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Effect of Polyvinyl Alcohol Concentration in Spinning Dope on Diameter, Beads and HHS of Produced Nanofibers

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Abstract: In this study it was aimed to produce nano fibers of poly vinyl alcohol PVA polymer via electrospinning ES method and to investigate the role of PVA concentration in the spinning dope on diameter, beads and HHS of produced fibers. Spinning dopes with different concentrations of PVA at rang of 5 to 20% (wt/wt on weight of solution) prepared. The requisite voltage for ES (30 kv) was supplied by a DC high voltage power supply and for collecting the fibers a grounded aluminum foil target placed 17 cm below the tip of capillary tube (spinning nozzle). The results showed that spinning dopes with PVA concentration between 8 to 12% (wt/wt) are capable of producing PVA fibers, but those below 8% (wt/wt) concentrations because of low viscosity formed droplets known as electrospray effect. Electrospinning suppressed at spinning dope concentrations above 12% (wt/wt) due to high viscosity of dope. According to the SEM photographs of electrospun PVA fibers it revealed that in higher concentrations produced nanofibers have larger diameter with less beads and HHS in compare with lower concentrations.

Key words: Electrospinning, polyvinyl alcohol, nanofibers, HHS, beads

INTRODUCTION

When the diameters of polymer fiber materials are shrunk from micrometers (e.g., 10-100 μm) to sub microns or nanometers (e.g., 10×10^{-3}-100×10^{-3} μm), there appear several amazing characteristics such as very large surface area to volume ratio (this ratio for a nanofiber can be as large as 10^3 times of that of a microfiber), flexibility in surface functionalities and superior mechanical performance (e.g., stiffness and tensile strength) compared with any other known from of the material. These outstanding properties make the polymer nanofibers to be optimal candidates for many important application such as filtration, medical, composite, military (Huang *et al*., 2003).

The concept of using static electricity to move fluids is over 500 years old. The term Electrospinning, derived from Electrostatic spinning, was used relatively recently (in around 1994), its fundamental idea dates back more than 60 years earlier. The electrospinning process was first developed by Zeleny. In 1971, Baumgarten made an apparatus to electrospin acrylic fibers with diameters in the range of 0.05-1.1 microns (Huang *et al*., 2003). In 1990's the electrospinning process was renovated by Reneker, as an economic and effective method for large scale fabrication of non-woven mats of nanofibers and since then has been widely studied by other researchers. A schematic diagram to interpret electrospinning of polymer nanofibers is shown in Fig. 1 (Gupta, 2004).

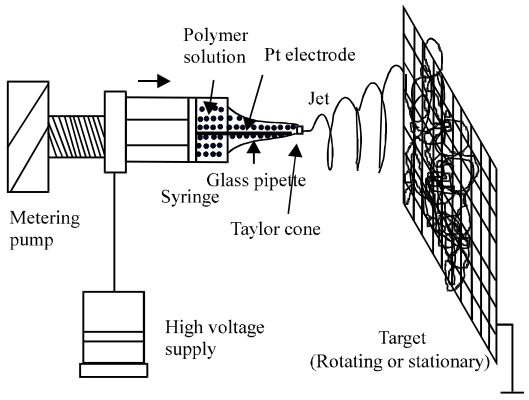

Fig. 1: Schematic diagram of electrospinning apparatus

There are basically three components to fulfill the process: A high voltage supplier, a capillary tube with a pipette or needle of small diameter and a metal collecting screen. In the absence of an electric field, the fluid forms a drop at the exit of the capillary, and its size is determined by surface tension. When an electric field is present, it induces charges into the fluid. These quickly relax to the fluid surface. The coupling of surface charge and the external electric field creates a tangential stress, resulting in the deformation of the droplet into a conical shape (Taylor cone). Once the electric field exceeds the critical value needed to overcome the surface tension, a fluid jet ejects from the apex of cone (Wan *et al*., 2004).

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In the electrospinning process, the polymer solution was poured in a syringe attached with a capillary tip, a high electric potential is applied to a droplet of solution at the tip of a capillary. A high voltage supply is applied for generating high DC voltage. The feeding rate, flow rate, back pressure for the precursor solution are controlled using a syringe pump. The capillary was tilted approximately 10° from the horizontal to maintain a droplet of solution and minimize a falling drop at the tip of the pipette. The positive electrode of a high voltage power supply was attached to a copper wire inserted into the polymer solution. The negative electrode was attached to collector. Charge is induced on the liquid surface by the electric field Mutual charge repulsion causes a force directly opposite to the surface tension. With increasing field, the free surface of the suspended drop changes to a cone, also commonly referred to as the Taylor cone. When the electric force from the applied field becomes larger than the surface tension of the droplet overcomes the surface tension and a charged jet of fluid is ejected from the tip of Taylor cone. The jet dries as the solvent is evaporated in the air.

The stretching process is accompanied by a rapid evaporation of the solvent, which leads to a reduction in the diameter of the jet. The dried fibers are deposited on the surface of the collection plate. The distance between the tip and the target was typically 5 to 25 cm. The process was carried out at room temperature. The electrospun fibers were held under vacuum at ambient temperature for 24 h in order to fully eliminate the solvent. The electrospun fibers retain a significant portion of their charge upon deposition. Prior to deposition on the collector, the jet may undergo whipping and bending instabilities that leads to accelerated solidification of the fluid jet and the collection of solid, charged fibers on the collector.

Polymers, molten in high temperature, can also be made into nanofibers through electrospinning. Instead of a solution, the polymer melt is introduced into the capillary tube. However, different from the case of polymer solution, the electrospinning process for a polymer melt has to be performed in a vacuum condition. The capillary tube, the fiber course, and the metal collecting screen must be encapsulated within a vacuum (Wan *et al*., 2004).

Poly vinyl alcohol is a water-soluble polymer produced industrially by hydrolysis of poly vinyl acetate. A number of grades of PVA are commercially available, which can be divided into two types: the fully hydrolyzed and the partially hydrolyzed PVA depending on the amount of acetate groups left in the backbone (Zhang *et al*., 2005).

In the presented study it was intended to produce nano fibers of poly vinyl alcohol, and to investigate the effect of polymer solution in the spinning dope on diameter, and conventional seen defects on produced nano fiber production namely beads and half hallow sphere.

MATERIALS AND METHODS

Materials: PVA (POLINOL®P-20) was purchased from DC chemical Inc. Distilled water prepared from Damavand Company. The morphology of electrospun fibers was observed by Scanning Electron Microscope (SEM; LEO, England) after gold coating. All the experiments were done in Research and Science Campus of Islamic Azad University in 2005.

Preparation of spinning solutions: Partially hydrolyzed POLINOL®P-20 dissolved completely after stirring for 30 to 60 min at room temperature in distilled water, and different concentrations of spinning dopes prepared from 5 to 20% (wt/wt on the weight of solution).

Electrospinning: Polymer solution was placed in an aluminum syringe (ID = 2.66 mm). The positive electrode of a high voltage power supply (30 kv) was attached to a copper wire (D = 1.8 mm) inserted into the polymer solution for charging the solution. The negative electrode was attached to grounded collector. The collector plate of aluminum foil was located at a fixed distance 17 cm below the needle tip.

The SEM photographs of electrospun PVA prepared after coating with gold. In SEM photographs of electrospun PVA three points were observed: diameter of electrospun PVA nanofibers, beads and Half Hallow Spheres (HHS) and alignment of nanofibers.

RESULTS AND DISCUSSION

In the presented study we chose polyvinyl alcohol for producing nanofibers according to its availability, nontoxic properties and ease of processing, beside it is soluble in water the most conventional solvent. Spinning dopes (viscose aqueous solutions) from 5-20% used to prepare fibers. At 5-7 wt% polymer solution, electrospinning was not enhanced due to the low viscosity. Fibers are not formed but only droplets are produced due to the jet breaks up into droplets known as the electrospray. At 8-12 wt% fibers formed in nano-scale. At 13-20 wt% because of high surface tension and high viscosity electrospinning is suppressed because it prohibits flow of a polymer solution continuously to the capillary tip.

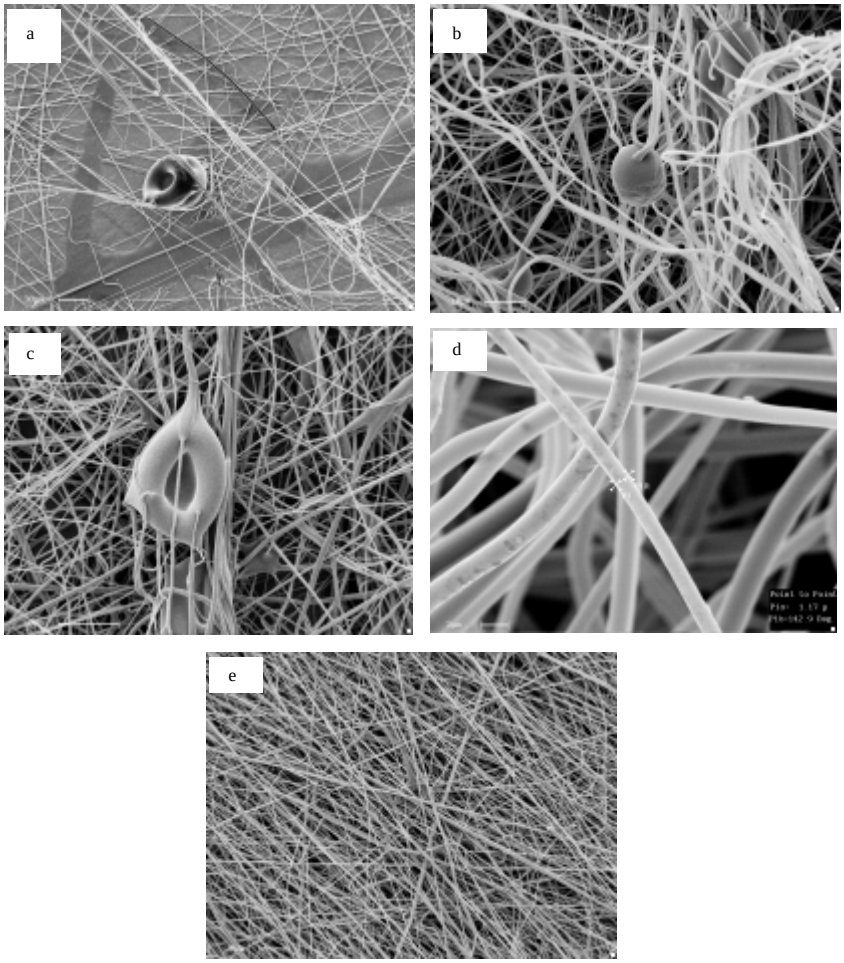

Fig 2: Effect of Polyvinyl Alcohol concentration in spinning dope on formation of Beads and HHS and ordering of fibers: a) 8%, b) 9%, c) 10%, d) 11%, e) 12% (wt/wt on weight of solution)

Table 1: Diameters of electrospun produced polyvinyl alcohol nano fibers of spinning dopes with different polymer concentrations.

	Concentration of PVA in spinning dope (wt/wt % on weight of solution)				
Fiber	8%	9%	10%	11%	12%
Diameters of produced nano fibers (nm)	208.99	351.08	316.54	345.60	359.11

It can be shown in Fig. 2 that with increasing the concentration of Polyvinyl Alcohol (PVA conc. >8-12%) or increasing viscosity (700-1500 cps) the beads (Fig. 2a-c) were completely disappeared and only fibers were formed (Fig. 2d-e). Also we can see the shape of the beads changed from spherical (Fig. 2a) to spindlelike (Fig. 2c) when the polymer concentration varied from low to high levels. With increasing concentration the aspect ratio of Beads and HHS (half hallow spheres) decreased and the order of produced network increased.

According to the diameters measuring of fibers by SEM depicted in Table 1 it can be seen that the electrospun produced PVA nanofibers had diameter between 200 and 359 nm, but with increasing the concentration of PVA in spinning dope the diameters of produced fibers increased.

CONCLUSIONS

In this study, Polyvinyl Alcohol nanofibers produced by Electrospinning. These nanofibers are suitable for filtration and medical applications. The diameters of produced nanofibers via electrospinning were in the range of 200-359 nm. Many parameters can influence on the quality and morphology of nanofibers such as polymer concentration. The best concentrations for producing nanofibers in this study were at the range of 8-12 wt%. In this range with increasing the concentration of polymer in

spinning dope the amount of Beads and HHS reduces considerably. SEM photographs of electrospun PVA fibers exhibited that in high concentration nanofibers have large diameter with less Beads and HHS.

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