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# Van der Waals interactions in ultrafine nanocellulose aerogels

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# VAN DER WAALS INTERACTIONS IN ULTRAFINE NANOCELLULOSE AEROGELS

A Thesis Submitted  
in Partial Fulfillment  
of the Requirements for the Designation  
University Honors

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Entitled: Van der Waals Interactions in Ultrafine Nanocellulose Aerogels

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## Introduction

Everyday objects, such as buildings or pencils, are on the meter length scale. For reference, a one story building is around 3 meters tall and a pencil is approximately 0.2 meters in length. Materials that are on the nanoscale, such as nanocellulose, are on the nanometer length scale, one billionth of a meter. The prefix nano relates to a value of  $10^{-9}$ . This makes one nanometer equal to  $10^{-9}$  meters. To better appreciate this length scale, imagine the diameter of a human hair. Hairs typically have diameters of a few micrometers, or microns, with the prefix micro relating to  $10^{-6}$ . Now, divide this single hair into one thousand pieces. That new piece of hair, just one-one thousandth the size of the original strand, has a length scale on the nanometer range. This is extremely small and could not be seen with the naked eye. Although extremely small, these nanoscale pieces can be combine in large amounts to make objects that can be seen and handled directly by a person.

At such a small scale, particles do not behave as one would typically expect. In the world of bulk materials with length scales greater than a few microns, objects are treated classically. This means Newton's Laws of Physics can be used to describe the movement and properties of objects. This area of research has been going on for hundreds of years. These theories have been developed and accepted by the scientific community. On the atomic level with lengths around 0.1 nm, objects such as electrons and protons need to be treated using quantum mechanics. This is a relatively new area of Physics that was introduced less than one hundred years ago. These particles behave differently than predicted if classical analysis methods are used. For example, electrons can tunnel through objects or seemingly jump between objects with nothing in between them. This allows the electrons to momentarily exist in regions that classical physics would not allow such as a gap between two objects. Tunneling electron microscopes use this to measure electrical current between to objects even though they are not touching because electrons have

the ability to jump across empty space which can only be described with quantum mechanics. Interestingly enough, objects on the nano level exhibit properties found both classically and quantum mechanically. Nanoscience is the term for these objects that exhibit interesting properties on the nanoscale (Lindsay, 2010).

One reason nanoscale objects exhibit interesting properties is due to the increased surface area to volume ratio. For a perfect cube, Equation 1 represents what the calculated surface-area-to-volume ratio is.

$$\frac{SA}{V} = \frac{6 \times L^2}{L^3} = \frac{6}{L} \quad (1)$$

Here, SA is the surface area and V is the volume of the cube. The end result is that the ratio is inversely proportionate to the size of the cube. This means that as the cube gets smaller, the surface-area-to-volume ratio gets larger. The result is that for small particles, a larger proportion of the atoms are present at the surface than in the interior of the particle. Since all interactions between objects takes place at the atomic level, more surface atoms allows for more interactions which can have positive and negative consequences. For example, in 2008 a sugar refinery in Georgia exploded due to sugar dust particles (Associated Press, 2008). Typically, sugar is not something that easily combusts. However, at the small length scale where most of the atoms are present on the surface, the sugar particles ignited resulting in an explosion. Other objects, such as a car's catalytic converter, are able to perform more efficiently when their surface-area-to-volume ratios are higher providing more locations to host chemical reactions.

Another interesting property found at the nanoscale is Van der Waals interactions. When materials are small enough, a weak force of attraction can be felt from another particle. This is caused by a permanent dipole, induced dipole, or electronically polarized molecules (Cao, 2008). Essentially, even though the particle is neutrally charged, the electrons can shift around to make

it seem as though one side is charge and the other is not. When two of these particles are near each other the charges of opposite sign are attracted to each other. This weak force of attraction can combine across many molecules producing a stronger effect than just one single molecule or particle. In 2002, it was shown that Van de Waals forces are actually what allow geckos to walk on walls because their feet contain millions of nanoscale fibrous structures that are small enough to feel this atomic attraction (Autumn, 2002).

There are many other materials that exhibit interesting properties on the nano level. An example of an interesting material at the nanoscale is cellulose. Cellulose is a natural material that can be found in algae and other green plants such as wood pulp and cotton fiber and is the most abundant organic polymer in the world (Klemm, 2005). In order to produce nanoscale materials, bulk cellulose must be refined. This process typically involves turning cellulose into microcrystalline cellulose which is then turned into nanocellulose. One popular refining method utilizes an acid hydrolysis technique to produce microcrystalline cellulose, a white powder material commonly used as a filler in medication tablets (Terinte, 2011). This material has a length scale of a few micrometers or roughly the same size as the diameter of a human hair. Cellulose and microcrystalline cellulose are safe for humans to touch and consume which allows other materials made of cellulose to also be safe for humans to handle. From microcrystalline cellulose, nanocellulose and nanoparticles can be formed.

It should be noted that there are primarily two methods for creating nanoparticles: a bottom-up approach and a top-down approach. The bottom-up approach uses individual atoms as building blocks to create the nanomaterial of interest similar to building a house with bricks, the building material is extremely small when compared to the end product. This method generally produces equally sized particles from the increased control in synthesis steps, but it requires a

greater knowledge of chemical synthesis techniques. The top-down approach uses a bulk material and breaks the material apart until nanosized particles are created. Using the brick and house analogy, imagine starting with a completely finished apartment complex. To create individual houses, the complex must be broken apart into the individual housing units. It may be easier to take a wrecking ball to the structure and break it apart, but there is not as much control in the exact size of each individual house that is created from the large apartment complex. Although top-down methods are typically easier to perform, there is not as much control on the nanoparticle size. This is due to the large degree of difficulty in knowing exactly how the material will break apart. However, many top-down methods have previously been shown to be effective in breaking down microcrystalline cellulose into nanocellulose including high pressure homogenation (Kettunen, 2011), high shear refining and cryosrushing (Chakraborty, 2005), and ultrasonication processes (Li, 2012). For this study, the ultrasonication process was used.

Nanocellulose, being a natural organic compound, is biodegradable and safe for humans. Previous studies involved using nanocellulose as the building block material to 3D bioprint a human ear (Markstedt, 2015). Essentially, Markstedt's group created a human ear that used nanocellulose instead of cartilage. Another study used cellulose nanocrystals as an additive in cement paste attempting to change the cement properties for different purposes (Cao, 2015). Other interesting uses for nanocellulose involve creating an aerogel out of the nanocellulose material which will have interesting chemical and physical properties. This study focused on nanocellulose aerogels.

Aerogels are ultralight materials with ultralow densities. They are typically poor thermal and electrical conductors due to their large pockets of air within the material. Aerogels also have large surface-area-to-volume ratios allowing them to be used in applications as a filter or catalyst

in chemical reactions. The aerogel starting material also plays a significant role in these properties. Aerogels are commonly created from particles evenly suspended throughout a solution, known as a suspension. Milk is a common suspension where tiny fat droplets are suspended throughout the rest of the liquid. If the fat droplets were too large, they would separate out just like oil does in water. To create the aerogel, the liquid is removed from the suspension while the particles are left in the same position. Two methods for replacing the water in the suspension with air are known to be supercritical drying and freeze drying (Sehauqi, 2011). Freeze drying freezes the liquid into a solid and then sublimates the solid out of the material by transitioning the frozen liquid directly to a gas at low pressure. The disadvantage to freeze drying is that when using water as the suspension fluid, the frozen water expands. This expansion process may cause damage to the structure of the suspended material. The advantage of freeze drying is that with most liquids, it is easier to freeze the liquid solid and sublimate the solid out using a vacuum pump. Supercritical drying involves bringing the sample to high temperature and high pressure which may also cause structural damage to the suspension and particles. Both methods have been used in creating aerogel materials (Sehauqi, 2011).

Creating aerogels out of nanocellulose has the potential to provide many useful applications. For the most part, the internal structures of nanocellulose aerogels contain nanosized fibers of cellulose in an interwoven fashion. Microscopic images show this fibrous network appearing similar to many spider webs woven amongst each other (Rohrback, 2014). When used as a base structure, it was shown that a hydrophobic coating could be placed on the nanocellulose aerogel to produce an oil absorbing material (Korhonen, 2011). Hydrophobic materials interact very weakly with water. The water molecules tend to stick together and form large drops similar to what water would do on a freshly waxed car. The hydrophobic coated



aerogel could be placed in a water and oil solution to absorb the oil and leave behind the water. Being low density, the bulk of the material is air allowing for many times its own weight in oil to be absorbed. What is useful about this is that the oil can either be extracted and the aerogel reused, or the nanocellulose and oil can be burned to release energy similar to a coal or natural gas power plant. This is possible because both materials are organic mainly made of carbon and hydrogen. This could potentially help clean up oil spills and provide a means to effectively dispose of the oil. Nanocellulose aerogels have also been modified with carbon nanotubes to create a pressure sensor with more interesting electrical properties (Wang, 2013).

Although there have been numerous studies involving the creation of nanocellulose aerogels along with potential uses, only a minority used the ultrasonication process. Even fewer studies show how varying the amount of initial nanocellulose can alter properties such as density, tensile strength, and adhesive strength when using this process.

### **Purpose**

The purpose of this study was to establish methods of synthesizing various compositions of nanocellulose aerogels using a top-down ultrasonication synthesis approach. Quantifying the effect composition has on physical properties was also be of interest due to the lack of scientific literature over this material. Microscopic techniques and physical experiments were used to determine how the internal structure of the aerogel material varies with composition and how the internal structure effects the physical properties. The main driving factor behind this study was that not much current scientific literature can be found about how the ultrasonic process effects the nanocellulose aerogels. It has not been shown that nanocellulose aerogels can exhibit Van der Waal interactions with other surfaces. Once this synthesis method is understood, this information

can be combined with other studies that have provided possible uses for nanocellulose aerogels which can be found in the current scientific literature such as oil absorption or catalyst support structures.

Overall, there are two primary questions to be answered by this study. First, how does the internal structure of the nanocellulose aerogel vary as the percent composition of the base material is changed using the ultrasonication synthesis method? Second, Do the different internal structures lend to different physical properties of the nanocellulose aerogels such as adhesive and tensile strength? Answering these questions would allow for a better understanding of this novel material and how it can be better controlled when synthesized. This knowledge could also point to ways the nanocellulose aerogel could be used in the future for things such as filtration devices or catalyst support mechanisms.

### **Methodology**

Five different samples of nanocellulose aerogels were created. First, a predetermined amount of microcrystalline cellulose powder was measured and combined with 100 mL of water. The exact amount of microcrystalline cellulose powder gives the percent concentration by mass of the samples. For example, a 10% aerogel sample contains 10% microcrystalline cellulose powder to water by mass (e.g. 10 g cellulose per 100 g water yields a 10% aerogel sample). Sample concentrations of 10.0%, 5.0%, 1.0%, 0.5%, and 0.2% were created. The source microcrystalline cellulose was produced by Alfa Aesar and had a similar look and texture to that of cooking flour.

The mixture of microcrystalline cellulose and water was then turned into a suspension using an ultrasonic probe. A Sonics Vibra-Cell model VC 505 was used for the ultrasonication process outputting 500 watts and can be seen in Figure 1. The ultrasonic probe emits ultrasonic sound waves that transfer energy into the microcrystalline cellulose breaking apart the crystals into nano-sized particles. When sufficiently broken apart, the microcrystalline cellulose particles did not settle to the bottom of the mixture creating the suspension. The ultrasonic probe was used until each of the five aerogel concentrations reached the suspension phase. Due to the different amounts of microcrystalline cellulose used in each sample, a different number of cycles were needed for each concentration. One cycle in this method is defined as having the Vibra-Cell set at 50% amplitude on a cycle of 8 seconds on and 2 seconds off for a total high power time of 30 minutes.

*Figure 1: Probe used during the ultrasonication process to create the suspensions.*



The ultrasonication process created large amounts of thermal energy requiring water to be circulated around the beaker containing the microcrystalline cellulose mixture. This was needed in order to keep the sample temperature down to not damage the ultrasonic probe or the microcrystalline cellulose. This is also why one cycle involves a 2 second pause allowing the sample to remain at a relatively low temperature. The water was chilled using a Fisher Scientific Isotemp 3006S Refrigerated Bath Circulator. The temperature for each cycle was set to 7° C. Table 1 indicates the number of cycles required for each sample to create the suspension. For

lower concentrations, six cycles was the limit to minimize contamination from the titanium ultrasonic probe due to the high power output.

*Table 1:* Number of cycles using the ultrasonic probe for each sample concentration.

<b>Sample Concentration</b>	<b>Number of Cycles</b>
10.0%	1
5.0%	3
1.0%	4
0.5%	6
0.2%	6

Upon creation of the suspension, each sample was frozen with liquid nitrogen and placed under vacuum and freeze dried for 72 hours. It was found that 48 hours worked for a few samples, but 72 hours ensured that all samples had their water sufficiently removed from the interior portions. If the water was not removed, the inside of the sample did not exhibit the intended aerogel properties as the sample would then dry through evaporation processes. A FreeZone Freeze Dry System model 77500 was used along with a Labconco model 117 vacuum system. The freeze dryer was set to a temperature of  $-40^{\circ}\text{C}$  and the pressure for the vacuum was set to 0.045 mbar closely matching previous studies (Sehaqui, 2011). This process removes all of the liquid from within the suspension without damaging the internal structure. This is necessary step to create the low density aerogel. The volume of the sample remains the same, but the mass drastically decreases due to air replacing where water used to be thus creating the aerogel.

After synthesis of the nanocellulose aerogels, multiple measurements and calculations of the samples were recorded including densities, percent air composition, adhesive strength, and tensile strength. Densities were measured using micrometers and mass scales. Percent air composition was calculated based off of the density, measured volume, and known density of

bulk cellulose. Adhesive strength was measured by compressing a piece of nanocellulose aerogel between two glass slides and measuring the force required to separate the two slides. There was a system that could raise and lower the sample to apply the force from the hanging mass. When raised, a mass was hung underneath attached to the glass slide providing a downward force. This device can be seen in Figure 2. Tensile strength of similarly compressed nanocellulose aerogel pieces was measured as well. This process involved using a Vernier Wireless

*Figure 2: Apparatus used to measure adhesive strength.*



Dynamics Sensor System which recorded data using Logger Pro allowing for a precise measurement of the force required for the sample to fail. The tested samples were 10 mm wide and clamped on each end leaving a 10 mm gap between each end.

Scanning electron microscope images were also captured of each sample concentration. This allows for images to be taken having resolutions of around 10 nm; much better than what any optical microscope can do. The collected images were then compared to each other in order to see how the internal structures varied between concentrations. These images were also used to relate the physical properties of each sample to the internal structure to determine how structure impacts the adhesive strength of the material.

Briefly, scanning electron microscopes work by taking advantage of the wave particle duality which is another impact of quantum mechanics that occur at small length scales. High energy electrons can behave as particles or as waves with sub-nanometer wavelengths depending

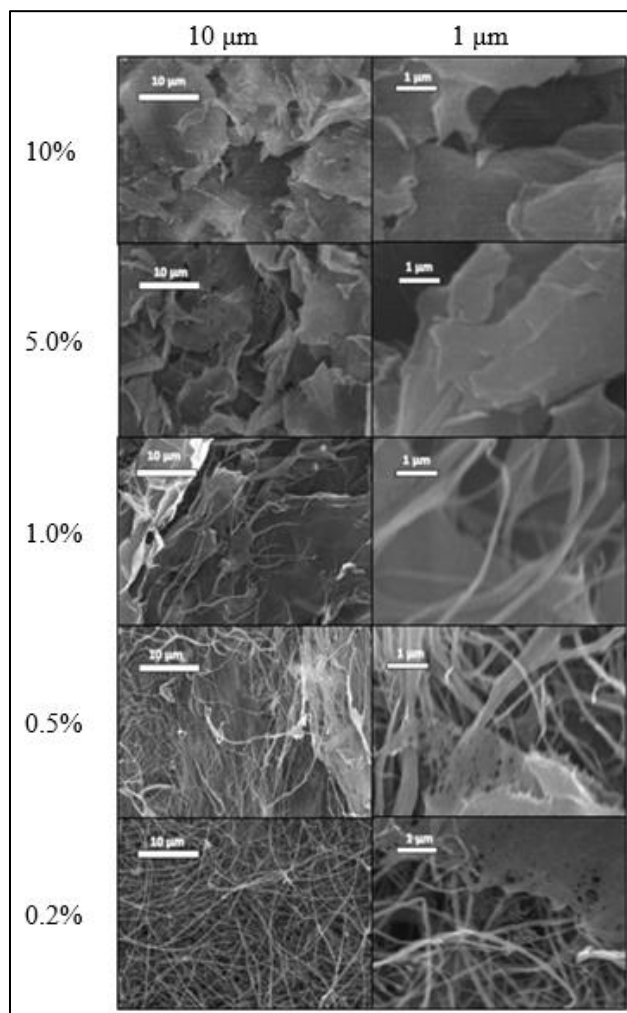
on the particle's momentum. Instead of using optical waves, a scanning electron microscope "views" the electron waves. An electron beam is directed at the sample and sensors detect how the electrons reflect in order to create the image. Because this microscopy technique uses electrons, there is the possibility that the electrons can collect on the sample, build up charge, and damage the sample. To avoid this, 10 nm of gold was sputter coated onto the nanocellulose aerogel sample to create a conductive coating decreasing the chance of charge build up. In an ideal case, the 10 nm would be a constant coating throughout, but the surface structure of the aerogel causes the gold to create uneven coatings of less than 10 nm.

## **Results and Discussion**

### **Scanning Electron Microscope Images**

Surface images were taken along with cross-sectional images. The surface images were used to determine the surface morphology to help determine if the adhesion forces of the nanocellulose aerogels was a mechanical feature, electrodynamic event, or due to Van der Waal interactions. Figure 3 contains the surface images. Note that as the concentration decreases, the internal structure of the sample changes. At high concentrations such as the 10% sample, there are more plate-like structures on the scale of a few microns. At low concentrations like 0.2% sample, the ultrasonication probe is able to run long enough allowing for a more thorough break down of the microcrystalline cellulose powder. This results in structure that is composed of cellulose nanofibers that have diameters of around 100 nm. The fibers randomly align but give a slight appearance to a spider web. For concentrations in between the two extremes, the morphology is a combination of the plate-like structures and the nanofibers.

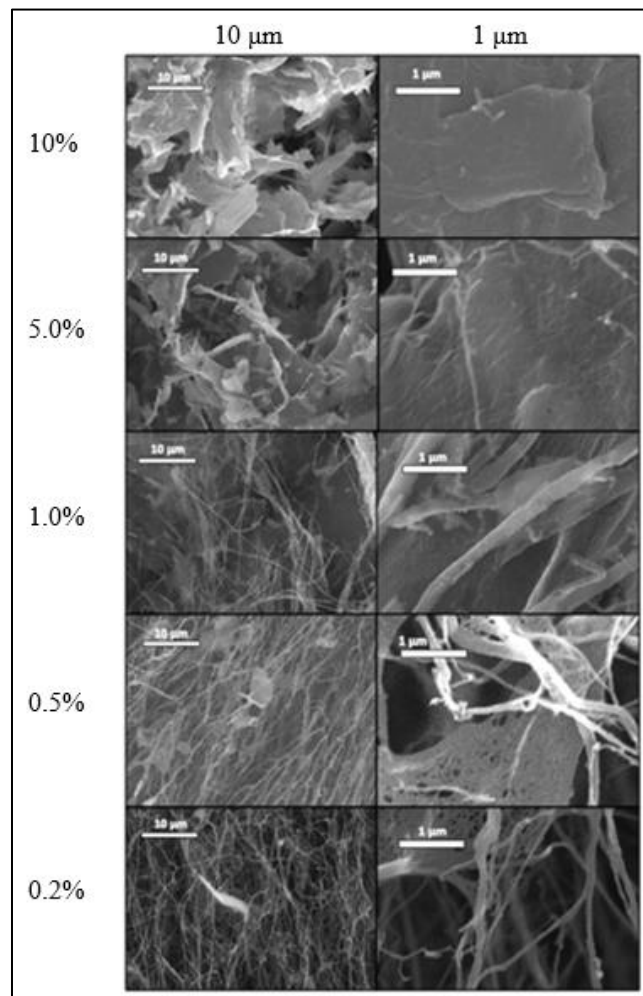
Figure 3: SEM surface images of the nanocellulose aerogel. The left column has a scale bar of 10  $\mu\text{m}$  and the right column has a scale bar of 1  $\mu\text{m}$ .



Similar structural characteristics can be seen in the cross-sectional images of the nanocellulose aerogels. Figure 4 contains the SEM images of the cross section. To obtain cross sectional images, pieces were cut apart using a sharp knife. Again, it can be seen that the higher concentrations contain more plate like structures with a low surface-area-to-volume ratio while the low concentrations consist of cellulose nanofibers. This indicates that different physical and chemical characteristics may be found at the lower concentrations. It also indicates that, for

future applications, the lower concentrations could be used as a catalyst support structure due to the increased surface-area-to-volume ratio. In total, the longer the ultrasonic probe is used in the microcrystalline cellulose and water mixture, the more fibrous the internal structure of the aerogel becomes. The fibers also have a much smaller length scale than the larger microcrystalline particles increasing the surface-area-to-volume ratio.

*Figure 4: SEM cross sectional images of the nanocellulose aerogel samples. The left column has a scale bar of 10  $\mu\text{m}$  and the right has a scale bar of 1  $\mu\text{m}$ .*





### Density and Percent Air

The density and percent air of each nanocellulose aerogel was calculated. The results can be found in Table 2. For calculating the percent air, a microcrystalline cellulose density of 1.46 g/mL was used (Sun, 2005). A total of five pieces were used for each sample concentration when measuring the density.

*Table 2:* Table containing the measured densities and percent air composition for each aerogel.

Sample Concentration	Density (g/mL)	Percent Air Composition (%)
10.0%	0.109 ± 0.008	92.5 ± 0.6
5.0%	0.054 ± 0.005	96.3 ± 0.3
1.0%	0.014 ± 0.002	99.0 ± 0.2
0.5%	0.0076 ± 0.0002	99.47 ± 0.03
0.2%	0.0061 ± 0.0004	99.58 ± 0.03

The percent air composition was calculated using the following formula

$$\left(1 - \frac{d_{AG}}{d_{MC}}\right) \times 100 = \text{Percent Air Composition} \quad (2)$$

where  $d_{AG}$  is the density of the aerogel found in the second column of Table 2 and  $d_{MC}$  is the density of microcrystalline cellulose which is known to be 1.46 g/mL. Note that Equation 1 holds in the limiting cases. If the sample is all air,  $d_{AG} = 0$  g/mL and the equation would output 100% air. If the sample was all microcrystalline cellulose,  $d_{AG} = 1.46$  g/mL and Equation 1 would output an answer of 0% air.

The densities found in Table 2 are what was expected for the higher concentrations. Imagine creating a 10.0% sample using 100 g of water and 10 g of microcrystalline cellulose. This results in the suspension having a volume of 100 mL after the sonication process. If all of the water is removed via the freeze drying process, the aerogel would still have a total volume of

100 mL but the only mass would come from the 10 g of microcrystalline cellulose from the beginning. This results in a density of 0.1 g/mL, close to what is seen in Table 2.

At smaller concentrations, the density begins to increase above what is expected. A possible explanation can be found when examining the synthesis method. Looking at Table 1, it can be seen that the lower concentrations require more cycles to break the microcrystalline cellulose up. During this process, the container is open to the air possibly allowing some of the water to evaporate. The process is very violent which could splash even more water out of the suspension. Although possible, the amount of water lost would be relatively small only accounting for a small percentage of the increase in density. To get better readings, a more precise way of measuring the volume of the aerogel needs to be used.

### **Van der Waal Strength**

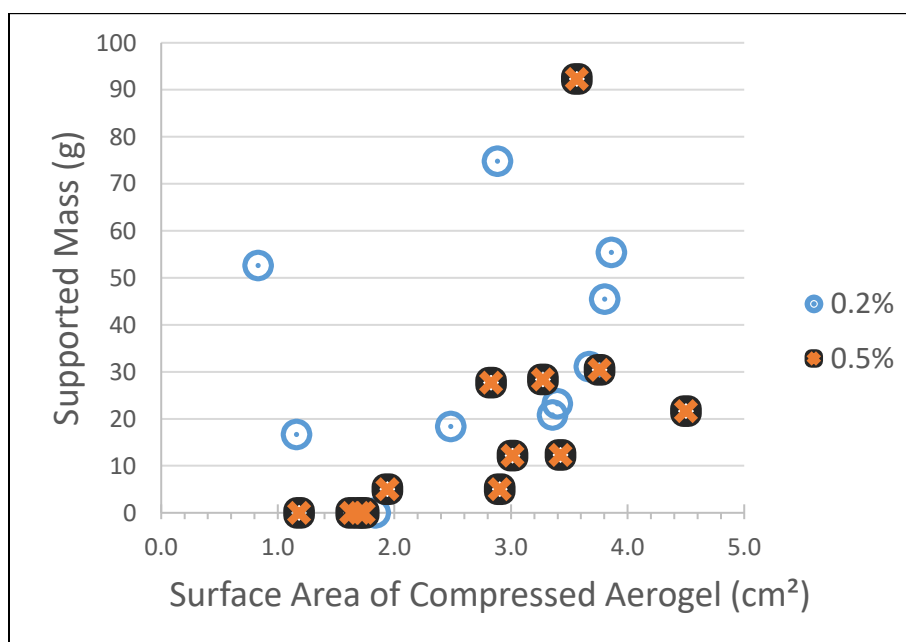
Adhesive strength measurements were then taken. The two contact surfaces for these measurements were glass microscope slides as seen in Figure 2. Only the 0.2% and 0.5% samples were able to support any mass which were the only two samples for which data was collected. These two samples had a more fibrous internal structure seen in the scanning electron microscope images than the other aerogels. Fibers have a larger surface area to volume ratio allowing more atoms in the material to be on the surface. More surface atoms can interact with atoms from other materials. These atomic interactions lead to the short range Van der Waal interactions causing the adhesive force.

Another indication that the force of attraction came from Van der Waals interactions and not something like the electrostatic force is that the aerogel stuck to many different types of materials, both insulating and conducting. If it was an electrostatic force, conducting materials

would not provide a suitable surface to adhere to. Conductive materials allow charge to flow. Any contact with the aerogel would cause any charge build up on the aerogel to dissipate. This results in the same charge being on both the conductor and the aerogel. Electrodynamics predicts that these two materials would then be pushed away from each other due to similarly charged particles repelling one another. Instead, the nanocellulose aerogels remained attached to the conductive material. Positively and negatively charged conductors also adhered to the low concentration aerogels in the same fashion indicating that an attraction from electrical charge was not the source of adhesion.

Figure 5 contains the data for the mass supported as a function of the surface area of the compressed sample. The surface area was found by taking a picture of the compressed sample and using Gwyddion, an image analysis software typically used for microscopy techniques, to calculate the surface area.

Figure 5: Data collected from the adhesive strength measurements.



From Figure 5, it can be seen that there were no real consistencies within these measurements. Ideally, there would be a linear relationship between surface area and supported mass. Increasing the surface area should increase the mass that can be supported. Instead, the data points appear to be random with both samples failing and both samples supporting a rather large force. It should be noted that the 0.2% samples failed to hold any mass 10% of the attempts and the 0.5% samples failed to hold a mass roughly 30% of the attempts.

The measurements were normalized to better compare directly from sample to sample. In this case, it can be normalized by taking the force required for the system to fail divided by the sample's surface area. In essence, this normalization creates a pressure measurement for each sample at which it fails. This allows for the direct comparison between all measurements. Figure 6 provides a look at the normalized force, or average pressure, required for the system to fail. This data set also excludes all of the failed samples in order to better understand how the two samples compare.

Figure 6: Average pressure required for the sample to fail for each concentration.

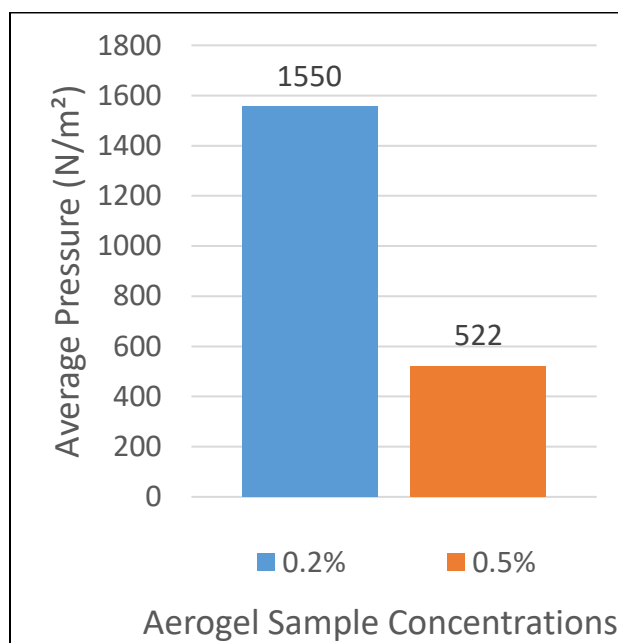
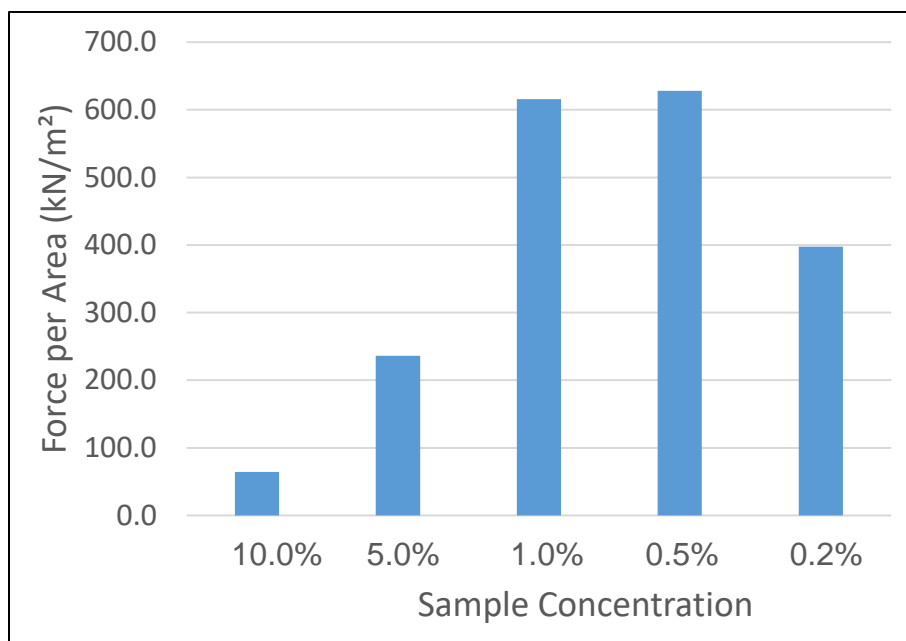


Figure 6 shows that on average, the 0.2% sample performs better when a sufficient seal is created between the two glass microscope slides. This indicates that there is an increase in the Van der Waals interaction for the 0.2% nanocellulose aerogel. This is expected since the 0.2% has a more fibrous structure leading to an increase surface area to volume ratio. There are more atoms to interact with the nearby glass slide allowing for a stronger interaction.

### Tensile Strength

Tensile strength measurements were also recorded. These measurements were used on a nanocellulose aerogel that had been compressed forming a thin, paper-like material and cut to have a width of 10 mm. Figure 7 provides the average values recorded. Note that the force required to break the compressed aerogel was normalized to the sample's cross sectional area.

Figure 7: Data recorded in the tension strength test normalized to the sample's cross sectional area.



It can be seen that the 1.0% and the 0.5% are the strongest samples requiring a force-per-area of around  $6 \times 10^5$  kN/m<sup>2</sup> to be broken. This equates to a value of about 87 pounds per square inch. The 0.2% samples were third strongest at about  $4 \times 10^5$  kN/m<sup>2</sup> required to be broken, or about 58 pounds per square inch. In theory, if there was a rope made of compressed 0.5% nanocellulose aerogel with a cross sectional area of 1 inch, it would be able to support over 80 pounds without breaking.

From this graph, it can also be seen that the plate-like structure of the 10.0% nanocellulose aerogel is not as strong as the lower concentrations. This specific concentration tended to be more brittle than the other concentrations. As soon as there was a slight bend in the sample, a crack would form which would then quickly cause the sample to fail. The higher concentrations were fairly ductile and were able to bend absorbing a torque if one was applied.

Interestingly enough, the 0.2% nanocellulose aerogel did not perform as well as it did in the Van der Waal test. It appears that a combination of plate-like structures and randomly oriented nanofibers provides the greatest mechanical interlocking tension strength as found in the 1.0% and 0.5% nanocellulose aerogel samples. Unfortunately, the 0.2% samples have such high percent air composition that they are only a few tenths of a millimeter thick. Any slight deformation or contamination within the sample could have caused a premature failure at these small scales. A thicker 0.2% sample could possibly be stronger under the tension test, but further tests will need to be conducted. For reference, a standard piece of printing paper is about 0.1 mm in thickness and the 0.2% samples compressed to a similar size.

### **Conclusion**

In summary, a novel method for the creation of nanocellulose aerogels was introduced. This method used an ultrasonication probe in a top-down synthesis approach to create the nanoparticles. The nanoparticles then formed a suspension which was freeze dried allowing for the removal of water without damaging the nanoparticle internal structure. The aerogel was created when the freeze drying process finished. This differs from previous methods that use chemicals to synthesize the aerogels in a bottom-up approach.

Different aerogel concentrations were easily produced by altering the initial ratio of microcrystalline cellulose to water ratio for the initial mixture. The mixtures were then turned into a suspension by running anywhere between 1 and 6 cycles with an ultrasonication probe. Physical characteristics such as density and percent air composition were measured and calculated. The aerogels had densities ranging from 0.109 g/mL to 0.0061 g/mL and percent air compositions ranging from 92.5% air to 99.58% air depending on the initial amount of

microcrystalline cellulose used in synthesis with the lower density and higher percent air composition coming from the 0.2% sample concentration. Altering the concentrations created aerogels with different physical properties that can, in the future, be tailored for specific applications depending on what is needed.

The low concentration aerogels, namely the 0.5% and 0.2% samples, also exhibited attractive forces when brought in contact with other objects. All other sample concentrations were not able to adhere to surfaces. Because of this, it is believed that the attractive force is due to Van der Waal interactions caused by the presence of nanofibers with a high surface-area-to-volume ratio in the aerogel. The nanofibers were only visible in the lower concentrations as can be seen in Figures 3 and 4. Although there was no general trend in the data points, it appeared that the 0.2% samples were slightly better at adhering to the glass microscope slide surface than the 0.5% when omitting failures.

Tension strength measurements were also recorded for all nanocellulose aerogel samples. The 0.5% samples performed the best followed closely by the 1.0% samples recording values of around  $6 \times 10^5 \text{ N/m}^2$ . Higher concentration samples were brittle allowing cracks to easily spread once a crack was started, but lower concentrations were slightly ductile allowing for the compressed sample to bend.

Future work for this study could entail varying the amount of microcrystalline cellulose even further to find the limits of what can be used and still create a useful aerogel. Lower concentrations could allow for the microcrystalline cellulose to be broken up into even smaller nanofibers possibly increasing the Van der Waals interactions even further. Future measurements should also be made with more precise pieces of equipment. One limitation for this study is that the adhesive strength apparatus was not the most precise. Samples may have also been



contaminated, so a miniature clean room environment may also increase measurement precision. It would also be useful to accurately measure the surface area of the sample using a surface area and pore analyzing machine to better understand the basic physical properties.

Cellulose is an extremely abundant material found in all places of the world. It is non-toxic allowing humans to safely handle it in many forms including particles on the micro and nano scales. It was previously shown that acid hydrolysis methods could be used to create useful ultralight nanocellulose aerogels, but an ultrasonication process is shown to be successful in the creation of similar nanocellulose aerogels. Derived from cellulose and made via a sonication and freeze drying process, this material is safe for humans to handle and still exhibits the interesting properties found in typical aerogels including ultralow densities and high percent air compositions. This material has potential to be useful for applications similar to filters or catalyst support structures in which current aerogels succeed.

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