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Electrospinning Process and Applications of Electrospun Fibers

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Abstract

An electric field is used to create a charged jet of polymer solution. As this jet travels in air, the solvent evaporates leaving behind a charged fiber that can be electrically deflected or collected on a metal screen. Fibers with a variety of cross sectional shapes and sizes were produced from different polymers. The diameter of these fibers was in the range of 0.05 to 5 microns. This paper describes the electrospinning process, the processing conditions, fiber morphology, and some possible uses of electrospun fibers.

1. Introduction

Various industrial applications of the production of liquid jets in the presence of intense electric field have been investigated. Some of these applications are listed in the literature [1-4]. One such application is the electrospinning process to spin microdenier synthetic fibers from polymer solutions. The electrospinning process has previously been employed for over three decades in industries to produce various products [5-13]. The present study is an attempt to characterize the electrospinning process, and identify the process parameters governing it. In this paper, the initial results of the study have been described.

In the electrospinning process, a polymer solution held by its surface tension at the end of a capillary tube is subjected to an electric field. Charge is induced on the liquid surface by an electric field. Mutual charge repulsion causes a force directly opposite to the surface tension. As the intensity of the electric field is increased, the hemispherical surface of the solution at the tip of the capillary tube elongates to form

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a conical shape known as the Taylor cone [14]. When the electric field reaches a critical value at which the repulsive electric force overcomes the surface tension force, a charged jet of the solution is ejected from the tip of the Taylor cone. Since this jet is charged, its trajectory can be controlled by an electric field. As the jet travels in air, the solvent evaporates, leaving behind a charged polymer fiber which lays itself randomly on a collecting metal screen. Thus, continuous fibers are laid to form a non-woven fabric.

The above description of the process suggests that the following parameters affect the process: solution properties including viscosity, conductivity, and surface tension; controlled variables including hydrostatic pressure in the capillary, electric potential at the tip, and the distance between the tip and the collection screen; and ambient parameters including temperature, humidity, and air velocity in the electrospinning chamber.

By appropriately varying one or more of the above parameters, fibers were successfully electrospun from water soluble polymers, biopolymers, and liquid crystalline polymers. Electrospun fibers may have an unusually small diameter, ranging downward from 5 microns to 0.05 microns. The small diameter provides a high surface area to volume ratio, and a high length to diameter ratio. These characteristics are useful in variety of applications, such as separation membranes, wound dressing materials, artificial blood vessels, in nano-composites, as a non-woven fabric, and many other applications. In the electrospinning process, fibers with a variety of cross sectional shapes and variations along their length may be produced.

2. Description of apparatus

The apparatus used in the electrospinning process is shown in *Figure 1*. It consists of a glass capillary tube with a 1.5 mm inside diameter tip mounted on an adjustable, electrically insulating stand. The capillary tube was filled with a polymer solution, into which a metal electrode was inserted. Hydrostatic pressure was established by an air pump, controlled by valves, and read on a manometer. The pressure was set to keep the solution at the tip of the tube, but not high enough that the uncharged solution dripped. The solution was charged by connecting the metal electrode to a high voltage power supply. Screens of different geometry were used as collecting devices for the charged fibers. The screen was mounted on an insulating stand so that its potential could also be controlled. The screen was grounded through a resistance so that the current flowing through the circuit could be observed.

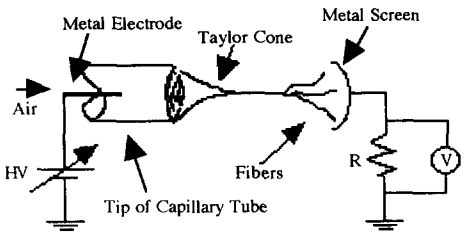

Fig. 1. Experimental set up of the electrospinning process.

3. Experimental method and material

The electrospinning process for poly(ethylene oxide) (PEO) having a molecular weight of 1,450,000 grams/mole is described in this paper. Aqueous solutions having concentration in the range of 1 % to 7 % (by weight) were prepared. Solution properties including conductivity, surface tension, and viscosity were determined.

Fibers were produced, as explained above, using the apparatus sketched in *Figure 1*, from PEO solutions. The electric potential, hydrostatic pressure, and the distance between the capillary tip and the collection screen were adjusted so that a stable jet was obtained, *Figure* 2. By varying the distance between the capillary tip and the collection screen, either dry or wet fibers were collected on the screen.

Since these fibers are charged, they can be deflected by a transverse electric field created between two metal plates. After the jet comes out of the transverse field region it travels in the deflected direction towards the metal collection screen. The dimensions of the deflecting plates were 7.5 cm x 7.5 cm separated by 12 cm. The typical value of the transverse electric field between the plates was in the range of 200 V/m to 500 V/m. Deflections of up to 15°were obtained.

The electric potential necessary to start the spinning process (V_{start}), i.e., to form a jet, was determined for all concentrations of the chosen molecular weight solution. This experiment was performed by slowly charging the solution in the capillary tube. The electric potential was increased until the cone formed and the jet was initiated from the apex of the cone. Similarly, the value of V_{stop}, i.e., potential at which the jet ceases was noted by forming the jet and then slowly decreasing the potential until the jet ceased. Hence, extreme values of V_{start} and V_{stop} were measured. This experiment was repeated five times and average values were obtained. In the above investigations, the solution in the capillary was at a positive potential, and was varied from 0 V to 12 kV, whereas the screen was at ground potential.

As the charged jet travels in air, its diameter decreases due to the simultaneous effect of stretching of the jet and evaporation of the solvent. The diameter of the jet was determined by a laser light (λ=632.8 nm) diffraction method [15]. As the jet diameter decreases, the surface charge density increases. The high repulsive forces from the increased charge density splits the jet and the smaller jets splay. The smaller jets then split and splay as their diameter is reduced further. This process may repeat several times to create many small jets which dry rapidly to form fibers with very small diameter.

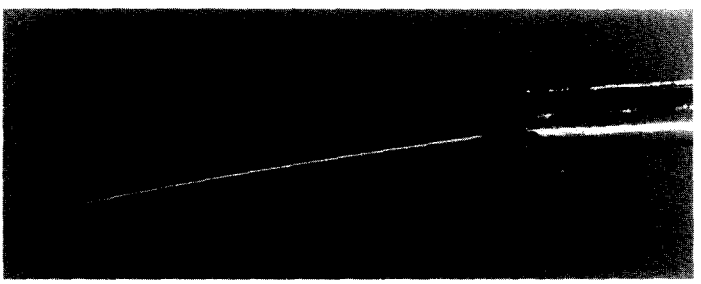

Fig. 2. Charged jet being ejected from the tip of the Taylor cone.

4. Results and discussion

The results for aqueous PEO system are considered as an illustration. The solution properties measured were as follows: surface tension 61 dynes/cm, viscosity between 30 to 6500 centipoise, and the conductivity around 400 μS/cm. Fibers were formed from solutions having a viscosity between 800 and 4000 centipoise. At a viscosity lower than 800 centipoise, the solution was too dilute to form a stable jet and the jet broke. At a viscosity higher than 4000 centipoise, it was difficult to form fibers due to the drying of the solution at the tip.

A typical concentration for electrospinning is 4 % by weight. As the electric field is increased, the solution at tip of the capillary elongates from an approximately hemispherical shape to a spheroidal shape and then to a cone shape. A further increase in the electric field to about 10^7 V/m, initiates ejection of the jet from the apex of the cone. A stable jet can be obtained by fine tuning the controlled variables. The electrospinning phenomena becomes complex when the electric field is increased further. The jet becomes unstable, and multiple jets are ejected from the tip. Sometimes jets were ejected from polymer carried by capillary attraction to the rim of

the capillary tube. This shows that the fiber forming process does not depend upon the shape of the liquid surface. The pressure due to the electric field can cause instabilities resulting in the formation of Taylor cones on and jets from liquid surfaces which are nominally flat, highly curved or at a meniscus.

In the electrospinning process, the polarity of the electric potential did not have any effect on the spinning process. Fibers could be spun from both positive and negative potentials. The current flowing through the resistance was varied as the electric field strength was changed. Typical current measured for above situation was about 100 nA. The electric field strength at the apex of the cone was determined using equation (1).

$$E = \sqrt{\frac{4\gamma}{\varepsilon_0 R}} \tag{1}$$

where E is electric field strength, γ is surface tension of polymer solution, ε_0 is the permittivity of the free space, and R is the radius of curvature of the rounded off cone apex. Dry fibers were collected on a metal screen of different size and geometry such as flat, curved, cylindrical, cubic etc. Particularly, a curved geometry was found to be the most efficient for fiber collection.

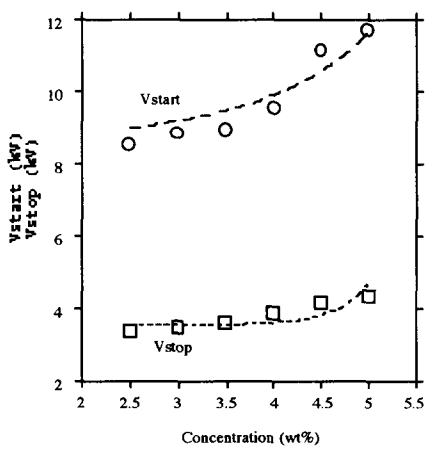

Fig. 3. Plot of electric potential to start and stop the electrospinning for various concentrations of the aqueous PEO solution.

In *Figure 3*, the applied electric potentials as a function of concentration is shown. These two curves represent possible extreme values for spinning. V_{start} shows the electric potential at which the jet was initiated and V_{stop} at which the jet ceased. The region between the V_{start} and V_{stop} is where a stable jet is obtained. As the solution concentration increased, the value of both V_{start} and V_{stop} increased, indicating that more force is required to form the jet from a highly viscous polymer solution. Below 2.5 % by weight concentration, the viscosity of polymer solution falls below critical value, and formation of stable jet becomes difficult and jet breaks down. Above 5 % concentration, the solution viscosity was to high, and it was difficult to form a stable jet.

Jet diameter was measured as a function of distance from the apex of the cone by laser light diffraction, at a constant tip potential and screen position. In *Figure 4*, a plot of jet diameter as a function of distance from the tip of the cone for 4 % by weight concentration PEO solution spun at 10 kV is shown. The distance between the capillary tip and the metal screen was set to 25 cm. The jet diameter was observed to decrease with increasing distance. The jet diameter decreased by about a factor of 5 at a distance of 10 mm from the tip of the cone, indicating a large amount of stretching and orientation of polymer molecules in the jet. The jet diameter was not measured beyond about 30 mm because the jet became too small and unsteady.

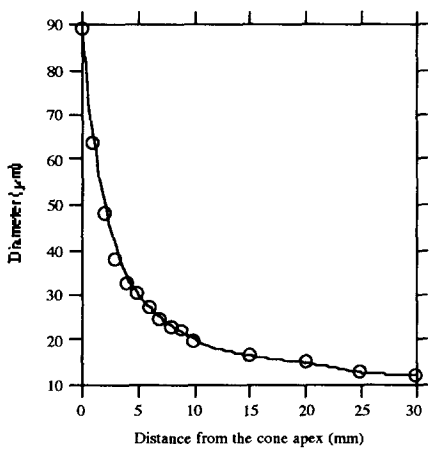

Fig. 4. Plot of jet diameter as a function of distance from the apex of the Taylor cone for 4 % by weight aqueous PEO solution spun at 10 kV.

The cone shape represented an order of magnitude decrease in the jet diameter within 1 mm from the capillary tip showing that elongational flow occurred. The magnitude of the strain rate in the cone was found to be of the order of $10^4\,s^{-1}$. This elongational flow tends to force the polymer molecules to orient in the extended form in the direction of elongation. Highly oriented fibers can be produced by crystallizing extended molecules inside the jet.

The splaying occurs after the splitting of the jet due to the forces from the increase in surface charge density as the diameter is reduced by evaporation is shown in *figure 5*. Some of the parameters contributing to this complex phenomena are surface charge, electric field strength, interfacial tension between air and the jet, surface tension, and viscous air drag.

Finally, fiber diameter and morphology were studied using an optical microscope, and a scanning electron microscope. *Figure 6* shows a scanning electron micrograph of electrospun fibers of PEO. Some of the fibers are in fact made up of a number of fibrils bundled together forming one thick fiber. The diameter of the fibers could be varied by changing the process parameters (i.e., electric field, distance between capillary and the collection screen, and the viscosity of the solution). As the applied electric potential increased from 8 kV to 10 kV, the deposition pattern of the fibers changed from a random orientation to a straight arrangement, as shown in *Figure 7*. The cross sectional shapes of these fibers were usually circular, but a variety of coils, loops, beads, and the like have also been observed.

Fig. 5. Photomicrograph of a single splayed jet

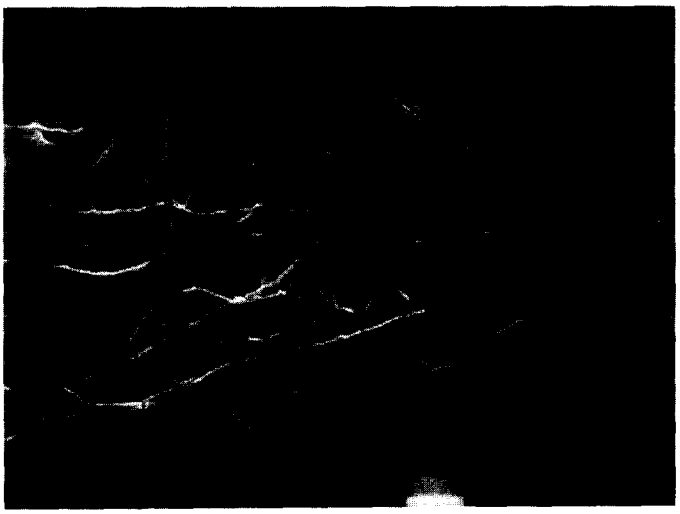

Fig. 6. SEM micrograph of electrospun PEO fibers spun from 4 % by weight solution at 8 kV.

Fig. 7. SEM micrograph of electrospun PEO fibers spun from 4 % by weight solution at 10 kV.

Forces from the charges on the fibers, with the help of the applied accelerating and deflecting electric fields, guided the fibers to form three dimensional objects with complicated shapes. Also, composite non-woven fabrics were produced by changing the composition of the spinning solutions successively so that layers of different polymers were deposited on top of each other. Some of the possible commercial uses of electrospun fibers are listed below:

1. As reinforcing fibers in composite materials.
2. As a non-wetting surface layer on ordinary textiles.
3. As a support for very thin polymeric separation membranes.
4. For application of insecticide on plants.
5. As a route to the production of non-woven fabrics.
6. As a wound dressing material.

5. Conclusion

Fibers from various polymers were successfully electrospun. Fibers with very small diameters can be spun from synthetic or biopolymers by forces from applied electric fields. The fact that these fibers are charged allows their trajectory to be controlled by electric fields. Fibers with a variety of cross sectional shapes and variations along their length were produced. Commercial applications of electrospun fibers in agriculture, medical, composite, and other areas are being investigated.

6. Acknowledgment

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7. References

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