

Effects of different parameters on the morphology of electrospun cellulose acetate/polyaniline nanocomposite

JEANETTE O. GRANDE-FLORES, BILJANA M. BUJANOVIC, AND GARY M. SCOTT

ABSTRACT: Cellulose is the most abundant naturally occurring polysaccharide. Its inherent mechanical stability, biocompatibility, biodegradability, and abundant hydroxyl groups available for derivatization provide benefits in the production of fiber-based materials such as conductive fibers. Cellulose derivatives, including cellulose esters such as cellulose acetate (CA), are readily generated in the fiber form following dissolution using a variety of solvents.

Electrospinning is one of the emerging technologies with an outstanding ability to regenerate fibers in the nanoscale range. Polyaniline (PANI) is a conductive polymer that is popular for its high chemical stability, nontoxicity, good processability, and stable intrinsic redox state.

This study explored the fabrication of a conductive PANI/CA nanocomposite through electrospinning. Working conditions, electrospinning variables, and solution parameters were established to produce coherent PANI/CA nanofibers. The effects of varying CA concentration, amount of PANI, molecular weight of CA, and feed flowrate on the morphology of the nanofibers were investigated.

Application: This paper can be useful to other researchers who are interested in the nanotechnology applications of cellulose. It can also be beneficial for industrial manufacturing engineers who utilize electrospinning for fabricating conductive nanothreads and similar applications.

Novel functional materials have attracted interest in recent studies, as they are engineered to combine material properties to perform specific functions. Advancements in technology coupled with increasing customer needs lead to demands in flexible electronic devices; hence, the birth of fiber-based electronic devices, also known as electronic textiles [1]. Depending on their electrical conductivity, there is a wide variety of applications for electronic textiles, such as special clothing, antistatic purposes, and electrostatic [2-5].

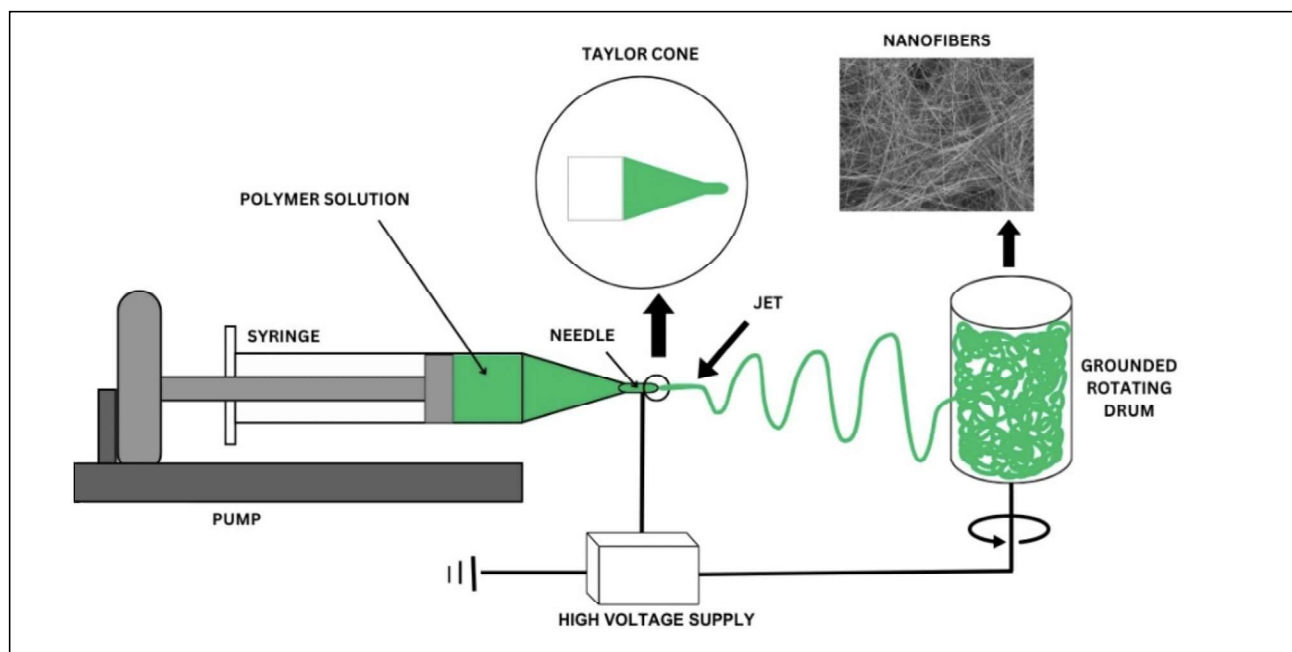
Fabrication at nanoscale dimensions is one way to engineer a material with desirable properties. Electrospinning is one of the newest technologies in producing nanoscale fibers by passing a polymer solution, either in dissolved or melt form, through a syringe to a metallic plate by means of electrical potential difference [6]. As the solution traverses the distance from the needle to the plate, the material stretches, gradually solidifies, and separates into superfine fibers. **Figure 1** illustrates an electrospinning system that was adapted and modified from [7].

Electrospinning can be used to produce nanocomposites, which are materials made from at least two nanosized components combined by promoting their interfacial interactions to achieve a balance of their individual beneficial properties. Polyaniline (PANI) is a conductive polymer that can be processed through electrospinning with a fibrous matrix. PANI is a popular antistatic coating because

of its high chemical stability, nontoxicity, good processability, and stable intrinsic redox state [8,9].

Cellulose, hemicelluloses, and lignin are the main chemical constituents that compose plant fibers. Among these, cellulose, a homopolysaccharide composed of β -D-glucopyranose (β -D-Glcp) units linked together by (1 $\rightarrow$ 4) glycosidic bonds, is the structural constituent responsible for the fibrous nature of the cell walls [10]. Cellulose has desirable inherent characteristics such as mechanical stability, biocompatibility, and biodegradability. However, a strong hydrogen bonding network renders cellulose insoluble in common solvents and obstructs melting at high temperatures. As a result, electrospinning cellulose presents significant challenges. An alternative approach is provided by cellulose acetate (CA), a class of cellulose esters that is soluble in a range of solvents, depending on its degree of substitution (DS), where DS = 3 designates the complete substitution of the hydroxyl groups on β -D-Glcp units. The bulky acetyl groups affect different properties of CA such as permeability, viscosity, and crystallinity. Hence, DS is also a key factor in electrospinning [11]. Electrospun CA nanofibers show a high surface area-to-volume ratio, good flexibility, high porosity, and excellent mechanical properties. In addition, CA offers other benefits, including low-cost synthesis, good biocompatibility, and biodegradability [12].

Using electrospinning, nanocomposites of CA and PANI can be fabricated. These nanocomposites can display both



1. Illustration of electrospinning system (adapted and modified from [7]).

the characteristics of CA and PANI and can be potentially used for applications involving outstanding electrical properties that require good biocompatibility and excellent mechanical properties. Prospective applications include sensors and actuators, energy harvesting devices, optoelectronics, drug delivery, tissue engineering, and nerve regeneration [13,14].

This study explored the fabrication of a conductive nanocomposite from PANI and CA through electrospinning. Specifically, the objectives of the study were to:

1. Establish the working conditions and parameters (e.g., voltage-to-gap ratio and humidity) for the electrospinning of PANI and CA solution to produce coherent PANI/CA nanofibers.
2. Investigate the effect of varying the concentration of CA on the morphology of the nanofibers.
3. Determine the maximum amount of PANI that can be electrospun with the CA polymer solution.
4. Determine the effect of varying the concentration of PANI on the morphology of the nanofibers.
5. Determine the effect of molecular weight on the morphology of the nanofibers.
6. Determine the effect of varying the electrospinning feed flow rate on the morphology of the nanofibers.

MATERIALS AND METHODS

Preparation of PANI/CA solutions

Two types of CA characterized by different molecular weights (MW), as determined by gel permeation chromatography (GPC), were purchased from Sigma Aldrich (St. Louis, MO, USA). One CA had a number average molecular weight (M_n) $\approx$ 30 kDa (CA1) and another had M_n $\approx$ 50 kDa (CA2), with the two CA having a comparable degree

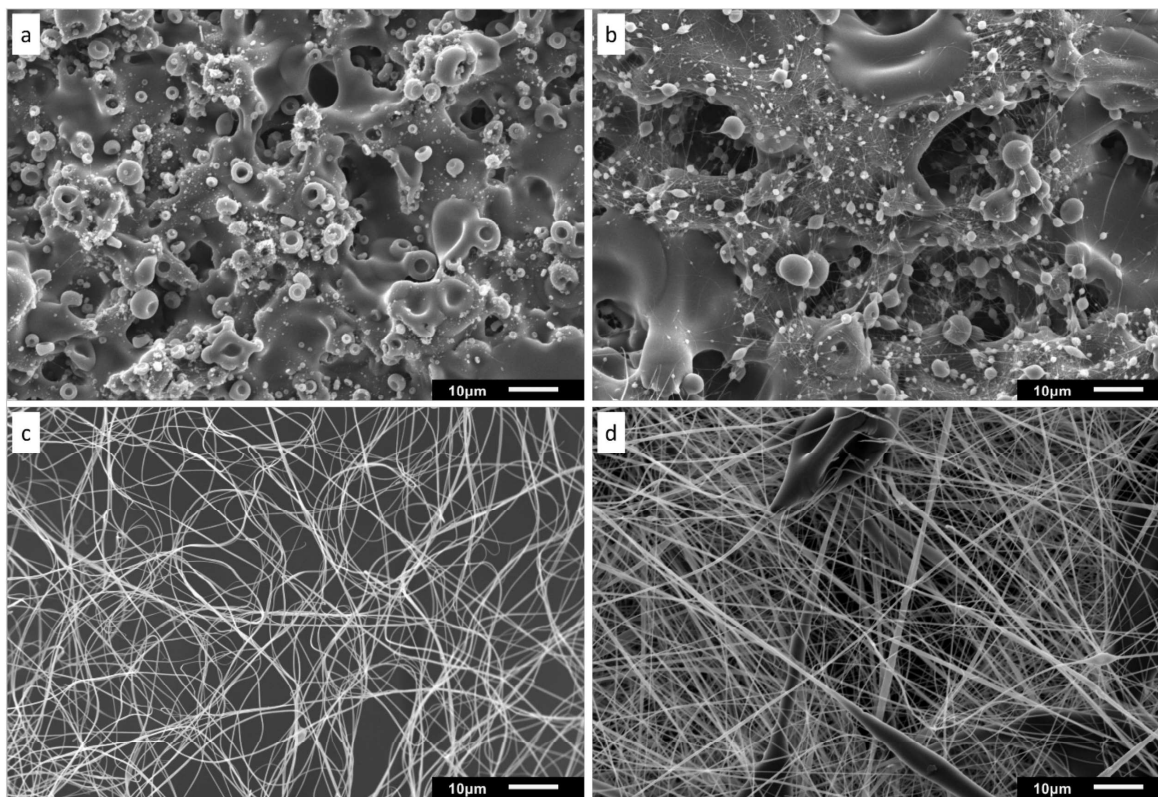
of acetylation at 39.8% and 39.7%, respectively. The CA was dissolved in dimethyl formamide (DMF)/acetone (2:1 v/v) to prepare 25% wt/v of CA-DMF/acetone. To this, varying amounts of PANI were added to produce different concentrations of PANI in CA (%wt): (a) 0.0% (pure CA), (b) 0.1%, and (c) 0.4%. Preliminary experiments were done to determine the relative amount of DMF and acetone, as well as the concentration of CA and amount of PANI to obtain coherent fibers at working conditions.

Electrospinning

The solution flow rate was adjusted at three different levels of 1 mL/h, 2 mL/h, and 4 mL/h. A total volume of 7.5 mL of the solution was electrospun using 22-gauge needles at a gap of 12 cm. The voltage was left constant at 12 kV (1 kV/cm). Each run was duplicated. Preliminary runs were conducted to determine the boundaries and conditions of the voltage-to-gap ratio, flow rate, and volume of the polymer solution to be electrospun. Relative humidity (RH) and temperature in the containment were kept at 50% RH and 24°C, respectively. The air velocity was controlled and remained the same. The rotation of the drum collector was set at 1000 rpm; translation was retained constant.

Analysis

The produced nanocomposite was subjected to scanning electron microscopy (SEM) to characterize the morphology of the resulting PANI/CA nanofibers. The SEM used was JEOL JSM-IT100LA (JEOL; Tokyo). ImageJ software was used to measure the diameter of the fibers. Two SEM images were taken for each product in the experimental runs, and 20 fibers on each image were measured.



2. Scanning electron microscopy (SEM) images of cellulose acetate 1 (CA1; ~30 kDa) electrospun solutions with 0.1% polyaniline (PANI) in 2:1 dimethyl formamide (DMF)/acetone at different concentrations of CA1: (a) 15%, (b) 20%, (c) 25%, and (d) 27.5%.

RESULTS AND DISCUSSION

Preliminary optimization

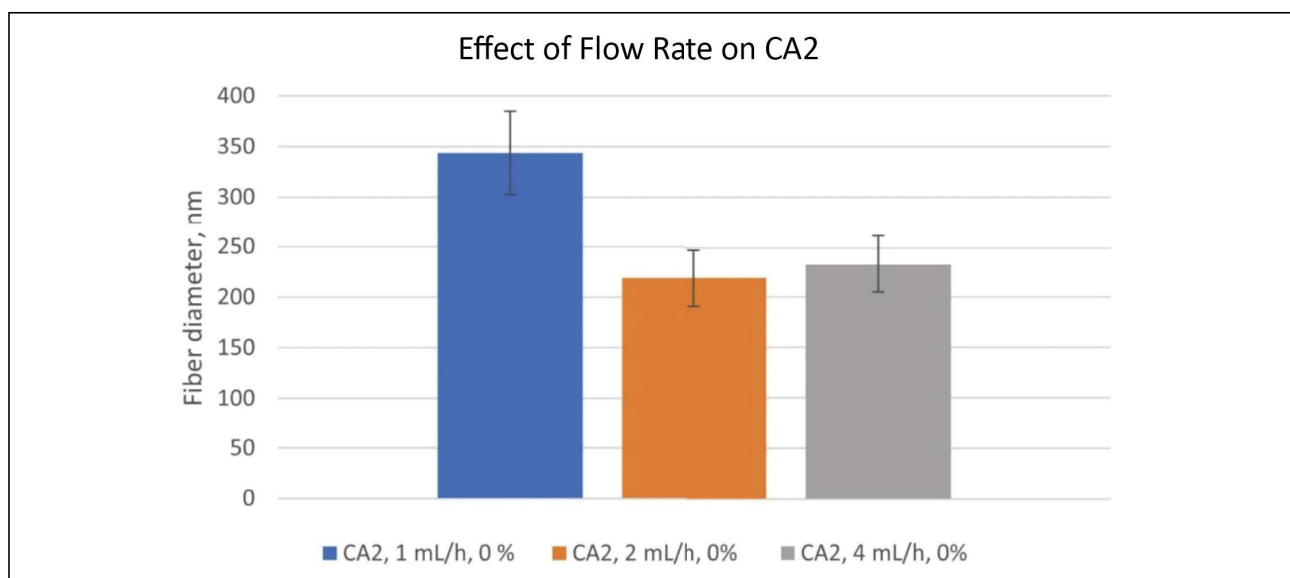
Electrospinning is a cost- and time-efficient technique for producing superfine fibers. However, various parameters need to be optimized first, including process (i.e., voltage, distance); ambient (i.e., temperature, humidity, air velocity); and solution (i.e., viscosity, conductivity, surface tension) [15,16]. The proper combination of these parameters determines the success of the electrospinning in producing superfine, smooth, and beadless nanofibers.

Effects of concentration of CA

The initial base polymer solution was electrospun at 10% CA (wt/v); specifically, CA1 of lower MW of ~30 kDa in 3:1 DMF/acetone (v/v). This solution was not sufficiently viscous to produce continuous fibers; hence, moist powders were collected on the metal plate. A critical concentration, above which polymer entanglement occurs, is the minimum needed concentration for the polymer fibers to be electrospun [17]. Viscosity is an important parameter, because it reflects the surface tension present on the Taylor cone. The Taylor cone refers to the stabilized hemispherical liquid drop suspended at the end of the needle (Fig. 1). Once the force provided by the potential difference exceeds the critical voltage (V_c) that balances the surface ten-

sion, the polymer solution starts to be ejected from the Taylor cone. However, in the case of low viscosity developed by the initial 10% CA1 in 3:1 DMF/acetone (v/v), the jet breaks up into droplets, resulting in electrospraying rather than spinning [18]. Hence, powders were collected, and no continuous fibers were formed. The concentration was increased to achieve the requisite viscosity. Increasing the concentration was necessary to reach the critical concentration where chain entanglement is sufficient to stabilize the jet and to form fibers. Additionally, the relative amount of DMF was decreased, increasing the fraction and evaporation rate of the more volatile acetone as the polymer solution traverses the distance between the needle spinneret and the collector. These adjustments facilitated the production of dry superfine fibers.

Figure 2 shows the SEM images of the electrospun solutions of different concentrations of CA1 with 0.1% PANI in 2:1 DMF/acetone (v/v). Increasing the polymer concentration affects the fiber morphology, and it follows the progression: (a) 15% CA: beads only; (b) 20% CA: beads with incipient fibers; (c) 25% CA: fibers only; and (d) 27.5% CA: globular fibers/macrobeads. A similar trend has been reported by Shenoy et al. [19] in describing the role of chain entanglement on the fiber formation during the electrospinning process; however, the “beaded fibers” morphol-



3. Effect of flow rates on the fiber diameters (CA2: cellulose acetate 2; ~50kDa).

ogy was detected between the (b) and (c) progression steps. This observation of fiber morphology is attributed to the competing effects of chain entanglement that stabilize the jet to form continuous fibers vs. the surface charge that overcomes the surface tension, furthering droplet subdivision. At lower concentrations (in the case of CA with 0.1% PANI <25%), the surface charge effect dominates, causing the droplet breakup, and hence, forming beads. As the polymer concentration is increased, chain entanglement becomes more and more sufficient to stabilize the electrospinning jet, preventing the droplet breakup. Further increase in the polymer concentration (in the case of CA with 0.1% PANI >25%) produces nonuniform fibers with larger diameters and microbead, because the electric field caused by the potential difference is not enough to stretch the jet consistently.

Effects of PANI concentration

Aside from polymer concentration of the solution, another important parameter in electrospinning is the solution conductivity. Polyaniline was selected for this study because it is one of the most popular conductive polymers due to its good processability, nontoxicity, and ease of synthesis. The conductivity of the PANI used in this study is 2–4 S/cm (as labeled by Sigma Aldrich). The initially proposed variation in the concentration of PANI with respect to CA was 0.0% w/w, 0.1% w/w, 0.5% w/w, and 1.0% w/w. However, preliminary experiments showed that electrospinning CA with as high concentration as 1.0% w/w caused tremendous splattering, with no fibers deposited in the collecting drum. Even at the PANI concentration of 0.5% w/w, occasional splattering still occurred, causing unstable electrospinning. Hence, the final concentration was lowered to 0.4% w/w of PANI/CA.

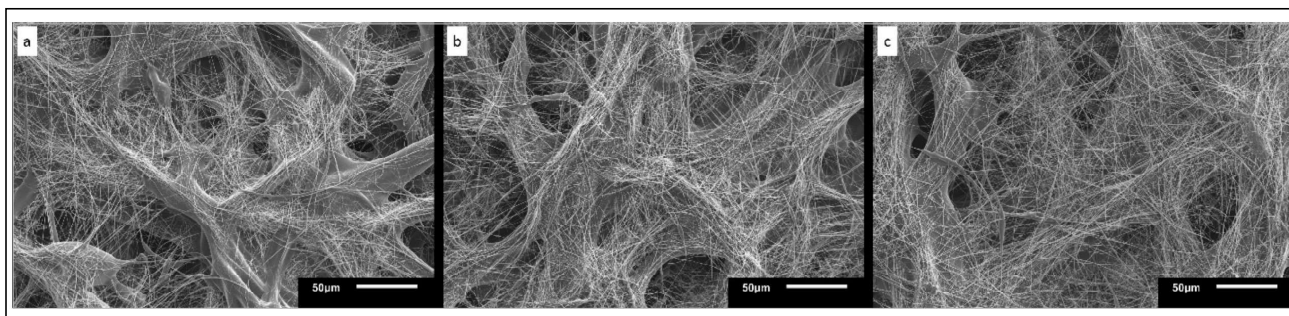
Experiment proper

Effects of flow rate

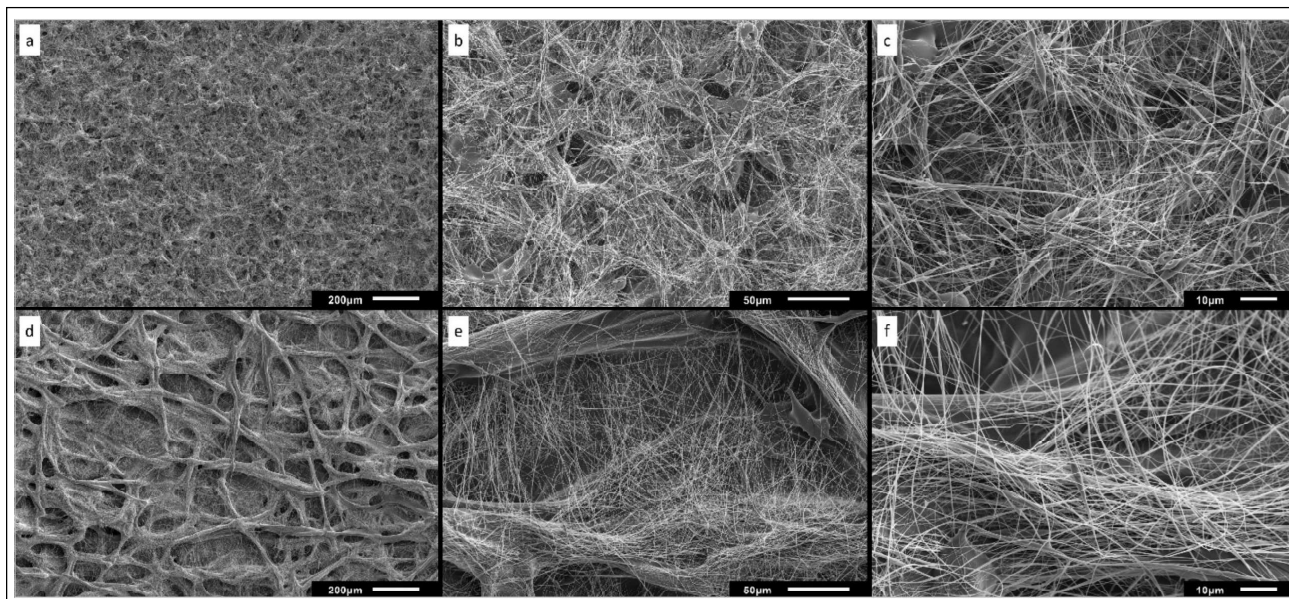
There is no observable difference in the morphology of the fibers collected from electrospinning CA with a high MW (CA2, Mn ~50 kDa) at different flow rates. The CA solution yields to non-nanosized diameters at the given conditions and parameters (**Fig. 3**). In this case, the viscosity was too high for the flow rate effects to become apparent. Electrospinning at a higher flow rate leads to the formation of beads and clusters as much as electrospinning the polymer solution above the critical concentration [20]. It was revealed that in the case of higher molecular weight, the electric field was lower than needed to produce beadless fibers from the very viscous solution at the polymer concentration of 25% wt/v. Surface tension exceeded the electric field, preventing the ejection of smooth, continuous fibers. Local accumulation at the tip of the needle spinneret occurs while the solvent evaporates, causing the deposition of large clusters. The smaller-diameter fibers were produced in between the ejection of these clusters (**Fig. 4**). To eliminate these problems, several factors or combinations of these factors can be adjusted such as: (a) the MW of CA (↓); (b) the concentration of CA (↓); (c) the electric field (↑); and/or (d) the flow rate (↓).

Effects of molecular weight

The viscosity of the solution is affected by both the MW and concentration of the polymer. These parameters influence the chain entanglement that is critical during the electrospinning process [19]. Chain entanglement provides the necessary surface tension that balances the critical voltage for stable and uniform ejection of the jet from the Taylor cone (Fig. 1). As previously mentioned, the too dilute solution leads to the breakup of the jet into droplets, while the too viscous solution leads to large diameter fibers with microbeads.



4. SEM fibers (25% w/v CA2) produced by electrospinning at different flowrates: (a) 1 mL/h, (b) 2 mL/h, and (c) 4 mL/h.

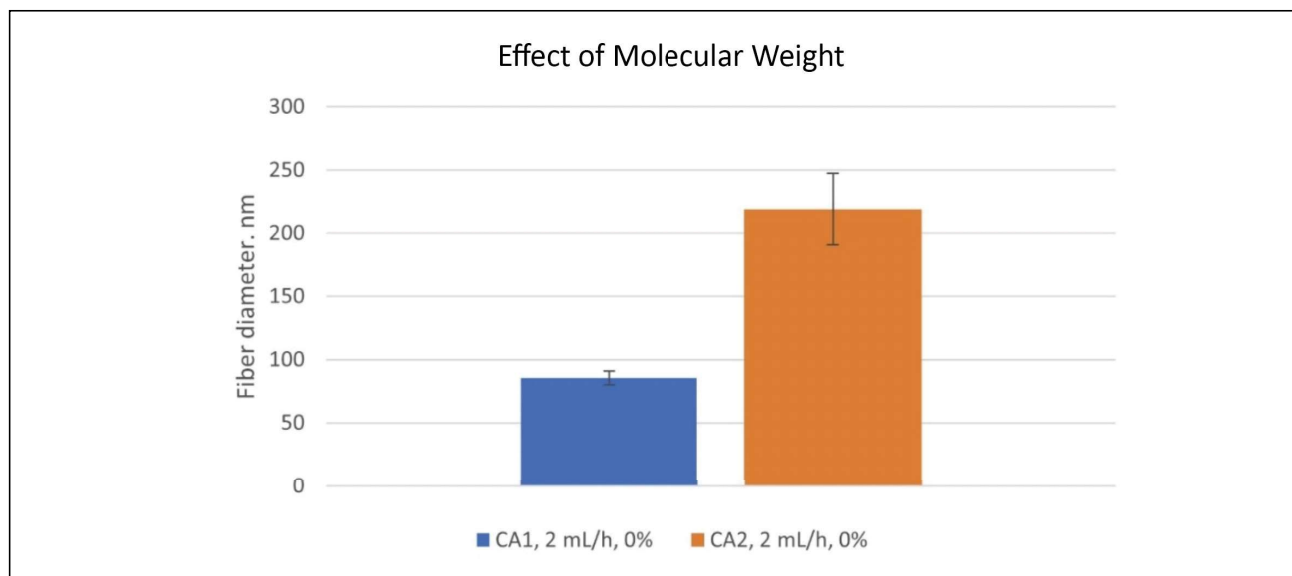


5. SEM images of electrospun 25% wt/v CA, 0% PANI, 2 mL/h at different magnifications: (a-c) CA1 (~30 kDa), and (d-f) CA2 (~50 kDa).

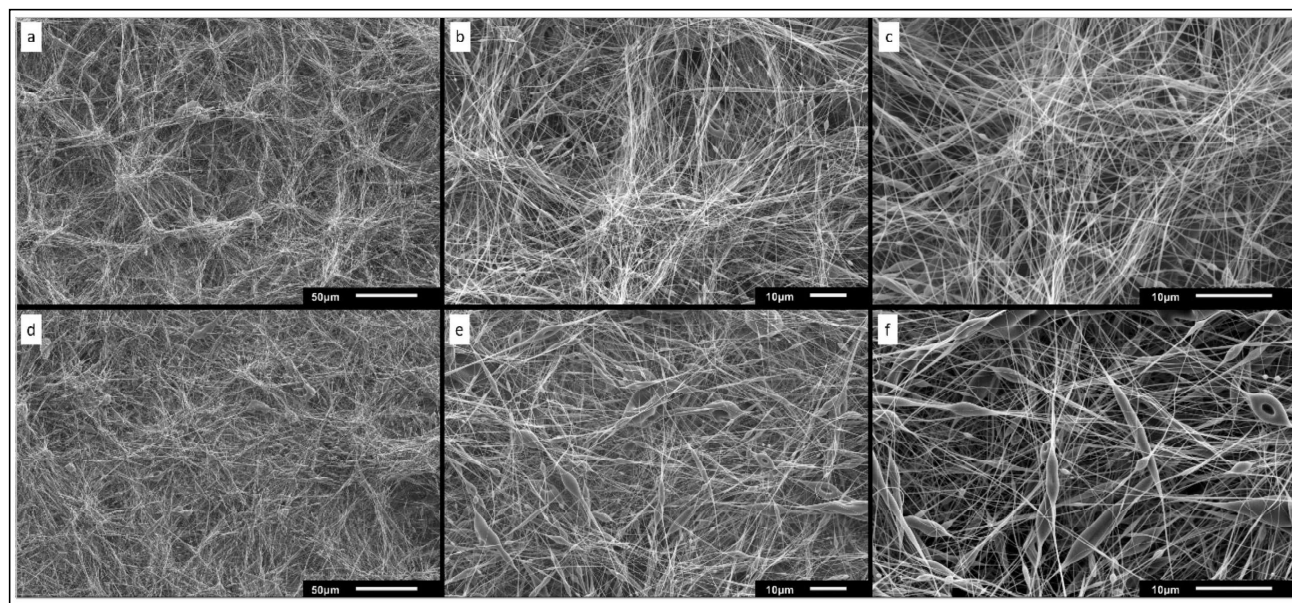
Figure 5 shows the fibers collected from electrospinning two different CAs, i.e., CA1 and CA2 with MW of ~30 kDa and ~50 kDa, respectively. These solutions were electrospun under the same conditions and were viewed under various SEM magnifications. During the preliminary experiment, the electrospinning executed using 25% w/v CA1 with 0.1% PANI at 15 kV yielded beadless and smooth nanofibers. On the other hand, electrospinning this CA1 sample with the same concentration but with 0% PANI and 12 kV resulted in nanofibers with microbeads and infrequent microclusters of the solution. Therefore, the appearance of the microbeads could be attributed to two possible factors: (1) conductivity effects, and (2) voltage and surface tension balancing constraints. Conductive solutions are generally preferred in electrospinning, because more charges are at the surface of the jet to react to the electric field, stretching the solution to prevent the formation of beads. Since there was no PANI in this sample, the lesser conductivity might explain the appearance of beads relative to the run from the preliminary experiment. Voltage and surface tension balancing constraints could also have affected the electrospinning. A higher voltage was required to eject smooth,

continuous fibers; the surface tension was greater than the electric field created by the 12 kV potential difference [21].

The electrospun CA2 polymer solution contained fibers of different sizes; both large diameter fibers and relatively small diameter fibers were produced (Fig. 5d–5f). This suggests an instability and non-uniform ejection of the polymer solution from the tip of the spinneret. Chain entanglement is too strong and the solution is too viscous for the electric field to overcome. During electrospinning, a large suspending droplet of Taylor cone is formed at the tip of the spinneret (Fig. 1). When the cone is large or heavy enough, it stretches, depositing large diameter fibers on the fiber collector. Smaller diameter fibers are generated during the separation of the large droplet of the solution before the next Taylor cone is formed to start a new cycle of the thick fiber deposition. **Figure 6** shows the effect of the molecular weight on the diameter of the electrospun fibers. Fibers electrospun from CA1 and CA2 had an average diameter of 86 ± 24 nm and 219 ± 127 nm, respectively. The average fiber diameter electrospun from CA1 was within the range of 1 to 100 nm, which is consistent with “nanoscale” as defined by the European Commission [22],



6. Effect of molecular weight of CA on the fiber diameter of electrospun CA solutions. Electrospinning was performed at the same conditions and parameters.



7. SEM images of electrospun 25% CA1 (~30 kDa) with: (a-c) 0.1% PANI, and (d-f) 0.4% PANI at 1 mL/h under different magnifications.

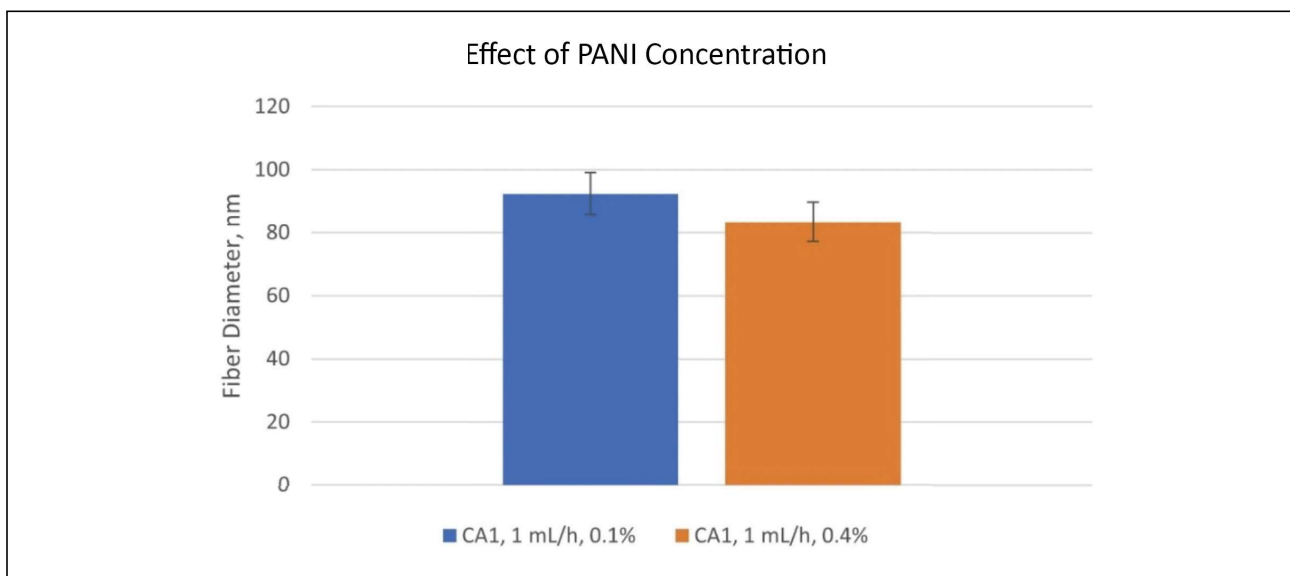
while the fibers electrospun from CA2 were not within that range. The chain entanglement in the CA2 solution is too strong for the electric field to overcome, resulting in large diameter fibers.

Some of the parameters that might resolve the inconsistency and the large diameter of the ejected fibers are: (1) voltage, and (2) polymer concentration. Increasing the applied voltage will provide a stronger electric field that can eject the jet from the Taylor cone in a spontaneous manner, in contrast to the formation of a large droplet suspended at the tip before ejection. With the decrease in the polymer concentration, the viscosity and surface tension of the solution are reduced, facilitating a continuous ejection of the jet.

Effects of PANI concentration

Figure 7 shows SEM micrographs of the CA1 fibers with different PANI concentrations that were electrospun under the same conditions. The PANI macroparticles were not noticed scattered across the fiber mats, indicating that PANI was completely embedded in the CA nanofibers. The PANI was uniformly dispersed by the solvent DMF/acetone in the polymer solution. The PANI nanoparticles were completely integrated in the CA, instead of just being mixed within the fiber mats [23].

The addition of PANI governed the success of the electrospinning process conducted at the same conditions. It was observed that polymer solutions without PANI did not



8. Fiber diameter of electrospun CA1 (~30 kDa) solutions with different PANI concentrations.

produce fibers. By contrast, polymer solutions with 0.4% PANI produced fibers, but they were heterogenous in size and contained elongated beads. Only polymer solutions with 0.1% PANI resulted in nanofibers with very small beads. An increase in the concentration of the conductive polymer in the solution increases the charge density carried by the jet and promotes stretching under a strong electric field. This helps in the production of smooth and beadless nanofibers [24]. However, it appears that further increases in the concentration resulted in the aggregation of the PANI, which caused the instability of the jet leading to the production of elongated beads. The electric field was not strong enough to overcome the cohesion within the polymer solution. **Figure 8** shows that there are no significant differences in the effect of increasing the concentration from 0.1% to 0.4% on the diameter of the electrospun nanofibers.

CONCLUSION

A PANI/CA nanocomposite was successfully fabricated using electrospinning. Electrospinning is an efficient technique for producing superfine fibers; however, the processing parameters and working conditions must first be established. Sufficiently viscous polymer solution was necessary to obtain smooth and continuous unbeaded fibers. A polymer solution that was too dilute led to the breakup of droplets because surface charge effects dominate, while a solution that was too concentrated resulted in nonuniform fibers with larger diameters and microbead because surface tension effects dominate.

There were no observable differences in the morphology of the fibers collected from electrospinning CA2 (MW=~50 kDa) at different flow rates. In this case, the viscosity was too high that flow rate effects were not apparent. Molecular weight affects chain entanglement, which directly influences the fluid flow dynamics of the jet. Electro-

spun CA1 resulted in nanofibers with microbeads and infrequent microclusters of the solution. Electrospun CA2 polymer solution had both very large diameter fibers and relatively smaller diameter fibers, suggesting growing instability caused by excessive chain entanglement.

The PANI was completely embedded in the CA nanofibers. An increase in the concentration of the conductive polymer in the solution increased the charge density, promoting stretching and facilitating the formation of smooth, beadless nanofibers. However, too high concentration of PANI caused aggregation that contributes to the instability of the jet, causing elongated beads to occur. A very high concentration of PANI resulted in significant splattering. **TJ**

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DECLARATION

This article is drafted based on part of the thesis manuscript [25] of Jeanette Grande as a partial fulfillment of the requirements for her MSc. in Paper Science and Engineering at SUNY-ESF.

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ABOUT THE AUTHORS

I (Grande Flores) pursued this research because of a keen interest in wood chemistry, nanotechnology, and development of smart materials. My bachelor's degree research in the Philippines was also related to this subject, but I had limited access to equipment, tests, and parameters at that time. When I was granted a Fulbright scholarship for my M.S. degree in the United States, I was determined to further explore this topic using the proper equipment.

The most challenging part of this study was navigating the electrospinning equipment, which was new to me. I eventually handled it well through repetitive usage.

Through this research, I discovered that electrospinning cellulose acetate with polyaniline requires an extensive optimization of different parameters to obtain a smooth and uniform morphology of the nanocomposite. The most interesting finding was that different parameters (voltage, distance, concentration, flow rate, etc.) should be balanced in order to obtain a smooth and uniform nanocomposite.

This research offers more understanding in the technology of electrospinning for producing value-added cellulose products in the smart textile indus-



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try. Aside from morphological characterization, I would like to perform more tests on the mechanical and conductive properties of the nanocomposites in the future.

Grande-Flores, who received her M.S. degree at State University of New York – ESF, is now assistant professor at the University of the Philippines in Los Banos. Bujanovic is project leader, Fiber and Chemical Sciences Research, at the U.S. Department of Agriculture Forest Products Laboratory, in Madison, WI, USA. Scott is director, Division of Engineering, and professor in the Department of Chemical Engineering, State University of New York – ESF, in Syracuse, NY, USA. Email Grande Flores at jogrande@up.edu.ph.

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