STRUCTURAL AND CHARACTERIZATION POLYACRYLONITRILE NANOFIBER FOR AIR FILTRATION ASSEMBLED BY ELECTROSPINNING

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Abstract

In electrospinning, the structure of nanofibers, which is affected by polymer solution parameters and processing conditions, influences the physical characteristics of nanofiber mats. In this study, under optimum conditions of electrospinning, the concentration of polyacrylonitrile (PAN) was changed 14, 16 and 18 wt %, and its effects on the nanofiber diameter and pore size of nanofiber mats were studied. The results showed that increasing the PAN polymer concentration enhanced the nanofiber diameter and pore diameter of nanofiber mats.

Keywords: *electrospinning; concentrations; pore size*

1. INTRODUCTION

Microfiltration is an important operation for biopharmaceutical processes, membrane pre-treatment, water and air purification, and food and beverage applications. The majority of commercial microfiltering media are inherently inhomogeneous (non-uniform in mass and thickness) at all locations. This affects the operational performance of the filtering media. Often the developmental objectives during fabrication of efficient microfiltration membranes are permeability, filtration performance and attaining uniformity in structure [Ma and Ramakrishna, 2008; Sang et al, 2008; Bjorge et al, 2009]. Recently, there have been a few attempts to prepare microfilter from nanofibers [Bazargan et al, 2010; Gopal et al, 2006; Homaeigohar, 2010]. Nanofiber mats offer unique properties such as high specific surface area (ranging from 1–35m2/g depending on the diameter of the fibers), good interconnectivity of the pores and the potential to incorporate active chemistry or functionality on a nanoscale [Ramakrishna et al, 2005; Reneker and Fong, 2006]. Unlike conventional fiber spinning techniques (wet spinning, dry spinning, melt spinning, gel spinning), which are capable of producing polymer fibers with diameters down to the micro meter range, electrostatic spinning, or 'electrospinning' is a process capable of producing polymer fibers in the nanometer diameter range [Chronakis, 2005; Theron et al, 2004; Pant et al, 2010; Homayoni, 2009]. Electrospinning is a novel and efficient fabrication process that can be utilized to assemble fibrous polymer mats composed of fiber diameters ranging from several microns down to fibers with diameter lower than 100 nm. This electrostatic processing method uses a high voltage electric field to form solid fibers from a polymeric fluid stream (solution or melt) delivered through a millimeter-scale nozzle. Nanofibers are the ultrafine solid fibers notable for their very small diameters (lower than 100 nm), their large surface area per unit mass and small pore size. Due to the inherent properties of the electrospinning process, which can control the deposition of polymer fibers onto a target substrate, nanofibers with complex, and seamless three-dimensional shapes could be formed. Construction of nanoscale composite fibers by electrospinning from a mixture of rigid rod polymer sand flexible polymers is also feasible. The electrospun nanofibers can even be aligned to construct unique functional nanostructures such as nanotubes and nanowires. Furthermore, depending on the specific polymer being used, a wide range of fabric properties such as strength, weight and porosity, surface functionality etc.can be achieved. The challenges confronted during fabrication of nanofiber mat by electrospinning are attaining: (1) homogeneity in the size (diameter) distribution of fibers in the mat, (2) uniformity in the deposition and orientation of fibers in the mat (thickness and structural indexes) and (3) durability of the fiber layers in the nanofibrous mat. The understanding the distribution, deposition and orientation of nanofibers would be extremely useful for preparation of uniform nanofibrous microfilters.

Hence, this investigation is aimed at studying the orientation of nanofibers in the electrospun mat and correlating the diameter of the nanofibers with the pore size of the mat.

2 EXPERIMENTAL
2.1 Materials
Polyacrylonitrile (PAN), N, N-dimethylformamide (DMF) and acrylamide (AM) were obtained from Aldrich Chemical and were used without further purification. Dope solutions were prepared by dispersing predetermined amount of silica nanoparticles (1 wt.% to PAN) into 14, 16 and 18 wt.% PAN solution in DMF. The dope was mechanically stirred for at least 24 h at 60 °C in order to obtain homogeneous silica dispersed PAN solutions [Mataram et al, 2010].

2.2 Method
The experimental set-up used for the preparation of nanofiber mat. A 50 mL plastic syringe was used to hold the electrospinning solution. The polyacrylonitrile solution was pumped at a constant rate of 2 mL/h with the help of a metering pump through a stainless steel needle of inner diameter 0.8 mm. A drum of diameter of 15 cm and length of 35 cm, connected to a variable speed motor, was used to collect the nanofibers. A high DC voltage was applied to the needle with the help of high voltage regulated DC power supply (Model ES 30P-5W, Gamma High Voltage Research, Ormond Beach, FL, USA). The applied voltage was limited at 21 kV and distance between the tip of the needle and the surface of the drum at 10 cm. The collecting drum was ground so as to generate the desirable electric field strength between the tip of the spinneret and the collector surface. The nanofibrous mat was carefully removed from the collector, and the residual solvent associated with nanofibers mat was removed by keeping the mat in oven for at least 2 days at 20 °C. The dried electrospun mats were stored in desiccators.

2.3 Fibers characterizations
Scanning Electron Microscopy (SEM) was used to measurements of PAN fibers. The pore size of the PAN Nanofibers membrane was determined using the bubble-point method. It is based on the measurement of pressure necessary to blow air through a liquid-filled membrane. The schematic of this is shown in Figure 1. The wet membrane at the end of the permeation experiment was placed in the cell supporting 5 cm^3 of distilled water and connected to a bubble-flow meter. Pressure was applied to the membrane base. At each pressure, the corresponding bubble (air) flow rate was measured. The relationship between the pore size and the corresponding pressure is given by the Young-Laplace equation [Mulder, 2003]:

$$\overline{} \tag{1}$$

where R is radius of the pore, ΔP differential pressure, γ the surface tension of the wetting agent and θ the wetting angle.

The static contact angle of the heat-treated Membrane was measured using a contact angle analysis system (VCA Optima, AST Products Inc, USA). 0.5 ml water droplet was dispensed on the membrane and the resultant angle measured.
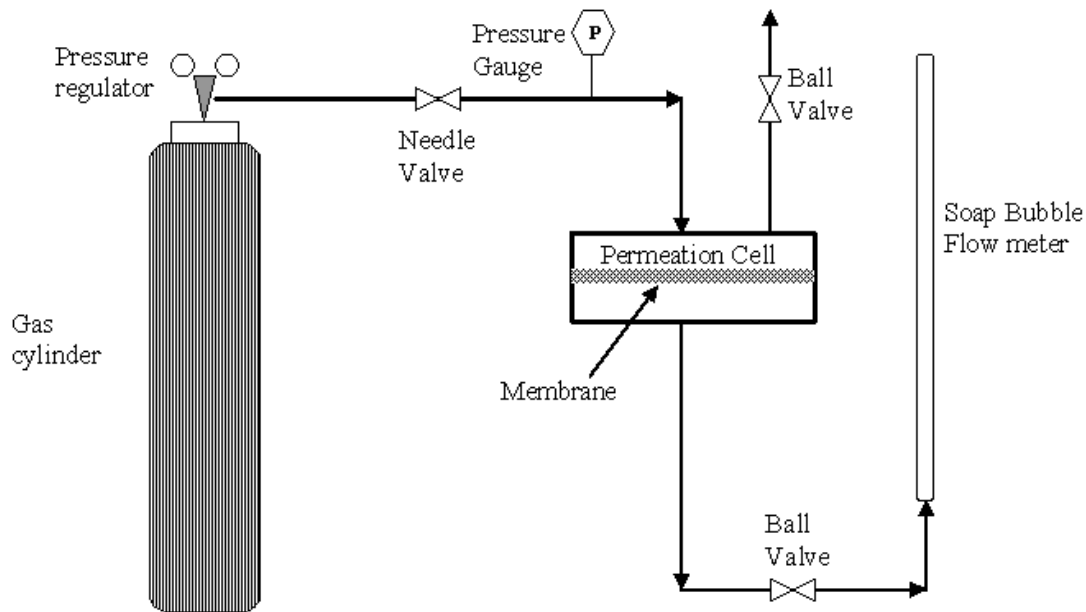
Figure 1. Bubble Point Method

3 RESULTS AND DISCUSSIONS

The molecule weight was increased from 14 to 18 wt.% while keeping a fixed feed rate (2 mL/h), rotational speed of collector (1.8 m/s), collecting distance (0.1 m) and electric field 21 kV. The average fiber diameter (calculated from 3 to 5 measurements using at least three SEM pictures of 1000× magnification) remained unchanged (Table 1). A shifting trend in the fiber distribution towards a higher fiber size was noted, and this indicates that the fiber diameter slightly increases as the molecule weight increases. The effect of molecule weight on fiber diameter is expected to be prominent in the case of highly viscous feed solutions. There was no noticeable change in thickness when the molecule weight was increased. The morphological changes in the membranes due to variation of the molecule weight are shown in Figure 2. Reduced number of beaded fibers and smaller sized beads were observed when the molecule weight was 14 wt.%. This finding is consistent with reports in the literature, that a low molecule weight favors the whipping instability and suppresses the axisymmetric instabilities, thereby suppressing the formation of beaded fibers [Zuo et al, 2005].

The drawing rate of the nanofibers can be enhanced by decreasing the molecule weight. It was hypothesized that an enhanced drawing rate would increase the number of fiber crossings and that the high extent of fiber crossing would reduce the pore size and improve the interconnectivity of pores. The pore size was measured to examine this hypothesis. Table 1 shows the effect of molecule weight on pore size. It was observed that the pore size decreased as the molecule weight decreased. This enhances repulsion between the fibers and changes the trend in fiber arrangement as evinced from the increased the pore size values. A considerable increase in accumulation of charge on electrospun polyacrylonitrile fibers has been noted when the molecule weight was increased [Kalayci et al, 2005].

Table 1 Effect of concentration of dope on the fiber diameter and pore size

No	Concentration of dope (wt.%)	Fiber diameter (µm)	Pore size (µm)
1	14	2,47 (0,38)	74,74
2	16	3,71 (0,93)	128,85
3	18	5,22 (0,38)	280,46

(14wt.%)

(16wt.%)

(18wt.%)
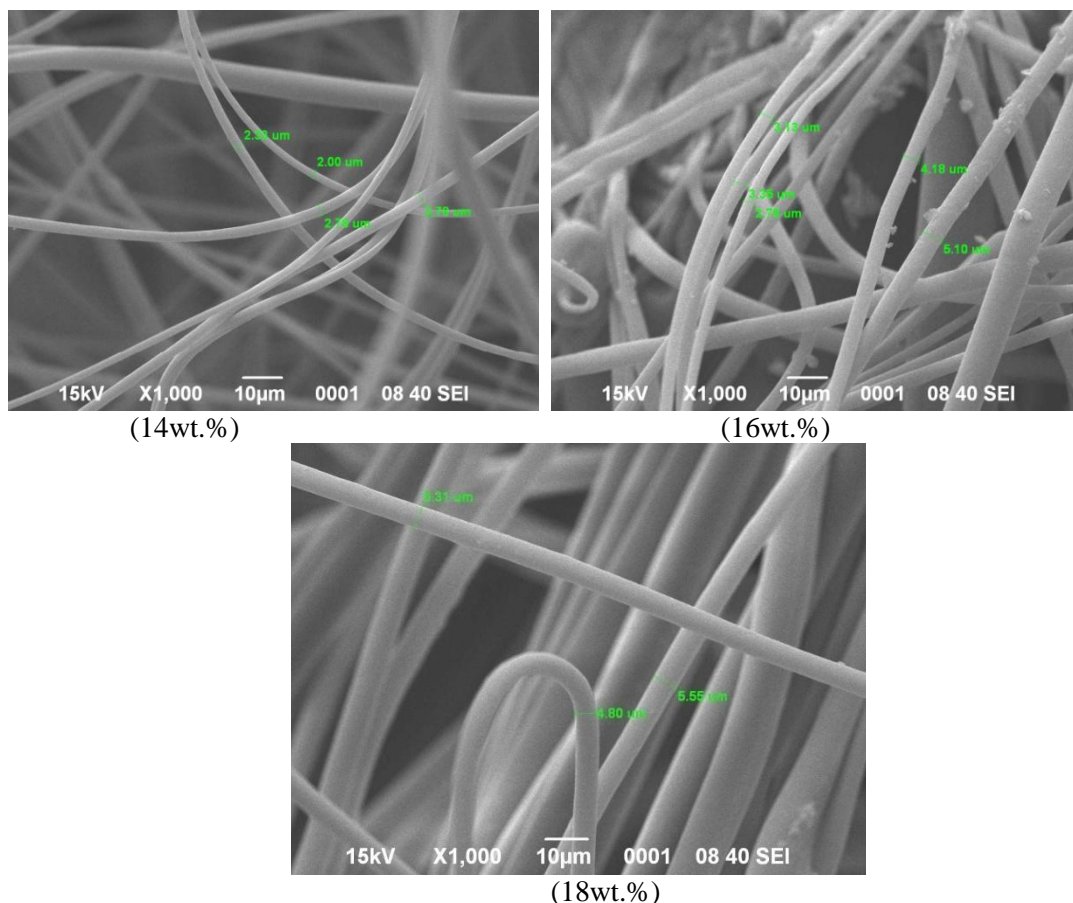
Figure 2 SEM micrograph show that diameter of fibers due to varying molecule weight

4 CONCLUSIONS

The fiber crossing and pore size can be optimized to attain improved structural (pore size distribution, pore interconnectivity and porosity) of the electrospun filtering media. Reduced number of beaded fibers and smaller sized beads were observed when the molecule weight was 14 wt.%. This finding is consistent with reports in the literature, that a low molecule weight favors the whipping instability and suppresses the axisymmetric instabilities, thereby suppressing the formation of beaded fibers. The pore size decreased as the molecule weight decreased. This enhances repulsion between the fibers and changes the trend in fiber arrangement as evinced from the increased the pore size values. A considerable increase in accumulation of charge on electrospun polyacrylonitrile fibers has been noted when the molecule weight was increased. In electrospinning, the structure of nanofibers, this is affected by polymer solution parameters and processing conditions, influences the physical characteristics of nanofiber mats. In this study, under optimum conditions of electrospinning, the concentration of polyacrylonitrile (PAN) was changed 14, 16 and 18 wt %, and its effects on the nanofiber diameter and pore size of nanofiber mats were studied. These demonstrate that control over the pore size distribution can be achieved by coordinating the drawing and collection rates. The results showed that increasing the PAN polymer concentration enhanced the nanofiber diameter and pore diameter of nanofiber mats.

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