ELECTROSPINNING OF POLYAMIDE 6 NANOFIBER USING WIRE ELECTRODES

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ABSTRACT

Electrospinning is one of the well accepted method to spin the fibers with diameter in nanometer range. In this spinning method, fiber spinning takes place between the electric field created by two electrodes. Various parameters such as polymer viscosity, potential difference, relative humidity and temperature of environment plays important role to decide fiber morphology and diameter. From the technology point of view, there are two types of instrument available i.e. needle type and needle less type. In the needle less method electrode may be cylinder or wire. Effect of parameters on needle type spinning method have been reported by many researchers. In this case, effect of different parameters on electrospinning of Nylon 6 using needle less electro spinning method with wire electrodes have been investigated. Fiber quality such as morphology and diameter have been characterized by scanning electron microscope (SEM). Pore size of the nanofiber mat has been analyzed by using porometer.

1. INTRODUCTION

Fiber having diameter 1 µm (1000 nm) or less, with aspect ratio more than 100:1 is known as nanofiber. Because of its extremely small features nanofibres have great potential to be used in various fields such as filtration, tissue engineering scaffolds, sensors, affinity membranes, catalyst & enzyme carriers, energy storage, release control and recovery of metal ions. A variety of processing techniques such as drawing [1], template synthesis [2,3], template melt extrusion or template assisted extrusion [4], phase separation [5], interfacial polymerization [6,7], self-assembly [8,9], melt blown [10], force spinning [11,12], and electrospinning have been used to prepare polymer nanofibers. All these methods have their own advantage, but electrospinning is a common and a fast-developing technique for the production of nanofibres due to its simplicity, efficiency and cost-effective setup. The industrial and scientific interest of electrospunnanofibre is due to its long length, small diameter and pore size [13-15]. During electrospinning, it is possible to control the fiber diameter and pore characteristics [26, 17]. Theelectrospinning setup consists of two electrodes of opposite polarity. One electrode is placed into the solution and another one on to a collector. Polymer nanofibres were formed from the solution by electrostatic forces between two electrodes of opposite polarity [16-20].The traditional electrospinning setup consists of a needle/nozzle in its jet outlet [21]. In recent years, electrospinning set up has improved much. Multiple needle electrospinning has been developed for enhancing the production rate. But the probability to encounter clogging and the electric field interferences generated by the numerous spinning heads are the disadvantages of multiple needle electrospinningsystem [22]. Needleless electrospinning system has overcome these problems. It has been found encouraging results for bulk scale production of nanowebs due to its multiple fiber forming jet formation of a suitable support (Rotating roller/ metal edge/ wire) from a polymer solution [23].

Process parameters such as polymer concentration, potential difference between electrodes, relative humidity, distance between the electrodes and chamber temperature plays important role during electrospinning. Effect of those parameters on fiber quality needle spinning method has been studied and reported by many researchers [24-26]. In the case of needleless technology, some parameters behave differently so this study is an attempt to study the effect of different parameters during the spinning of Nylon 6 using wire electrode.

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2.0 EXPERIMENTAL

2.1 Materials

Nylon 6 is selected for electrospinning, was purchased from the local market of Mumbai (India). Acetic acid (MW 60.05 g/Mol) and formic acid (MW 46.05 g/Mol) obtained from Merck life science PvtLtd., Mumbai (India), was used as it is without further purification. Polypropylene spunbonded non-woven fabric was procured from Techfab (India) Industries Ltd, Daman (U.T). and the Marcy land, soil was procured from soil lab, BTRA.

2.2 Methods

Spinning solution preparation

The measured amount of solvents like Acetic acid and formic acid 2:1, was taken in a conical flask. The polymer was added slowly with 500-700 RPM stirrer speed and set the temperature of the heating plate at 70˚C. The solution was heated and stirred until the polymer dissolved.

Electrospinning

The nanofibrous mats were prepared using electrospinning machine from ELMARCO (NS IS500 U) needleless technology.Nylon 6 nanofibrous mats were deposited on the spunbondedpolypropylene fabric, for preparing uniform fibers without beads, the parameters such as concentration of polymer, bottom electrode voltage, top electrode voltage, electrode distance and relative humidity was studied.

2.3 SEM analysis

Morphology of Nylon 6 nanofibrous mats was examined by Scanning Electron Microscope (SEM JEOL JSM 5400) a voltage of 15kV. All samples were sputter coated with gold prior to SEM analysis. Fiber diameter was measured by image software called Image J (NIH, http://rsbweb.nih.gov/ij/), based on images obtain from SEM analysis. As many as 150 fibers were selected for each sample on different positions. Nanofiber diameter of each sample was estimated from statistics from those fibers.

Pore Size Analysis

Quantachrome's 3G porometer with standard test method ASTM D 6767, operating under windows ® the 3G win software was used for the analysis of pore size. This method is based on pore size characteristics of Geotextiles by capillary flow test. Atmospheric conditions were 21±2˚ C temperature and 60±5 % R.H.

3. RESULTS AND DISCUSSION

3.1 Effect of polymer concentration on Electro-spinning

Effect of polymer concentration on spinnability and fiber diameter was studied by varying the concentration of Nylon 6 in the solution from 11-18 wt%. During the spinning, other parameters such as distance between electrodes, voltage of top electrode, voltage of bottom electrode, width of deposition, carriage speed, RH %, temperature and deposition time were kept fixed. Fiber quality and morphology was investigated by SEM and diameter was measured by image J software. The SEM images of fiber at different concentrationand plot of the polymer concentration against diameter and pore are given in Figure 1 and Figure 2 respectively.In the large area,few beads were observed at 11% concentration whereas fibers were bead free from 12 to 18 wt% of polymer concentration. The diameter of the fibers found to be increased with an increase in polymer concentration. As pore size of the mat increases with increase in fiber diameter, for small pore size initial concentrations 11-14% was suitable but based on fiber morphology and uniformity of fiber diameter distribution, 13 wt% kept fixed for further set of experiments.

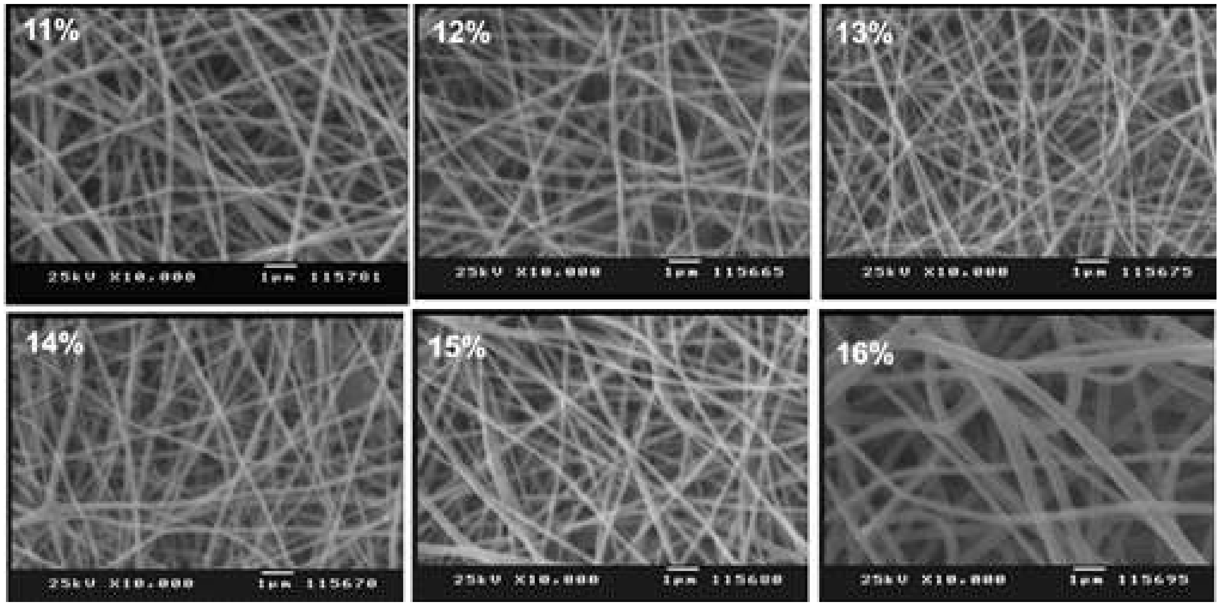
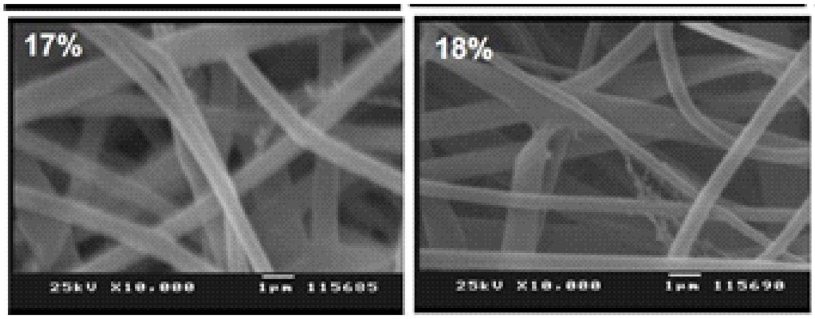


Figure 1: Scanning electron micrograph of nanofiber at different concentration of Nylon 6



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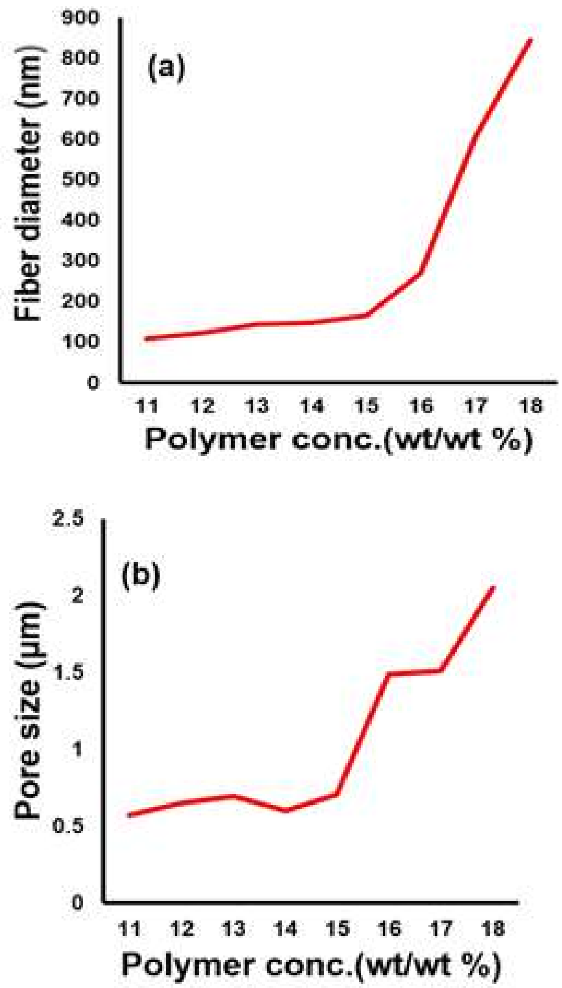

Figure 2: Effect of polymer concentration on (a) fiber diameter and (b) pore size of mat

3.2 Effect of positive voltage on fiber diameter and pore size

The effect of positive voltage on the diameter of the nanofiber and pore size of the deposited nanofiber mat was investigated by varying the voltages from 32.5 kV to 50 kV at interval of 2.5 kV. During spinning, other parameters such as concentration, -ve voltage, distance between electrode, relative humidity, temperature and deposition time a was kept fix at 13wt %, -15 kV, 160 mm, 45%, 21±2˚C and 5 min respectively. Fiber morphology was investigated by SEMand the diameter of the fiber was measured using image J software. SEM images at different voltages, plot of voltage verses fiber diameter and pore size are shown in Figure 3 and Figure 4 respectively. Morphology of the fibers found bead free at all the cases but decrease in diameter was observed up to 42.5 kV due to high stretching force on the spinning jet. Beyond the 42.5 kV again increase in fiber diameter was observed up to 50 kV due to reduction in flight time of spinning jet because of high potential difference. Though decrease in fiber diameter was found up to 42.5 kV but after 35 kV this decrease was not significant so 35 kV was considered as optimum and kept fix for further set of experiments.

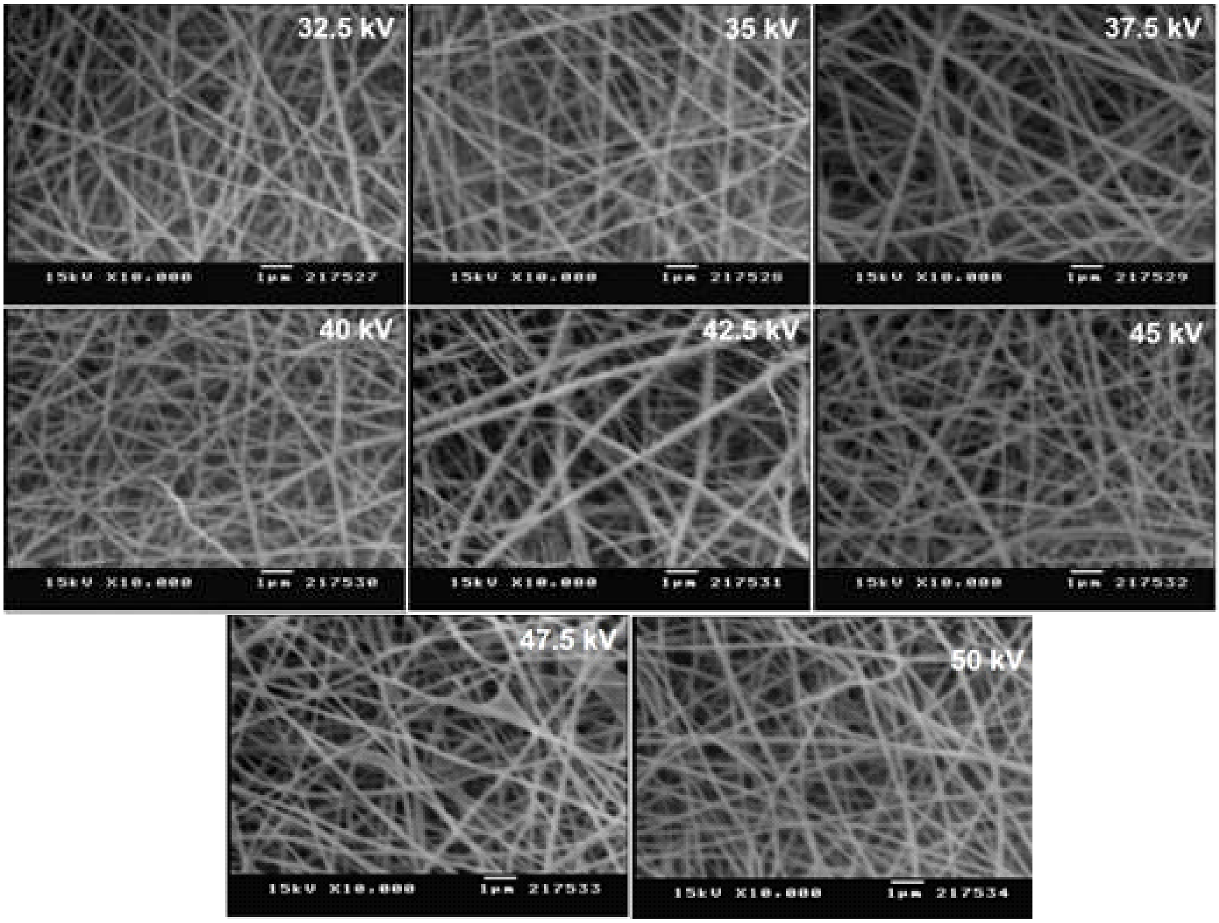

Figure 3: Scanning electron micrograph of nanofiber at different positive voltage

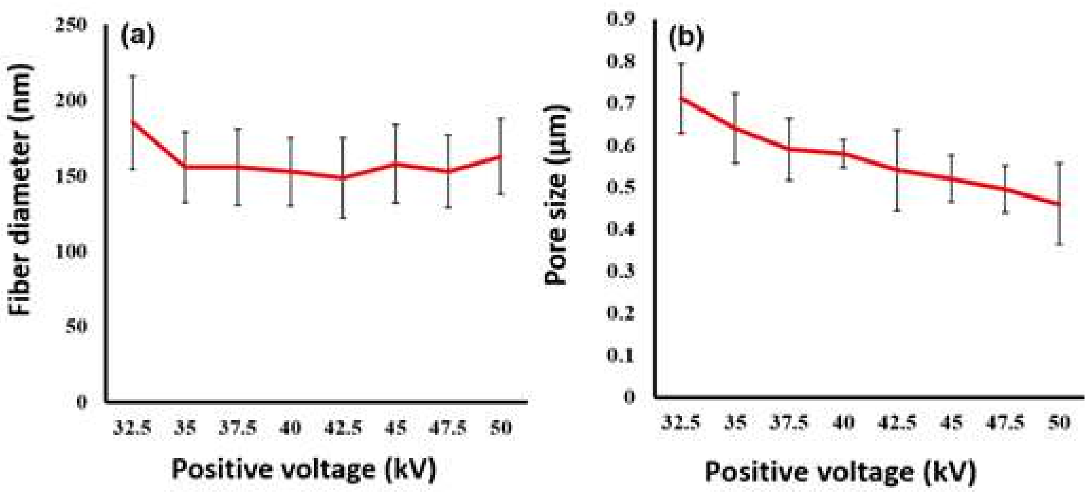

Figure 4: Effect of positive voltage on (a) fiber diameter and (b) pore size of mat

3.3 Effect of negative (collector) voltageon fiber diameter and pore size

Effect of negative voltage on spinnability, fiber quality and fiber diameter wasinvestigated by changing the negative voltage from 0 to 15 kV at interval of 2.5 kV. During this study, other parameters such as polymer concentration, positive voltage, distance between electrode, relative humidity, temperature and deposition time were kept fixed at 13 wt%, 35 kV, 160 mm, 45%, 21±2˚C and 5 min respectively. SEM images at different voltages, plot of voltage verses fiber diameter and pore size are shown in Figure 5 and Figure 6 respectively.

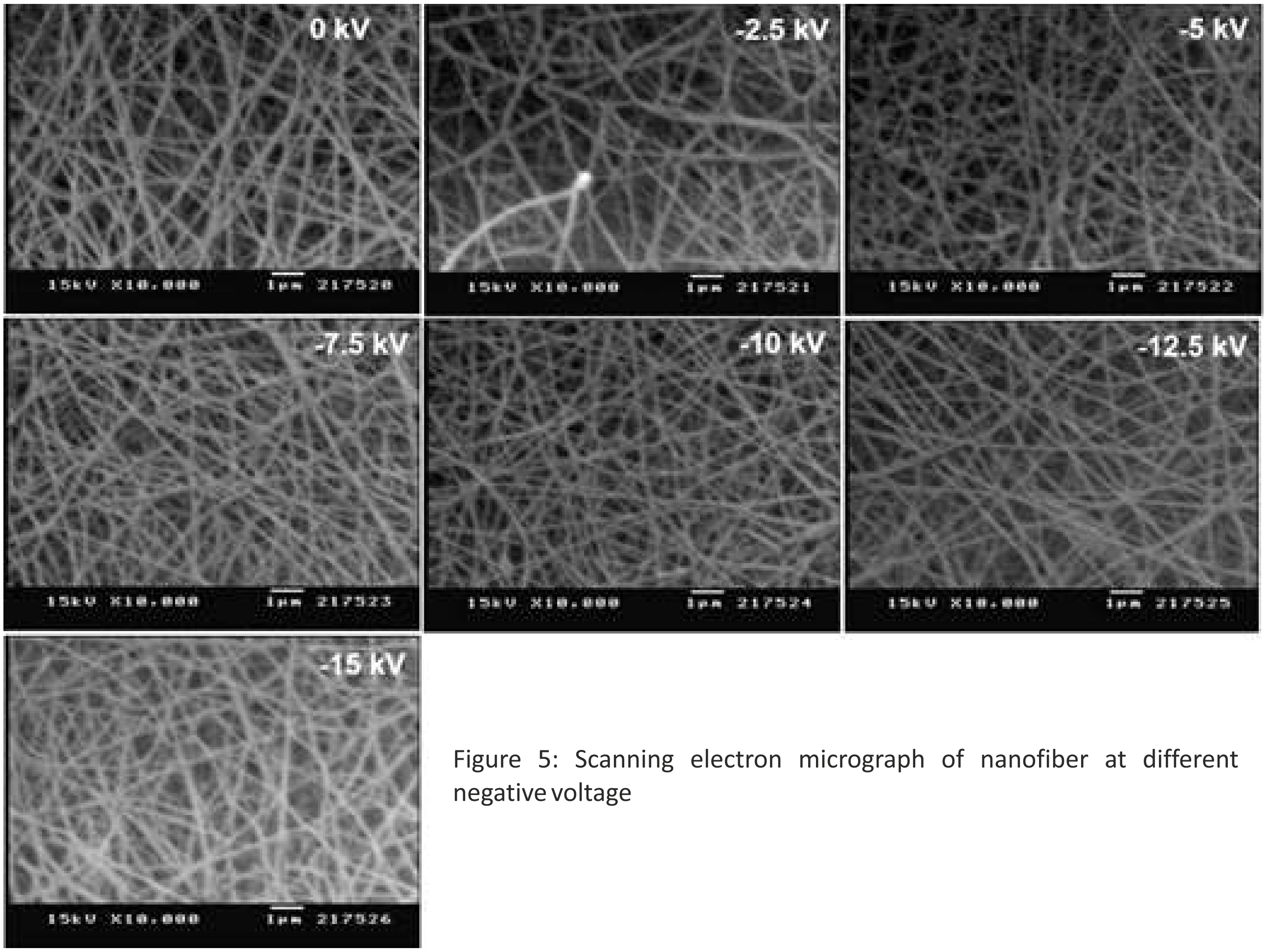

Figure 5: Scanning electron micrograph of nanofiber at different negative voltage

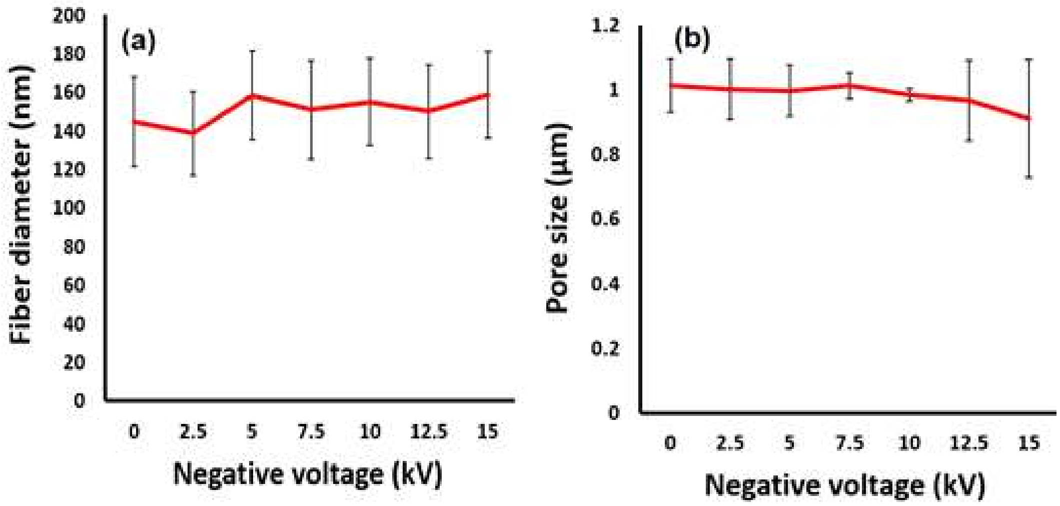

Figure 6: Effect of negative voltage on (a) fiber diameter and (b) pore size of mat

From the given data it can be inferred that there is no significant effect of negative voltage on fiber diameter, whereasslow decrease in pore size was observed with an increase in negative voltageafter -7.5 kV. Higher the collector voltage beyond a threshold value lowers the spread area so fiber density is high at that area. This increase in deposition density at higher collector voltage causing the reduction in pore size. To achieve the small pore size, -15 kV was kept fix for further set of experiments.

2.4 Effect of Distance between electrodes

Further the effect of distance between the two electrodes on fiber quality, diameter and pore size of the nanofiber mat during spinning was investigated by changing the distance from 130 to 190 mm at interval of 10 mm. During this study, other parameters such as concentration, bottom electrode voltage, top electrode voltage, relative humidity percentage, temperature and deposition time was kept fix at 13wt %, +35 kV,-15 kV,45%,21±2˚C and 5 min respectively. SEM images at different distance, plot of voltage verses fiber diameter and pore size are shown in Figure 7 and Figure 8 respectively.

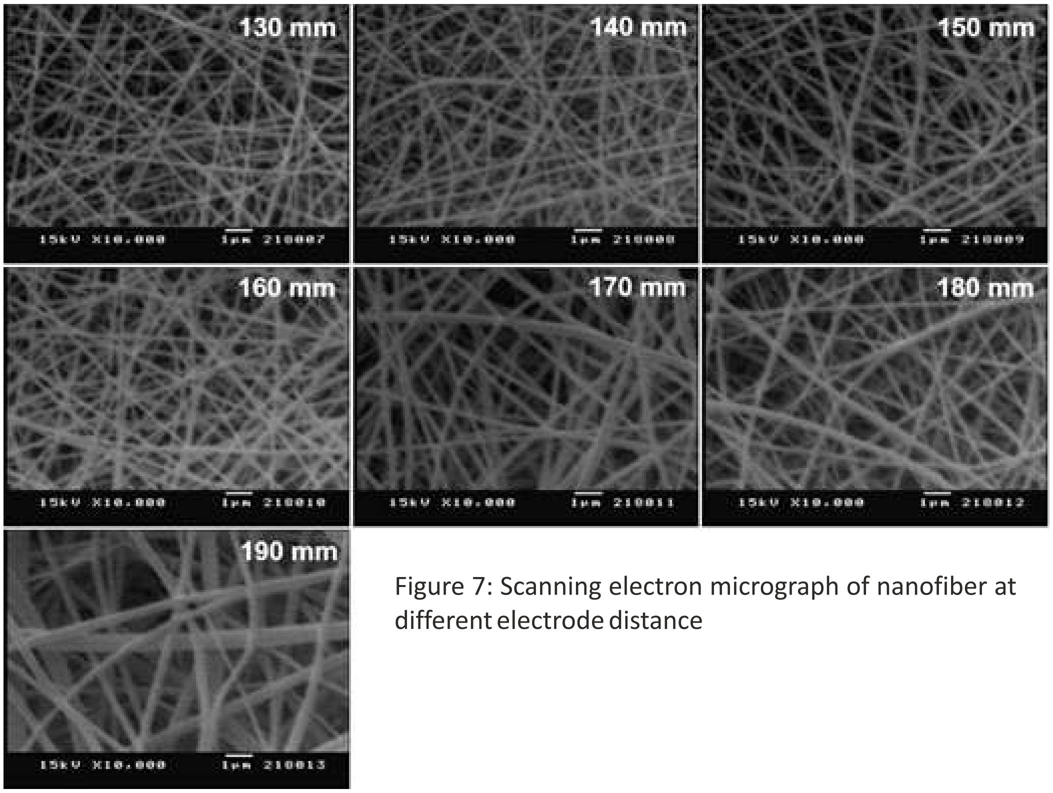

Figure 7: Scanning electron micrograph of nanofiber at different electrode distance

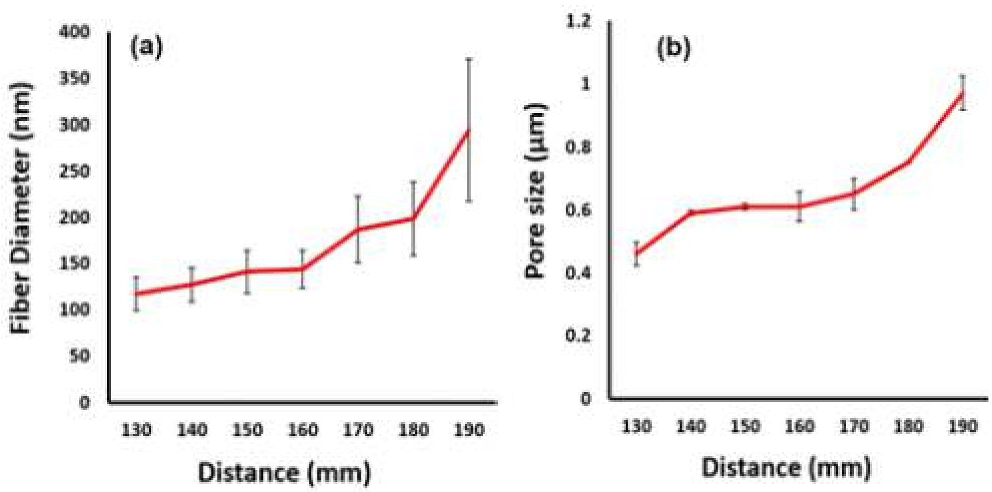

Figure 8: Effect of distance between electrode on (a) fiber diameter and (b) pore size of mat

When the voltage between electrode is fix, fiber diameter increases with an increase in distance between electrodes because strength of electric field reduces at higher distance which applies a low stretch on the solution. Increase in fiber diameter also leads to increase in pores size of the mat.

3.5 Effect of Relative Humidity

Effect of environmental relative humidity percentage on the spinning and fiber quality was investigated by changingrelative humidity percentage from 45 to 70 % inside the spinning chamber at interval of 5%.During this study, other parameters such as polymer concentration, positive electrode voltage, negative electrode voltage, distance between the electrodes, temperature and deposition time was kept fix at 13wt%, +35 kV, -15 kV, 130 mm,21±2˚C and 5 min respectively. SEM images at different RH%, plot of voltage verses fiber diameter and pore size are shown in Figure 9 and Figure 10 respectively.

Fiber quality and morphology was found similar in the above range of RH% because Nylon6 is well spinnable within that range. At 70% RH small increase in diameter and significant increase in pore size was observed. The charge transfer between environment and solvent also helps for the evaporation of solvent somore the water vapor more is the charge diffusion. Thus, the fibersolidifies before further elongation and causes thicker fiber.Pore size is increasing with the lower web density and web density of nanofibres generally reduces with increase in fiber diameter.

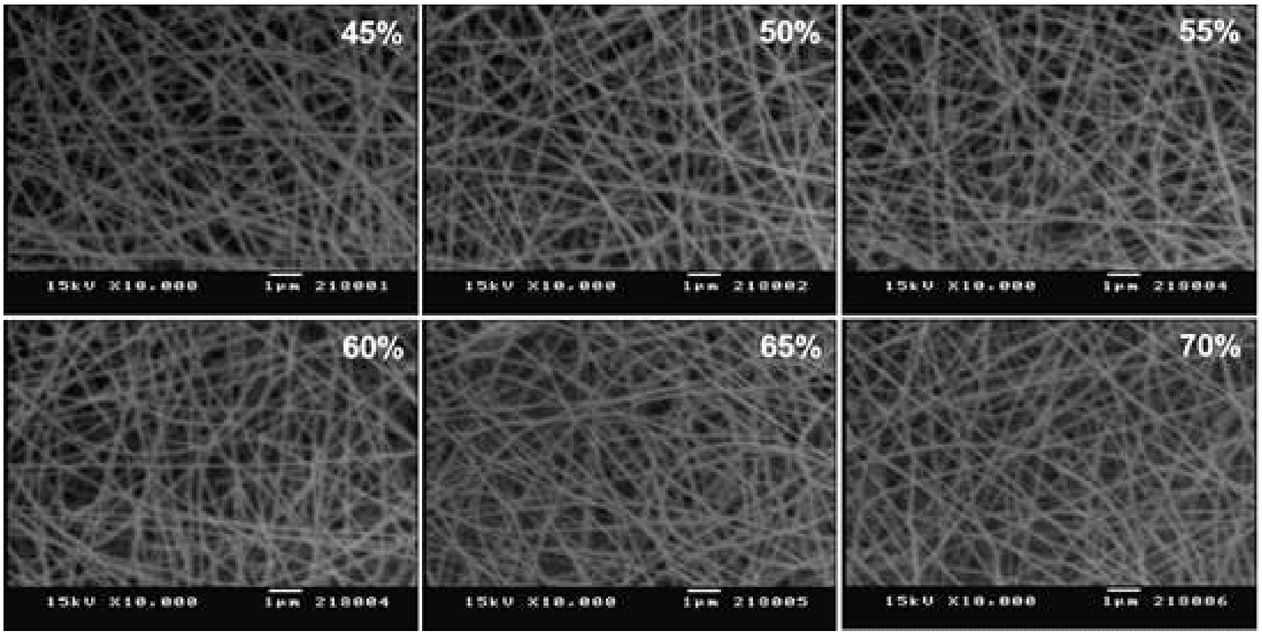

Figure 9: Scanning electron micrograph of nanofiber at different relative humidity percentage

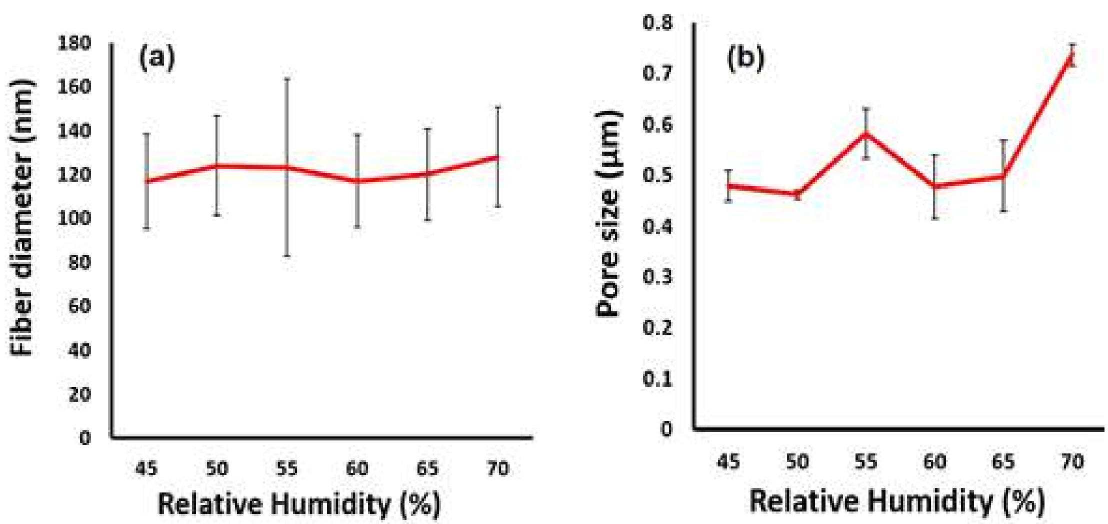

Figure 10: Effect of RH% on (a) fiber diameter and (b) pore size of mat

3.6 Effect of deposition time

The pore size of the nanofiber web depends on fineness of fiber and web density.To reduce the pore size up to minimum level, fiber diameter and web density need to be standardized. After standardization of related parameters to spin fine fibers, web density was standardized by changing the deposition time from 0.5 to 5 min. At 5 min of deposition pore size was found 0.5 µm compared to 48 µm in existing microfiber media. Pore size values at different time is given in Table 6 and plot of pore size against deposition time is given in Figure 13. There were small changes in pore size after 2 min of deposition.

Table 6. Pore size at different deposition time

Time of deposition (min)	Pore size (µm)
0.5	9.96
1.0	1.63
2.0	0.70
3.0	0.63
4.0	0.61
5.0	0.50

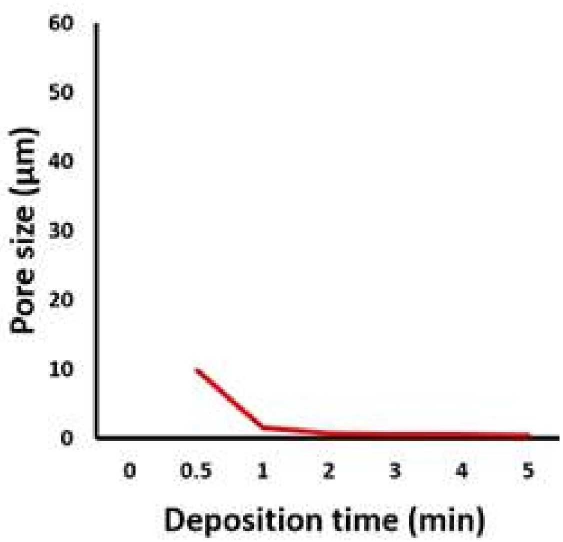

Figure 11. Effect of deposition time on pore size

4. CONCLUSION

Polyamide 6 solution in acetic acid and formic acid solvent system was successfully spun using electrospinning system with wire electrode. Increase in fiber diameter as well as pore size was observed with increase in polymer concentration from a certain percentage at which fiber morphology was found acceptable. At standardized concentration, increase in positive electrode voltage could not help to make the fiber finer after a threshold value due to the increase in force and reduction in time for molecular chain relaxation. There was no significant effect of negative electrode voltage on fiber diameter but decrease in pore size was observed at higher voltage due to less spreading of fibers. Increasing trend in fiber diameter was found by increasing the spinning distance because of reduction in charge density per unit volume. Polyamide 6 was found spinnable within a range of relative humidity. As usually reduction in pore size value was observed with increase in deposition time. For the used polymer at 13% concentration, positive voltage, negative voltage, distance between the electrodes and relative humidity was 35kV, -15kV, 130mm and 45to70% respectively for getting finer fiber and pore size.

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