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Effect of polymer concentration on morphology of polyacrylonitrile (PAN) nanofibers prepared by electrospinning

R.Sandhya, P.K.Panda*
Materials Science Division, CSIR-National Aerospace Laboratories, Bangalore-560017, (INDIA)
E-mail : pkpanda@nal.res.in

ABSTRACT

Polyacrylonitrile (PAN) nanofibers were prepared by electrospinning homogeneous viscous solutions of PAN in DMF of concentrations 9%, 12% and 16% (by wt.). The electrospinning was carried out at 9kV DC with tip to collector distance (TCD) of 7cm. The morphology of nanofiber webs characterized by scanning electron microscope (SEM) indicates smooth and cylindrical nature of fibers with aspect ratio >1000. The fiber diameter was found in the range of 50-320 nm and the average fiber diameter increases linearly with PAN concentration. The morphology of nanofibers was good for higher PAN concentration while some beaded or swollen structures were present in fibers prepared at low PAN concentration.

KEYWORDS

Polyacrylonitrile (PAN);
DMF;
Nanofibers;
Electrospinning;
Morphology.

INTRODUCTION

Carbon fiber is a very important structural material due to its light weight, high specific strength and modulus, high thermal and corrosion resistance etc. Therefore, carbon fibers are used for variety of applications such as in aerospace, automobile, chemical industry, general engineering, missiles, as reinforcement in composite materials and textiles[1]. Polyacrylonitrile (PAN) is used as the precursor for preparation of carbon fibers[2]. The fibers required stabilization for conversion of linear polymer to a cyclized, highly condensed thermally stable structure. The long heating time required for stabilization and carbonization of the precursor fibers add to the high cost of carbon fiber production. Oxidation occurs simultaneously with the cyclization reaction and is a diffusion controlled process. The rate of oxidation depends upon the diameter and chemical composition of the fiber in addition to the temperature and atmosphere. A decrease in the fiber diameter of the precursor fiber would result in a decreased stabilization time[3-4]. For example, the stabilization time can be 1000 times faster if the diameter of the precursor fibers reduced from few mm in case of conventional production to few nanometers by electrospinning method. Also, the fibers prepared by electrospinning have diameters so small that skin-core effects caused by differential stabilization are eliminated.

Electrospinning is a very simple and versatile technique for synthesizing nanofibers or fiber mats from broad range of organic polymers[5-15]. The very large surface area to volume ratio, flexibility in surface functionalities, superior mechanical performance and versatility of design are some of the characteristics that make the polymer nanofibers optimal candidates for many important applications such as composites, pro-

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tective clothing, catalysis, electronics, bio-medicine including tissue engineering, implants, wound dressing membranes, drug delivery, vessel engineering, ultra filtration and agriculture. The process involves the application of a strong electrostatic field to a polymer solution or a homogeneous polymer and oxide precursor sol placed in a container with a metal capillary such as a syringe with metallic needle. The positive terminal of a high DC source is connected to the needle while the negative terminal is connected to the counter electrode, in the form of a metal plate or aluminium foil placed at a distance of about 10cm. Under the influence of the electrostatic field, the solution experiences repulsive force. As the voltage surpasses a threshold value, electrostatic forces overcome the surface tension, and a fine charged jet is ejected. The jet moves towards the counter electrode, subdivides into large numbers due to high repulsive force, finally deposits in the form of nanofibers on the counter electrode.

The concentrations of polymer phase in the solvent influence morphology of nanofibers. A detailed study on the morphology of PAN nanofibers as a function of its concentration in solution is missing. Therefore, efforts are made in this paper to study the morphology of PAN fibers at different PAN concentration and their size distribution.

EXPERIMENTAL

Materials

Poly acrylonitrile [$(C_3H_3N)_n$] powders with average molecular weight of ~1,50,000 and N,N dimethyl formamide (DMF) were procured from M/s. Sigma-Aldrich. The above materials were used as starting materials.

Preparation of homogeneous viscous PAN solutions

Three different solutions of PAN in DMF such as 9%, 12% and 16% were prepared by dissolving 0.9g, 1.2g and 1.6g of PAN in 9cc of DMF respectively. The homogeneous viscous solutions were obtained after constant stirring for 2h.

Electrospinning of PAN nanofibers

About 5 cc of the viscous PAN solution was taken in a syringe with fine capillary metallic needle. The electrospinning was carried out by maintaining Tip to Collector (TCD) distance of 7 cm and at a DC voltage of 9 kV. The positive terminal of the high voltage source was connected to the metallic needle and the negative terminal connected to the flat metallic plate covered with aluminum foil. The flow rate of the solution was maintained at 1.3 mL / 1 h and the humidity of the chamber was maintained in the range of 50–60%. The voltage was gradually increased till the liquid came out through the needle and split into web of fibers collected on the aluminum foil. A schematic drawing of the electrospinning set up is presented in Figure 1. The solution preparation methodology and electrospinning conditions are summarized in TABLE 1.

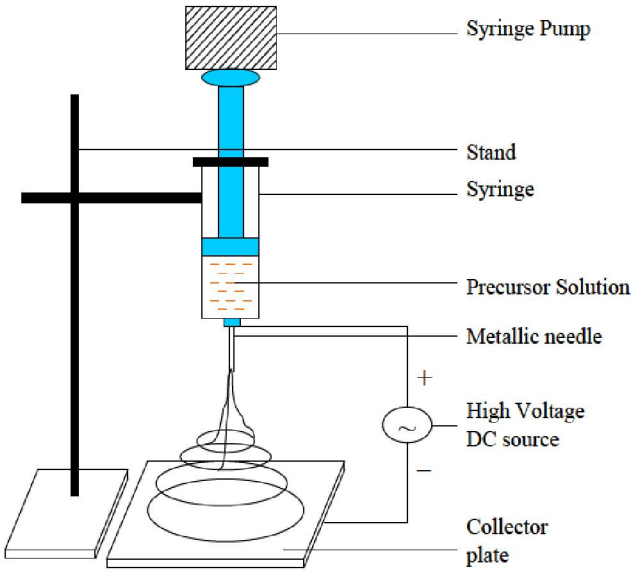

Figure 1 : A schematic diagram of electrospinning set-up.

CHARACTERIZATION

The morphology of as-spun nanofibers was observed under a scanning electron microscope (Carl Zeiss Supra 40 VP, Germany) after vacuum coated with a thin layer of gold. The morphology of the as-spun PAN nanofibers and their distribution of diameter with variation in PAN concentration are shown in Figure 2(a-c). The average fiber diameter and the distribution of fibers were determined using ~ 20 randomly selected fibers taken from SEM micrograph. The average fiber diameter vs. concentration of PAN solutions is presented in Figure 3.

TABLE 1 : PAN solution preparation and electrospinning conditions.

Chemical precursors	Preparation of Solution	Electrospinning Conditions
(i) Poly-acrylonitrile (PAN) (Mw, ~1,50,000) (ii) Dimethyl formamide (DMF)	9%, 12% and 16% PAN solutions prepared in DMF at room temperature with constant stirring for 2h.	Nozzle dia. (internal): 0.5mm, Tip to collector distance (TCD): 60-70mm, Voltage:9kV, Humidity: 50-60%

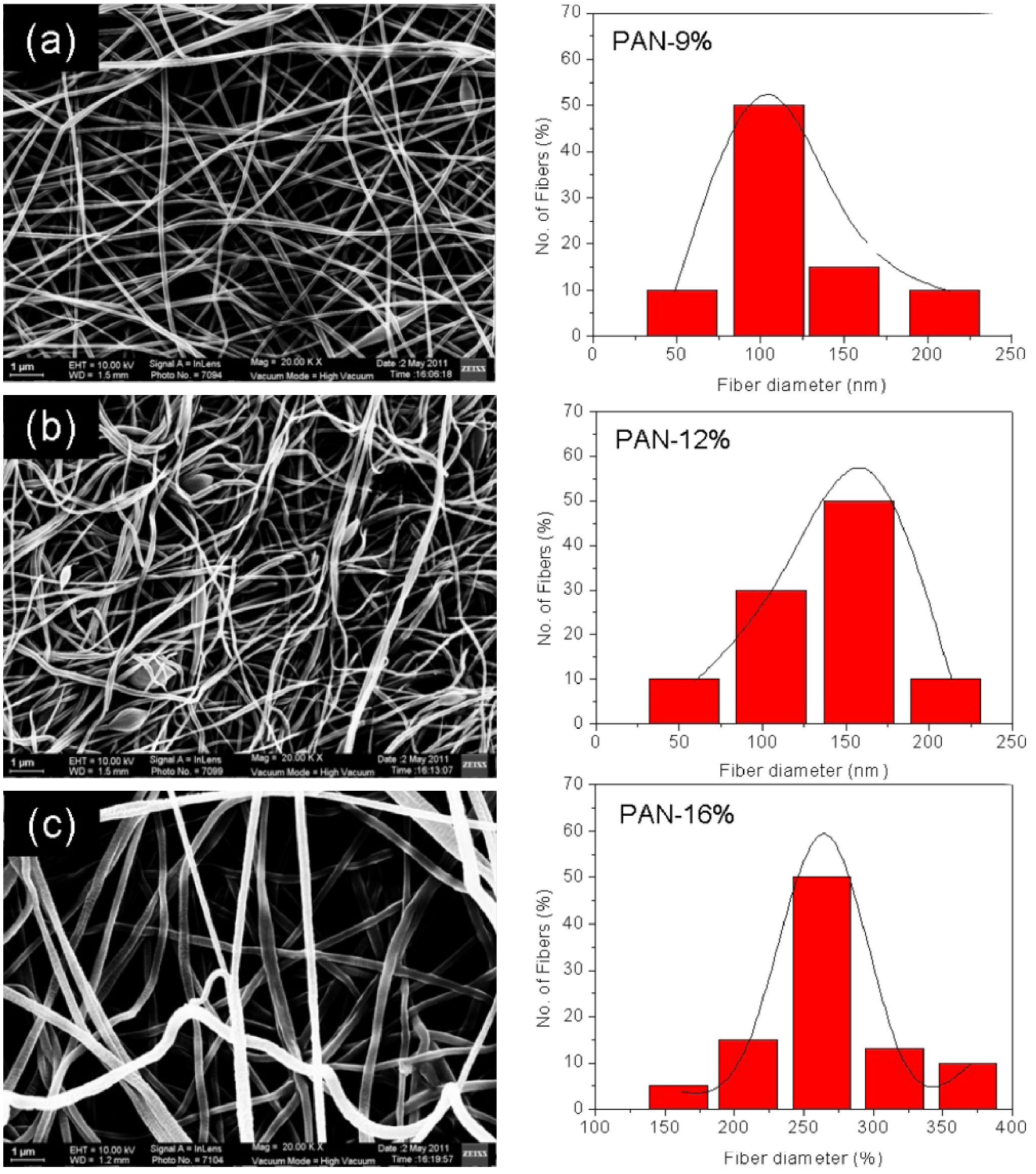

Figure 2 : SEM micrographs of PAN nanofibers and their diameter distributions at (a) 9 %, (b) 12 % and (c) 16 % of PAN solution in DMF.

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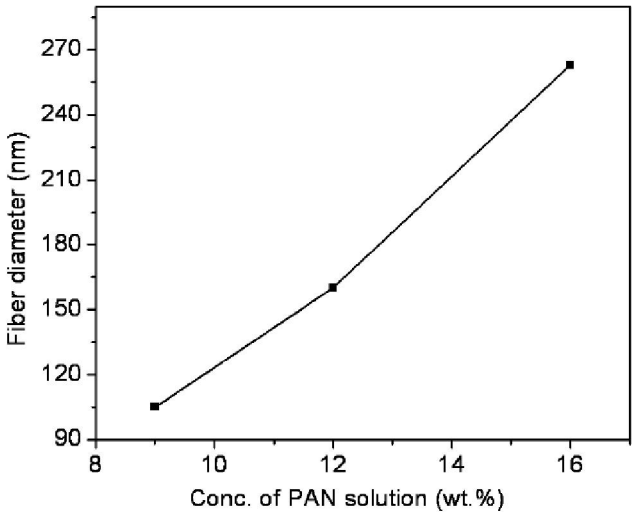

Figure 3 : Fiber diameter Vs. conc. of PAN solution.

RESULTS AND DISCUSSIONS

From the SEM photographs Figure 2(a-c), it is observed that the fibers are cylindrical and are smooth due to its amorphous nature. The average fiber diameter increases with PAN concentration (Figure 3). For example, the fiber diameter increases from 105 ± 25 nm for 9% PAN to 263 ± 25 nm for 16% PAN concentration. This is in similar line of observation made by Ji-Huan He et al.[14]. This is due to high viscosity of the solution at higher PAN concentration. In general, the morphology of fibers and smoothness are better for higher % PAN solutions due to higher content of organics. At lower PAN concentration, presence of beaded or bulb structures was noticed.

CONCLUSIONS

Polyacrylonitrile (PAN) polymer nanofibers were successfully prepared by electrospinning technique using three different solution concentrations of PAN in DMF. The fibers were found cylindrical with smooth surface and the aspect ratio was more than 1000. The average fiber diameter increased linearly with the increase in PAN concentration. The morphology of nanofibers was good for higher PAN concentration while some beaded or swollen structures were present for lower PAN concentration.

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