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Electrospinning of polyaniline-polyacrylonitrile blend nanofibers

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Abstract: Electrospinning of emeraldine base polyaniline/polyacrylonitrile (PANI/PAN) blends with different composition ratios were performed using N-methyl-2-pyrrolidone (NMP) as solvent. The blends were electrospun at various electrospinning temperature and electric fields. Morphology and fibers diameters were investigated by scanning electronic microscopy (SEM). The average diameter of nanofibers and their distributions were determined from 100 measurements of the random fibers with image analyzer software (manual microstructure distance measurement). Electrical conductivity of the prepared mats was characterized using standard four point probe method. The fibers with diameter ranging from 60 to 600 nm were obtained. The PANI/PAN blends containing up to the PANI content of 30% could be electrospun into the continuous fibrous structure, although pure PANI solution was not able to be electrospun into the fibrous structure. Average of fiber diameter decreased with increasing PANI content and electrospinning temperature. The electrospun PANI/PAN fibers at 50 °C and 75 °C showed smaller diameters with much better uniformity than those electrospun at 25 °C. The electrical conductivity of the mats increased with the increase of PANI content in the blend with percolation threshold of 0.5%.

Introduction

Polymers that exhibit high electrical conductivity have been synthesized in the last few decades [1]. These electrically conductive polymers have increasing number of applications in different areas of microelectronics and chemical analysis. The most classical formulation for the calculation of a metal resistor can be shown as:

$$R = k \frac{L}{A} \quad (1)$$

where R is the resistance of a conductor, A is the section area, L its length and k is the resistance coefficient. This equation is only valid for metal conductors where plenty of electrons exist in the conductor. In order to be able to accurately describe the polymer conduction, one can use modified version of this equation based on allomeric scaling law as described in detail in literature [2].

Among all conducting polymers, polyaniline have been of particular interest because of its environmental stability, controllable electrical conductivity and interesting redox properties associated with the chain nitrogen. Polyaniline also exhibits solution- or counterion-induced processability. Furthermore, the electrical properties of polyaniline can be substantially improved through secondary doping. The excellent processability, together with the presence of a number of intrinsic redox states, has enhanced the potential applications of aniline based polymers for use in practical

devices [3]. Polyaniline can be used in diodes, Field Effect Transistors (FETs), and different type of sensors like biosensors, gas sensors, humidity sensors and etc [4-8].

In nano-scale, the electrical conductivity of conductive polymers may increase [9]. It should be noted that systems in nano-scale may possess entirely new physical and chemical characteristics. As an example; higher electrical conductivity arises when the size of a wire is reduced below certain critical thickness (nano-scale). Such property raises the wide potential application of electrospinning. Nevertheless, it is impossible at the level of the visible world to make fibers at the nano-level with unusual properties [9]. Using nanofiber structure of conducting polymers, due to high surface to volume ratio of nanofibers, the performance of conducting polymers can be enhanced. It has been reported that optical sensors based on electrospun nanofibers showed sensitivity up to three orders of magnitude higher than that obtained from thin film sensors for the detection of nitro compound, ferric and mercury ions [10-12]. The higher sensitivities reported for these electrospun nanofibers can be attributed to their high ratios of surface area to volume.

To date, the most successful method of producing nanofibers and nanoporous materials is through the process of electrospinning [13, 14]. The electrospinning process uses high voltage to create an electric field between a droplet of polymer solution at the tip of a needle and a collector plate. When the electrostatic force overcomes the surface tension of the drop, a charged, continuous jet of polymer solution is ejected. As the solution moves away from the needle and toward the collector, the solvent evaporates and jet rapidly thins and dries. On the surface of the collector, a nonwoven web of randomly oriented solid nanofibers is deposited.

Several researches have been done in order to produce nanofibers of polyaniline. Reneker and Chun reported that polyaniline fibers could be successfully electrospun from sulfuric acid into a coagulation bath. A similar work was done by MacDiarmid et al. in which the average diameter has been reported as $\approx 139\text{nm}$ [15, 16]. Later on, works have been done on electrospinning polyaniline/polyethylene oxide (PEO) blend. PEO was added to assist in fiber formation. Norris et al. reported conducting ultrafine fibers with diameters less than $2\text{ }\mu\text{m}$ using electrospinning process [17]. Leon reported electrospinning of polyaniline/polystyrene with the diameters below 100 nm [18]. Fabrication of polyaniline-based nanofibers with diameter below 30 nm is also reported [19].

In the present work we used polyaniline/ polyacrylonitrile blend to form a nonwoven mat. Polyaniline exists in a large number of intrinsic redox states. The half oxidized emeraldine base is the most stable and widely investigated state in the polyaniline family that can be dissolved in N-methyl-2-pyrrolidone (NMP). Polyaniline emeraldine base/polyacrylonitrile blend solution in NMP was prepared and then it was electrospun with different blending ratio. Fibers diameter, fibers morphology and electrical conductivity of the mats were analyzed and discussed.

Results and discussion

Published literature have shown that in the electrospinning process, the system configuration and operation conditions differ vastly from one material to another, depending on the material and the choice of solvent. Physical and chemical parameters of polymer solution such as viscosity, electrical conductivity, surface tension and air temperature can determinedly affect the formability and morphology of electrospun fibers. In the following sections effects of some electrospinning

parameters on the fiber formation and morphology of PANI/PAN blend solutions were discussed and the best condition for obtaining PANI/PAN fibers was examined.

Effect of PANI Content

We were not able to obtain the fibers from the pure PANI solution because a stable drop at the end of the needle was not maintained. Figure 1 shows SEM micrographs of PANI nanoparticles electrospun from pure PANI solution. As seen in Figure 2, most of PANI particles have a round shape on a scattered layer of the PANI particles, while the fibrous structure is not observed. The major complication in electrospinning of PANI is the poor solubility of PANI. At low polymer concentration, the solution does not contain sufficient material to produce stable solid fibers. With increasing polymer concentration, insoluble PANI particles in the solution increase rapidly, resulting in unspinnable solution. Therefore, we prepared PANI/PAN blend solutions with different PANI content using NMP as solvent. At PANI content above 30% regardless of electrospinning conditions drops were formed instead of fibers. A series of experiments were carried out when the PANI weight percent was varied from 10% to 30%. The applied voltage was 20 to 30 kV and the chamber temperature was held at 25, 50 and 75 °C. Figure 1 shows the SEM micrographs and the surface morphology of obtained fibers at 25 °C and 25 kV. At a solution containing 30% PANI, the fibrous structure was not completely stabilized and a bead - on - string structure with non uniform morphology was obtained. The fibers between the beads had a circular cross section, with a diameter typically between 60 nm and 460 nm and mean fiber diameter of 164 nm. As the PANI content decreases to lower than 20%, a fibrous structure was stabilized. At 20% PANI content, fibers mean diameter increased to 425 nm with some beads on the fibers. At 10% PANI content, continuous fibers without beads resulted regardless of electric field with the mean fiber diameter of 602 nm at 25 kV. Smooth and uniform fibers with average diameter of 652 nm were electrospun from PAN solution at the same electrospinning condition. These results reveal that as the PANI contents in the blends increase up to 30% the average diameter of blend fiber gradually decreases from 602 to 164 nm and its distribution becomes significantly broader with higher standard deviation as shown in Fig. 1. It is also observed that fibers with not uniform morphology are electrospun at 25 °C. Figure 3 shows SEM photomicrographs of electrospun PANI/PAN blend fibers at 50 °C at various blend ratios. This figure shows that fibers with uniform morphology without remarkable beads are formed regardless of PANI content. It is also observed that at 50 °C average diameter of electrospun fibers decreases from 194 nm at 10% PANI content to 124 nm at 30% PANI content at 50 °C. Similar to the results obtained at 25 °C fiber formation from pure PANI solution and blends containing more than 30% PANI was not possible.

In electrospinning, the coiled polymer chains in the solution are transformed by the elongational flow of the jet into oriented entangled networks. Experimental observations in electrospinning confirm that for fiber formation to occur, a minimum chain entanglement is required [22]. Below this critical chain entanglement, application of voltage results beads and droplets due to jet instability. The gradual increase in fiber diameter with content of PAN in the blends may be explained by the increase of solution viscosity due to higher viscosity of PAN solution. The ranges of shear viscosity of the PANI/PAN blends are shown at Table 1.

It is obvious that shear viscosity of the solutions decrease with PANI content in the blends. Therefore, as the concentration of PAN in the blend is increased the solution

viscosity and resulted polymer chain entanglements increase significantly. During electrospinning, the stable jet ejected from Taylor's cone [13] is subjected to tensile stresses and may undergo significant elongational flow.

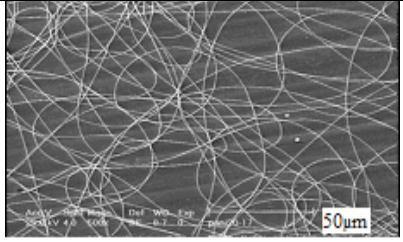
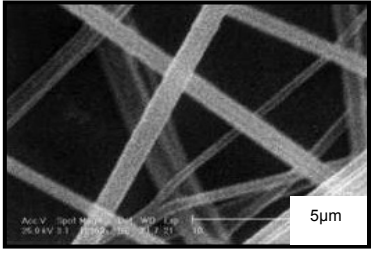
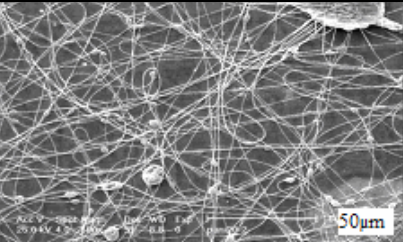
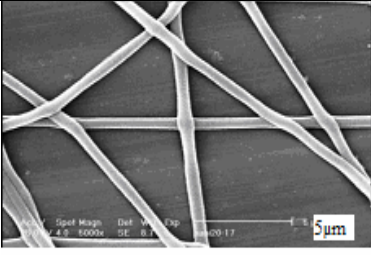
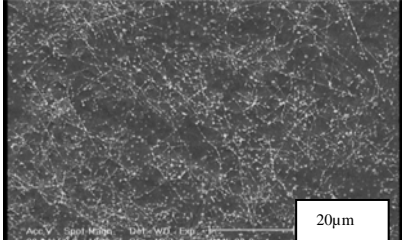
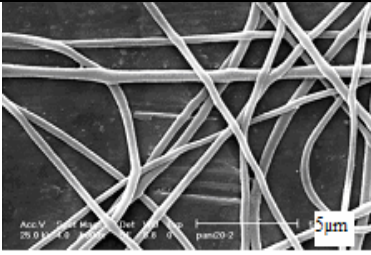
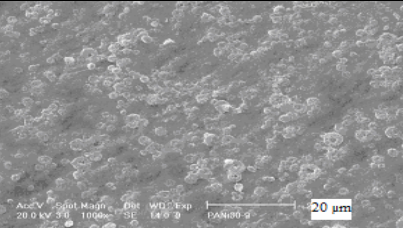
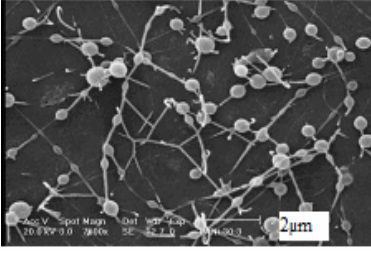
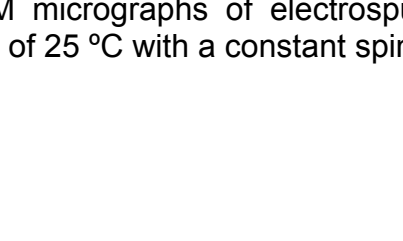
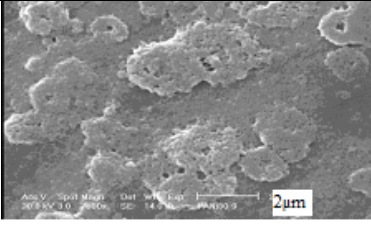
0% PANI content			Average fiber diameter =652 nm Standard deviation=65 nm
10%PANI content			Average fiber diameter =602 nm Standard deviation=40 nm
20%PANI content			Average fiber diameter =425 nm Standard deviation=56 nm
30%PANI content			Average fiber diameter =164 nm Standard deviation=75 nm
100 %PANI			

Fig. 1. SEM micrographs of electrospun fibers at applied voltage of 25 kV and temperature of 25 °C with a constant spinning distance of 10 cm.

The nature of this elongational flow may determine the degree of stretching of the jet. The characteristics of this elongational flow can be determined by elasticity and viscosity of the solution. The results show that viscosity of the PAN solution is higher than PANI solution. Hence viscosity of the blend solution decreases with an increase in PANI content.

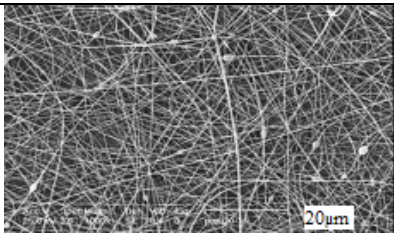
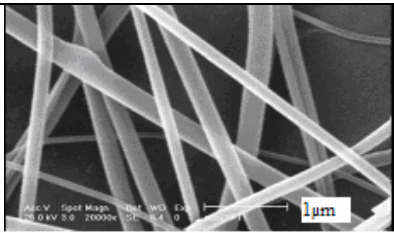
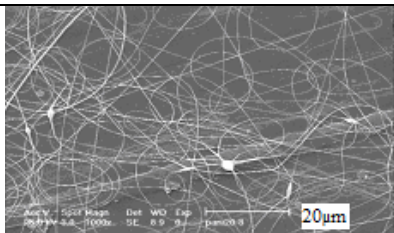
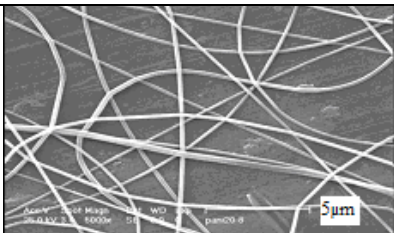
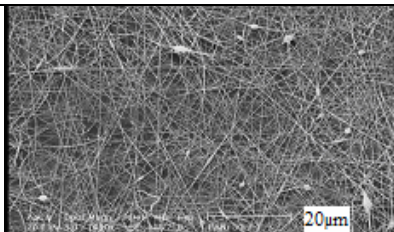
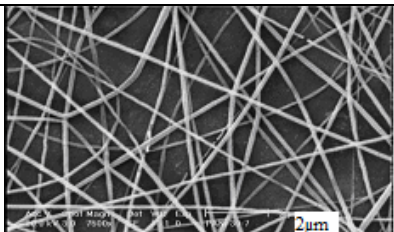
10%PANI content			Average fiber diameter =194 nm Standard deviation=62 nm
20%PANI content			Average fiber diameter =180 nm Standard deviation=26 nm
30%PANI content			Average fiber diameter =124 nm Standard deviation=37 nm

Fig. 2. SEM micrographs of electrospun fibers at applied voltage of 25 kV and temperature of 50 °C with a constant spinning distance of 10 cm.

Therefore, jet stretching during the electrospinning is more effective at higher PANI content. As a result, the fibers diameters decrease with increasing PANI content in the blends. On the other hand, at the high PANI content, an insufficiently deformable entangled network of polymer chain exists and the ejected jet reaches the collector before the solvent fully evaporates. Therefore, at low solution viscosity ejected jet breaks into droplets and a mixture of beads and fibers is obtained. This explains the formation of droplets and beads at high PANI content. Effect of electrospinning temperature is discussed in the following section. He and coworkers [23] showed that the diameters of electrospun nanofibers are greatly affected by solution viscosity, and solution viscosity has an allometric relationship with its concentration. Our results shows that the electrospun nanofibers diameters (d) of PANI/PAN blends has a relationship with PANI content in the form of

$$d \propto (PANI\%)^2 \quad (2)$$

Effect of electrospinning temperature

Studies on the electrospinning show [13, 24-26] that many parameters may influence the transformation of polymer solution into nanofibers. Some of these parameters include (1) the solution related properties such as viscosity and surface tension, (2)

process variables such as electric potential at the capillary tip and (3) ambient parameters such as air temperature in the electrospinning chamber.

Tab. 1. Shear viscosity of the PANI/PAN blend solutions at 22 °C and shear rate of 500 s^{-1} and average diameter of electrospun fibers.

PANI/PAN blend ratio% (w/w)	Shear viscosity (Pa.s)	Average nanofiber diameter (nm)
100/0 (5% solution)	0.159	No fiber
30/70	0.413	164
20/80	0.569	425
10/90	0.782	602
0/100	1.416	652

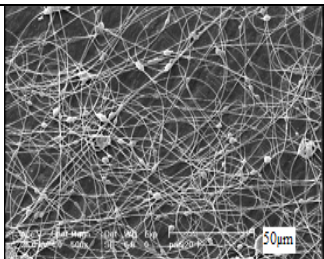
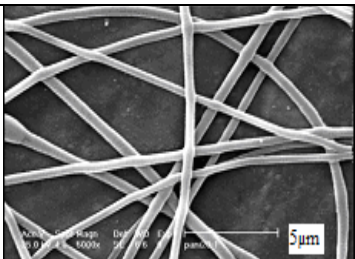
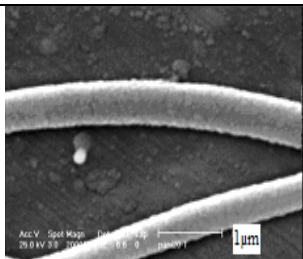
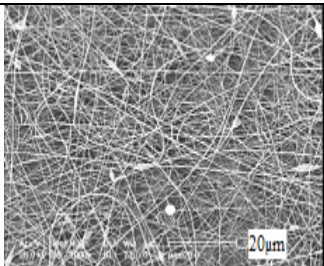
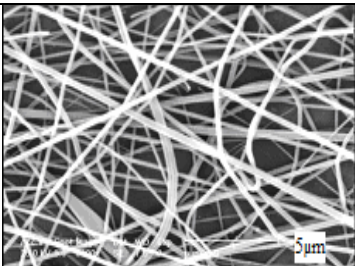
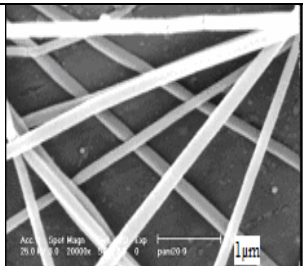
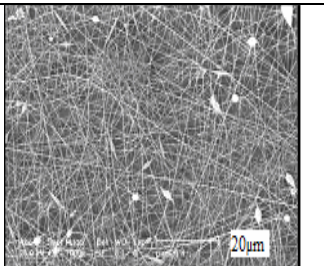
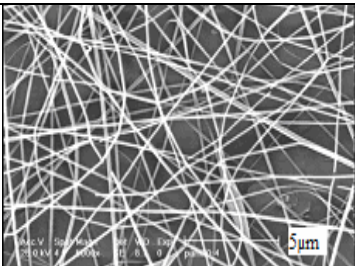
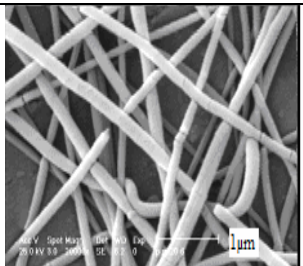
Electrospinning Temperature °C			
25			
	Average fiber diameter=500 nm	Standard deviation=86 nm	
50			
	Average fiber diameter=205 nm	Standard deviation=29 nm	
75			
	Average fiber diameter=110 nm	Standard deviation=25 nm	

Fig. 3. SEM micrographs of electrospun fibers at applied voltage of 20 kV and PANI content of 20% with a constant spinning distance of 10 cm.

In order to study the effect of electrospinning temperature on the morphology and texture of electrospun PANI/PAN nanofibers, solution containing 20% PANI was electrospun at temperatures 25, 50 and 75 °C. SEM micrographs of electrospun fibers at 20 kV are shown in Figure 3. The electrospun sample at 25 °C shows fibers with several beads and not uniform surface morphology as shown at lower magnification in Figure 3. Interestingly, the electrospinning of the solution shows bead free fiber morphology at 50 °C and 75 °C, whereas fibers with large beads are observed at 25 °C especially at high PANI contents (Figure 3). With an increase in electrospinning temperature fibers morphology changes gradually from mixture of beads and fibers through uniform fibers. As shown in Figure 3 at 50 °C continuous fibers with uniform morphology were obtained while increasing the electrospinning temperature to 75 °C caused bead free but fragile and cracked fibers. Diameter measurement of electrospun fibers at 25 °C showed a size range of approximately 400 to 700 nm with 480 nm being the most frequently occurring. They were within the same range of reported size for electrospun PANI/PEO nanofibers [18]. With increasing the electrospinning temperature to 50 °C, fiber diameter was decreased to a range of approximately 110 to 290 nm with 170 nm the most occurring frequency. At 75 °C, fibers dimensions were 70 to 170 nm with 110 nm the most occurring frequency. It was obvious that diameter of electrospun fibers were decreased with increasing of electrospinning temperature. The distributions of fibers diameters electrospun at 25, 50 and 75 °C are shown in Fig. 4. At 25 °C broad distribution of fiber diameters was obtained, while a narrow distribution in fiber diameters was observed at 50 and 75 °C.

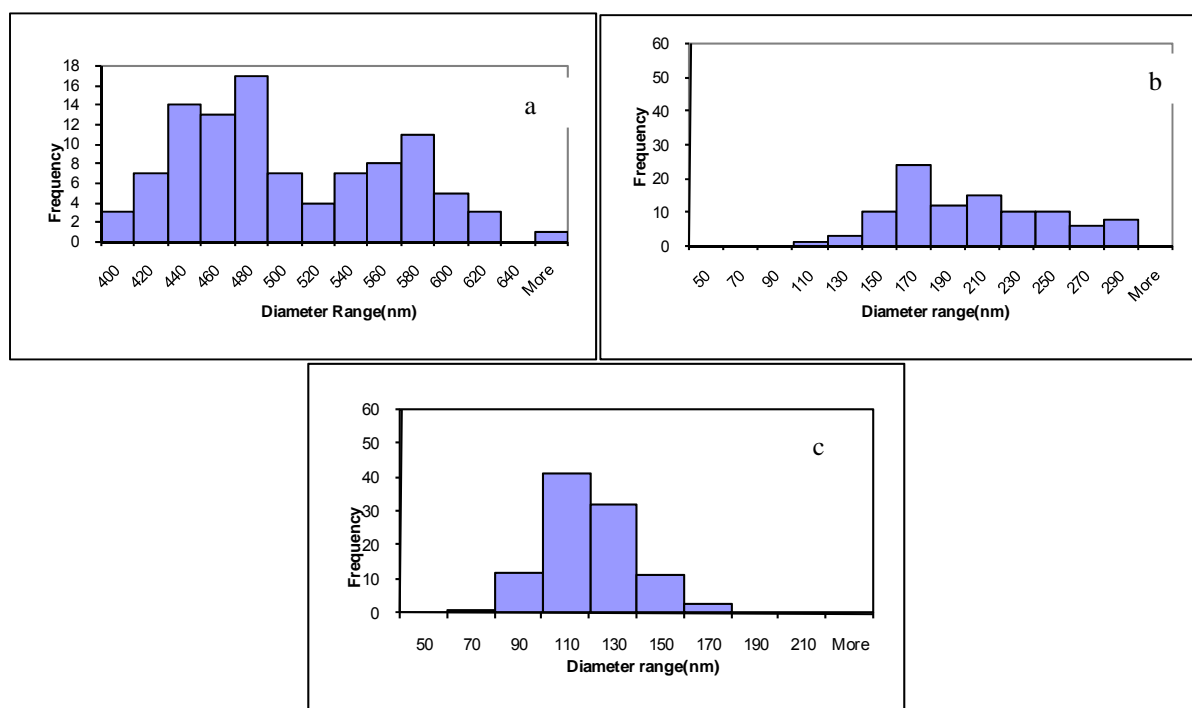


Fig. 4. Distribution of fiber diameter electrospun at PANI content of 20%, applied voltage of 20 kV, spinning distance of 10 cm and electrospinning temperature of (a) 25 °C, (b) 50 °C and (c) 75 °C.

Several factors with PANI/PAN blends may explain the effects of electrospinning temperature and PANI content on morphology of the electrospun fibers. Since nanofibers resulted from evaporation of solvent from polymer solution jets, the fiber diameters will depend on the jet sizes, elongation of the jet and evaporation rate of the solvent [24]. At a constant PANI content, as the electrospinning temperature is increased, the rate of solvent evaporation from the ejected jet increases significantly. In the case of electrospinning at 25 °C due to the high boiling point of NMP (approximately 202 °C), the fibers with relatively high solvent content travels during electrospinning process and reach the collector. Therefore, the collected fibers have irregular morphology due to contraction of the fibers during the electrospinning and on the collector.

At higher electrospinning temperature rate of solvent evaporation from the ejected jet increases significantly and a skin is formed on the surface of the jet, which results collection of dry fiber with smooth surface. Presence of a thin, mechanically distinct polymer skin on the liquid jet during electrospinning has been discussed by Koombhongse et al. [27]. On the other hand higher electrospinning temperature results higher degree of stretching and more uniform elongation of the ejected jet due to higher mobility and lower viscosity of the solution. Therefore fibers with smaller diameters and narrower diameters distribution will be electrospun at higher electrospinning temperature.

Effect of applied voltage

In order to study the effects of applied voltage, the blend solutions were electrospun at various applied voltages and temperatures. From the results shown in Fig. 5, it is obvious that the diameter of electrospun PANI/PAN fibers at 50 °C decreased as the applied voltage increased.

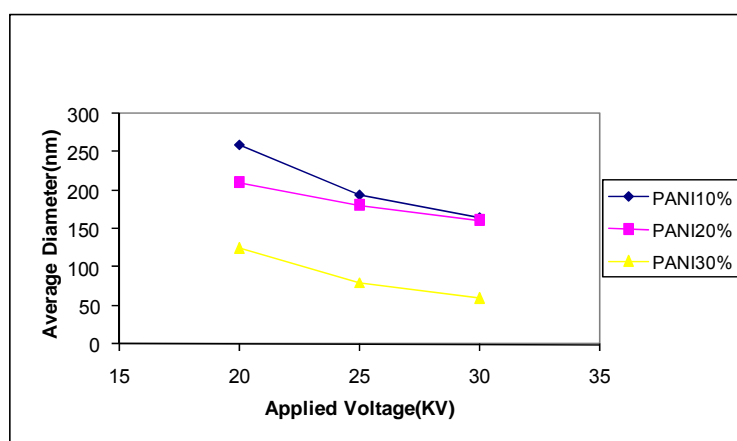


Fig. 5. Average fiber diameter of electrospun fibers at various applied voltages and PANI content at temperature of 50 °C and electrospinning distance of 10 cm.

Similar results were observed for electrospun fibers at 25 and 70 °C (results were not shown). The same results were found by Fenessey et al. [28] and Ding et al. [29] and others [30-31].

Electrical conductivity

Figure 6 shows electrical conductivity of the electrospun mats at various PANI/PAN blend ratios. As expected, electrical conductivity of the mats was found to increase with an increase in PANI content in the blends. Figure 6 shows that the electrical conductivity of the mats increases sharply when the PANI content in the blends is less than 5% , after which it will gradually reach to 10^{-1} S/cm at higher PANI content. This result is in agreement with the observations of Yang and co workers [35] which reported the electrical conductivity of PANI/PAN blend composites. Yang et al. [35] proposed the classical law of percolation theory, $\sigma(f)=c(f-f_p)^t$, where c is a constant, t is critical exponent of the equation, f is the volume fraction of the filler particle and f_p is the volume fraction at percolation threshold. The results of Figure 6 indicate that the conductivity of the mats follows the scaling law of percolation theory mentioned above as shown in equation 1 which results in a value of 0.5 wt% of PANI for f_p . This value for the percolation threshold is much lower than that reported by Yang et al. [35] which may be due to the difference in the studied sample form. Their measurements were performed on the prepared films whereas our measurements were performed on the nanofiber mats. It is worth noting that the classical percolation theory predicts a percolation threshold of $f_p = 0.16$ for conducting particles dispersed in an insulating matrix in three dimensions [35] which is in agreement of our finding.

$$\sigma = 9 \times 10^{-7} (f - 0.5)^{3.91} \quad R^2 = 0.99 \quad (3)$$

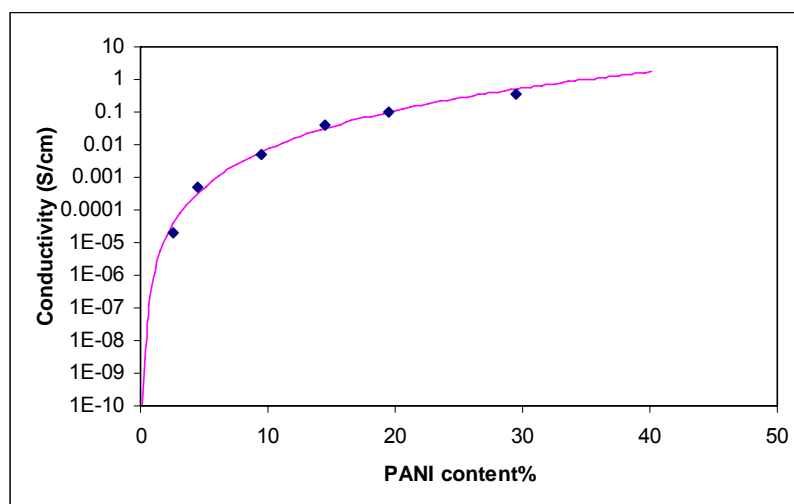


Fig. 6. Electrical conductivity of electrospun mats at various PANI contents.

Conclusions

The electrospinning of PANI/PAN blend in NMP was processed and fibers with diameter ranging from 60 to 600 nm were obtained based on electrospinning conditions. Morphology of fibers was investigated at various blends ratios and electrospinning temperature. At 30% PANI content and 25 °C fibers with average diameter of 164 nm were formed with beads (droplets of polymer over the woven mat) and not uniform morphology. At this condition solution viscosity and chain entanglements may not be enough, resulting in spraying of large droplets connected with very thin fibers. Averages of fiber diameters decreased with PANI content in the solutions but PANI/PAN solution containing more than 30% PANI did not form a stable jet regardless of applied voltage and electrospinning temperature. For pure PANI solution, since the viscosity is too low to get stable drops and jets, we could not

get the fibers. It was found that at 25 °C fiber morphology was changed to beaded fibers when PANI content was higher than 20%. With increasing the electrospinning temperature, the morphology was changed from beaded fibers to uniform fibrous structure and the fiber diameter was also decreased from 500 nm to 100 nm when the electrospinning temperature changes from 25 °C to 75 °C. The mean of fiber diameter is the smallest and the fiber diameter distribution is the narrowest for the electrospun fibers at 75 °C. However some cracks are observed on the surface of the electrospun fibers. There was a slight decrease in average fiber diameter with increasing applied voltage. It is concluded that the optimum condition for nanoscale and uniform PANI/PAN fiber formation is 20% PANI content and 50 °C electrospinning temperature regardless of the applied voltage. The conductivity of the mats follows the scaling law of percolation theory which predicts a value of 0.5 wt% of PANI as percolation threshold for the blend of PANI/PAN.

Experimental

Materials

Commercial polyacrylonitrile (PAN) polymer containing 6% methacrylate with molecular weight (M_w) of 100000 was supplied by Polyacryl Iran Co (IRAN). N-Methyl-2- pyrrolidone (NMP) was from Riedel-de Haën. Aniline from Merck was vacuum distilled prior to use. The polyaniline (PANI) used was synthesized in our laboratory.

Sample preparation

Polyaniline was synthesized by the oxidative polymerization of aniline in acidic media. 3 ml of distilled aniline was dissolved in 150 ml of 1N HCl and kept at 0-5 °C. 7.325 g of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ was dissolved in 35 ml of 1N HCl and added drop wise under constant stirring to the aniline/HCl solution over a period of 20 minutes. The resulting dark green solution was maintained under constant stirring for 4 hrs. The prepared suspension was dialyzed in a cellulose tubular membrane (Dialysis Tubing D9527, molecular cutoff = 12,400, Sigma) against distilled water for 48 hours. Then it was filtered and washed with water and methanol. The synthesized polyaniline was added to 150 mL of 1N (NH_4) OH solution. After an additional 4 h the solution was filtered and a deep blue emeraldine base form of polyaniline was obtained (PANIEB). The synthesized polyaniline was dried and crushed into fine powder and then passed through a 100 mesh. Intrinsic viscosity of the synthesized polyaniline dissolved in sulfuric acid (98%) was 1.18 dl/g at 25 °C.

The PANI solution with concentration of 5 % (W/W) was prepared by dissolving exact amount of PANI in NMP. The PANI was slowly added to the NMP with constant stirring at room temperature. This solution was then allowed to stir for 1 hour in a sealed container. 20% (W/W) solution of PAN in NMP was prepared separately and was added drop wise to the well-stirred PANI solution. The blend solution was allowed to stir with a mechanical stirrer for an additional 1 hour.

Various polymer blends with PANI content ranging from 10 wt% to 30 wt% were prepared by mixing different amount of 5% PANI solution and 20% PAN solution. Total concentration of the blend solutions were kept as 12.5%.

Electrospinning

Polymeric nanofibers can be made using the electrospinning process, which has been described in the literature and patented [20-21]. Electrospinning uses a high electric field to draw a polymer solution from tip of a capillary toward a collector. A voltage is applied to the polymer solution, which causes a jet of the solution to be drawn toward a grounded collector. The fine jets dry to form polymeric fibers, which can be collected as a web.

Our electrospinning equipment used a variable high voltage power supply from Gamma High Voltage Research (USA). The applied voltage was varied from 1- 30 kV. A 5-ml syringe was used and positive potential was applied to the polymer blend solution by attaching the electrode directly to the outside of the hypodermic needle with internal diameter of 0.3 mm. The collector screen was a 20×20 cm aluminum foil, which was placed 10 cm horizontally from the tip of the needle. The electrode of opposite polarity was attached to the collector. A metering syringe pump from New Era pump systems Inc. (USA) was used. It was responsible for supplying polymer solution with a constant rate of 20 $\mu\text{L}/\text{min}$.

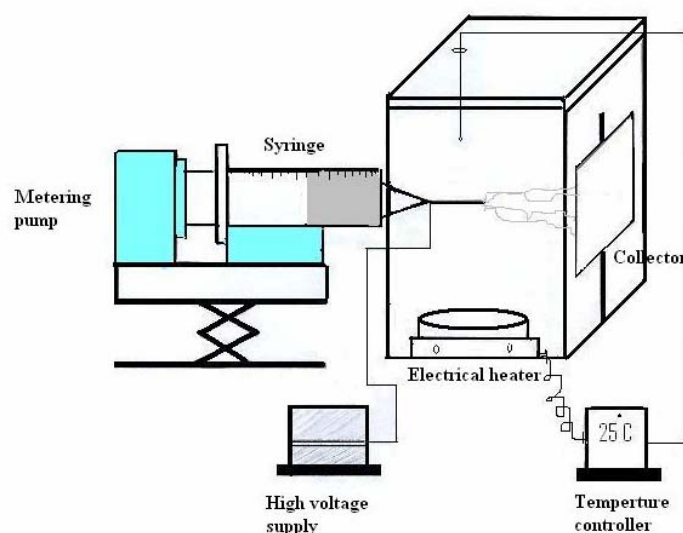


Fig. 7. Schematic diagram of electrospinning apparatus.

Electrospinning was done in a temperature-controlled chamber and temperature of electrospinning environment was adjusted on 25, 50 and 75 °C. Schematic diagram of the electrospinning apparatus is shown in Figure 7. Factorial experiment was designed to investigate and identify the effects of parameters on fiber diameter and morphology (Table 2).

Characterization

Shear viscosities of the fluids were measured at shear rate of 500 s^{-1} and 22 °C using a Brookfield viscometer (DVII+, USA). Fiber formation and morphology of the electrospun PANI/PAN fibers were determined using a scanning electron microscope (SEM) Philips XL-30A (Holland). Small section of the prepared samples was placed on SEM sample holder and then coated with gold by a BAL-TEC SCD 005 sputter coater. The diameter of electrospun fibers was measured with image analyzer

software (manual microstructure distance measurement). For each experiment, average fiber diameter and distribution were determined from about 100 measurements of the random fibers. Electrical conductivity of the electrospun mats was measured by the standard four point probe method after doping with HCl vapor.

Tab. 2. Factorial design of experiment.

Factor	Factor level
PANI Content(wt%)	10,20,30
Electrospinning temperature(°C)	25,50,75
Applied voltage(kV)	20,25,30

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