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Nanofiber Fabrication and Characterization for the Engineering Education

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Abstract

Electrospinning is a novel process of producing sub-micron and nano-sized fibers that consist of higher physical properties (e.g., surface area, porosity and flexibility). In a typical electrospinning process, a jet is ejected from a charged polymer solution when the applied electric field strength overcomes the surface tension of the solution. The ejected jet then travels rapidly to the collector target located at some distance from the charged polymer solution under the influence of the electric field and becomes a solid polymer filament as the jet dries. This communication presents the fabrication and characterizations of nanofibers and devices for undergraduate and graduate students to enhance their hands-on laboratory experiences.

KeyWords: Nanotechnology, fabrication and characterization of nanofibers, electrospinning.

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1. INTRODUCTION

Electrospinning is relatively an easier and direct process of fabricating a non-woven mat of polymer fibers compared to the conventional methods, such as melt spinning, wet spinning, extrusion molding, etc. It offers the distinct advantage of forming fibers in the micro to nanometer range, as well as a high surface area to volume ratio compared to the conventional fiber forming techniques [1-5]. This is not a new technique of manufacturing fibers. It has been known since the 1930s; however, it never gained industrial importance in the last decades, owing to low productivity and application possibilities. However, some special needs of biomedical, filtration, absorption and military applications reinvigorated its interest in these days [6-10].

In electrospinning process, a polymer dissolved in a suitable solvent is sufficiently charged using a high voltage source. The charge could be applied to the solution by directly inserting the electrode in the solution or applying it to the needle through which the solution is ejected. A jet is then ejected from the surface of a charged polymeric solution, when the applied electric field or electrostatic repulsion overcomes the surface tension. In other words, when the intensity of the electric field is increased, the hemispherical surface of the solution at the tip of the capillary tube elongate to form a structure known as Taylor cone [3]. During its flight to the target, the jet undergoes a series of electrically driven bending instabilities that give rise to a series of looping and spiraling motions. In order to minimize the bending instability caused by the repulsive electrostatic charges, the jet elongates to undergo large plastic stretching that finally leads to a significant reduction in diameter.

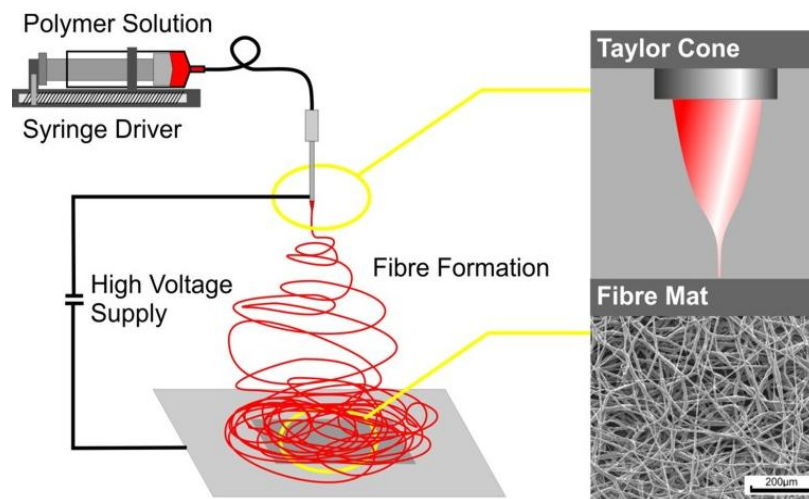


Figure 1: A set up of an electrospinning process, Taylor Cone and a SEM image of the produced nanofibers.

The electrospinning process consists of a polymer, solvent, digital syringe pump, a high voltage power supply, a ground screen, and an electrode. Prior to electrospinning, the viscosities of the solutions are usually measured. Figure 1 shows a set up of an electrospinning process and a scanning electron microscopy (SEM) image of the produced nanofibers [2].

In this paper we briefly describe the electrospinning method in both theoretical and practical aspects such as parameters and characterizations of the material and then provide a summary of how Dr. Asmatulu taught his PhD level nanotechnology course in Spring 2007 semester and how he will incorporate the electrospinning method as an experiment in his fall 2007 undergraduate level course as a lab experimentation.

2. THEORY

As is stated, the electric field reaches a critical or threshold value when the repulsive force surpasses the surface tension and a continuous jet of a liquid polymeric solution is ejected from the tip of the cone. Initially, the jet travels in a linear path, then slowly tends to bent away from the linear path and form complex round shape during its flight to the target. The length of the jet is proportional to the applied voltage. The jet undergoes large reduction in cross-sectional area and spiral loops grow from the jet. This phenomenon can be referred to as “bending instabilities”. Figure 2 shows the jet profiles providing a remarkable record of the transition from pressure-driven flow to electrically-driven flow. The change of shape of the Taylor cone from convex to concave is in accord with changes in charge density as the field strength is varied. Taylor studied the electric field used in electrospinning process and identified a critical or threshold voltage at which electrostatic repulsion overcomes the surface tension as [3]:

$$V_c^2 = 4 H^2 / L^2 \{ \ln 2L/R - 3/2 \} (0.117 \pi \gamma R) \quad [1]$$

where V_c is the critical voltage, H , the distance between the tip- target, L , the length of the capillary, R , the radius of the capillary, and γ is the surface tension of the polymeric solution.

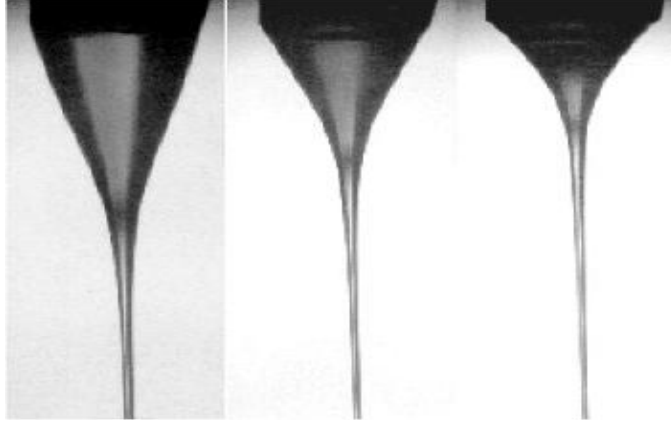


Figure 2: Optical photograph showing the electrospun fiber jets at 0.5 ml/min pump speed under different applied field strengths (from left to right: 3.67 kV/cm, 4.33 kV/cm and 5.0 kV/cm).

3. ELECTROSPINNING PARAMETERS

Several parameters are tested to achieve different sizes and shapes of nanofibers for the industrial applications. System parameters include polymer and solvent types and structures, while process parameters include electric potential, flow rate and polymer concentration; distance between the capillary and collection screen; temperature, humidity, and air velocity effects in the chamber; and conductive nanoparticle (Fe, Au, Ag, Pt) additions for polymer charge capacity improvements during the electrospinning process. Figures 3 and 4 show the effects of screen distance, electric potential, flow rate and concentration on fiber diameter [8-10]. As is seen, by adjusting those parameters, any size and shape nanofibers can be produced for various applications.

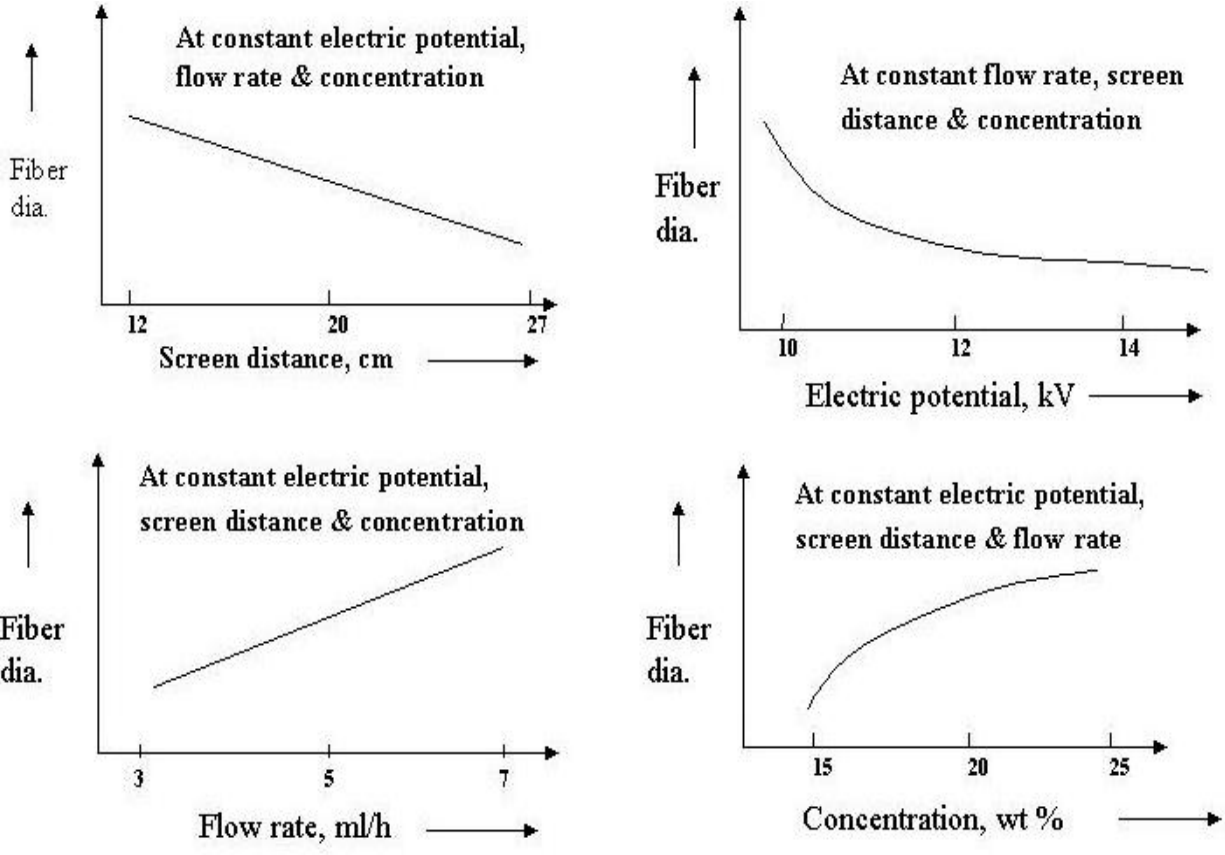


Figure 3: Effect of spinning distance, electric potential, flow rate and concentration on the electrospun fiber diameter.

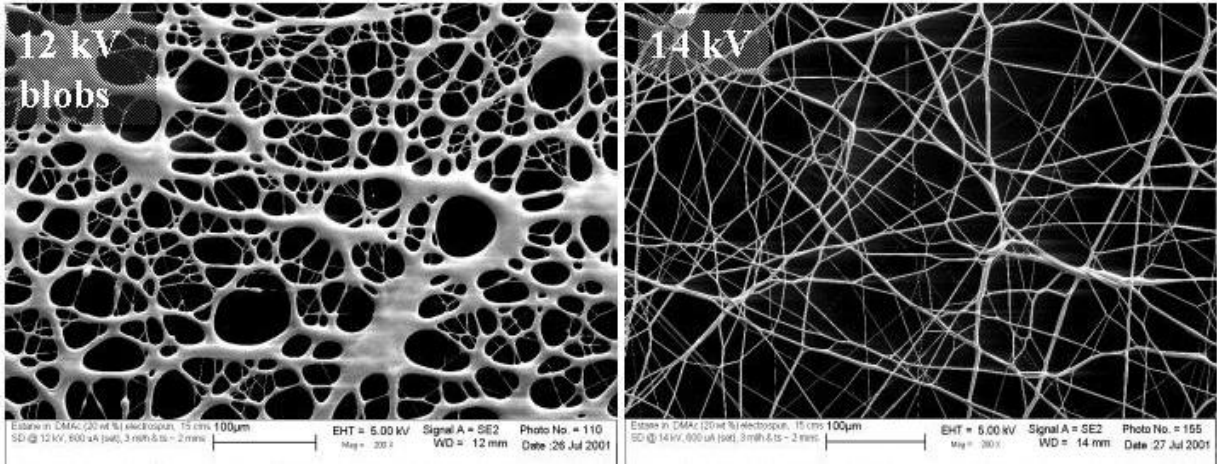


Figure 4: SEM images showing the effect of DC voltage on the diameter and morphology of the polymer nanofibers obtained at 12 kV and 14 kV using Estane polymer dissolved in *N,N*-dimethylacetamide (DMAc).

4. CHARACTERIZATION TECHNIQUES

Several characterization techniques including (but not limited to) SEM, transmission electron microscopy (TEM), atomic force microscopy (AFM), small-angle x-ray scattering (SAXS), and optical microscopes are utilized to identify the electrospun micro and nanofibers. For example, SEM micrographs of several electrospun fibers reveal a structure that consists of pores, beads, fibers, or a combination of pores, beads, and fibers. The beads may be spherical or elongated (spindle-like) and pores may be round, while the fibers may be round or flat. The combination of elongated beads, pores, and fibers, known as the bead-on-string morphology is observed under many conditions. The fibers may exhibit a variety of coils, loops, and bends resulting in different size and shape fibers [10].

The produced fibers are so small that even a human eye can not distinguish their sizes and shapes. Figure 5 shows the SEM images for comparisons of human hair and pollen sizes with different size electrospun nanofibers [9,10]. Because of their size and shape, there are several applications in various fields, and it will be extended in the near future.

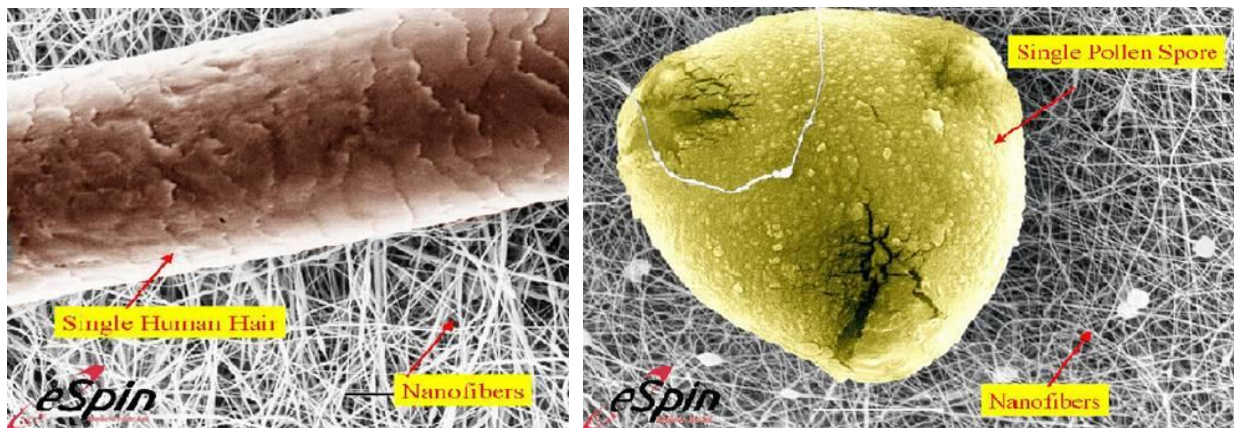


Figure 5: Comparisons of human hair and pollen sizes with different size electrospun nanofibers.

5. EDUCATION

Nanotechnology education is offered either as an option or required courses by many universities around the world. It usually involves a multidisciplinary natural science education with courses in engineering, physics, chemistry, math and biology. Additionally, nanotechnology is also considered to be taught as part of science studies at high schools and middle schools. This emerging field of science and technology is leading to a technological revolution in the new millennium [8-10].

The corresponding author has developed two new courses, namely Nanomaterials Fabrication and Characterization (PhD level) and Introduction to Nanotechnology (undergraduate level). In the first nanotechnology course, nanoparticles, nanofibers, nanofilms, nanotubes and nanocomposites fabrication techniques and their applications were given in detail. There were 35 students in the Spring 2007 class, most of which were Mechanical Engineering students. The teaching mode was utilizing PowerPoint slides to describe the above processes. Homework sets involved the evaluation of recently published nanotechnology papers. In the term project, the students prepared on an applications of nanomaterials and devices, such as nanocomposite manufacturing for aircraft industry, nanoelectromechanical systems, microelectromechanical systems and nanotechnology applications for fuel and solar cells.

In the Introduction to Nanotechnology course which is offered in Fall 2007, the similar topics will be covered at introduction level. There will be homework, term project, exams and laboratory sessions. A nanotechnology laboratory is being developed in the Department of Mechanical Engineering at Wichita State University, and dedicated to do a number of nanotechnology experiments for students. We plan to have undergraduate students work on the electrospinning method in the nanotechnology laboratory. Students will produce nanofibers using the described electrospinning method and then characterize properties such as fiber size, porosity, sound absorption, etc. utilizing the laboratory equipment available in the Mechanical Engineering Nanotechnology Laboratory. This will provide them a first hand experience on how nanomaterials can be produced and later how their properties can be characterized.

6. CONCLUSION

Electrospinning is a unique method to produce nanosize polymeric wires/fibers that consist of higher surface area, porosity and flexibility. A number of parameters including system and process affect the nanofiber size, shape and morphology, and by controlling those well defined fibers can be produced for various applications. This method will improve the knowledge of the students on how to design, analyze and manufacture nanomaterials and devices using electrospinning method.

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