ELECTROSPINNING OF CELLULOSE AND CARBON

NANOTUBE-CELLULOSE FIBERS FOR SMART APPLICATIONS

A Junior Scholars Thesis

by

ALEXANDER MORGAN PANKONIEN

Submitted to the Office of Undergraduate Research
Texas A&M University
in partial fulfillment of the requirements for the designation as

UNDERGRADUATE RESEARCH SCHOLAR

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Major: Aerospace Engineering

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Approved by:

Research Advisor: Zoubeida Ounaies
Associate Dean for Undergraduate Research: Robert C. Webb

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ABSTRACT

Electrospinning of Cellulose and Carbon Nanotube Cellulose Fibers for Smart
Applications (April 2008)

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Cellulose is one of the Earth's most abundant natural polymers and is used as a raw
material in various applications. Recently, cellulose based electro-active paper (EAPap)
has been investigated for its potential as a smart material. The electrospinning method of
fiber production is not a new way of fabrication; however, it has attracted a great deal of
attention as a means of producing non-woven membranes of nanofibers due to its simple
methodology and the advent of nano applications. Electrospinning occurs when the
electrical force on a polymer droplet overcomes its surface tension, and a charged jet is
ejected. As the liquid jet is continuously elongated and the solvent is evaporated, the
fibers of sub-micron size or nano size are formed, depending on the conditions. In a
previous study, a cellulose mat was electro-spun and tested for piezoelectric
characteristics. This aligned, electrospun cellulose mat showed a possibility as a
promising smart material. Additionally, carbon nanotubes have been considered for the
versatile nano-applications due to their superior material properties such as low density
and high aspect ratio. Parametric studies were conducted to find optimum conditions for

electrospinning. Various ways of reducing surface tension of solutions were investigated including radiative and convective heating of the solution. Pre-examination of solution is very important in consistent, uniform fiber formation.

In this study, cellulose and CNT-cellulose composite fibers were prepared via electrospinning. The optimal experimental conditions for fiber generation were found so that the mechanical strength of both the composite and the pure cellulose fibers could be compared in future tests. Eventually, this fiber will be interwoven into the CNT-cellulose mat and be used as an electro-active paper sensor and actuator. The CNT-cellulose electrospun mat will be widely applicable to the fields of sensors, filters and reinforcements in composites because of its intrinsic properties of porosity, light weight, flexibility, and large surface area. To be used in the aforementioned applications, piezoelectric properties of this composite will also be tested in the next step.

DEDICATION

To my family and friends who've always believed in me.

ACKNOWLEDGMENTS

NOMENCLATURE

CNT	Carbon Nanotubes
DMAc	Dimethylacetamide
LiCl	Lithium Chloride
Pa	Pascals (Pressure) PAN Polyacrylonitrile
PLA	Polylactic acid
PVDF	Polyvinylidene Fluoride
PZT	Lead Zirconium Titanate
SWNT	Single-Walled Nanotubes
TrFE	Trifluoroethylene
V	Volts (Potential Difference)
hr	Hour (Time)
m	Meters (Distance)
mL	Milliliters (Volume)
wt	Weight Percentage in Solution

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CHAPTER I

INTRODUCTION

Cellulose

Cellulose, a natural occurring polymer, is known to exhibit piezoelectric characteristics, albeit weakly. In the last few years there has been a renewed interest in cellulose research and application, sparked mostly by technological interests in renewable raw materials and more environmentally friendly and sustainable resources. Cellulose is the most abundant natural polymer on Earth. Cellulose can be found in the cell walls of plants around the world. Primary sources for cellulose include cotton, flax, hemp jute, straw and wood pulp.[1] Cellulose is also a high molecular weight polysaccharide. Consequently, it consists of consists of glucose-glucose linkages arranged in linear chains, where every other glucose residue is rotated in opposite direction (Fig 1).[2]
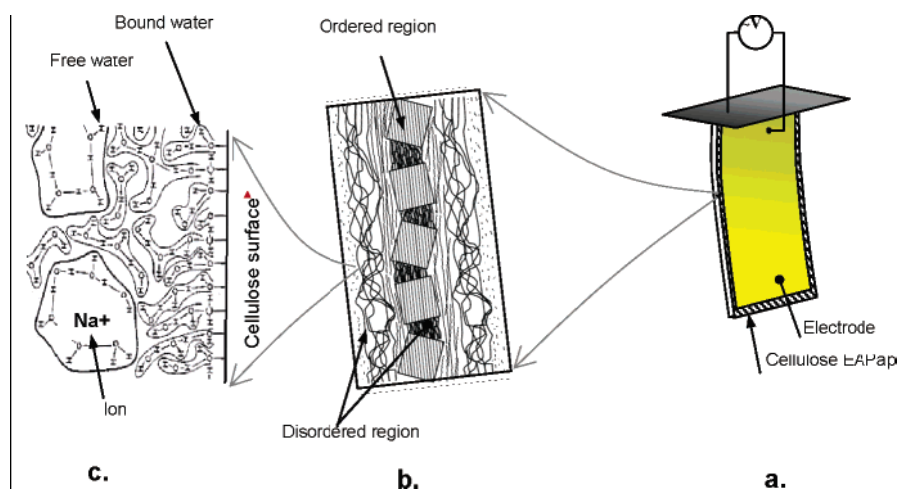

Figure 1. Cellulose Polymer Orientation [3]

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This thesis follows the style and format of the International Journal of Nanoscience.

Then, in 1955, Fukada verified the piezoelectric coefficients of wood and its electromechanical coupling effect. He also demonstrated that the oriented cellulose crystallites were responsible for the observed piezoelectricity.[2] Currently, the most technologically important piezoelectric ceramics are lead-based, such as the most commonly used ceramic (PZT, lead zirconium titanate) and are consequently harmful to the environment. Also, the only commercially available piezoelectric polymer is PVDF which is both synthetic and requires labor-intensive processing Kim et al. showed that cellulose paper can act as a smart material by exhibiting a large displacement with low actuation voltages and low electrical power consumptions. Consequently, spun-fiber cellulose, which is abundantly occurring as a primary structural component in green plants, shows promise as a bio-friendly piezoelectric alternative.[2]

Electrospinning

Electrostatic fiber spinning, also known as "electrospinning," forms submicron scale diameter fibers using electrostatic forces in a charged polymeric solution. The typical setup for electrospinning includes a syringe, needle and pump to hold and control the solution flow rate (1-5 mL/hr). A high voltage power supply (5-30 kilovolts) is connected to the needle and a collector plate is grounded and placed 5-20 cm away from the needle tip.[5] The relationship between the solution's properties, the voltage required and the distance between the needle and the target are specific to each solution and at this point in time must be experimentally determined and verified for each solution due to the complexity of the electrical instabilities. The solution flows through the needle,

forming a drop at the tip. As the voltage is raised, the electric force deforms the shape of the droplet until a critical voltage is reached. Then, the electric force overcomes the viscoelastic forces in the fluid and a jet is formed in the shape of a Taylor cone.[6] Extensive research, including the seminal work by Reneker, has shown the critical shape of the Taylor cone to have a half angle of 33.5° which is independent of fluid properties for Newtonain fluids.[7] The jet of fluid originally emanates from the Taylor cone in a straight line but then begins to whip wildly further from the needle due to electrohydrodynamic instability.[8] The whipping jet finally deposits the solution on the grounded target, which, if static, results in an unwoven mat of fibers (See Figure 2). The charge applied to the solution while forming the jet also aligns the cellulose molecules' electric properties. As verified by Renker and Chun, electrospinning has proven to be a valuable method of polymer fiber production for fibers in the 40-2000 nm diameter range.[9]
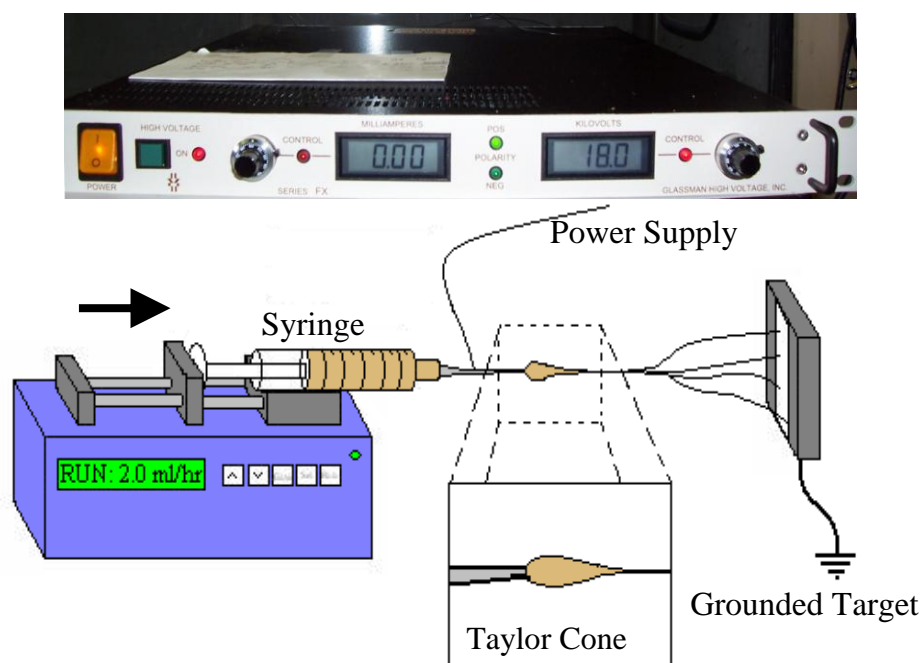
Figure 2. Electrospinning Setup

Past production methods for cellulose fibers included mixing cellulose with chemical binders or the use of cellulose acetate.[10] The use of other binding agents was also often accompanied by submerging the fiber formation in a chemical bath. The fibers were collected as they emerged in a process called "wet-spinning". The processing of cellulose fibers then included the use of intermediate salts or other ionic compound in the mixture. Kim et. al. showed that these fibers did not result in well-aligned and ordered fibers even when proper elongation was achieved. Because the piezoelectric properties of a material depend heavily on the structure and composition of the material, the introduction of intermediate compounds and the degree of alignment severely alter

the inherent piezoelectric properties of the solution.[8] This should not be surprising because cellulose exhibits unique properties in that it is not readily soluble in water or other ordinary solvents.[1] Exotic solvents such as liquid ammonia, chlorinated alkanes, and Dimethyl acetamide with Lithium chloride have been shown to effectively dissolve the cellulose. Ciferri et. al. determined that of these solvents, the DMAc + LiCL solvent does not result in the degradation of the cellulose.[11] By electrospinning cellulose fibers from a DMAc + LiCl solution on a moving target, an electrically and mechanically aligned cellulose fiber can be created. Kim et. al produced submicron-sized cellulose fibers with a 3% weight Cellulose solution dissolved in the DMAc + LiCl solvent. They also showed that 1% weight and 2% weight solutions led to the formation of irregular clusters on the fibers. Mimicking the reported test conditions of an electric field strength between 1 and 4 kilovolts per centimeter should allow for the production of cellulose fibers via electrospinning.[12]

Electrospinning of Polymer Nanotubes

Carbon nanotubes (CNTs) have recently come to the forefront of modern science because of their anisotropic mechanical and electrical properties. A typical single carbon nanotube can have an elastic modulus higher than one TPa and a tensile strength of 10 GPa.[13] Resultantly, there has been a significant effort to incorporate CNTs into polymer materials. Kymakis et. al. showed a 5 order of magnitude increase in the electrical conductivity of films when CNT concentration was raised from 0% to 20% wt in the films.[14] However, the commonly used single-walled carbon nanotubes (SWNTs), a type

of carbon nanotube, interact with one another due to their van der Waals forces to form entangled clusters. Many different research methods including magnetic field suspension, mechanical stretching of a polymer/Nanotube composite film, and use of a substrate have attempted to reliably and uniformly align the nanotubes.[15] Meanwhile, electrospinning has generated renewed interest in this area because of its promising ability to also align the SWNTs during jet formation. The SWNTs' alignment and dispersion problems should theoretically be fixed by the high shear forces and electric forces while spinning.[13] Several papers have reported this method as a reliable way to create composite fibers and yarns with carbon nanotubes and several different polymers. For example, carbon nanotubes have been completely dispersed in both polylactic acid (PLA) and polyacrylonitrile (PAN), and electrospun at a voltage of 25kV with the needle 15cm away from the target.[16] However, one problem gained with this approach is the random orientation of the fiber associated with the wild spinning of the jet. The first and most widely used way to compensate for the variation in orientation is to project the fibers onto a rotating drum. This method rolls the deposited fibers in the same direction as they are deposited, thereby uniformly aligning the fibers. Several other methods involve the alteration of the electric field between the syringe and the grounded target by methods such as placing the drum between two charged electric plates to separate different diameter fibers.[13] Consistency of fiber diameter is not the only concern of composite solutions. Length of fibers is directly dependent upon the sustainability of the Taylor cone formation. Also, electrospun fibers tend to form "beads" when the viscous effects of the solution within the Taylor cone are insufficient to adequately orient the

SWNT or polymer within the solution (See Figure 3).[11] All of these factors, including choice of solvent and thus viscosity, must be carefully considered in the process of electrospinning polymers with nanotubes.
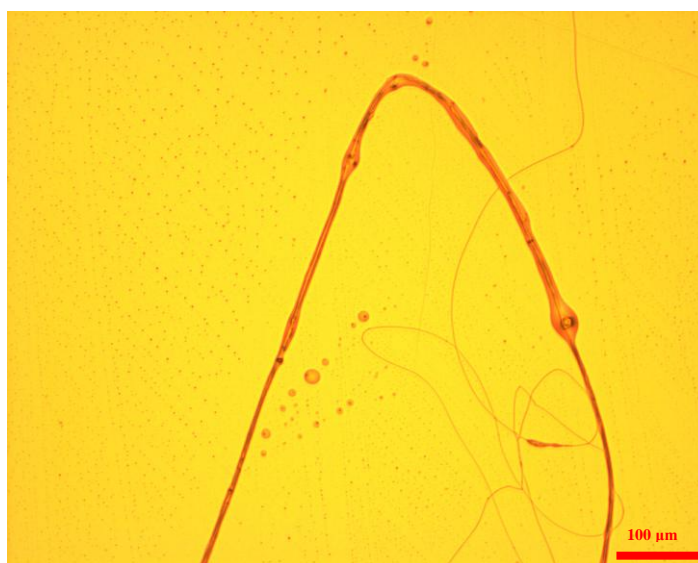

Figure 3. Beading on Electrospun Fibers

CHAPTER II

METHODS

Initial Cellulose Solution Setup

Upon receiving the 2% wt cellulose solution from a production lab in South Korea, the solution was determined to be too viscous to electrospin with a conventional setup as depicted in Figure 2. The target was placed 7 cm horizontally from the needle tip with a glass slide attached to collect the fibers for later inspection and the voltage was raised to the maximum allowable setting on the power supply, 30 kV. The high voltage caused a great deal of electrical sparking between equipment, but a few microfibers resulted (Fig. 3). Many different combinations of parameters were tried, including voltages ranging from 5 kV to 30 kV, distances to target ranging from 4 cm to 15 cm, and solution flow rates through the needle from 0.5 mL/hr to 5 mL/hr. However, every combination of these parameters resulted in the solution forming viscous drops instead of a Taylor cone.

It was determined that the equilibrium between the viscous and electrical forces in the Taylor cone could not be established. The viscous forces were too high to effectively and safely be overcome by available instrumentation. Adding to the difficult of the problem, the content of the solution could not be altered significantly without affecting the solubility of the Cellulose in the solution. Resultantly, several setups were immediately drafted to lower the viscosity of the solution without drastically changing its chemical composition by heating the solution.

Preheated Cellulose Solution Setup

The first attempted setup relied on heating the solution before it was place in the syringe.

The cellulose solution was placed on a hot plate in a beaker and heated to approximately

80°C, just below the boiling point of DMAc. Heating the solution to this temperature

allowed for the greatest reduction the viscosity without evaporating the solvent. The

solution was then drawn into the syringe without using the needle to facilitate solution

movement because the solution was still quite viscous. The syringe was then cleaned to

prevent electrical conduction along the outside of the plastic syringe and the needle was

screwed onto the needle. The syringe with the heated solution in the needle was then

placed on the syringe pump and the electrospinning test was run (See Figure 4).

Figure 4. Preheated Cellulose Solution Setup, "Setup A"

This setup, which came to be known as "Setup A", produced more fibers than the solution that did not heat the solution. However, even at the highest voltage setting, 30kV, and a target distance of 10cm, far fewer fibers were produced than expected. It was noted that the solution properties had probably changed slightly as some DMAc solvent evaporated during the heating process. In addition, many of the fibers' diameters varied along the length of the fibers. The varying fiber diameters were most likely caused by the uneven cooling of the solution as it moved through the syringe. The solution cooled during its transfer from the beaker to the syringe. Also, the syringe was unavoidably in contact with a metal pump on the bottom and cool air on the top and at the needle. Because each test was run at a set voltage and distance, the parameters could not be configured for the changing viscosity of the solution as it cooled. Preheating the solution had allowed the solution to reach the desired temperature quickly without conductively trying to heat the electrified setup during the actual test. Recognition of the need for safely heating the solution to a constant and controllable solution temperature for the production of homogenous fibers led to the creation of a third setup.

Radiatively Heating Cellulose Solution Setup

In an attempt to keep the cellulose solution heated throughout the experiment, a heat lamp was used to heat syringe without contacting and potentially grounding the highly electrified setup. The setup was nearly identical to the initial setup without heating; however, a heat lamp's radiation was directed near the end of the syringe. The infrared light heated the solution as it neared the needle in the syringe, passed through the needle

and then exited the needle to form the Taylor cone. Once again the parameters were

varied with the voltage ranging from 5 kV to 30 kV, the distance to the grounded target

set at 10cm, and the solution flow rate ranging between 0.5 mL/hr and 5 mL/hr. A

thermo probe was used to measure the temperature of the solution exiting the tip of the

needle before the power supply charged the needle (see Figure 5).

Figure 5. Radiatively Heated Cellulose Solution Setup, "Setup B"

This setup, designated as "Setup B", provided sustainable heating of the solution in the needle. However, electrospinning occurred occasionally and intermittently, interspersed between periods where viscous forces still allowed the solution to only form globules. The temperatures achieved by using the heat lamp to heat the needle were still not high enough for consistent fiber production. The solution only reached a maximum temperature of approximately $35^{o}C$ within the needle, far below the desired solution temperature of $70^{o}C$. The infrared radiation from the heat lamp was not heating the needle and thus the solution high enough to reach the viscosity levels needed for electrospinning. However, the complete containment of the solution within the syringe while heating solved the problem of evaporating DMAc changing the solution's concentration while heating. After several more trials, it was determined that the heat lamp could not be brought close enough to heat the solution to the desired temperature without the electricity arcing and shorting out the lamp.

Convectively Heated Cellulose Solution Setup

A fourth setup, also called "Setup C" was created that used a much higher temperature heat source, a heat gun. The potentiometer on the heat gun allowed for flow temperatures up to $500\ ^{o}C$. However, the high airspeed from the heat gun threatened to disturb the formation of the Taylor cone and the nano-scale fibers. Thus a shield of cardboard was installed between the airflow and the tip of the needle (see Figure 6), allowing the needle to be heated without blowing away the forming fibers.

Further, to increase the heating effects of the hot air on the needle, a heat sink was made from aluminum foil and wrapped around needle. This heat sink increased the surface area exposed to the hot air flow, thereby increasing the heat transferred into the needle by the hot air. By adjusting the temperature and distance of the heat gun from the heat sink around the needle, the temperature of the solution within the needle was carefully calibrated to higher temperatures. Setup C also conserved the solution's concentration of solvent by allowing for heating within the syringe and thereby preventing DMAc evaporation, similar to Setup B. The resultant setup required a complete change in equipment. The needle length was increased from 5/8" to 2". A carboard shield was installed between the pump and the needle tip. A heat sink and heat gun were set up in the area between the cardboard shield and the solution pump to allow for heating the solution. The syringe was changed from a disposable plastic needle to a reusable glass needle that would not melt from the higher airflow temperatures (see Figure 6).

Figure 6. Convectively Heated Cellulose Setup, "Setup C"

The solution temperature within the needle was measured with a thermoprobe as the solution emerged from the tip of the needle. The solution temperature within the needle was varied between 20 ^{o}C and 45 ^{o}C. Not only did Setup C produce fibers of consistent diameter, but in addition it consistently produced fibers with smaller diameters than the other setups. The lower viscosity of the solution also allowed for the establishment of a stable Taylor cone at lower voltages. The distance to the grounded target from the needle was fixed at 10 cm while the voltage ranged from 5 kV to 25 kV. The tested solution flow rates ranged from 0.5 mL/hr to 5 mL/hr.

Cellulose with Carbon Nanotubes Solution Setup

The solution made from the cellulose solution mixed with single-walled carbon nanotubes dissolved in DMAc presented an additional degree of difficulty in electrospinning. The concentration of SWNT needed to be high enough so as to affect the piezoelectric properties of the nanofibers in the end. By adding more SWNT to the solution to increase their effect on the solution's properties, the amount of DMAc was also increased, decreasing the viscosity of the solution. This change in viscosity changed the parameters at which the solution electrospun, decreasing the required voltage and the solution flow rate, and eliminating the need for heating the solution. Consequently, the original, unheated cellulose setup was used with the Cellulose SWNT solution, colored black because of the carbon nantoubes, instead of the original cellulose solution (see Figure 7).

Figure 7. Cellulose with SWNT Simple Electrospinning Setup

The distance to the grounded target was set to 10cm and the voltage was varied between

5 kV and 30 kV. The solution flow rate ranged from 0.5 mL/hr to 5 mL/hr. The original

solution production method involved adding 10% wt more DMAc to a 2% wt Cellulose

solution to achieve dispersion of the SWNT. As a result, the determination of solution

properties and its production rather than the determination of the electrospinning

parameters posed the greatest design challenge for the Cellulose with SWNT solution.

10% wt added DMAc prevented the solution from being viscous enough to electrospin.

However, 5% wt DMAc added complicated the achievement of proper SWNT

dispersion. The final solution decided upon for its relative ease of production and

appropriate viscous quantities used 8% wt DMAc added to the solution. The resultant

cellulose with SWNT properties are listed in Table 1.

TABLE 1: Cellulose with SWNT Solution Properties

Compound	Weight Percentage
DMAc	90.71
LiCl	7.35
Cellulose	1.83
SWNTs	0.1

CHAPTER III

RESULTS

The attempt to produce nanofibers from the 2.5% wt cellulose solution via horizontal electrospinning without heating failed to produce a Taylor cone. The solution was too viscous to form a Taylor cone and any size fibers at room temperature despite the voltage being increased or any other change in the distance or solution flow rate. The following generation of setups relied on heating the solution in various ways to reduce the viscosity.

The preheated solution setup resulted in, at best, sporadic production of variable diameter fibers. The fibers ranged in diameter from approximately 20 micrometers up to 100 micrometers as shown in the optical microscope picture in Fig. 8.
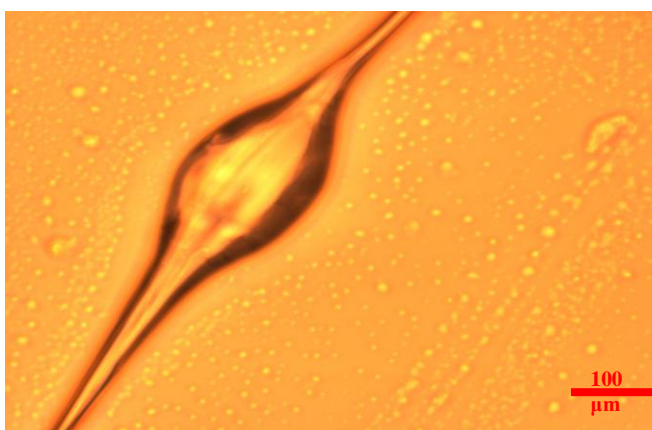

Figure 8. Varying Fiber Diameter from Setup A

The main cause of inconsistent Taylor cone formation and thus variable fiber diameter was the solution cooling in the syringe. Only at the highest allowable voltage by the power supply, 30 kV, did a Taylor cone form from the cooling solution for a few seconds (see Table 2 for optimal conditions). Several trials resulted in the conclusion that the varying temperature conditions of the solution prevented repeatable fiber production using this setup.

TABLE 2: Setup A Optimal Fiber Conditions

Condition	Value
Solution Preheated to	65 ^{o}C
Potential Difference between Needle and Target	30 kV
Distance between Needle and Target	10 cm
Solution Flow Rate	2 mL/hr
Needle Gauge	22

Setup B produced fibers with more consistent diameters as shown in Fig. 9. However, the fiber diameter still varied between approximately 5 and 20 micrometers, too large to produce nanofibers. When a thermoprobe measured the temperature of the solution in the needle, the problem of the large fibers was revealed to be a still relatively unstable Taylor cone formation caused by insufficient heating. The infrared radiation from the lamp was not being absorbed in high enough levels in the needle to heat the solution.

The heat lamp setup could not generate the temperatures within the needle to reduce the

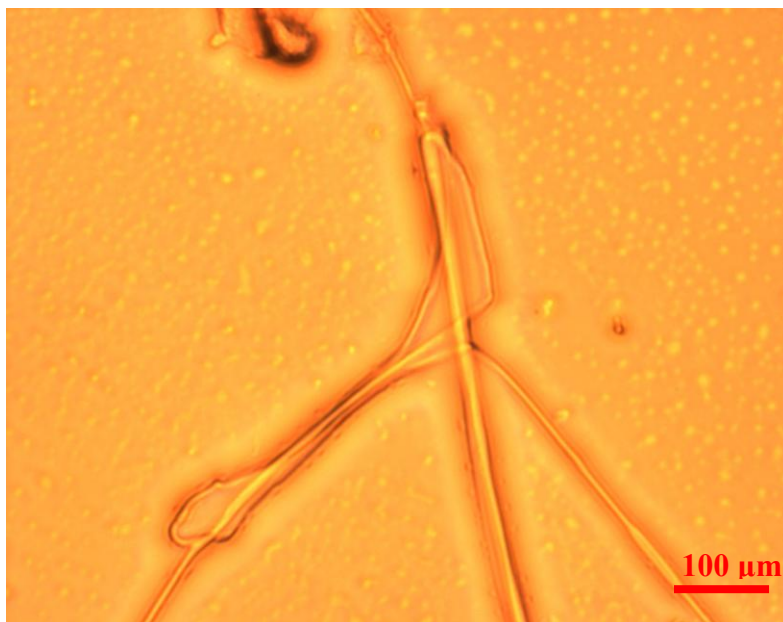viscosity to the necessary levels for stable Taylor cone formation.

Figure 9: Consistent Fiber Diameter from Setup B

Even at the highest recorded temperature of 35 $^{\circ}$C with the maximum voltage setting, the

viscous forces were still too strong to allow for the formation of a stable Taylor cone.

However, this setup still allowed for more control over the solution's concentration

because no DMAc could evaporate from the solution as it was being heated in the

syringe. The conditions used when the produced fibers had the most consistent and

smallest diameter are recorded in Table 3.

TABLE 3: Setup B Optimal Fiber Conditions

Condition	Value
Temperature Achieved in Needle	35 $^{\circ}$C
Potential Difference between Needle and Target	30 kV
Distance between Needle and Target	10 cm
Solution Flow Rate	2 mL/hr
Needle Gauge	22

Setup C, which used the heat gun to convectively heat the needle, combined the high

temperatures in the needle from Setup A with the continuous heating from Setup B. This

combination resulted in the reduction of solution viscosity while maintaining

consistency in the solution's solvent concentration. By maintaining better viscosity

levels than the other setups, more consistent Taylor cones were maintained for longer

time periods. The resultant fibers had consistent diameters that were less than 5

micrometers in diameter (as depicted in Fig. 10). Also, as seen in the images, there was

nearly no electrospraying, which causes small beads to appear in addition to the fibers.

Nearly all of the solution that came from the needle went directly into nanofiber
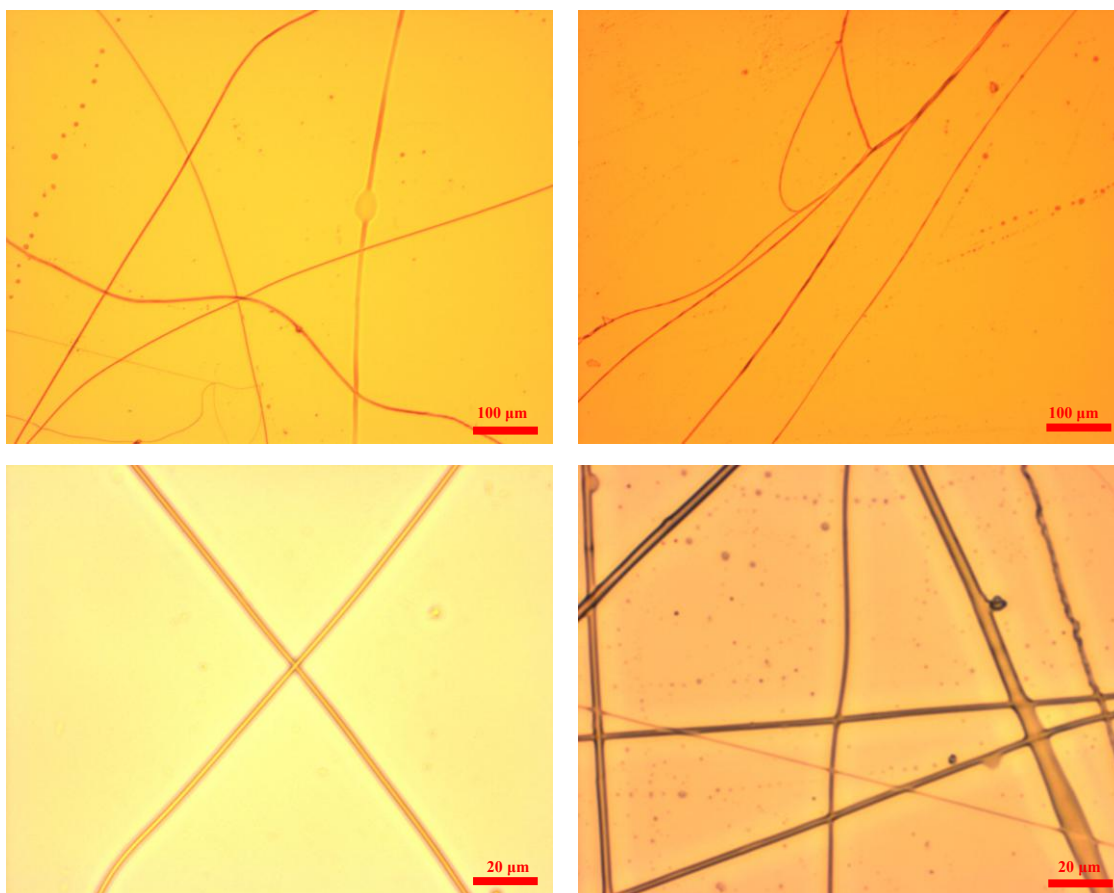
production.

Figure 10: Consistent Nanofiber Production from Setup C

The above optical microscope images show the nanofibers produced using the heat gun/heat sink setup. The presence of fibers less than 1 micrometer in diameter show that the setup effectively blocked the fiber production from the high airspeeds associated with the hot air from the heat gun. Even though some of the fibers appeared watery with somewhat ill-defined borders, their diameters remained relatively consistent. The relatively small of electrospraying and large fiber production establishes this setup as a reliable and reproducible method for continuous nanofiber production. The lower

viscous effects in the cellulose solution due to more heating also allowed the power

source to be turned down to lower voltages, decreasing the power requirements for the

experiment to be run (as seen in Table 4). A slightly larger diameter (smaller gauge)

needle was used to assist in the production of a larger Taylor cone that was less

susceptible to inconsistencies in the solution's homogeneity.

TABLE 4: Setup C Optimal Fiber Conditions

Condition	Value
Temperature of Airflow from Heat Gun	100 $^{\circ}$C
Temperature of Airflow around Heat Sink/Needle	70 $^{\circ}$C
Temperature Achieved in Needle	42 $^{\circ}$C
Potential Difference between Needle and Target	18 kV
Distance between Needle and Target	10 cm
Solution Flow Rate	2 mL/hr
Needle Gauge	20

The Cellulose with SWNT solution presented several problems with CNT dispersion in

solution production. As seen in the initial dispersion of Figure 11, the larger CNT

agglomerates, which had dimensions as large as 100 micrometers, originally interfered

with the production of homogeneous fibers from the Cellulose with SWNT solution.
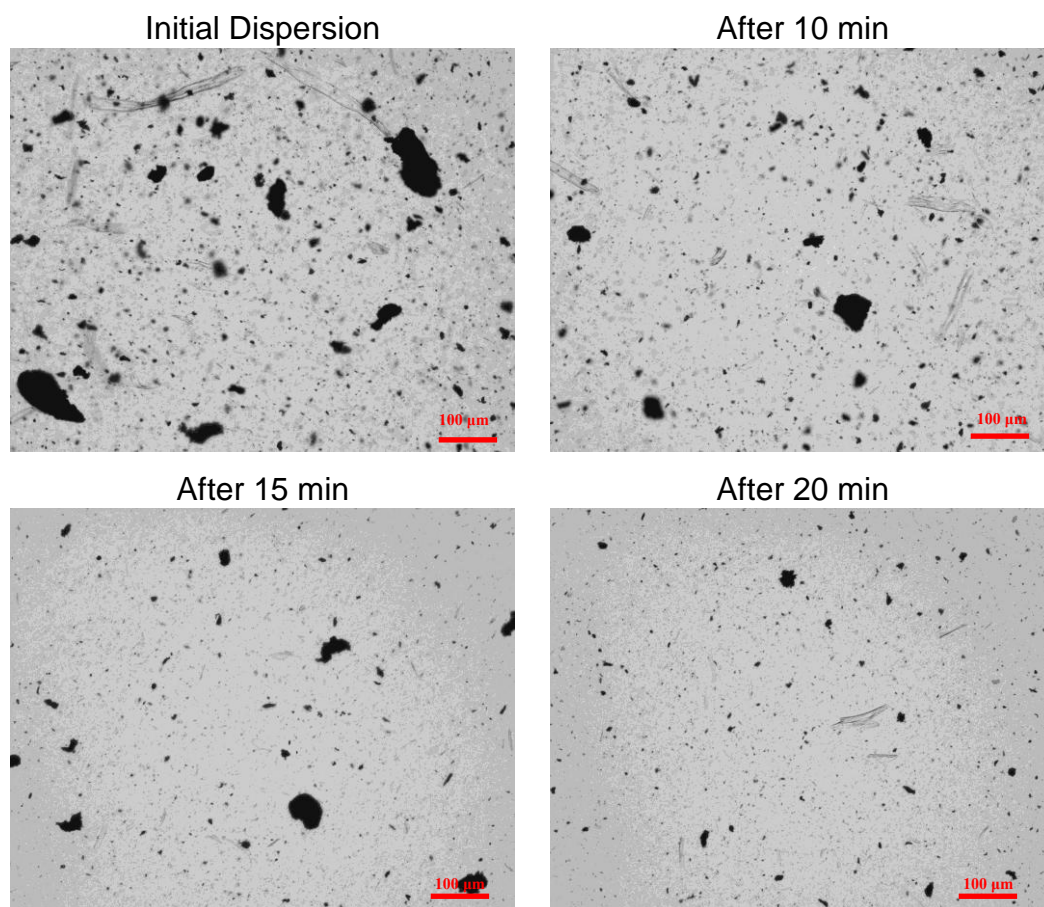
Figure 11: Dispersion of CNT in Cellulose with SWNT Solution via Sonication

Large beads were interspersed along the length of the fibers. It was theorized that these beads were caused by both electrospraying as well as some of the larger CNT agglomerates passing through the Taylor cone into the fibers. To reduce the agglomerate size and impact on fiber production a sonication horn was used on the Cellulose with SWNT solution just before electrospinning the solution. After 20 minutes of sonication, the maximum agglomerate size was reduced to less than 5 micrometers in diameter, as seen in Fig. 11, greatly reducing most of the beads' size.
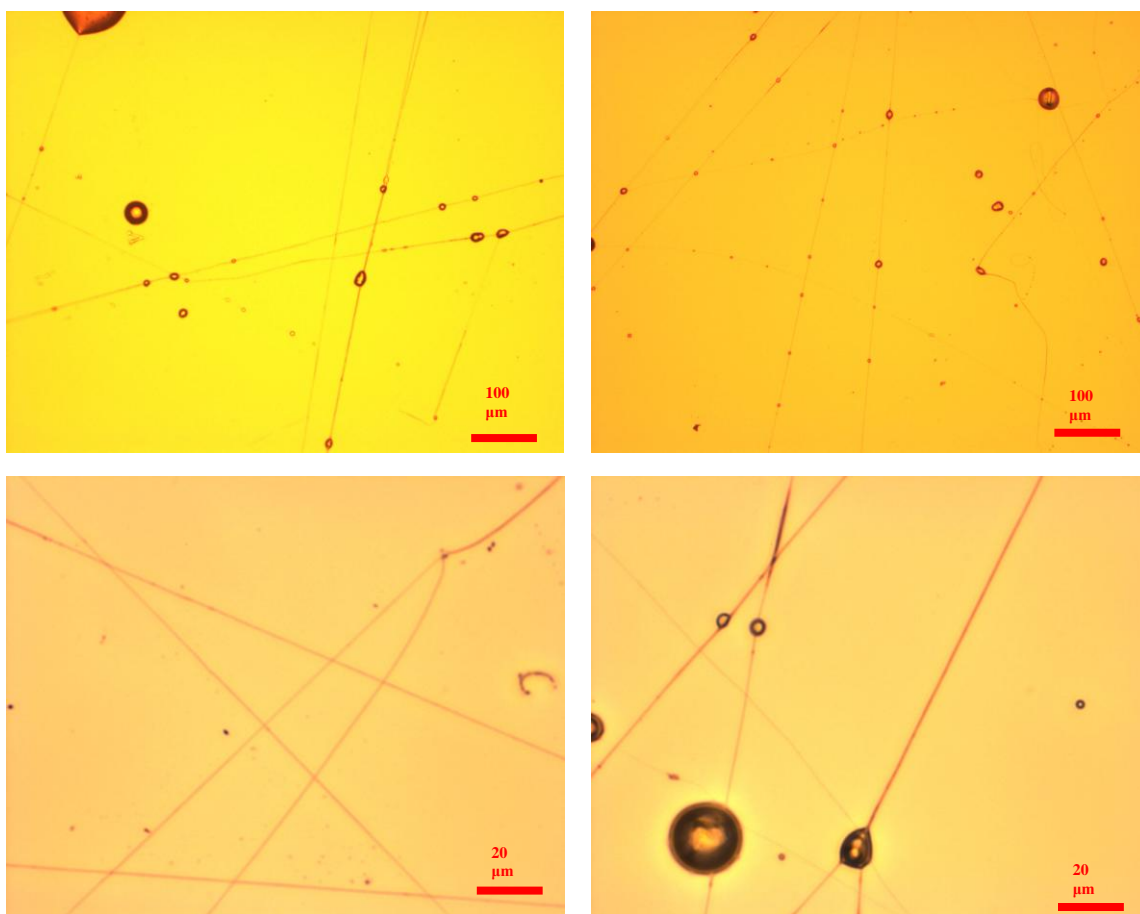
Figure 12: Cellulose with SWNT Nanofibers

When the sonicated Cellulose with SWNT solution was electrospun, the resultant fibers

still had some beads interspersed along their lengths, as seen in Fig. 12. However, the

beads were relatively uncommon and small in diameter. Also, the average fiber diameter

was less than 2 micrometers and remained remarkably consistent between all of the

fibers. The Cellulose with SWNT solution was much less viscous than the original

cellulose solution. Resultantly, the required voltage was significantly lower, the flow

rate was decreased and a smaller diameter (larger gauge needle) were used to obtain the

optimal fiber conditions listed in Table 5.

Table 5: Cellulose with SWNT Optimal Fiber Conditions

Condition	Value
Room Temperature	22.6oC
Potential Difference between Needle and Target	11 kV
Distance between Needle and Target	10 cm
Solution Flow Rate	1 mL/hr
Needle Gauge	25

CHAPTER IV

SUMMARY AND CONCLUSIONS

Summary

The 2.5% wt cellulose solution could not be electrospun at room conditions because of its high viscosity. So, three different setups were used to heat the solution, thereby lowering its viscosity.

The first setup, Setup A, used a hot plate to preheat the solution before it was loaded into the syringe to be electrospun. The fibers produced using Setup A had inconsistent diameters because the solution cooled quickly from the initial temperature and became viscous again. The cooling solution didn't allow for the establishment of a stable Taylor Cone. Accordingly the fibers ranged from a few micrometers to about 100 micrometers.

The second setup, Setup B, used a heat lamp setup to radiatively heat the solution while it was in the syringe. This heating method kept the solution a constant, elevated temperature. However, the needle did not absorb enough heat to achieve a temperature high enough to significantly reduce the solution's viscosity, resulting in the formation of a Taylor cone for only a few seconds at a time. The fiber diameter was more consistent, but the Taylor Cone did not form long enough to produce many fibers. The resultant fiber diameter ranged from approximately 5 micrometers to 20 micrometers.

The final setup, Setup C, used a heat gun blowing on the heat sink attached to the syringe to keep the solution in the syringe at a constant temperature, high enough to maintain a Taylor Cone and produce many uniform fibers with consistent diameters. The resultant average fiber diameter was less than 5 micrometers.

The 2% wt Cellulose with SWNT was electrospun after sonicating for 20 minutes to increase SWNT dispersion (for solution concentration see Table 1). The solution was electrospun at room temperature and the parameters were altered until a Taylor Cone formed, producing uniform fibers with diameters consistently less than 2 micrometers.

Conclusions

Through comparing average fiber diameter and homogeneity as well as parameter reproducibility the optimal setup was determined. The heated cellulose heat gun / heat sink setup and the cellulose with SWNT setup resulted in the best reproducible conditions for fiber diameter less than 5 micrometers with minimal electrospraying. The tested range and optimal settings for the parameters in these setups can be found in Table 6. The fibers from these setups will be aligned into mats and mechanically tested in future experiments.

Table 6: Optimal Setup and Parameter Values for Both Solutions

2.5% wt Cellulose Solution (Setup C)

Parameter	Tested Range	Optimal Value
Distance	4→13 cm	10 cm
Voltage	10→30 kV	18 kV
Solution Flow Rate	0.5→3 mL/hr	2 mL/hr
Temperature (Heated)	20→45 ^{o}C	45 ^{o}C

2% wt Cellulose with SWNT Solution

Parameter	Tested Range	Optimal Condition
Distance	7→10 cm	10 cm
Voltage	5→25 kV	11 kV
Solution Flow Rate	0.5→3 mL/hr	1 mL/hr
Temperature (Room)	21→23 ^{o}C	22 ^{o}C

REFERENCES

1. "Cellulose" *Columbia Encyclopedia.* Sixth Edition. (New York, Columbia University Press, 2007)

2. J. Kim and S. Yun, Z. Ounaies. *Macromolecules,* **39**,12 (2006).

3. E.-H. Lee, H.-M. Kim, K.-S. Kim, and I.-J. Chin. *Preparation of Electrospun Cellulose Membrane for EAPap Actuator.* Manuscript in preparation, Inha University (2008).

4. J. Kim and Y. B Seo. *Smart Materials and Structures.* **11,** 355 (2002).

5. Y. M. Shin, M. M. Hohman, M.P. Brenner, and G.C. Rutledge. *Polymer.* **42**, 9955 (2001).

6. R. Samatham, and K. J. Kim. *Polymer Engineering Science.* **46**, 954 *(2006)*

7. A.L Yarin, S. Koombhongse and D.H. Reneker. *Journal of Applied Physics.***90**, 4836 (2001).

8. C.-W. Kim, D.-S. Kim, S.-Y. Kang, M. Marquez, and Y. L. Joo. *Polymer.* **47**, 5097 (2006)

9. D. H. Reneker and I. Chun. *Nanotechnology.* **7**, 216 (1996).

10. H.Q. Liu, and Y.-L. Hsieh. *Journal of Polymer Science.* **40**, 2119. (2002)

11. E. Bianchi, A. Ciferri, G. Conio, A. Cosani, and M. Terbojevich. *Macromolecules.* **18**, 646 (1985)

12. C.-W. Kim, M. W. Frey, M. Marquez, and Y. L. Joo. *Journal of Polymer Science Part B: Polymer Pysics.* **43**, 1673. (2005).

13. Z.-M. Huang, Y.-Z. Zhang, M. Kotaki, and S. Ramakrishna. *Composites Science and Technology.* **63**, 2223 (2003).

14. E. Kymakis and G. A.J. Amaratunga. *Journal of Applied Phyiscs*, **99**, 084302 (2006)

15. Y. Dror, W. Salalha, R. L. Khalfin, Y. Coehn, A. L. Yarin, and E. Zussman. *Langmuir.* **19**, 7012 (2003)

16. J. N. Coleman, U. Khan, W. J. Blau, and Y. K. Gun'ko. *Carbon*. **44**, 1624 (2006).

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