero with no real trend, avoid this. This station is where dry testing was often done as well, refer to 3.1.2.

Figure 3.3.3 Picture of the four probe station with its viewer and extra lighting. (A) On the left and right are the four probes with their x, y, and z movement capabilities, (B) in the middle is a moveable stage, (C) in the upper right is extra lighting, and (D) at the top of the image is the viewer with 2.0x magnification capabilities.
3.4 Annealing

The annealing process is used to improve the sensitivity of graphene devices. This is done to remove defects in the substrate (base) of the device as well as the graphene. First the device is heated to 400 °C and then allowed to cool slowly, this burns off unwanted residue or dirt on the graphene and silicon base of the device. This cleaning of the device decreases charge carrier trap sites on the device and thereby increases sensitivity.

![Figure 3.4 Ideal structure of a single atomic layer of carbon (graphene). This shows the 2-D hexagonal lattice structure of graphene.](image-url)
Section 4

Analysis

4.1 Effect of Concentration on Capacitance

Since as shown in section 2.2 the graphene liquid interface is characterized as capacitive, there is a motivation to test how different electrolyte concentrations affect the capacitance of the device. To investigate this effect, the device is tested with a current applied across the graphene and an electric bias sent through the liquid to influence the graphene; further explanation in section 1.4. This data is converted into capacitance. Specifically the double layer capacitance of the device, explained in section 2.1.2, is what is being compared to changes in concentration.

Capacitance shifts due to concentration can also be calculated theoretically. This is done using the Debye length and specific dielectric constants that change due to concentration. The Debye length is used to calculate the theoretical double capacitance on the graphene device and can then also be compared to experimental data and the concentration of the electrolyte.

Table 4.1 shows the data for the shift in double layer capacitance of graphene devices due to different concentrations of a potassium phosphate solution and the correlating Debye Length.

<table>
<thead>
<tr>
<th>Concentration</th>
<th>Debye Length</th>
<th>$C_{dl}$ (Theory)</th>
<th>$C_{dl}$ (Experimental)</th>
<th>$\pm \Delta C_{dl}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>moles/Liter</td>
<td>M</td>
<td>F/cm$^2$</td>
<td>F/cm$^2$</td>
<td>F/cm$^2$</td>
</tr>
<tr>
<td>(DI Water)</td>
<td>$1.05 \times 10^{-8}$</td>
<td>$9.48 \times 10^{-6}$</td>
<td>$7.48 \times 10^{-7}$</td>
<td>$7.48 \times 10^{-7}$</td>
</tr>
<tr>
<td></td>
<td>$5.00 \times 10^{-4}$</td>
<td>$1.37 \times 10^{-7}$</td>
<td>$5.16 \times 10^{-5}$</td>
<td>$1.33 \times 10^{-5}$</td>
</tr>
<tr>
<td></td>
<td>$5.00 \times 10^{-7}$</td>
<td>$4.34 \times 10^{-8}$</td>
<td>$1.63 \times 10^{-4}$</td>
<td>$1.54 \times 10^{-4}$</td>
</tr>
<tr>
<td></td>
<td>$5.00 \times 10^{-6}$</td>
<td>$1.37 \times 10^{-8}$</td>
<td>$5.13 \times 10^{-4}$</td>
<td>$2.43 \times 10^{-6}$</td>
</tr>
<tr>
<td></td>
<td>$5.00 \times 10^{-5}$</td>
<td>$4.27 \times 10^{-9}$</td>
<td>$1.60 \times 10^{-3}$</td>
<td>$2.53 \times 10^{-6}$</td>
</tr>
<tr>
<td></td>
<td>$5.00 \times 10^{-4}$</td>
<td>$1.33 \times 10^{-9}$</td>
<td>$4.99 \times 10^{-3}$</td>
<td>$2.90 \times 10^{-6}$</td>
</tr>
</tbody>
</table>

In this table d is the Debye length in terms of meters and $C_{dl}$ is the double layer capacitance in terms of farads. The double layer capacitance is the majority of the overall capacitance. Quantum capacitance is part of the overall capacitance of a graphene device; however since it is an intrinsic property of graphene, electrolyte concentration should not affect it. Therefore to see the change in capacitance due to concentration, double layer capacitance should be the topic of importance.

It is clear that in Table 4.2 that concentration has a positive correlation with double layer capacitance and an inverse relationship with Debye Length. This is expected since a decrease in Debye length allows charges to flow more easily into the device increasing capacitance. However, this table also shows that the theoretical double layer capacitance increases at a greater rate than the experimental does. This discrepancy between theoretical data and experimental data may be due to the experimental data being calculated assuming that the mobility of the device is constant.
Figure 4.1 The effect of electrolyte concentration on double layer capacitance. The theoretical curve is shown against the experimental data. There is a clear discrepancy between the theoretical and experimental data in this chart. A table of the experimental best fit line and error is also shown.

Due to the plot above, it must be assumed that mobility is not an intrinsic value of the device. This is most likely due to charge scattering from the liquid on top of the device, which would not allow the ideal amount of charge to pass into the device. Also, the theoretical data assumes a random statistical distribution for water molecules, which is also not the case. This distribution just means that water molecules are theoretically calculated to be less structured than they generally are. Furthermore, the theoretical data is also taken assuming perfectly ambient conditions for the temperature of the electrolyte solutions which may have not been the case. Therefore, a large discrepancy between the theoretical curve and experimental data would be logical.
4.2 Effect of Concentration on Mobility

Viewing the difference between the theoretical capacitance of the graphene device and the experimental, showed that electrolyte concentrations have a clear effect of the device. Since the mobility of the device is correlated to the sensitivity, there is a direct motive to look into how to improve sensitivity in graphene devices by changing electrolyte concentrations on it.

Table 4.2 The mobility, dependent on the slope of changing conductivity over changing gate voltage and how that correlates to electrolyte concentrations. The concentration of the electrolyte is shown on the far left, then the respective slopes of the conductance curves, and the motilities with respect to the curves for electrons and holes.

<table>
<thead>
<tr>
<th>Concentration</th>
<th>Left: $\frac{d\sigma}{dV_g}$</th>
<th>Right: $\frac{d\sigma}{dV_g}$</th>
<th>Mobility Holes</th>
<th>Mobility Electrons</th>
</tr>
</thead>
<tbody>
<tr>
<td>moles/Liter</td>
<td>S/(m*V)</td>
<td>S/(m*V)</td>
<td>cm$^2$/V*s</td>
<td>cm$^2$/V*s</td>
</tr>
<tr>
<td>(DI Water) 8.50*10$^{-9}$</td>
<td>24.5</td>
<td>26.0</td>
<td>1.01*10$^3$</td>
<td>1.07*10$^3$</td>
</tr>
<tr>
<td>5.00*10$^{-8}$</td>
<td>40.4</td>
<td>39.6</td>
<td>1.61*10$^3$</td>
<td>1.58*10$^3$</td>
</tr>
<tr>
<td>5.00*10$^{-7}$</td>
<td>45.7</td>
<td>41.5</td>
<td>1.82*10$^3$</td>
<td>1.65*10$^3$</td>
</tr>
<tr>
<td>5.00*10$^{-6}$</td>
<td>64.9</td>
<td>65.6</td>
<td>2.58*10$^3$</td>
<td>2.61*10$^3$</td>
</tr>
<tr>
<td>5.00*10$^{-5}$</td>
<td>67.0</td>
<td>68.3</td>
<td>2.66*10$^3$</td>
<td>2.71*10$^3$</td>
</tr>
<tr>
<td>5.00*10$^{-4}$</td>
<td>73.8</td>
<td>70.0</td>
<td>2.93*10$^3$</td>
<td>2.78*10$^3$</td>
</tr>
</tbody>
</table>

In chart 4.2 the mobility goes up in accordance with the slope of the conductance curves. This is expected as spoken about in section 1.3, when the slope of $d\sigma/dV_g$ is greater so is the sensitivity and mobility of the device. Also, the mobility of the electrons and holes seem to be on a similar order. This is not the case with most materials, however since graphene is ambipolar and has a symmetric band structure this is ideal.

![Figure 4.2 Conductivity vs. Gate Voltage plot of two different electrolyte concentrations. The concentrations are in units of milimolar and an increase in mobility due an increase in concentration is shown.](image)
As expected the 5 mM solution produces a steeper conductivity curve for the graphene device than the 500 μM solution did. This change in the curve as shown in equation 13 correlates to an increase in mobility. Below in Fig. 4.2.1 the change in mobility due to concentration is investigated from de-ionized water to 500 mM. Furthermore, the mobility values for holes and electrons are compared and fitted to logarithmic functions.

![Graph showing mobility vs concentration with logarithmic fit for electrons and holes.](image)

**Table 4.2** Mobility vs. Concentration Plot. A correlation between the electrolyte concentration and the mobility of graphene is shown. The mobility values of electrons and holes are compared and fitted.

<table>
<thead>
<tr>
<th>Model</th>
<th>Log3P1</th>
<th>Reduced C hi-Sqr</th>
<th>Adj. R-Square</th>
<th>Value</th>
<th>Standard Error</th>
<th>Model</th>
<th>Log3P1</th>
<th>Reduced C hi-Sqr</th>
<th>Adj. R-Square</th>
<th>Value</th>
<th>Standard Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>Holes</td>
<td>a</td>
<td>3.06*10^3</td>
<td>2.39*10^2</td>
<td>1E-3</td>
<td>1E-4</td>
<td>Holes</td>
<td>a</td>
<td>3.12*10^3</td>
<td>1E-3</td>
<td>1E-6</td>
<td>1E-6</td>
</tr>
<tr>
<td>Holes</td>
<td>b</td>
<td>-1.51*10^2</td>
<td>39.1</td>
<td>1E-6</td>
<td>1E-6</td>
<td>Holes</td>
<td>b</td>
<td>-1.51*10^2</td>
<td>39.1</td>
<td>1E-6</td>
<td>1E-6</td>
</tr>
<tr>
<td>Holes</td>
<td>c</td>
<td>8.72*10^7</td>
<td>5.08*10^6</td>
<td>1E-7</td>
<td>1E-7</td>
<td>Holes</td>
<td>c</td>
<td>5.95*10^9</td>
<td>1E-7</td>
<td>1E-9</td>
<td>1E-9</td>
</tr>
</tbody>
</table>

The mobility of the device with either majority carrier goes up logarithmically due an increase in concentration. Also, both majority carriers react similarly to changes in the electrolyte concentration. This is ideal due to graphene’s ambipolar and symmetric band structure properties. However, the electron mobility values seem to correlate better logarithmically than the mobility values of holes do. This may be due to holes having a larger effective mass than electrons or a factor that is unseen. Regardless the two majority carriers line up fairly well and definitely show a positive correlation between mobility and concentration.
4.3 Annealing
Annealing is a heating method used to increase the sensitivity of graphene devices. This process removes defects and charge carrier traps in the material. Therefore an increase in mobility is expected after the annealing process.

![Graph showing conductivity vs. gate voltage](image)

**Figure 4.3** Conductivity vs. Gate voltage plot, showing the change in mobility due to annealing. Post and Pre annealing data is compared tested with a 5 mM electrolyte concentration.

The figure above shows the benefits of annealing devices. There is an increase in the slope of the conductivity over the gate voltage for the device in a 5 mM electrolyte concentration. This slope as shown in equation 13 correlates to the mobility of the device, meaning an increase in the slope signifies an increase in mobility. This increase is very significant and shows higher values than any non-annealed devices that were tested. The annealing process went as expected and removed defects as well as charge carrier trap sites. Since sensitivity is characterized by mobility this plot shows that annealing the devices should lead to increased sensitivity in the device.
Section 5

Discussion

The motivation was increase the sensitivity of graphene biosensor devices in biological environments. This is due to the fact that graphene devices have historically had lower mobility values in liquid (biological environments) than in vacuum. Since sensitivity is characterized by mobility in these devices this is an important issue to fix. To try to increase sensitivity the graphene biosensors were tested in different electrolyte concentrations creating different biological environments. The devices were also annealed (sections 3.4 and 4.3) to see if that would further improve the sensitivity of the devices.

First, the capacitance of the device created by the graphene liquid interface was calculated. This was done to see what effect changing the electrolyte concentration would have on the capacitance since capacitance is a term that is part of mobility (Eq. 2). It was found that capacitance went up with higher electrolyte concentrations, which theoretically makes sense due to the Debye length decreasing (Table 4.1). However, when looking at figure 4.1 it is very clear that the theoretical data assuming mobility as a constant value of the device does not fit our experimental data. They both show a positive correlation with electrolyte concentration but the experimental data increases at a significantly slower rate than the theoretical data. However, this may be due to scattering occurring in the top gated liquid of the device, which would not allow all the charges expected to pass into the graphene lowering the increase of capacitance and mobility. These data imply the possibility that mobility may not be constant and is changing due to scattering.

Then, the effect of electrolyte concentration change was compared to the mobility of the device. This was done by characterizing the slope of conductance curves to mobility and comparing that to the concentration changes. In table 4.2 there is a clear increase of mobility the higher the electrolyte concentration was, from 1,140 to 2,607 cm²/V*s; these data agree with the theory and Eq. 2. The mobility of the device due to a changing concentration was plotted, along with the difference between the mobility of the two majority carriers, electrons and holes. The comparison of the mobility with respect to the electrons and the holes is very similar. This is due to graphene’s properties of being ambipolar and having a symmetric hexagonal band structure.

Overall, the data showed a clear increase in sensitivity of graphene biosensors with a shift in electrolyte solutions and through the annealing process. These methods show how to improve the sensitivity of these biosensors with current testing methods. However, data clearly shows that either the mobility is changing or it is not being measured correctly. In either case it is clear more exploration needs to be done to find out what the case is. Charge scattering due to top gating with liquid may explain the issues with this correlation [3]. One method of dealing with charge scattering may be to use the Hall Effect to align the charges and independently measure the mobility of these graphene devices. Dr. M. S. Crosser, as well as Ethan Minot and Morgan Brown from Oregon State University are working to measure the mobility of these devices independently at this moment.
Section 6

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I would like to thank all my physics professors at Linfield over the past four years. Professor Murray sparked my intrigue for physics in introductory courses and somehow convinced me that typing endless lines of code to model a system was enjoyable. Professor Xie for helping me with helping and correcting me on any physical project I made in the machine shop. Professor Mackie for applying class concepts to methods companies use around the greater Portland area. Professor Heath for introducing me to my favorite subject, circuits. Finally, a special thanks to Dr. Michael Crosser who picked me up for research instead of letting me paint houses all summer. Again thank you to all my professors for making me a better student and teaching me about the most important branch of science.

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