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Developing Sub-Nanofibers in Electrospun Nylon-6 Web by Controlling the Parameters of the Process

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Summary: The aim of the present work is to subject the polyelectrolyte solutions of nylon-6 in formic acid undergoes an electrospinning process. To form sub-nanofibers (<50 nm in diameter) within the electrospun nanofibers mats, polymer concentration, applied voltage, solution temperature and environmental condition were chosen as variables. Scanning electron microscope was used to probe the presence of sub-nanofibers into the electrospun mat. To appear sub-nanofibers significantly, during the process of electrospinning, an optimum condition was obtained by finding a proper concentration of solution, applied voltage and temperature of solution which were 15 wt.%, 22 kV and 25 °C, respectively.

Keywords: electrospinning; electrospun mat; nanofiber; nylon-6; sub-nanofiber

Introduction

Electrospinning is a modern technique by which a very fine and continuous fibers are formed from an electrically charged jet of polymer solutions when the electrostatic forces overcome the surface tension of the polymeric fluid. A wide variety of parameters including solution properties, process conditions and other ambient parameters affect the process of electrospinning and properties of forming fibre mat.[1] Nylon-6 is an engineering materials in which its electrospun mats have found wide applications in industry such as water/air filtration media,[2] biomedical,[3] protective clothing,[4] and tissue engineering.[5] Because of their potential, many researchers are following to disclose the new aspect of the process and the application by different types of modifications.

Formation of high aspect ratio fibers in sub-nano scale has attracted considerable attention in between the various morphologies of electrospun nylon-6 mats. Barakat et al.[6] revealed that sub-nanofibers can be prepared by addition of strong ionic salt to the polar polymer solutions. Pant et al shown that, not only the addition of some additives such as Titanium dioxide (TiO_2) nanoparticles (NPs),[4] lactic acide monomer,[7] methoxy poly (ethylene glycol) oligomer[8] and graphene oxide[9] but also successive electrospinning as well as solvent degradation of nylon-6 could cause the formation of sub-nanofibers in electrospun nylon-6 mat.[2,10]

Temperature of solution as a significant factor, by which it causes the rate of solvent evaporation from electrospinning jet, has been issued in this paper, to investigate the formation of sub-nanofibers. Polymer concentration and applied voltage were also optimized to develop sub-nanofibers considerably into the electrospun nylon-6 nanofibers.

Electrospinning

Nylon-6 with molecular weight of 35 kDa from Sigma-Aldrich was used to make a concentration of 15, 18 and 22 wt.% in formic acid provided from Merck. All materials were used as received without further purification.

An electrospinning device equipped with temperature and humidity controllers

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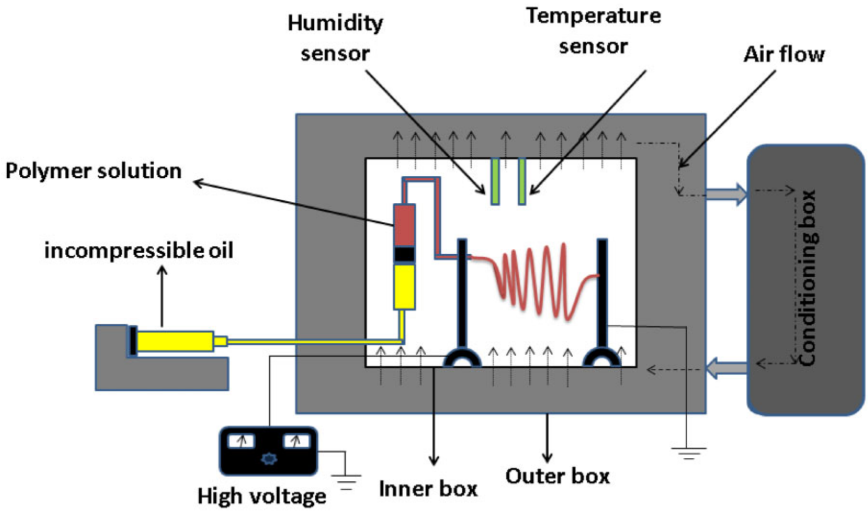

Figure 1.
Schematic representation of electrospinning chamber.

was employed for producing the samples. The temperature is controlled by a cooling coil and a heater in the conditioning box. The schematic diagram of electrospinning setup is shown in Figure 1. As seen in Figure 1, an incompressible oil was employed in a cylindrically glass vessel located between syringe pump (JZB 1800 Double channel syringe pump from china) and a glass syringe contained polymer solution that was placed in the inner box. Therefore the polymer solution at a desired temperature for electrospinning was delivered by syringe pump to the needle at fixed flow-rate. A high electrical voltage was applied to the needle by a high-voltage source (Gamma high voltage ES 50P-10W) to provide a sufficient electric field for electrospinning. To build a needle-to-plate electrode configuration, a plate wrapped with aluminum foil was used as a collector for the fibers at a fixed tip-to-collector distance of 15 cm. To form sub-nanofibers within the electrospun nanofibers mats, polymer concentration, applied voltage, solution temperature and environmental condition were chosen as variables. The obtained electrospun nanofiber mats were dried at room temperature for 24 h to remove the residual solvents.

Figure 2 shows the microphotographs of nylon-6 mats electrospun from solution of

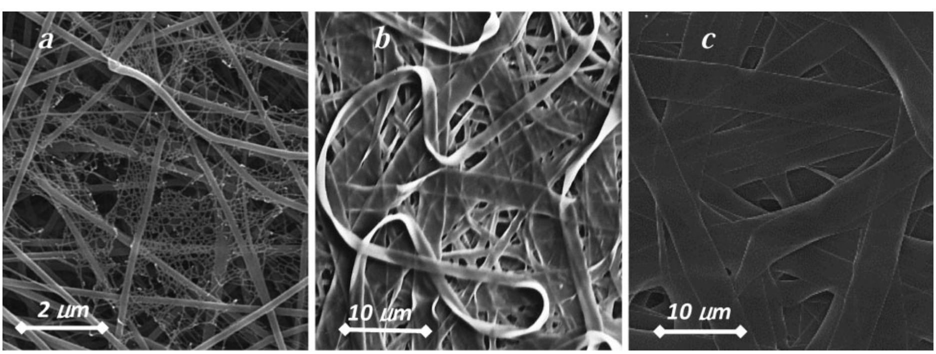

Figure 2.
SEM images of electrospun nylon-6 nanofibers mats at voltage 22 kV, solution temperature 25°C and relative humidity 35% and different concentrations: (a) 15 wt.%, (b) 18 wt.%, (c) 22 wt.%.

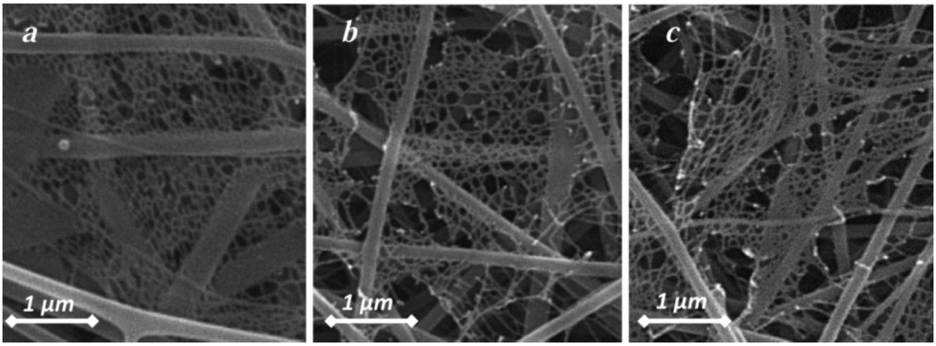

Figure 3.
SEM images of electrospun nylon-6 nanofibers mats at cocentration 15 wt.%, solution temperature 25°C and relative humidity 10% and different voltages: (a) 20 kV, (b) 22 kV, (c) 24 kV.

different concentrations. As seen, a transition in cross-section of electrospun fibers was happened from round-like to ribbon-like form by varying thepolymer concentration. This is in agreement well with that reported by Tsou et al.[11] All other parameters including voltage, temperature, humidity, and distance were kept constant. According to our observation, sub-nanofibers with diameter less than 50 nm could be often observed by solution of 15 wt.%, while in higher concentration they were disappeared.

Furthermore the formation of sub-nanofibers in nylon-6 was investigated as a function of applied voltage. At all applied voltage i.e. 20, 22, and 24 kV, the polymer solution was completely ionized to form sub-nanofibers in between nanofibers as shown in Figure 3. Polymer concentration and solution temperature were chosen to be 15 wt.% and 25 °C, respectively. According to the experiences, a critical voltage about 22 kV can be considered for sustaining in sub-nanofiber development. This is in agreement with that reported by Nirmala et al.[3] Increasing and/or decreasing the applied voltage from 22 kV causes to reduce the sub-nanofibers. Besides, the diameter of nanofibers was affected by changing the applied voltage; it become thinner when the applied voltage is increased.

The temperature of solution was the third factor we changed from 25 to 45°C to probe the formation of sub-nanofibers. Applied voltage and polymer concentration were chosen to be 22 kV and 15 wt.%, respectively. In contrast to the applied

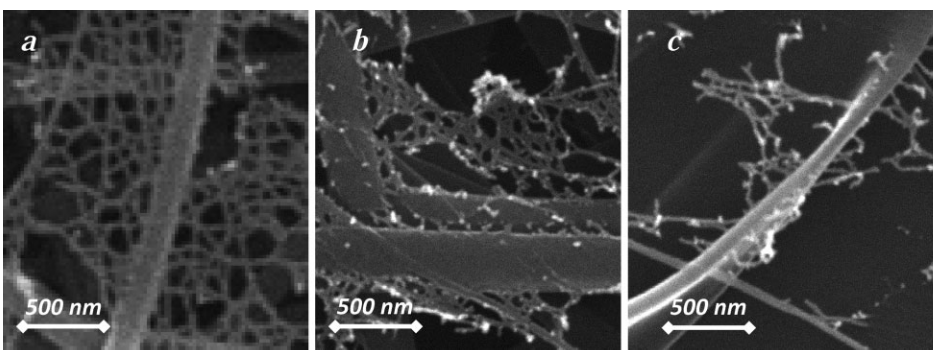

Figure 4.
SEM images of electrospun nylon-6 nanofibers mats at concentrations 15wt.%, voltages 22kV and relative humidity 10% and different solution temperature: (a) 25°C, (b) 35°C, (c) 45°C.

voltage, the solution temperature was an effective factor. Increasing the temperature of the process decreased the density of sub-nanofibers within the electrospun fibers. Figure 4 shows the SEM microphotographs of the samples electrospun at 25, 35, and 45 °C. To appear the sub-nanofibers, the most appropriate temperature of solution was obtained at 25 °C. Higher temperature of solution decreased the formation of sub-nanofibers and increased the diameter of nanofibers until ribbon-like structure became appear.

Conclusion

The effect of polymer concentration, applied voltage and solution temperature in the process of electrospinning on morphology of electrospun nylon-6 mat was investigated in this study. Exploring the SEM microphotographs shown that sub-nanofibers with diameter lower that 50 nanometers can be developed within electrospun nylon-6 fibers, if appropriate condition has been provided. An appropriate polymer concentration, applied voltage and solution temperature for formation of sub-nanofibers were obtained by 15 wt.%, 22 kV and 25 °C respectively. This structure developed in electrospun mat can be potentially applicable in a wide variety of filtration field like air filter media.

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