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Independent Study Final Report
Characterize the Wettability of Graphene

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Abstract

The purpose of this independent study is to continue the research on the wettability of graphene. Since all previous research were done in ambient conditions and in short period, this research filled the gaps by analyzing how the contact angle of graphene varies with respect to temperature and environment over long term. In this experiment, Wilhelmy plate method was used. A glass sample was inserted into five liquids: water, glycerol, hexadecane, diiodomethane, and ethanol glycol; the interfacial tension of each liquid was calculated from 25-85℃ and a temperature-interfacial tension relationship for each liquid was derived. Afterwards, the graphene sample was inserted into liquids using the same method, and a temperature-contact angle relationship was derived for each liquid. Since experimental interfacial tensions in water at different temperature corresponds to theoretical values, we assumed our experimental method was accurate. The results showed that contact angles of graphene in water and glycerol did not vary too much with time. Contact angles of graphene in hexadecane were zero, but the advancing contact angles in diiodomethane were in a decreasing tendency. Due to the time constraints, the experiment on ethanol glycol has not been finished. Our next steps will be finishing all experiments and deriving a surface energy equation for graphene.

Introduction

Graphene, a one-atom-thick sheet of carbon atoms arranged in a honeycomb-like pattern, is considered to be the thinnest, strongest and most conductive material in the world. All of these special characteristics of graphene are attracting researchers and businesses in a variety of fields, such as electricity, energy generation, and even aerospace.

Because of its good thermal conductivity and small thickness, people were interested in it as an application for enhancing heat transfer, either for pool boiling or dropwise condensation. In order to validate this potential application, the wetting behavior of graphene is important. However, there are gaps in previous studies that try to address the heat transfer enhancement application: all research are done in ambient conditions and over the course of few hours. Thus, our group hope to fill the gaps by analyzing how the contact angle of graphene varies with respect to temperature and environment (gas, liquid) over many days and weeks. My independent study mainly focused on how the contact angle of graphene vary with respect to temperature, which uses Wilhelmy plate analysis.

Wilhelmy plate method is used for "measuring the surface tension of a liquid, the interfacial tension (IFT) between two liquids and the contact angle between a liquid and a solid." [1] As shown in Fig. 1 below, a plate is inserted into the liquid and contact angle formed between the liquid and the plate.
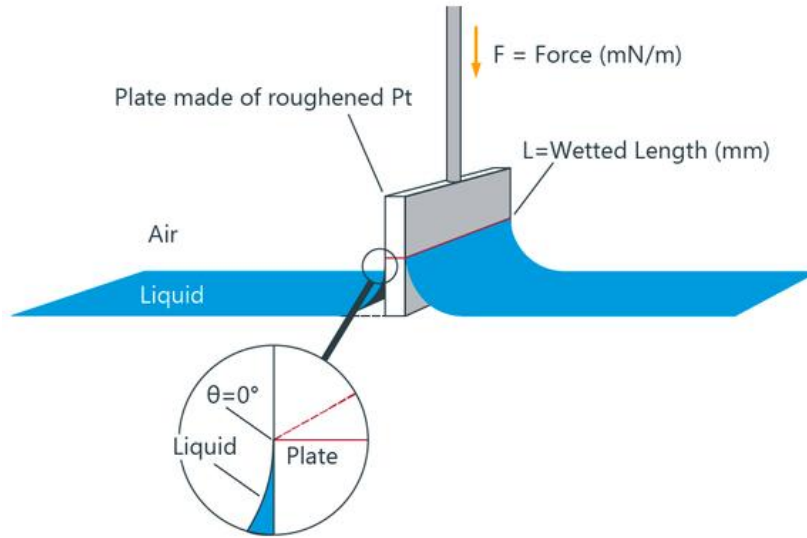
Figure 1. Schematic diagram of the Wilhelmy plate method

The force F correlates with the surface tension or interfacial tension σ and contact angle θ according to the following equation [1]:

$$\sigma = \frac{F}{L \cdot \cos\theta} \qquad (1)$$

,where L is the wetted length.

Since the interfacial tension of a liquid is a constant, we should calculate the interfacial tension first with the plate of a known contact angle, and then calculate the contact angle of graphene using the same method.

Apparatus & Procedure

The main objective of this research is to measure the interfacial tension of liquids and measure the contact angle of graphene. The experimental setup are shown in the Fig. 2 and table 1. below:
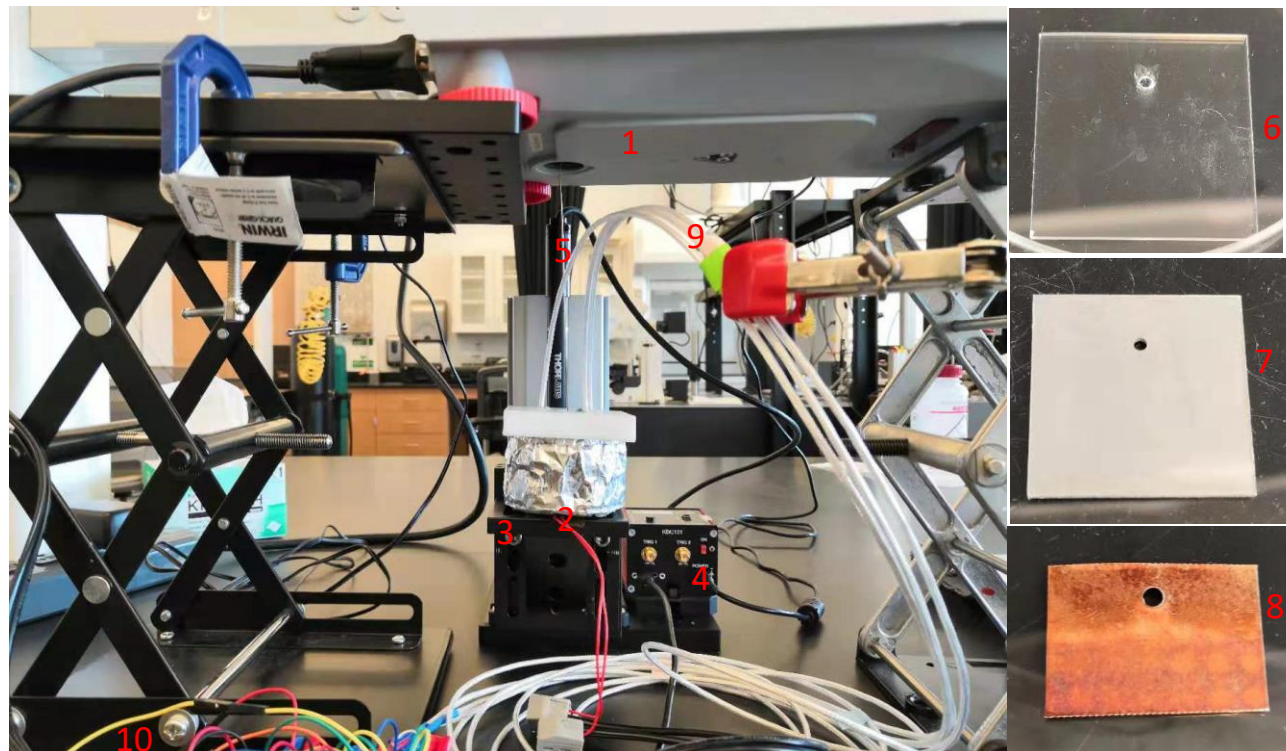
Figure 2. Experiment setup

Table 1. Equipment used to conduct the experiment

	Equipment	Description
1	Microbalance	OHAUS 026.02.00.0D11
2	Heater	Mcmaster 19055, 2 inches in diameter, 8000-10000 Volts in resistance, 200℃ max temperature
3	Stage	Thorlabs stage: Z825B
4	Motor	Thorlabs Kinesis KDC101
5	Hook	-
6	Glass Slide	25mm x 25mm x1 mm
7	Boehmite Slide	25mm x 25mm x1 mm
8	Graphene Slide	25.5527mm x 22.019mm x0.4064 mm
9	Thermocouples	-
10	Arduino	MCP23017-E/SP 1728HQ8

As shown in Fig. 2, a container with a heater at the bottom was placed on the stage, which was controlled by the motor to move vertically. Holes were drilled on the lid of the container, so that the thermocouples could be inserted into the container to measure the temperature of the liquid

and ambient atmosphere. One end of the hook was attached to a glass/boehmite/graphene slide, and the other end was hang to the microbalance. Arduino was used to control and record the temperature of the liquid.

Since glass has very high surface energy, we assume its contact angle is 0. Thus, we can use glass as a reference material to measure the interfacial tension of liquids. However, the surface of glass can be easily contaminated, so we should do plasma cleaning before each measurement. After a 2-minutes plasma cleaning, the glass slide was attached to the hook and inserted into the container. We then zeroed the microbalance, so that only the change in mass would be recorded. By using Kinesis software, we moved the stage up with a velocity of 0.1 mm/s and an acceleration of 0.05 mm/s. If the slide touch the liquid surface, the mass measured by the microbalance would change. Then we continued inserting the slide with the same velocity until it reached the immersion depth of 3mm. After stayed at the 3mm depth for one minute, the stage moved back down for 3mm with the same velocity and waited for another one minute. Finally, the stage moved back to the home position. We repeated the procedure for five liquids: water, glycerol, hexadecane, diiodomethane, and ethanol glycol, and for at least five temperatures between 25-85℃. After we calculated the interfacial tension of each liquid at different temperature, a liner relationship between the interfacial tension and the temperature was derived for each liquid. We could then change the glass slide to graphene slide and repeated the procedure to measure the contact angle of graphene.

Result

Beginning with glass sample inserted in water at 25℃, the mass change was recorded by the microbalance. As the immersion depth increased, the buoyancy force F_b also increased, which can be calculated with the equation [2]:

$$F_b = \rho g V \tag{2}$$

,where ρ is the density of liquid, g is gravity and V is the immersion volume.

The total surface tension F, can be calculated by the equation:
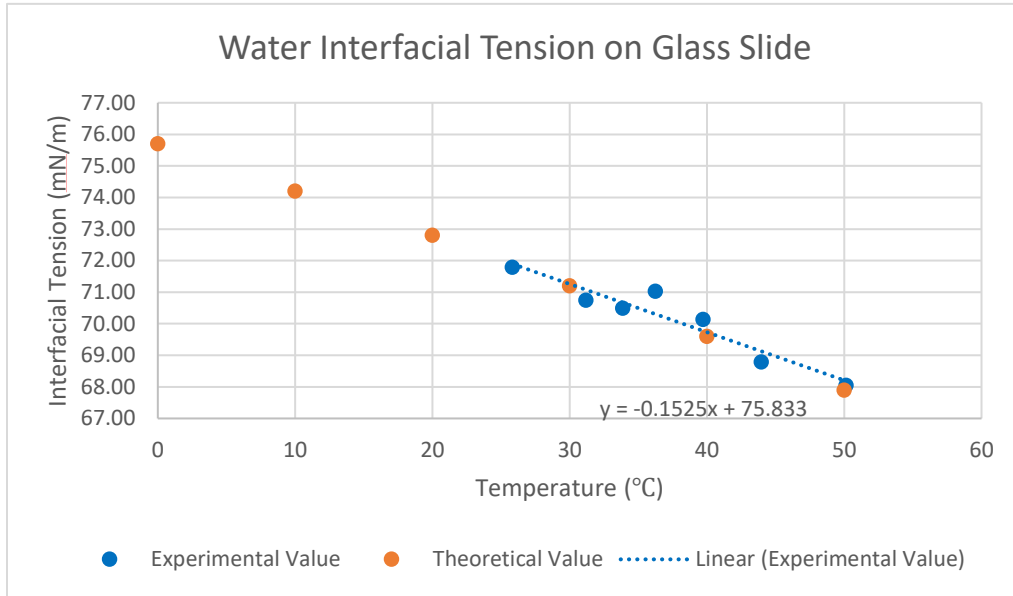
$$F = F_m + F_b = mg + \rho g V \tag{3}$$

,where F_m is the force given by the microbalance and m is the mass measured by microbalance.

The wetted length of slide was the diameter of bottom surface:

$$L = 2 \times (0.025 + 0.001) = 0.052m \tag{4}$$

With Eqn. 1, the interfacial tension can be calculated.

The relationship of interfacial tension and temperature for water is shown in Fig. 3 below:

**Figure 3. The relationship of interfacial tension and temperature for water**

After knowing the interfacial tension of water, we switch to the graphene sample. Since graphene slide has a different dimension, the wetted length became:

$$L = 2 \times (0.0255527 + 0.0004064) = 0.0518668m \qquad (5)$$

Contact angle can be derived by converting Eqn. 1:

$$\theta = arccos(\frac{F}{L \cdot \sigma}) \qquad (6)$$

The change of contact angle of graphene with respect to temperature is shown in Fig. 4 below:
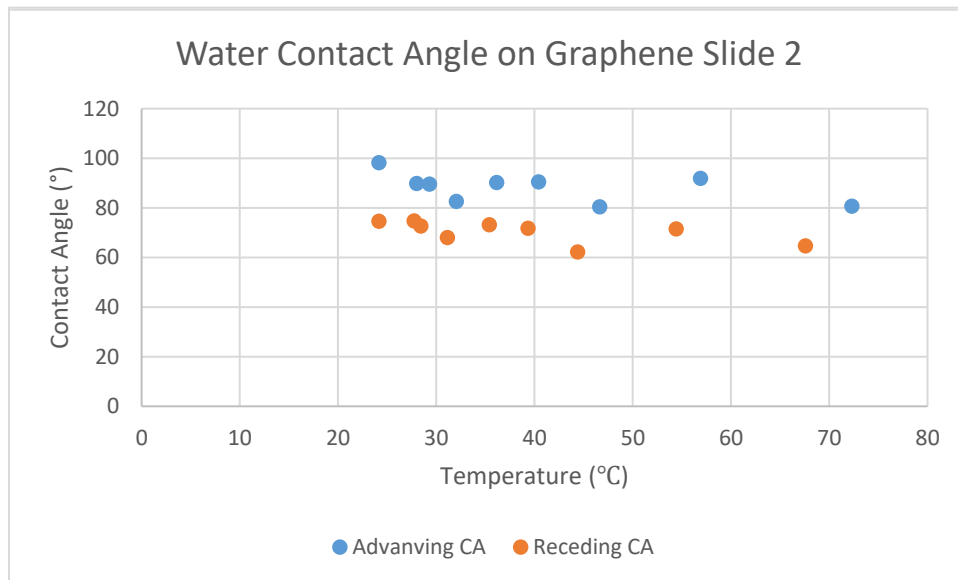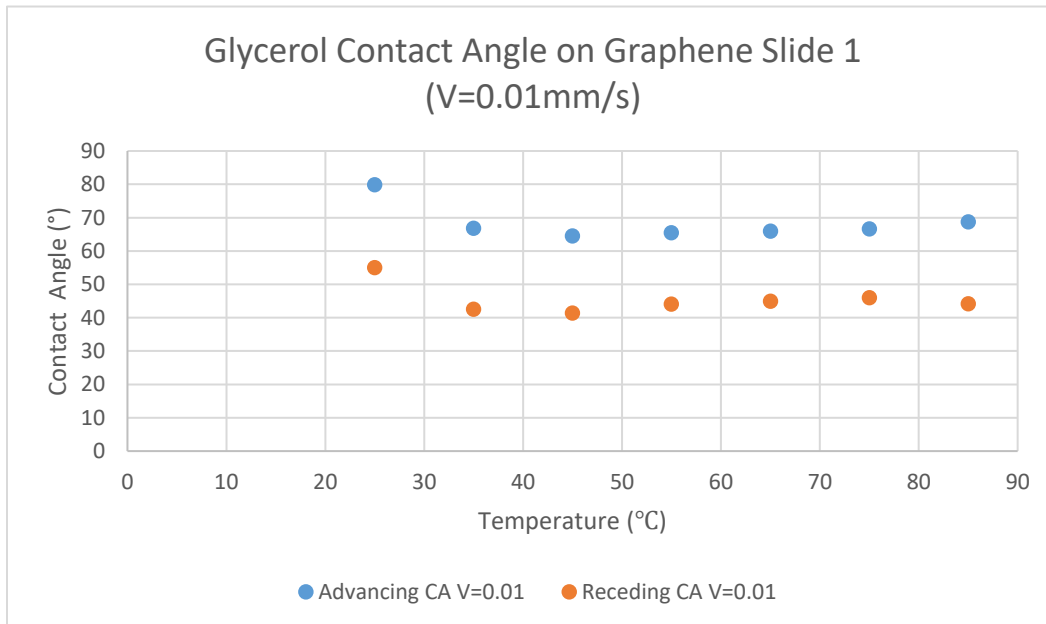

Figure 4. Water contact angle of graphene slides with respect to temperature

Following the same method described above, the interfacial tensions of glycerol, hexadecane, diiodomethane and ethanol glycol vs. temperature, and graphene contact angle in each liquid vs. temperature are shown in figures below.
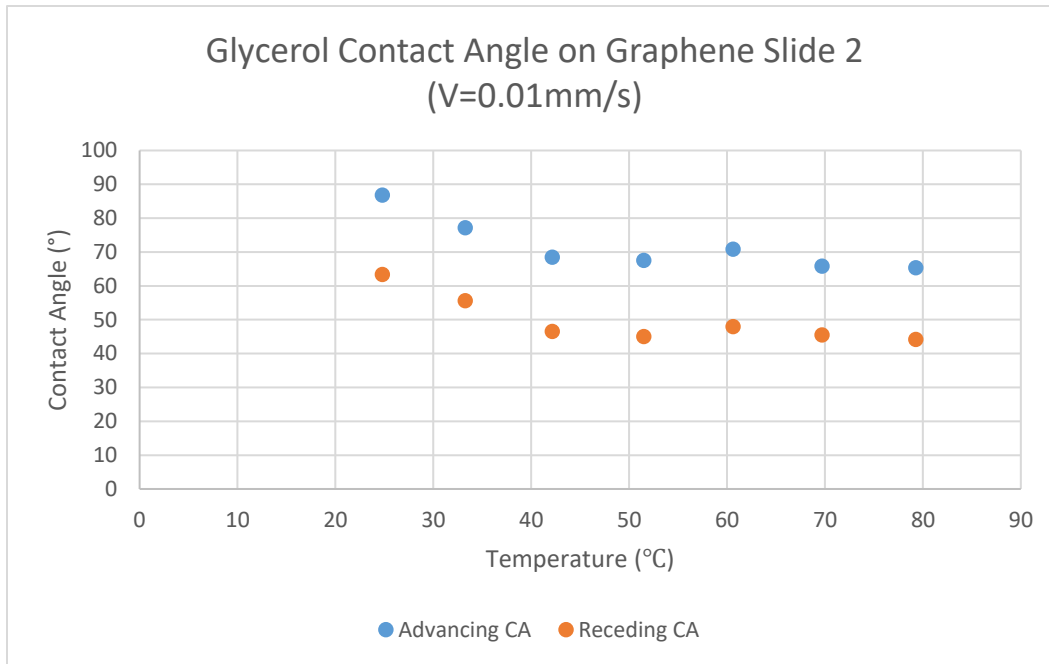
Glycerol:
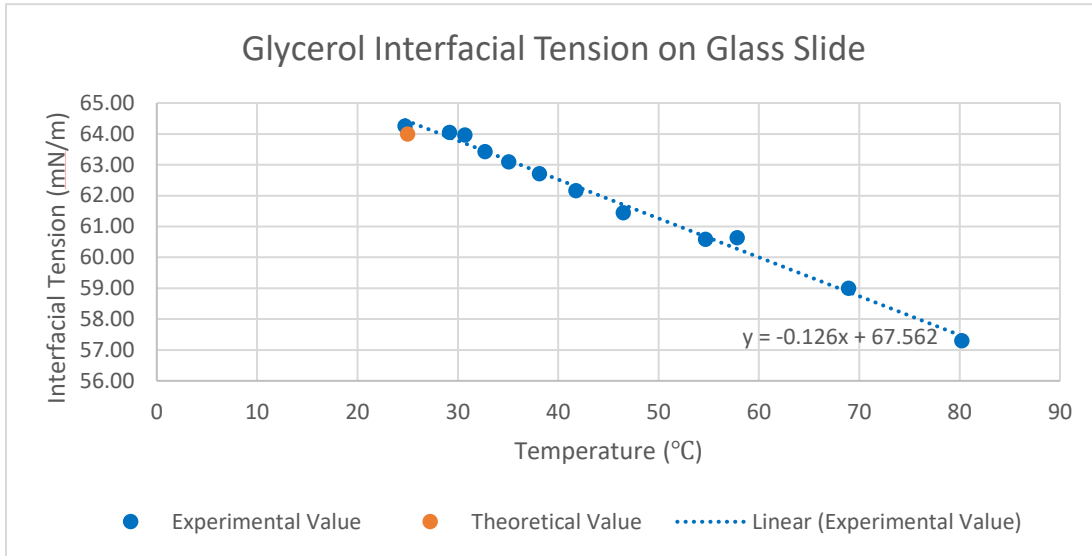
Figure 5. The relationship of interfacial tension and temperature for glycerol
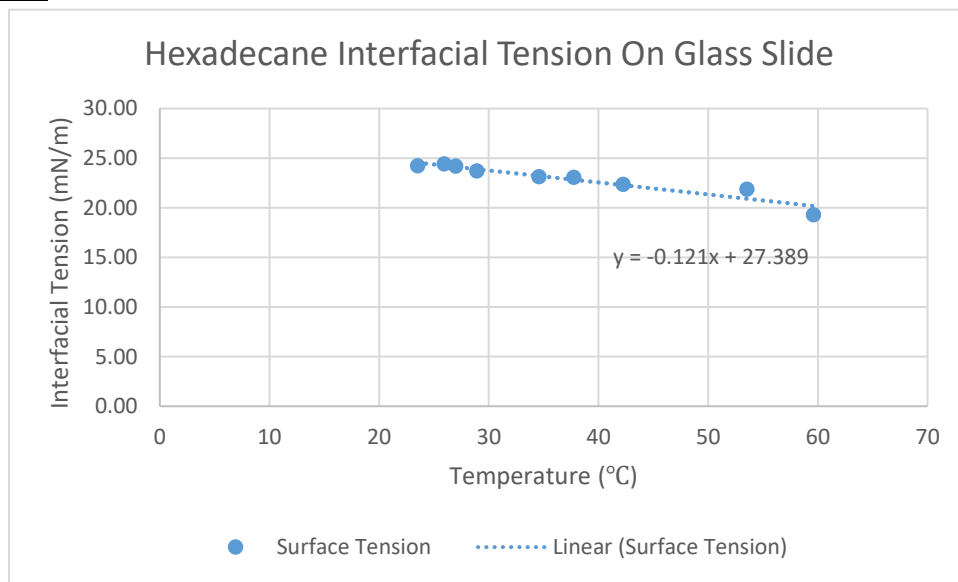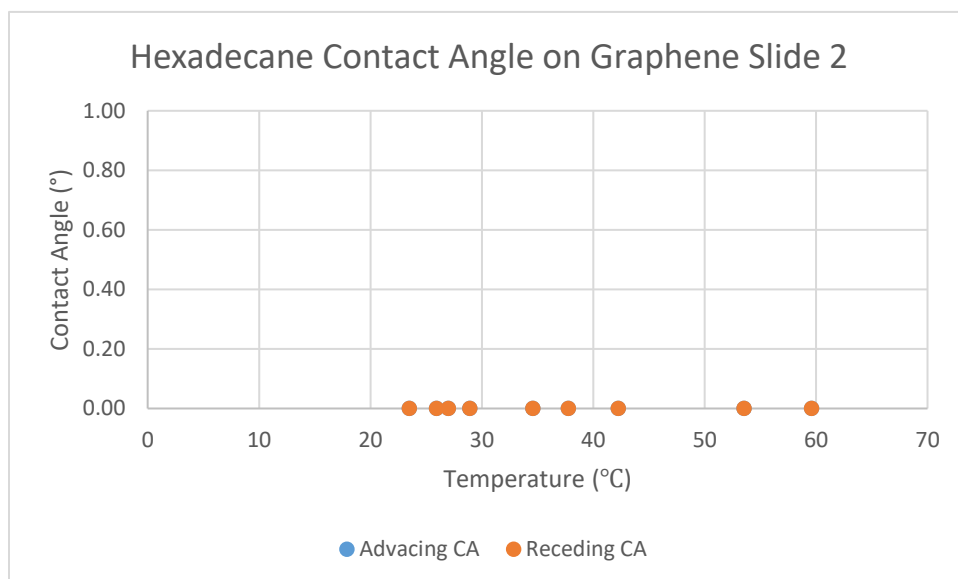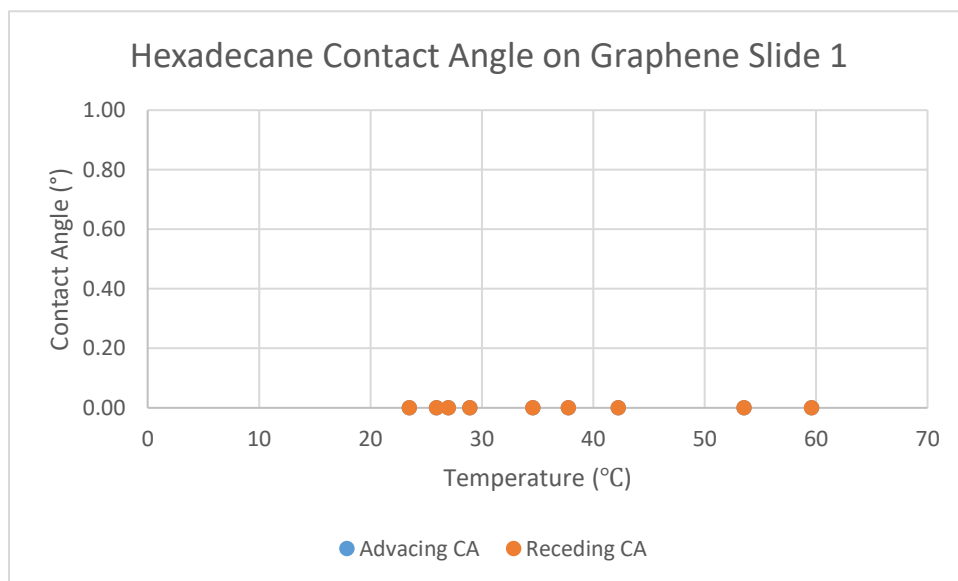
Figure 6. Glycerol contact angle of graphene slides with respect to temperature
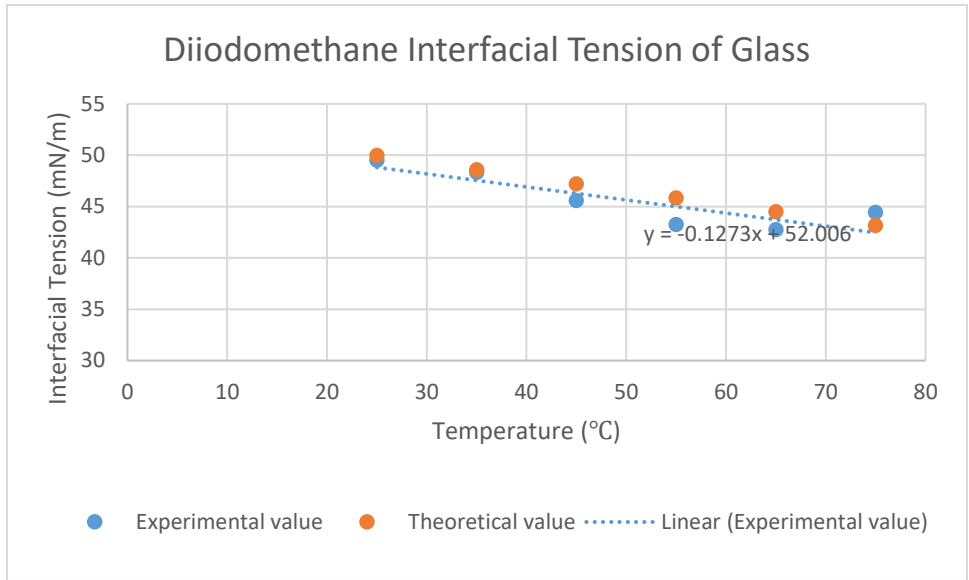
Hexadecane:

Figure 7. The relationship of interfacial tension and temperature for hexadecane

Since the hexadecane has low surface energy, both advancing and receding contact angles were 0°.
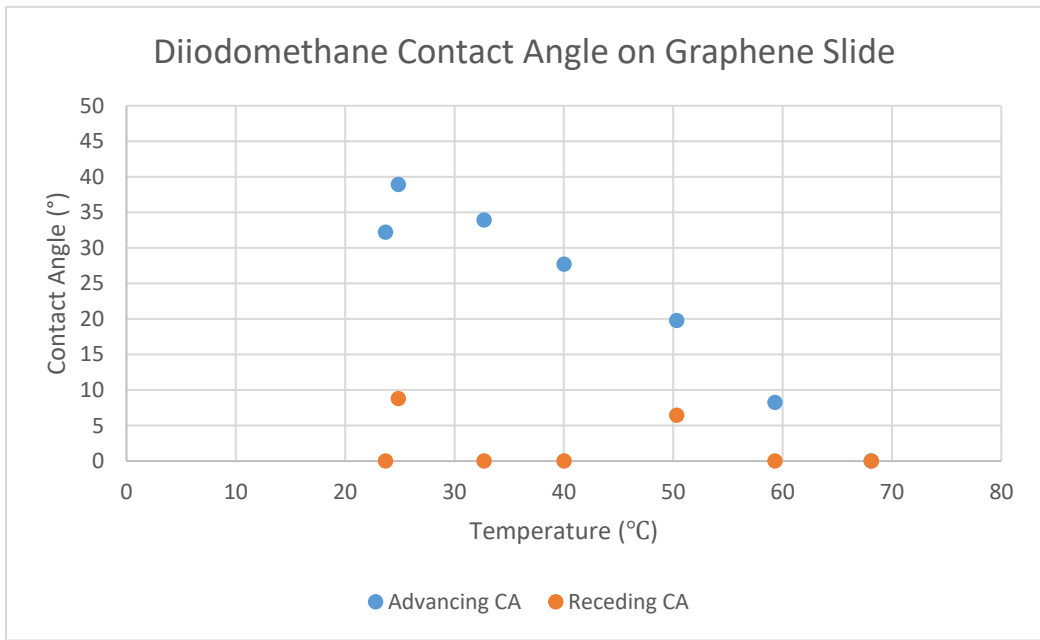

Figure 8. Hexadecane contact angle of graphene slides with respect to temperature

Diiodomethane:

Figure 9. The relationship of interfacial tension and temperature for diiodomethane

Figure 10. Diiodomethane contact angle of graphene slides with respect to temperature

Ethanol glycol:

Due to the time constraint, the measurement in ethanol glycol has not been finished.

Discussion

Since the theoretical surface tensions of water at some temperatures were provided by Engineering Toolbox[2] and the values are in a linear relationship, the experimental values are compared with the theoretical ones. As shown in Fig. 3, the experimental linear relationship line almost collapsed with the theoretical values. Thus, we could assume our data were accurate and the experimental method was reasonable. For glycerol, only the surface tension at 25℃ was provided. As shown in Fig. 5, that value was on the linear relationship line, so we could assume the temperature-interfacial tension relationship we derived was accurate. Since no theoretical value for Hexadecane was provide, no comparison was made in Fig. 7. Similar to water, theoretical values for different temperatures of diiodomethane were provided. The collapse in Fig. 9 of lines showed that the relation we derived for diiodomethane was accurate.

As shown in Fig. 4 and Fig. 6, the contact angles of water and glycerol did not vary too much with respect to temperature. The contact angle of each liquid at 25℃ was compared with the value from sessile drop measurement. The differences were both with in 10%, making this experiment more reasonable.

Both advancing and receding contact angles in hexadecane were 0° in Fig. 8. This showed that the surface energy of graphene should be higher than the surface energy of hexadecane. The receding contact angles in diiodomethane were 0°, but the advancing contact angles were in a decreasing tendency in Fig. 10. We might assume that the surface energy of graphene was closer to that of diiodomethane.

Conclusion

In this independent study, the contact angles of graphene were derived in four liquids: water, glycerol, hexadecane, and diiodomethane from 25℃ to 85℃ using Wilhelmy plate method. We realized the experimental interfacial tensions for water at different temperature corresponds to theoretical values. Thus, we assumed our experimental method was accurate. The contact angles of graphene in water and glycerol did not vary too much with time. Contact angles of graphene in hexadecane were zero, but the advancing contact angles in diiodomethane were in decreasing tendency. Due to the time constraints, the experiment on ethanol glycol has not been finished. There might be some errors in our experiment. Since the samples were easily contaminated by air, the temperature-interfacial relationship might not be absolutely accurate. The air flow in the fume hood caused a little vibration of the hook, which might influence the contact point. Only two groups of experiments were done, so it was not sufficient to prove the experiment was repeatable. Our next steps will be finishing experiments on ethanol glycol, repeating this experiment on more samples, and deriving a surface energy equation for graphene.

Reference

[1] KRUSS GmbH, *Wilhelmy Plate Method*, https://www.kruss-scientific.com/services/education-theory/glossary/wilhelmy-plate-methodAlireza Mo/

[2] The Engineering ToolBox, *Surface Tension*, https://www.engineeringtoolbox.com/surface-tension-d_962.html