A Characterization and Measurement System for Propylene Glycol Properties (Means of Attitude Control in Space)
A Characterization and Measurement System for Propylene Glycol Properties (Means of Attitude Control in Space)

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I. Abstract

Recently, engineers have been able to design smaller satellites called Nano-satellites. Nano-satellites (1-10kg) also known as “Cube Sat” are based nominally on multiples of a unit cube of 10 centimeters on each side. The basic cube shape is known as 1U CubeSat. A major obstacle when designing the nano-satellites is to choose a propulsion system that would be safe, effective, low cost, and could easily function with a resistojet. The proposed system uses Propylene Glycol (C₃H₆O₂) as a safe propellant for a resistojet. In order to predict the behavior of the propellant, the property to be characterized is its surface tension at different concentration levels, 0 % to 99% C₃H₆O₂. Studying the properties of the propellant will provide design parameters for the resistojet propulsion unit for a nano-satellite. In the process, a first device was built which measures two required parameters for computing the surface tension. The device measures the height and the radius of curvature of the meniscus of the liquid through a capillary tube. Experimental results were obtained for the mass concentration of the solution ranging from 0 to 40% C₃H₆O₂ (diluted in de-ionized and filtered water, H₂O). It was expected that the denser the solution was, the higher its surface tension. A second measurement system that is more accurate and has a better repeatability than the first has been completed. The system has been subjected to several testing which consist of locating and displaying the pixel location of an object that the template was programmed into the system. The freezing point of the propylene glycol was also obtained. Concentrations from 10% to 99% V/V of C₃H₆O₂ at intervals of 10% were experimented carried out and the result show that the lowest freezing point temperature attainable by the fluid is approximately -60 °C. The data obtained were at first glance correct until the physical appearance of the experimented samples displayed a white icy precipitate.
II. Background

Satellites are the central cutting edge of technology that is influencing our everyday life. Examples such as the Internet, GPS, satellite broadcasting services, and many others are ubiquitous in our daily life. Yet, their associate cost is exorbitant. One of the up-coming technologies that could potentially reduce these costs is the Nano-satellite. As their name indicates, Nano-satellites are satellites that are thousands of times smaller and lighter than regular satellites (about 1-10kg for Nano-satellites). One of a common form factor for a Nano-satellite is the “CubeSat” which is generally a cube of 10 centimeter long in each side and weighs up to 1.33 kg, known as the 1U CubeSat. Their small size and light weight are central to the cost-effectiveness argument. By minimizing the cost, it would be a matter of time before the satellite-based internet or television could be affordable for every single individual on the planet earth. One key limitation to the Nano-satellites currently in space is the lack of propulsion capability to maintain or control their orbital trajectory. The pragmatic propulsion solution for Nano-satellites has to be low cost and require minimum power. Low cost is required in order not to make such class of satellites a simple extension of the already expensive larger satellites, while low power is limited to the low effective surface area available for solar cells (typically on the order of a few to tens of watts of averaged power). Solutions currently available in literature include ideas such as micro scale combustion thruster, field emission electric propulsion (FEEP), micro ion thruster, and several others from the MEMS solid propellant micro thruster documentation (Solid Propellant Micro-Thruster: an Alternative Propulsion Device for Nano-Satellite). All those systems are great concepts, yet none of them are simple solutions with safe propellant, low energy consumption, and low cost. The proposed idea here is to make a micro thruster that is highly efficient, with safe and non-toxic propellant, and at low cost for effective orbital maneuvering of Nano-satellites.
III. Nomenclature

\( \gamma \): Surface tension in Newton (N) or pound-force (lb)

\( h \): capillary liquid height in the tube in millimeters (mm) or inches (in)

\( g \): local gravity in m/s\(^2\) or ft/s\(^2\)

\( R \): radius of curvature of the meniscus in millimeters (mm) or inches (in)

\( \rho \): Density of the liquid in kg/m\(^3\) or lb/ft\(^3\)

IV. Introduction

There are several ways to measure the surface tension of a liquid or an aqueous solution. We have for example the sessile drop test, which measures the angle of contact and height of a droplet which then used for computing its surface tension. The measurement method that we opted for uses a capillary tube where the height of the column and the angle of contact between the liquid and the wall of the capillary tube, would be the measured entities. The challenge is to obtain accurate and precise measurements using a system that will require minimum human intervention. Several existing devices are used to measure surface tensions of objects and liquids. Though all those methods are highly accurate, very few devices would use capillary tubes and/or measure multiple samples simultaneously. A second goal is to design a system that will accurately measure the surface tension of multiple similar or different samples all at once with one automated device. The device is composed of a High Definition (HD) camera, a moving table, a capillary tube support system (that will support five capillary tubes at once for five consecutive measurements), programming software (LabView) that is used to automate the system, and a support bench that will hold the entire system in place. In order to record the freezing point temperature of the propylene glycol, one particular method considered was to plot the cooling curves of all the samples that were considered. Thus a thermocouple backed with a USB DAQ 6211 (data acquisition) from National instrument and LabView program are used to record the temperature variation of the solution as the environment temperature is brought to -70 °C.
V. Theory

1. Surface Tension Measurement:

The Young-Laplace equation describes the phenomenon of capillary action. The following expression was obtained and modified from the Young-Laplace equation:

\[ \gamma = \frac{\rho ghR}{2} \]  

(1)

\( \gamma \) is the surface tension of the liquid-air, \( h \) is the height of the liquid in the capillary tube (from the leveled surface of the liquid to the bottom of the meniscus), \( \rho \) is the density of the liquid, and \( R \) is the radius of curvature of the meniscus.

![Figure 1: Capillary column Meniscus](image)

The radius of curvature is given by the following expression:

\[ R = \frac{a}{\cos(\theta)} \]  

(2)

Where \( a \) is the inside radius of the capillary tube.

2. Freezing Point Temperature:

It is known that the phase diagram of a liquid can be obtained by examining the cooling curves’ freezing point of different composition of the liquid when plotting its temperature variation with respect to time for various concentrations. Most phase diagrams are graphed with temperature as a function of concentration of the elements. A phase diagram should involve two different materials (or solutions) that are being mixed together at different volume to volume concentrations. Our purpose here is to match the phase
properties of other scholars to the various concentrations that interest us. The respective physical properties are also noted after the phase properties are recorded. An example of phase diagram is as follow [1]:

![Phase Diagram of Propylene Glycol](image)

**Figure 2: % vol. Propylene Glycol phase diagram [1]**

The phase diagram above indicates that a volume-% concentration of 60% has a freezing point of -60 °C. Those results along with several others would be utilized as basis for the understanding of the propylene glycol freezing point temperature for different volume-% concentrations.
VI. Preliminary Design and Device Set up

Figure 3: Preliminary Experimental Set up

Prior to automating the measurement device, a first simple device was designed which measures the height of the column in the capillary tube of one tube at a time. It requires an operator to constantly press on the camera to capture images of the capillary tube. The experimental set up consists of a camera, a support (for the capillary tube), a capillary tube, a container, and a base support. The first support is holding the capillary tube over the container, which contains the aqueous solution. The camera is mounted on a screw that allows the user to remove and replace the camera without changing the experiment set up or the view area of the camera. The objective to making this first set up was for quantifying an approximate accuracy of the measurement method. It is in the purpose of this research to not only measure precise data but also minimize repeatability error by reducing accuracy error to near zero. The following image represents the entities that were used to measure the height of the column in the capillary tube.
Figure 4: Preliminary Capillary Set Up

Once the images are taken, they are processed using LabView Vision Assistant. The process using Vision Assistant (LabView Vision Assistant) was implemented to reduce, if not eliminate, random measurement errors. LabView Vision Assistant is programmed to automatically measure the water column from the images previously captured. It accelerates the analysis process and increases the accuracy of the results.

Several data have been obtained through the process explained above. Note that it is only an example analysis of the 10% mass% propylene glycol column height. Another image that was acquired in order to compute the surface tension was also measured by the set up was highly inaccurate thus abandon. The image in question is the angle that the liquid forms with the walls of the capillary tube (angle of contact). It is captured and submitted to the screening and measurement process of Vision part of the program.

Figure 5: Meniscus Angle Acquisition
VII. Current Measurement Design and Set Up

Further progress has been done to improve the first capillary measurement process. It consists of fully automating the capillary measurement process by allowing the camera to find and capture the images by itself. This was done because the first data obtained were unsatisfactory and contained irregularities that were noticeable on several images. It was deduced that the action of pressing on the camera rendered the images asymmetric, thus convoluting the data obtained.

1. Capillary tube support

This simple set up allows the system to hold and analyze up to 8 capillary tubes. It consists of a bridge, that is used to clamp the capillary tubes, and a trunk that supports the bridge with two forks. The bridge can be removed from the forks to allow us to replace the used capillary tubes with unused ones. This set up will be fixed on the base support so that it maintains one position throughout measurements.

Figure 6: Capillary Tubes Folder
2. Moving Camera Set up

![Image of moving camera set up]

**Figure 7: Moving Camera Mounted on Longitudinal Table**

The moving camera set up is designed to eliminate human error from the measurement process. It is fully automated and is operated by a computer program (LabView) which directs the camera to find and capture the required images. It is programmed to travel the whole bridge length, find the wanted threshold, capture it, move to the next image then repeat the same steps until all capillary heights from all capillary tubes are obtained. The collected data is processed then sent to an Excel file where it is stored. A motor is mounted below the moving table which is connected to a screw rod by two gears. The screw rod spins within a plastic nut connected to the table allowing it to move longitudinally. Two cylindrical rods (one in each side) guide the longitudinal. The current through the motor is controlled by an Op Amp (there is 12 volts across the Op Amp) with a gain of 1 (voltage follower). The Op Amp receives signals from a DAQ card which receives commands from the programmed LabView computer software.

3. Base Support

The base support is acting as an anchor for the whole system. It is added to the device to prevent any relative motion of the other components. Also, both the moving camera and the fixed camera will be
programmed with respect to a fixed reference frame, thus making any unplanned relative motion intolerable.

4. Final and Current Set up

After several design modification the system is now complete and functional. The new set up consists of all the part (elements) explained above except for the camera which was replaced with a simpler and more affordable camera (a Microsoft live-cam). An extended LabView program was also concocted to fully automate the system.

![Measurement Apparatus](image)

Figure 8: Measurement Apparatus.

5. Freezing Point Temperature Set up

In order to obtain the propylene glycol phase diagram, the volume-% concentration of the mixture propylene glycol – H₂O was experimented in which the cooling temperature was recorded as function of time. Concentrations from 0-100% v/v at intervals of 10% were tested in a first hand. A set up consisting of a climate control apparatus, a USB DAQ NI, and a k-type thermocouple was used to carry out the project.
VIII. Equipment

- **Capillary height measurement set up**
  - Camera (Microsoft Live-Cam, Man # Q2F-00001)
  - Longitudinal Table:
    - Horizontal and vertical Platforms
    - Screw rod
    - Two guide rods and two support rods
    - Plastic gears
  - DC- Brushless Motor
  - Capillary Tubes (Heparin Free)
  - Capillary Tube holder
    - Bridge
o Trunk

Petri dish

• Freezing point temperature set up

➢ USB DAQ NI 6211 (S/N- E71CFB)

➢ Climate Control Chamber (S/N – MC0814460)

➢ K type thermocouple

IX. Data Acquisition Process

• Capillary Data acquisition

  o Place capillary tubes on capillary tube holder

  o Input number of tubes in the program (a Labview NI. program)

  o Edit / add a template to the program image acquisition process.

  o Run program

• Freezing Point Temperature Data acquisition

  o Prepare desired concentration for any desired range (in this case 0-10-100% v/v)

  o Place solution in Climate Control Chamber

  o Run Program
X. Results / Data Acquired

1. Freezing Point Result

The freezing point temperatures of ten different concentrations were obtained at a first stance. Every sample was submitted to an environment of -70°C. In this experiment, the environment was assumed to be constant thus eliminating errors due to the environment temperature variation. Note that all the mixture was done using de-ionized water.
Figure 10: First Set of Measured Data

It was noticed that all samples from different concentrations were reaching a stable phase at approximately -55°C to -60°C. The phenomena was occurring at around 40% V/V concentration and would repeat for the
remaining higher concentrations. This should not have occurred as it was predicted based on the documentation that were used as references. To try to understand what was causing the samples to display such characteristics, the freezing point temperatures were obtained at the concentrations of 32.5, 35, 37.5 % V/V C₃H₈O₂.

![Graphs for 32.5%, 35%, and 37.5% V/V C₃H₈O₂](image)

<table>
<thead>
<tr>
<th>Temperature, °C</th>
<th>Time, s</th>
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<tbody>
<tr>
<td>40</td>
<td>0</td>
</tr>
<tr>
<td>20</td>
<td>1000</td>
</tr>
<tr>
<td>0</td>
<td>4000</td>
</tr>
</tbody>
</table>

Figure 11: Second Set Measured Data

Note that all three plots once obtained and compared looked as follow:

All three graphs were stabilizing at about -55°C. That is an indication that the change may have occurred in between 37.5% and 40% V/V for that everything before the 37.5% concentration, including the 37.5%
solution, would have the same behavior as the 30% V/V concentration. The conclusion is that the change has occurred somewhere between the 37.5% and 40% V/V concentration.

2. Capillary Height and surface tension measurement

2.1 Preliminary design results:

As explained in the set up earlier, the credibility of the experimentation was tested to ensure that appropriated data would result from the experiment. The experiment followed with a plot that is as follow: There were four different concentration of sample that were used for testing that are 10%, 20%, 30%, and 40% mass% diluted into the appropriate mass of de-ionized water.

![Graph](image)

Figure 12: Preliminary Column Height Measured Data

It was now evident that the height of the fluid in the capillary tube would indeed vary with the concentration and that it would inversly vary with respect to the mass % concentration of the solution.

2.2 Current a Final design progress:
From the previous and much simpler set up, great improvement have been done in the process of measuring the height of the fluid column. In addition the set up was also modified to measure the angle of contact of the column’s meniscus. The set up has been completed but is still under testing to ensure its functionality and repeatability. Also, the equipment used in the system has certain limitation that is inherent the full functionality of the set up. That may or may not be fixed due to financial reasons. Of all the testing that were done on the system, one of them is the capability of the system to find and log in the capillary tubes’ position. Once the template has been inputted in the program, the system will search, on its workspace, find, and graph the object location to confirm that it has located and recorded the objects desired information. The plot is as follow:

![Graph Pixel](image)

**Figure 13: Position Graph Pixel**

The green dots on the graph above represents the pixel location of four capillary tubes (note that a pixel size varies with the photographic frame it is located into). As it can be observed on the graph, several uncertainty related to the precision of the camera creates a cluster of points that renders the acquired position unprecise.
XI. Discussion and Data Interpretation

1. Freezing Point Result Interpretation

Understanding the importance of obtaining consistent an adequate data is essential to proceed with the interpretation of the data obtained. The information obtained from this experiment will likely be utilized to characterize a propellant used for the propulsion system of a Cube-Sat. Determining the properties of the propellant is not only important due to the fact the system is highly costly but also that it will be carried in a carrier that has more and much more expensive cargo. Last but not the least important aspect is that the credibility of the scholars in charge of the project may be jeopardized.

It may seem relatively simple to obtain the freezing characteristics of the propylene Glycol \( \text{C}_3\text{H}_8\text{O}_2 \) by either retrieving the information from other literatures or by simply estimating the behavior of the fluid using a theoretical approach. First, the data given from other experiment may reflect an aspect of the fluid that is not relevant to the information sought. It is also to take a huge risk on trusting information that was provided from other sources due to doubts on the reliability of the experiment process. Second, mathematical approaches to understanding a problem is always advised before putting the theory to test through real life experimentation. It would be unprofessional to certify that a system would be functional in certain conditions without real life testing but just relying on the perfect world assumption of the mathematical approaches. To support these statements, the simple experimentation done on the different concentrations of propylene glycol /de-ionized water proved to show different results than what the reference data, shown in the introduction, was suggesting. The reference was showing that 60% was the concentration at which the lowest freezing point temperature was recorded (-60 °C). This is contested in the results above which clearly show that the lowest freezing is, although indeed about -60 °C, is the same for all concentrations above 40%, shown below in Figure 14:
Figure 14: Compilation of all the First Measured Data

Also, while running the experiment the 40% sample image was captured after freezing had occurred and several hours had elapsed:

Figure 15: 40% Sample after Freezing Process is Complete
Figure 15 indicates that a physical phenomena has occurred while the fluid was being brought to its freezing point. As one can see, there are white icy precipitates at the surface of the sample which is not normal (or known) physical conditions of the fluid. In the effort to explain that phenomena several hypothesis were brought up:

- By assuming that the white icy precipitate is frozen water, one could hypothesize that the H₂O molecules are being segregated from the rest of the sample thus pouched up top on the surface which is then rapidly frozen and solidified (if not instantly).

- A second hypothesis which also assumes that the white icy precipitate is water, would be that the mixing method was not adequate thus causing the molecules in the sample to separate prematurely. There are presumptions that the temperature variation, the settling time of the sample prior usage, and the volume of the sample tested may have further increased the possibility of the molecules separation before and during the experiment. In order to resolve this issue, an idea was proposed by Dr.-Ing Katsuaki Shirai, Assistant Professor, Chair of Thermo-Fluids Dpt. of Mechanical Engineering, to use a different and more durable mixing method. The mixing method consists of subjecting the solution to special vibration frequencies in order to increase the shearing force between the two elements present in the solution. This could be rephrase as to increase to bonding force between the molecules in the solution thus increasing the bonding time by several factors and also prohibiting the elements in the solution to separate prematurely. This is, of course, a hypothesis and can be proven wrong by other resolution methods.

Beside the proposed hypothesises and solutions, a completely different method of characterizing the freezing properties of the propyene glycol could be considered. That method consists of measuring both the environment and the solution temperatures during the experiment. Then a differential temperature plot will be done with the two data recorded which should indicate the behavior of the solution with respect to the environment it is subjected to. Also, adding a control for each experiment
that is known to have a repeatable and predictable behavior to insure the consistency of the experiments over time (H₂O is being considered)

2. Capillary height (data acquisition system)

The capillary height is needed for computing the surface tension of the different concentration as explained in the theory part above. For similar reasons as the freezing point temperature acquisition, the accuracy and repeatability of the acquisition system is essential for this project.

- The preliminary design showed the predicted patterned that the solution would have as the concentration changes. According to the surface tension equation the height of the column in the capillary tube is inversely proportional to the surface tension of the solution in question. Although not accurate, the plot had a negative slop when plotting the data obtained from 10% to 40% mass% concentration. Note that in the course of the experimentation it was noticed that it would be simpler and more accurate to make volume to volume (V/V) concentrations instead of mass % concentration. It also reduced the solution preparation time by several minutes.

- A second data acquisition method was developed to implement a system that is repeatable and accurate. The system is complete but is still under testing to certify that it is easily controlled by any users and would output as accurate and precise data as possible. Several tests on the localization of objects by the system were done (as shown in the results) to confirm the clear positioning of the object sought, or template, by the camera. The number of objects to find is also a critical parameter that the system has. It is indeed capable of receiving information, from the user, on the number of objects to scan for and effectively carry out the task. Although not precise, the system has been able to complete all the tasks commanded by the user. Several other tests still need to be done on the system before it can be effectively used for acquiring the data it is meant for. While thinking ahead, a major modification to the system would be to obtain a camera that
has a much higher resolution than the one currently used. It will eliminate the precision errors that are in the current system.

**XII. Conclusion and Future Work**

The goal here was to obtain accurate information about the properties of propylene glycol. It was more important to accurately measure the data than to actually obtain any sets of data to characterize the fluid. This explains the concentration of all efforts on building a system that would execute the data acquisition as accurately and repeatedly as possible. In what concerns the freezing point, implementing the modifications to the system proposed in the discussion and insuring that the system is consistent through all experiment would be an essential step in comprehending the data obtained earlier. It could also be possible to obtain useful information about the thermal and mechanical relationship of the propylene glycol at different concentrations and temperatures. In another hand the surface tension acquisition system will require more ample testing before it can be deemed functional and ready to be used for acquiring data. More work is needed to be done before the system can be complete and fully functional.
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Appendix

1. Freezing Point Plots 20% through 99% V/V C₃H₆O₂ at intervals of 10%
Freezing Point Plots 32.5%, 35%, and 37.5% V/V C_3H_8O_2
### Nomenclature

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Definition</th>
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<tbody>
<tr>
<td>p.p</td>
<td>pixel point (coordinates)</td>
</tr>
<tr>
<td>avg</td>
<td>average</td>
</tr>
<tr>
<td>ref</td>
<td>reference</td>
</tr>
<tr>
<td>tru</td>
<td>actual</td>
</tr>
<tr>
<td>H</td>
<td>height</td>
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### 10% Propylene Glycol solution being tested: Results

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<tr>
<td>avg ref p.p</td>
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<tr>
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<tr>
<td>ref p.p tru H</td>
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</tr>
<tr>
<td>p.p factor</td>
<td></td>
<td>0.001116</td>
</tr>
<tr>
<td>p.p H</td>
<td></td>
<td>840.885</td>
</tr>
</tbody>
</table>

*water column height (in inches)* = 0.938362 inch
4. Pixel Point Localization of objects