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## Conductive AgNW/Tempo CNF Thin Film

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CONDUCTIVE AGNW/TEMPO CNF THIN FILMS

by

David Flewelling

A Thesis Submitted in Partial Fulfillment  
of the Requirements for a Degree with Honors  
Biomedical Engineering

The Honors College

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## ABSTRACT

Cellulose is a strong, readily available biologically sourced polymer with a wide potential for applications in many fields. Its high degree of biodegradability makes it an excellent candidate for environmentally friendly sensors, but it is necessary to devise a method by which to render it conductive. Silver nanowires are highly conductive nanoparticles with many potential applications in sensing, as well as being on a similar order-of-magnitude in size as cellulose derivatives, which makes them a prime candidate for use in this application. Methods of casting cellulose nanofibrils (CNF), cellulose nanocrystals (CNC), and TEMPO-Oxidized CNF (TEMPO CNF) films were explored, and the resulting products were treated with silver nanowires by both surface deposition and homogenization to develop conductive films. It was found that the results of this process had low sheet resistances, beneficial for use in this application.

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# CHAPTER I

## BACKGROUND

### On Cellulose

While wood has had a role as a material of human construction and creation for thousands of years, it is only recently that we have begun to realize the incredible potential for and versatility of a key component of its structure: cellulose. Figure 1 shows the chemical structure of the cellulose monomer residue unit, a disaccharide of D-glucose. Note the six alcohol groups, which allow hydrogen bonding within and between strands of cellulose, a major cause of the material's strength. The existing applications for cellulose at this time are broad: cellulose has been proposed for use in medicine as a unique basis for wound dressing and skin substitution[1], electronic papers and audio membranes of high quality[2, 3], chemical filtration and hydrophobic coatings[4], and even an aid to the production of carbon nanofibers[5].

There is growing concern about the environmental impact of many common consumer and industrial goods. While plastic is not the sole offender in environmental effects, it is certainly the most well known. Plastic-based materials are heavily reliant on crude oil extraction, the impact on the environment of such being well documented, as well as being having a rate of renewal that is effectively zero on human

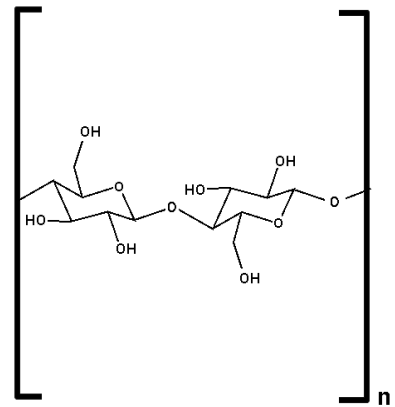


Figure 1 – The molecular configuration of cellobiose, the repeating unit of cellulose. Hydrogen bonding between fibers can occur at the alcohols, which contributes to the high fiber strength of cellulose.

timeframes. This is in addition to the long times needed for plastic to degrade in the environment, which make its presence a constant hazard for wildlife and potential source of environmental toxins. There is therefore a large push to develop materials which can replace plastic while also having milder impact when extracted and shorter lifespans after disposal. Cellulose is a strong candidate for this. In fact, cellulose degradation in the environment is faster than its sourcing from wood would suggest, since the decay rate of wood is slowed by the presence of lignins, which are generally not present in processed and delignified cellulose[6].

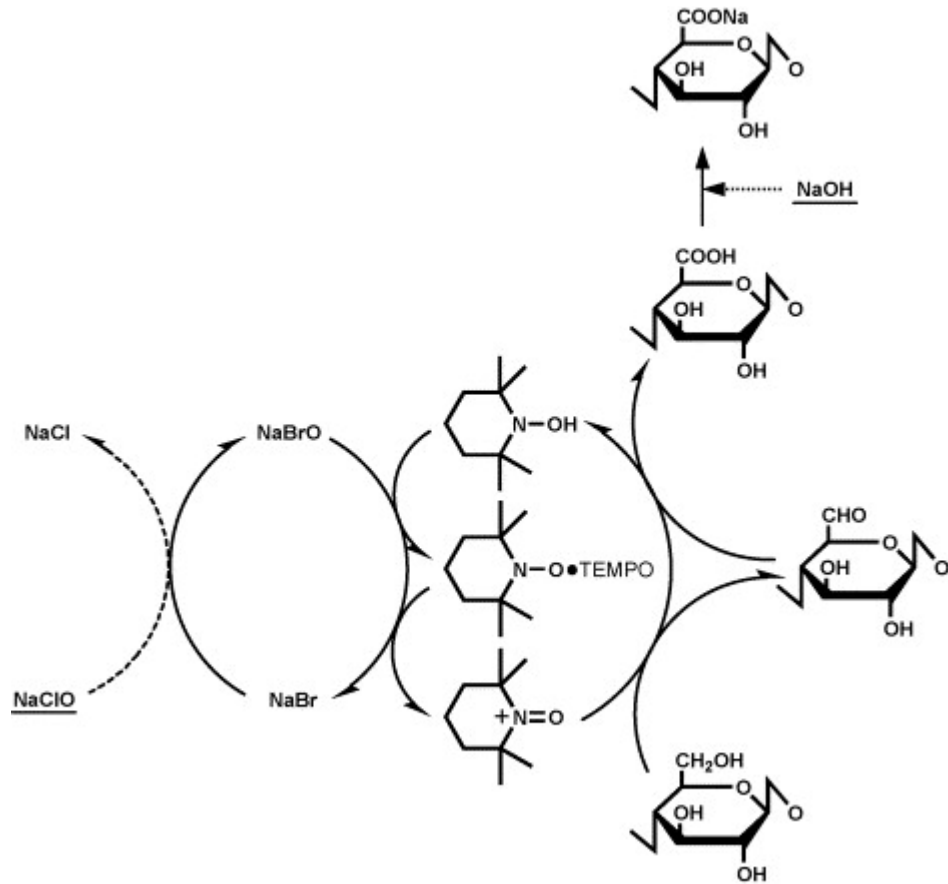


Figure 2 – The reaction scheme of the TEMPO oxidation process. The native cellulose alcohol is oxidized to a carboxylic acid, which is then converted to carboxylate.[7]



Approaching cellulose as a broad category glazes over many key differences between subcategories of material. There are three primary methods of processing cellulose into fibers: mechanical attrition, chemical processing, and bacterial synthesis[8]. Of relevance in the research discussed here is 2,2,6,6-Tetramethylpiperidin-1-yl)oxyl (TEMPO)-oxidized cellulose nanofibrils (TEMPO CNF), a particular kind of chemically-processed nanocellulose with extremely small fiber length and diameter. Particle size is controlled both by covalent bonding and the balance of bonding between fibers and hydrogen bonding to water. Cellulose nanofibril(CNF) slurries, as shown in Figure 3, have small particle sizes but do not completely dissociate into a homogenous suspension and instead flocculate into a slurry. TEMPO oxidation encourages complete dissociation by converting alcohol groups of unprocessed cellulose to carboxylate, which are highly polar and have a high charge affinity for water. As a result, the fiber length of TEMPO CNF is 0.5 to 100  $\mu\text{m}$  with diameters between 3 and 5 nm, while CNF lengths vary from 1 to 100  $\mu\text{m}$  with diameters between 5 and 50 nm,[9] a dramatic difference in both variance and size. The reduction in size and in variance of TEMPO CNF allows the formation of a relatively homogenous suspension, and thus changes the material properties of the final film.[7] The most relevant factors are the difference in mechanical properties and introduction of optical transparency. Comparisons of TEMPO CNF/polymer films to cellulose/polymer has been documented as significantly increasing both wet and dry tensile strength of resulting gels and dried forms[10]. The smaller particle size and reduction in variance also allows for visible light to pass through solutions of TEMPO CNF, shown in Figure 3. Key to the applications in films and sensors, these factors should compound to

allow thinner films which use less material to retain the same strength, while allowing visual inspection of material deposition.

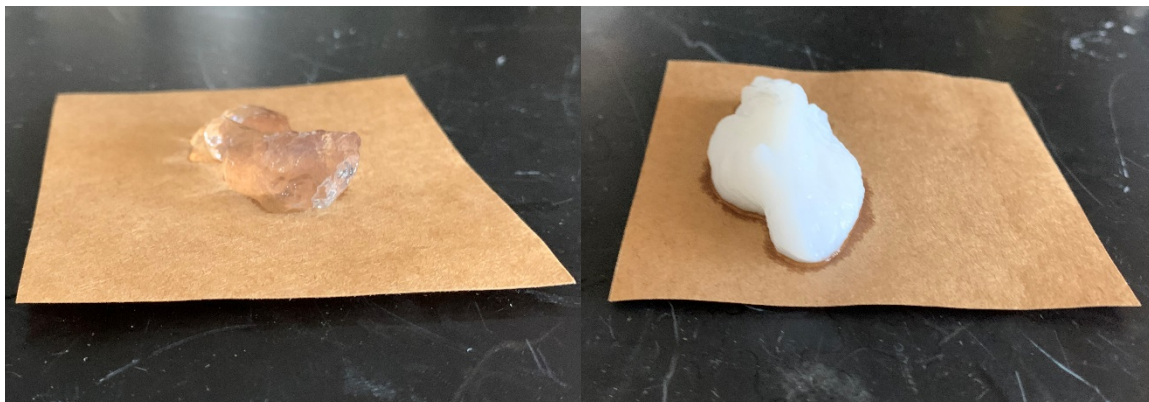


Figure 3 – TEMPO-mediated oxidation has clear and significant impacts on the structure of cellulose nanomaterials. On the left, TEMPO-treated cellulose, which is clear and “Jell-O-like” in consistency. On the right, cellulose nanofibrils, which have a thick and almost cloud-like texture.

### Silver nanowires

Metal nanoparticles are a broad category of materials which can be used for different purposes. While there are many methods of synthesizing nanoparticles, the method used for the research here is based on nucleation and proceeding growth. This process is generally theorized to proceed with the sudden appearance of small metallic nuclei which then condense into a number of large particles due to the rate mechanics of the particle growth process. Formally, these two phases are known as a LaMer Burst and Ostwald Ripening respectively[11]. Metal ions can be induced to form nanoparticles by dissolving a salt containing the relevant metal, then following with the addition of a reducing agent, and finally quenching the reaction. A classical example is the reaction of silver salts with sodium borohydride, a common reducer[12]. This produces a colloidal

solution of silver nanoparticles, generally spheres, of sizes controlled mainly by the ripening time.

It is also possible to control the relative shape of the particles resulting from this process through the use of directing agents. These are molecules which block off certain faces of the growing nanoparticle, limiting growth to specific conformations or directions. Nanowires and Nanorods are 1D nanoparticle structures whose growth has been constrained on all but one axis. This results in wires and rods (hence the names) which may be anywhere from dozens of nanometers to several microns in length. There are many proposed synthesis procedures for the creation of silver nanowires[13], such as polyol synthesis using Ethylene Glycol (EG) as a reducing agent[14, 15], wet synthesis relying on pH manipulation,[16] and microwave-driven polyol synthesis using chloroplatinic acid[17] as examples. These reactions are generally small batch or one-pot methods that are useful in a laboratory setting where small amounts of the material in question are needed.

### Objective of Research

The relatively comparable sizes of nanowires and TEMPO CNF fibrils leaves open the possibility for interweaving of the materials together. This is especially useful for high-conductivity materials such as silver or copper, which have the possibility of forming a continuous network across and beneath the surface of a film. Using cellulose as an inexpensive scaffold, silver nanowires (AgNWs) could be added to create a conductive cellulosic membrane or film. Similar designs have been proposed[18–22], though it appears that these generally rely on nano-scale patterning rather than nanoparticles, are focused on specific applications on one component, and/or primarily use non-cellulosic

materials. There is therefore a gap in establishing that a general-purpose conductive film can be created using simply nanocellulose and nanowires. More specifically, this thesis investigates if it is possible for nanowires to integrate into the film structure of nanocellulose in such a way that the film is continuously conductive, without complex or prohibitively expensive processes. During this research, a reliable process for synthesizing silver nanowires of micron-scale length was developed, and several methods of integrating this material into a network for cellulosic fibrils were tested to determine that a conductive film incorporating cellulose and silver nanowires is possible using simple methods.

## CHAPTER II

### METHODS

#### Synthesis of Metallic Nanowires

Nanowires were synthesized using a polyol process previously outlined and tested.[23] 5mL propylene glycol (PG) was heated to 170°C for one hour, stirred continuously in a round bottom flask to form propionaldehyde, which acts as a weak reducer to facilitate nucleation. 1.5mL of a 114mM polyvinylpyrrolidone in propylene glycol (PG) was then added, followed shortly by 1.5mL of a 94 mM AgNO<sub>3</sub>/PG solution. The solution was then allowed to stir and react for an extended time, up to 1 hour, before being removed from heating and allowed to cool. Although previous results had been reported using this process, it was found that it was extensively difficult to replicate the initial results of the paper, and it was necessary to conduct an extended investigation to determine the cause of reaction failure.

Testing determined that replication was possible when the  $\text{AgNO}_3$  solution was created immediately before use in the reaction vessel, and the synthesis protocol was amended to include this detail going forward. Resulting solutions were then centrifuged and a solvent exchange from PG to a polar solvent with high evaporation rate (generally methanol or acetone) was performed. Several washing steps were included, due to the need to remove accumulations of PVP on the surface of the particles in question. The resulting solution was then evaluated via UV-spectroscopy to obtain an absorbance curve, and

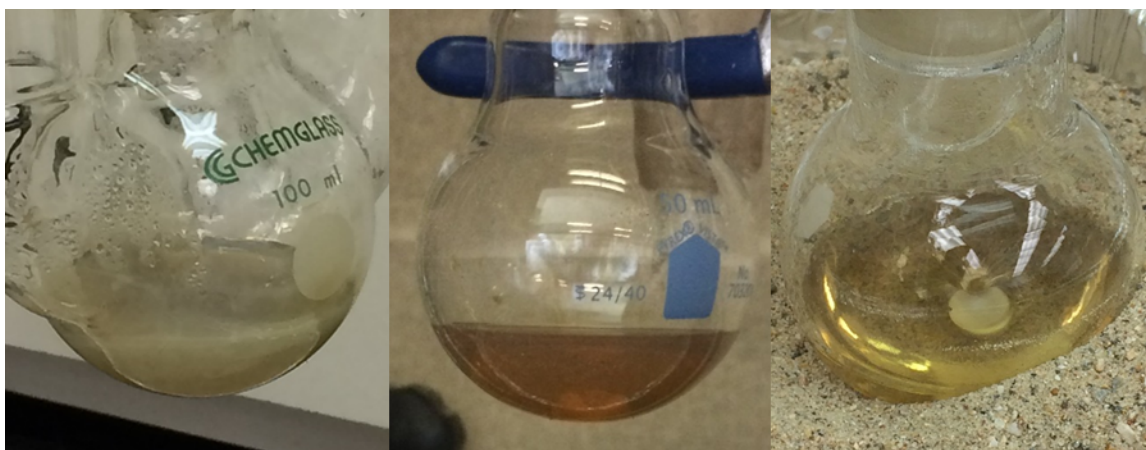


Figure 4 – From left to right: A successful batch synthesis of silver nanowires, an unsuccessful batch synthesis, and an incomplete reaction. The opacity of the successful synthesis is likely due to the long “fibers” of nanowires which may interact, and scatter light differently depending on orientation. The left two syntheses are both nanospheres at different growth stages, the right being an early stage where the particulates are small, and the middle where they have grown in size and undergone a shift in absorbance.

imaged via Transmission Electron Microscopy (TEM) to precisely verify composition and characterize the resulting particles. In addition, silver nanowires have a visibly unique appearance which allows for ready confirmation of synthesis. (See figure 4)

## Preparation of CNF and TEMPO Films

Due to the high cost of TEMPO, initial tests were performed to determine if CNF could be used as a mechanically equivalent to TEMPO as the basis for a flexible, transparent, and mechanically strong film. To achieve this, water was removed from a CNF slurry via vacuum filtration using a glass filter to achieve a disc-like shape. These CNF were notably opaque even at low volumes, and often required excessively high drying times despite the use of vacuum filtration to aid water removal. Drying tests therefore continued using cellulose nanocrystals (CNC), a chemical modification of native cellulose which possesses similar mechanical and optical properties to TEMPO CNF, but with larger fiber sizes.

Samples of CNC were diluted at varying concentrations and volumes and placed in laboratory petri dishes to dry under fume hood ventilation. It was found that drying using this method posed issues due to the tendency of the dried cellulose to adhere closely to the bottom of each petri dish, which made effectively removal impossible. Larger samples were replaced with small plastic sample dishes which could be cracked and broken inexpensively to allow access to the samples, and this method proved to be successful. Unoptimized test films were created by diluting CNC in water to 0.25 wt%, and petri dishes were filled to approximately 1 cm (+- 0.1 cm) before drying. Four samples were obtained from this process and it was found that they were mechanically sturdy enough to avoid easy tearing while maintaining optical transparency. As such, this process was retained for use in AgNW deposition investigations.

## Deposition of AgNW onto TEMPO Films

Two methods were explored to integrate AgNW into TEMPO CNF. The primary success marker was determined to be conductivity across patches, since this would indicate that a large surface or sub-surface network of nanowires had formed and allowed for a detectable mean-free-path across the material.

Initial attempts at creating conductivity were attempted using spotting/dropping, or the direct addition of a solution containing silver nanowires onto the surface of the dried film, both common techniques. It was found that this process was successful in inducing conductivity in patches when high concentrations of nanoparticle solution were used (1.4x dilution of reaction-concentration), but the deposition process was not homogenous even if the entire surface was covered in nanoparticle solution. This resulted in small patches of conductivity, and a uneven optical transparency due to accumulations of silver particulates in specific areas. A new approach to the drying process was testing by homogenizing TEMPO CNF slurries with the AgNW reaction solution prior to drying. The produced films had better optical transparency and more even particle distribution, but higher concentrations of nanowire solutions were needed to induce conductivity via this method.



## CHAPTER III

### RESULTS

#### Nanowire Characterization

Obtained nanowire solutions underwent UV spectroscopy measurements to determine absorbance spectra, as the signature of AgNWs is distinct from those of spheres or other similar particles (See Figure 5) on account of the multiple non-symmetric axes possessed by the wire shape.[24, 25] As a result, peak absorbance spectra changes depending on whether the radial or longitudinal axis of the wire is acting as the absorptive agent, and creating the appearance of a “humped” spectra. The bottom spectra of Figure 5 corresponds to results from the successful and adopted synthesis process. Additionally, synthesized solutions suspected to contain nanowires were tested using TEM to obtain direct visual confirmation of presence. Obtained wires varied in size, generally 100-200 nm in diameter and 10-50  $\mu\text{m}$  long, or aspect ratios of 50 to 500. Yield purity was not characterized quantitatively, but images do suggest the presence of various silver particulates with uncontrolled growth. Despite several washing steps, PVP also appears in

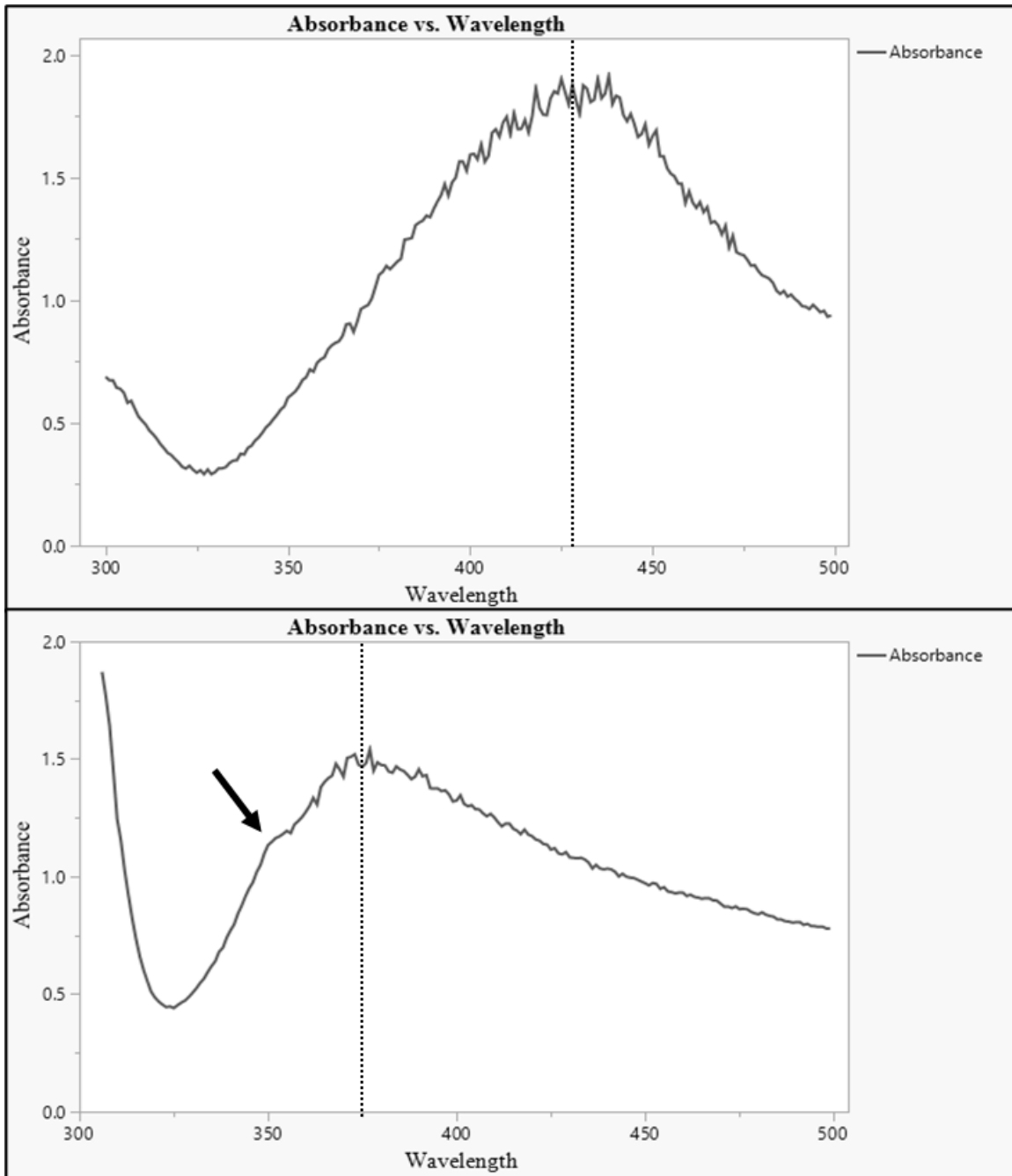


Figure 5 – Absorbance spectra of two samples obtained via synthesis. Top sample is a result of the failed synthesis, while the bottom sample is a successful silver nanowire synthesis. Note the blueshift of peak absorbance (dotted line), and the formation of a plateau at 350nm (arrow). The plateau or “humped” feature is a common signal of AgNW formation.

most images taken as a dark cloudiness around the particulates. Visual characterization necessarily also occurs due to the unique appearance of AgNWs (see Figure 4). As a final

note, the synthesis has presented several issues down the road due to the appearance of clumps in the reaction solution. While these have not been characterized, reactions with high degrees of clumping appear “thinner” than previous solutions that do not exhibit this behavior.

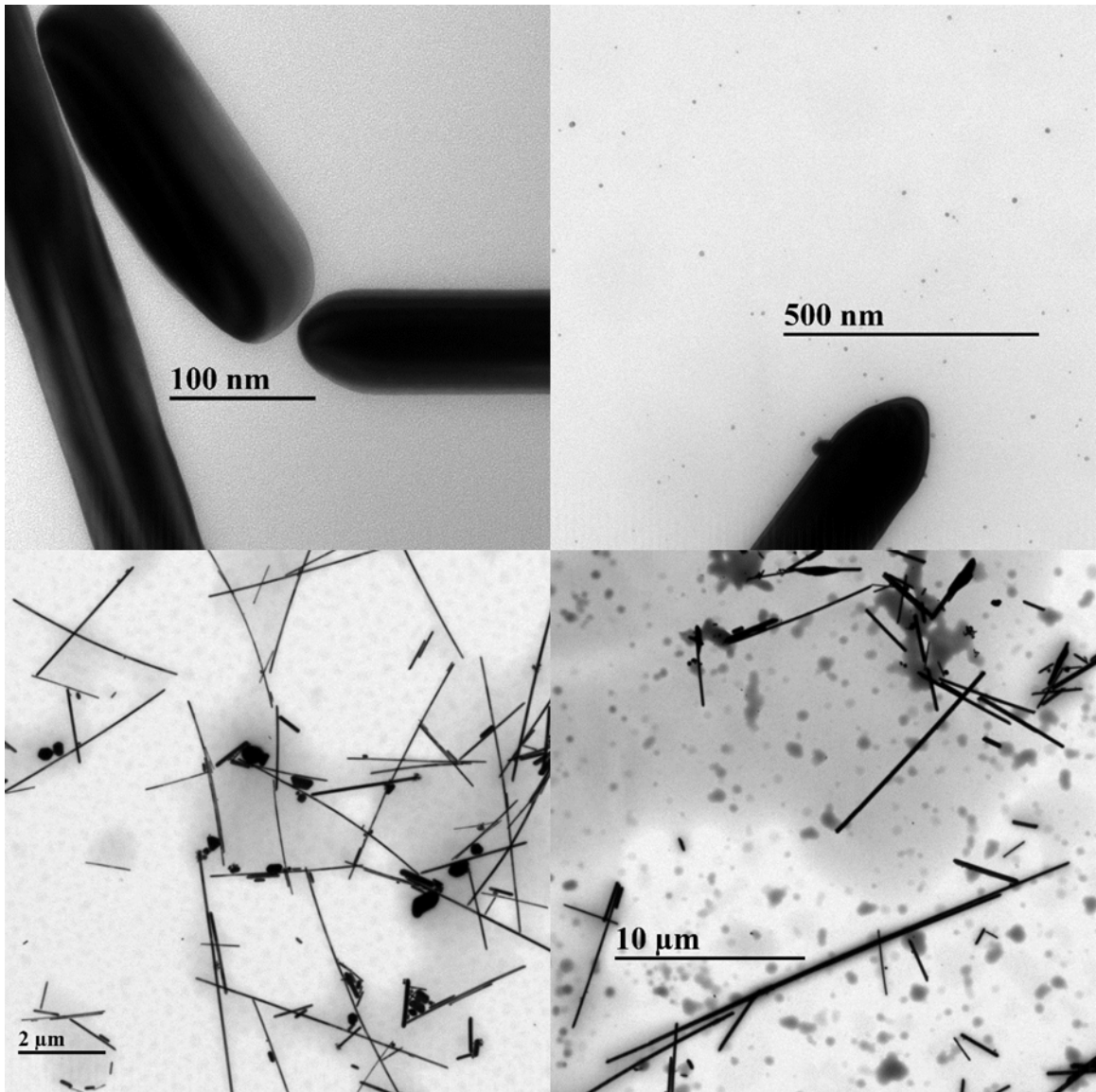


Figure 6 – TEM imagery of successful nanowire synthesis methods. Solution is a mixture of wires and silver agglomerates and particles of various shapes. Dark cloudiness in background is likely the result of incomplete washing leaving PVP residues on samples prior to imaging.

## Film Drying Process

A large number of films were produced as a result of testing the drying process. Initial assessments of material viability were made by simple visual and tactile assessment of film qualities, principally looking for optical transparency, uniformity, frequency and ease of breakage, and flexibility (See Table 1). Of these factors, transparency and flexibility were key, as a tendency to rips or breaks can be largely compensated for by increasing the amount of material. TEMPO was chosen for a film basis as it has high optical transparency and flexibility, at the cost of being somewhat fragile, a factor which can be corrected for by increasing film thickness. CNC was transparent with a good balance of stiff without losing flexibility, and was therefore less vulnerable to breakage. CNF was very resistant to breakage but not optically transparent. Methods of drying CNF also required pulling water from the slurry via vacuum filtration, which resulted in films that were noticeably thicker in the center than on the rims.

Table 1: Qualitative ranking of materials used for film production

<b>Material</b>	<b>Transparency*</b>	<b>Uniformity</b>	<b>Resistance to breakage</b>	<b>Flexibility*</b>
<i>CNF</i>	1	1	3	1
<i>CNC</i>	2	2	2	2
<i>TEMPO</i> <i>CNF</i>	3	3	1	3

Note: All features measured on a scale of 1-3, 1 being lowest-performing material, 3 being best-performing material. Stars denote features of critical importance



Figure 7 – A CNF film produced via vacuum filtration and removal of water from a raw slurry. The film is stiff and resilient, but the optical transparency is low, and the accumulation of material into spots is notable, especially on the upper portion of the film which is notably lighter.

## Integration of AgNW

### Confirmation of conductivity

was induced using a simple multimeter set to signal the presence of an electric current between the positive and negative probes, followed by a measurement of the resistance across the confirmed distance. Films with added AgNW were tested continuously on patches with apparent silver to determine if a significant

conductivity was present, and this signaled the success of the process. Ideally, a significant conductivity across a large distance, 1-2cm, would be detected. Only small patches of continuously conductive film were found, on average 2 mm in diameter. This distance is too small for more formal distances like four point tests to be performed, although simple multimeter tests were still possible (See Figure 8). The likely culprit behind this is the uneven distribution of dried AgNW solution on the surface of the material: most dried films had notable concentrations of silver into single areas, due to the warping of the film as it dried. This effect seems to be a natural result of the dropping process, and additional steps would likely need to be taken to correct for it, such as hot pressing TEMPO films before they have completed drying. Homogenizing of TEMPO CNF slurries and AgNW solutions prior to drying had promising results (See Figure 9). These films were more even, but additional material was required to induce conductivity in samples, although it could be achieved with large enough amounts of nanowire solution. As quantitative measurements of nanoparticle concentrations in the solutions used here haven't been conducted, any

number named would be conjecture. Notably, what could be a corrosion effect was observed on films treated with dropping several weeks post-treatment. The cause of this is not certain, but it's possible that it is simply oxidation of the surface of free silver. Silver tarnish. This effect is not visible in homogenized films.

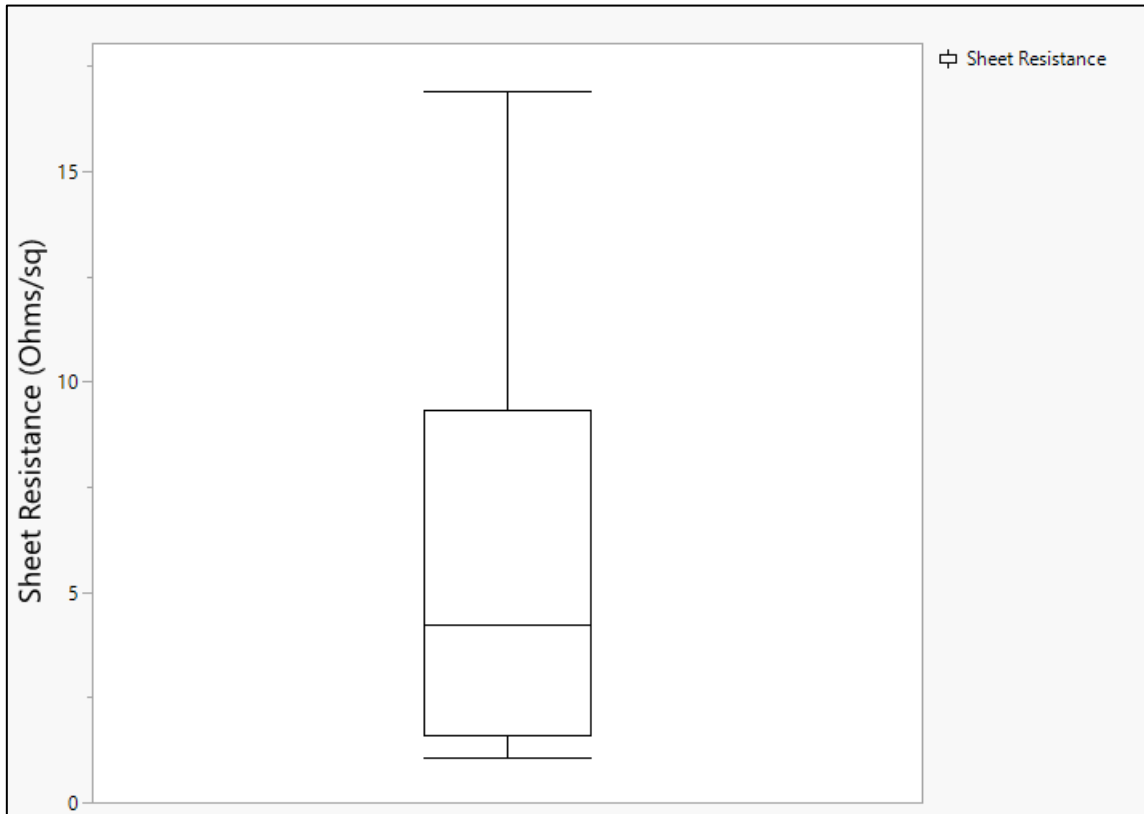


Figure 8 – Box chart of sheet resistivity measurements obtained using a multimeter. Sheet length was measured as the distance between multimeter probes, with an estimated 0.5mm width of probe diameter.



Figure 9 – Clockwise from top-left: TEMPO film treated by the dropping method, with extensive corrosion. A second film treated by dropping, note the wrinkling and light corrosion. A homogenized TEMPO/AgNW film, with no corrosion, even color, and relative lack of wrinkling. Lastly, a film treated with dropping, but corrosion is absent.

## CHAPTER IV

### DISCUSSION AND CONCLUSION

#### Are Improvements Needed to the Nanoparticle Synthesis?

The data available on the AgNW synthesis confirm that the process is successfully producing nanoparticles, but it is likely that this is not sufficient for final use in a device. The factor that contributes most to this assessment is the continued clumping of material in the reaction vessel. It's likely that this is an issue of improper cleaning of reaction vessels leading to accumulation of silver around contaminants, but extensive use of Contrad-70 and 5M nitric acid on glassware and stirring equipment does not seem to have alleviated this issue. This is a subsection of a larger issue in device creation, which is the relatively low purity of solutions, and the current inability to control aspects of the finished particulate. TEM images show wires primarily in a mixture of particulate, and of varying sizes. It would be ideal to homogenize the resulting particles. For the sake of process control and convenience, it would also be advantageous to determine the concentration of reaction solutions and if scaling of the reaction is possible. That said, the data available now suggests that the current process is successful in creating usable reagents, and is reliably usable. This is not an area in urgent need of attention to improve the quality of conductivity, or at least the data does not suggest so.



## Improving Characterization of Films

Simple air-drying of films does appear to create materials usable for the creation of sensors, but the lack of mechanical strength is an issue. While flexibility allows a device some room to avoid direct tears by simply giving, it would be beneficial to look into ways to prevent tears altogether. While flexibility was emphasized in the feature analysis (Table 1), it's possible that resistance to breakage is a more important feature. It would be ideal to perform some kind of standardized strength testing on the materials together to quantify these values in a way that allows for more direct analysis. Instron measurements are an accessible option for materials testing and would be an ideal follow up to the mostly-qualitative analysis of strength presented here.

Optical transparency should also be assessed for future development. This feature has a significant advantage in a testing environment as it allows visual assessment of the spread of silver across a material. It would be more difficult to evaluate the effectiveness of AgNW integration in an opaque film compared to the CNC and TEMPO CNF films tested. However, this may not be needed after a method of integrating AgNW has been established as effective, and visually checking the deposition is no longer needed. Therefore, in applications of conductive films that do not require transparency, CNF may be a more ideal material, especially considering the lower cost of CNF compared to TEMPO CNF. This applies doubly if the proposed revisit of flexibility vs. strength finds that strength is a more important feature.

## Improvements to the Integration of Nanoparticles

While the data presented confirm that conductive films can be established by direct integration of AgNWs into TEMPO CNF films, several questions remain which are key to the design of such a device. The most central of these is how to quantify the amount of nanoparticles needed to establish conductivity. This is difficult to perform due to the variable nature of the synthesis method. Additions of AgNWs were recorded primarily in their relative concentration from the reaction solution, but the concentration of nanoparticulate in these solutions is variable and not precisely known. It will be necessary to characterize reaction concentrations on average, then correlate this to a threshold level needed to establish conductivity to answer this question.

This is especially complicated by the fact that film warping occurs during both the casting and dropping process, which leads to uneven additions of material to the surface of the films. This problem would be accounted for by switching preferentially to the homogenized AgNW/TEMPO CNF solution before drying, and notably this effect was not observed in the films prepared in such a way (see Figure 9). Homogenization also appears to correct for the corrosion observed on the films. If this is the case, it could be that most wires are on the interior of the film and not exposed to air, which may reduce surface oxidation. Films with suspected corrosion were not measured for conductivity, but since mean free paths depend on surface contact between particles, corrosion may impact the conductivity of completed films.

## Conclusion

Research conducted up to this point confirms that cellulosic membranes can be made conductive through simple direct addition of silver nanowires. Work remains to be done to quantify important parameters of film function, most importantly the concentration and volumes of AgNWs needed to induce conductivity, and the degree of conductivity induced. In addition, better characterization of physical parameters is needed to optimize the design and production processes. Finally, it would be advantageous to develop a functional sensor to design the technology around. Initial attempts were made into using the design as a humidity sensor, but usable results were not obtained and are therefore beyond the scope of this discussion. Nevertheless, the purpose of this investigation, to confirm that conductivity can be induced through simple methods of integrating nanoparticles into a surface has been confirmed.

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