Morphology, Mechanical Properties and Surface Characteristics of Electrospun Polyacrylonitrile (PAN) Nanofiber Mats

Zafarulla Khan, Feras Kafiah, Hafiz Zahid Shafi, Fayez Nufaiei, Sarfaraz Ahmed Furquan, Asif Matin

Abstract— This paper explores the effect of solution and electrospinning parameters on the morphology, mechanical properties and surface characteristics of Polyacrylonitrile (PAN) electrospun nanofiber mats. PAN/DMF (Dimethylformamide) solutions with different concentrations were electrospun under various parameters. The results show that the average fiber diameter increase from 208 nm to 881 nm with an increase in PAN concentration from 6 wt% to 12 wt%. Feed rate has inconsistent trend on the fiber diameter; however with increasing feed rate from 0.8 ml/hr to 1.4 ml/hr, the average fiber diameter more than doubledfrom400nm to 895nm. Average fiber diameter decreased slightly from 383 nm to 332 nm up to a certain threshold value of voltage, and then increased significantly to 750 nm when voltage was increased beyond this threshold. Somewhat surprisingly, when the distance between needle tip and collector was increased from 100mm to 150 mm, average fiber diameter increased almost four times (200 to 750 nm).Increasing the needle diameter was found to decrease average fiber diameter and has a direct effect on Taylor cone shape and jet length. The increase in PAN concentration from 6 to 12% increased the tensile strength, failure strength and ductility of electrospun nanofiber mats by 346%, 229% and 504%, respectively. PAN concentrations have a significant effect on the wettability of the nanofiber mats as determined by the contact angle measurements. The electrospun mats became increasingly more hydrophobic with increase in PAN concentration.

Index Terms—PAN, electrospinning, nanofiber morphology, solution and process variables, mat.

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I. INTRODUCTION

Electrospinning is considered as the most powerful technique to produce micro- and nanofibers [1],[2]. The nonwoven ultrafine fiber mats find applications in a vast range of engineering, biomedical and industrial fields such as membrane technology [3], filtration media [4],[5], energy-related needs (harvesting, transmitting and storage), tissue engineering [6]-[8], medical prostheses [9], drug delivery [10]-[12], and wound healing [11], [13], [14]. Fibers required in such applications may be produced by many techniques. Of these, electrospinning is a particularly simple, low cost and versatile method. Furthermore, this process gives a better flexibility in controlling the porous mat characteristic properties such as fiber diameter, porosity and fibers alignment. During electrospinning, a conical fluid structure called the Taylor cone[15] formed at the tip of the needle. The Taylor cone is an important feature of electrospinning as it allows the polymer solution to leave the tip of the needle and drawn rather down to a much smaller fiber diameter. This requires a critical voltage at which the repulsive force of the charged polymer overcomes the surface tension of the solution and a charged jet erupts from the tip of the Taylor cone. A phenomenon called Rayleigh instability occurs if the applied voltage is not high enough, under such a condition the jet will break up into droplets. If the voltage is sufficiently high, a stable jet will form near the tip of the Taylor cone. Beyond the stable region, the jet is subject to bending instability[16], that results in the polymer being deposited on the grounded collector via a whipping motion[17]. These stable shapes result only from equilibrium of the electric forces and surface tension in the cases of in viscid, Newtonian and viscoelastic liquids[18]. There is a multitude of process parameters that control the structural morphology, size, geometry and physical and mechanical properties of electrospun fibers. Fabrication of these fine fibers require careful consideration and control of various process parameters (such as applied voltage, solution feed rate, needle diameter, distance between the needle tip and the collector), material properties (polymer chemistry and molecular weight), solution properties (such as conductivity, viscosity and surface tension) and environmental parameters (humidity, temperature, air and vacuum in the spinning chamber). Almost any soluble polymer can be electrospun if its molecular weight is high enough. By appropriately selecting these parameters, wide range of materials including natural polymers, polymer blends, ceramic precursors and metal or metal oxides have been electrospun into submicron

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and nanofibers to produce different fiber morphologies, such as beaded [19],[20], ribbon [20],[21], porous [22],[23], core–shell [23], and aligned [24] fibers. Several researchers have investigated the effect of solution[25], [26] and process parameters [25], and found that polymer concentration, viscosity and solution conductivity play important role on the outcome of electrospinning. C.Henriques et al. [25], have found that a low concentration the solution led to the formation of beaded fibers where as an intermediate concentration yielded good quality fibers. Hazim J. Haroosh et al. [27], studied the effect a solution viscosity and conductivity and suggested that the viscosity of polymer solution played a dominant role when compared to electrical conductivity of solution. Amir Houshang at el. [26], found that solution concentration is an important parameter that affects the homogeneity of nanoweb and evenness of diameter of spun nanofiber. In the recent past, many studies have been conducted to evaluate and understand the effect of process parameters on the morphology of electrospun fibers and porous mats. These studies help to some extent but do not elucidate how exactly the process parameters affect the morphology of the mats. For example, it has been reported that increasing spinning voltage increases the fiber diameters of poly vinyl alcohol (PVA) [28], whereas Wannatong et al. [29], found that the diameter of electrospun polystyrene (PS) initially decreases with increasing voltage to a certain value, then increases with increasing the voltage. Another important electrospinning process parameter is the needle tip to collector distance (hereafter referred to as distance). Several researchers have investigated the effect of distance on the morphology and fiber diameter of electrospun mats and/or fibers, but the results are rather contradictory. For instance, Qiang Li et al. [30], found that varying distance have no obvious effects on fiber morphology of polyvinyl alcohol(PVA) nanofibers. They have, however, observed that the average fiber diameter slightly decrease with increased needle to collector distance. Bin Ding et al. [31], studied the effect of distance on the size of electrospun PVA micro/nano fibers and found that fiber diameter increases slightly from ~160 to ~250 nm with increased distance from 4 to 12 cm, respectively. Pitt Supaphol et al. [32], have investigated the effect of distance on the size of as-spun mats and morphology of as-spun fibers. Their results suggested that the size of spun mats increased with increasing distance whilst fiber diameters slightly decreased from 220nm to 160 nm with varying distance from 5 to 20 cm. They have also shown that at lower distance (5 cm) beaded fibers with fused fibers at the touching points were formed indicating incomplete drying whereas at higher distance (15 cm) smooth fibers without beads were obtained. Polyacrylonitrile (PAN) offers many excellent properties such as the higher hydrophobicity and insolubility in a wide range of solvents [5], [33], and is the most extensively used in electrospinning [34]. Although a number of researchers have investigated the electrospinning of PAN [34]-[41], a comprehensive work in which the effect of main parameters on the electrospun PAN nanofibers has not yet available in the literature. This work investigates the effect of key solution and process parameters on the morphological behavior, surface characteristics and mechanical properties of electrospun PAN nanofibers.

II. Experimental Procedure

A. Materials

Polyacrylonitrile (PAN) having Mw =150,000g/mol and density of 1.184 g/cc was supplied from Sigma Aldrich. N, N-Dimethylformamide (DMF) having density of 0.948g/cm3 supplied from Alfa Aesar was used as a solvent to dissolve PAN. Different concentrations 6, 8, 10 and 12% (wt/wt) of PAN-DMF solutions were prepared by magnetically stirring the mixture for one hour at 85 ºC until a homogeneous clear PAN-DMF Solution was obtained. Viscosity was calculated by gravity based rheology measurements using the Gilmont apparatus. A tantalum ball was used in measuring the time taken to travel the specified path due to gravity. Density of the tantalum ball was considered as 16.6 gm/ml, with the ball constant parameter of 0.35 for calculations.

B. Electrospinning

Electrospinning was carried out using NANON-A1 apparatus (Mechanics Electronics Computed Cooperation.LTD. Japan). The process started by preparing a syringe (5ml) with a specific amount of solution of different concentrations. Syringe was then fixed inside Electrospinning chamber and other process parameters (voltage to be applied, distance and feed rate, etc...) were finally adjusted before electrospinning commenced.

C. Morphological Characterization

PAN nanofiber mats produced were characterized by checking their morphologies and average fiber diameter. Morphology was observed after gold plating the samples by using SEM (Jeol 4600 instrument) at different magnifications. Average nanofiber diameter were determined from high magnification SEM micrographs using ImageJ software (http://rsb.info.nih.gov/ij/) in which, averages have been taken for around 250 reading of every mat and their standard deviations, minima and maxima were calculated.

D. Surface Property Characterization

Wettability of the electrospun polymeric mats was determined using the Kyowa-DM 501 contact angle instrument. De-ionized water was considered for contact angle measurement between the water droplet and the electrospun mat. A half angle sessile drop method was used for measuring the water contact angle. Static measurements and contact angle image were captured after 100 ms of the droplet being placed on the surface.

E. Mechanical Properties Characterization

Tensile tests of electrospun mats were carried out using Electroforce instrument (Bose). The samples were heat treated at 220°C for 30 minutes before the tensile test. The tests were carried out in accordance with ASTM D 1822. The samples were cut in a rectangular shape with a gauge length of 15mm. A 225 N load cell was considered for the tensile test study with a constant strain rate of 0.1 mm/sec.

III. Results and Discussion

A. Solution Parameters (Effect of Solution Concentration and Viscosity)

Polymer solution concentration has a significant effect on the morphology and mechanical properties of electrospun mats

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and fibers. In the present work we have studied the effect of two important solution parameters (namely viscosity and polymer concentration) on the morphology, wet-ability and mechanical properties of electrospun mats. Structural morphology of the spun mats with different PAN concentration (6, 8, 10 and 12%)were investigated by SEM taken at images high magnification while fixing other process parameters as shown in Fig. 1.

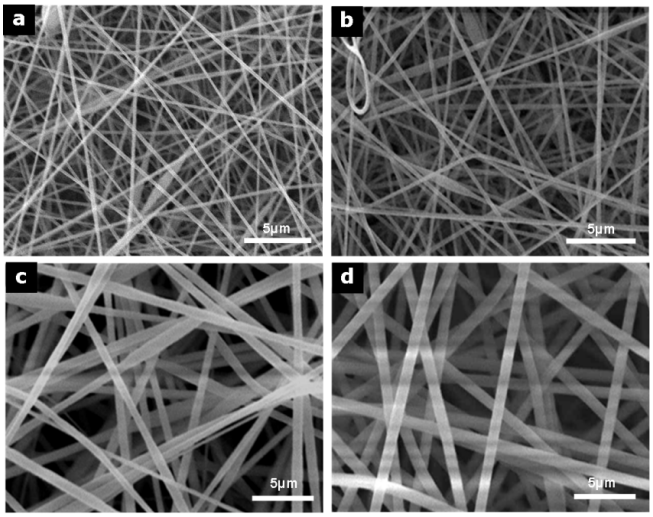

Fig. 1. SEM micrograph of PAN nanofiber at 5000x magnification(a) 6wt%, (b) 8wt%, (c) 10wt%, (d) 12wt%. All the mats were electrospun at: spinning voltage of 21 kV; needle collector distance of 150 mm and 1.5 ml/hr feed rate (scale bar = 5µm).

It was observed that a small increase in PAN concentration considerably reduces the bead formation phenomenon during the electrospinning process. This is mainly due to the increase in viscosity of the solution with increase in PAN concentration, Fig.2. The viscosity increased from 148 cp to 2472 cp with increase in PAN concentration from 6 to12 wt%, respectively. The fiber diameter was observed to increase from 208 nm to 881 nm with increase in PAN concentration from 6 to 12%respectively, Fig. 3.

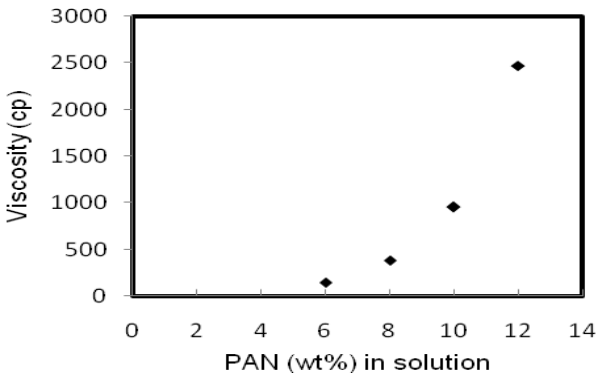

Fig. 2. Viscosity of (6 - 12) wt% PAN/DMF solutions.

This significant increase in fiber diameter is attributed to increased viscosity by increasing the polymer (PAN) concentration from 6 to 12 % in the solution. Increased viscosity results in more chain entanglements which cause hindered flow of the solution jet during its flight to the collector at fixed process parameters resulting in an increased diameter. On the other hand, the increase in PAN concentration leads to reduced bead formation with no beads observed for higher PAN concentrations of10 and 12 %.As mentioned earlier, at lower concentration, viscosity of the solution is also low and thus viscoelastic forces in the polymer solution present in the jet that cannot withstand the larger columbic repulsive forces and hence results in breakup of charged jet into small jets leading to formation of droplets. Our findings are in line with the theoretical findings of Heikkilä et. al.[42], Fong et. al.[19] and experimental findings of Qian Li et. al. [30].

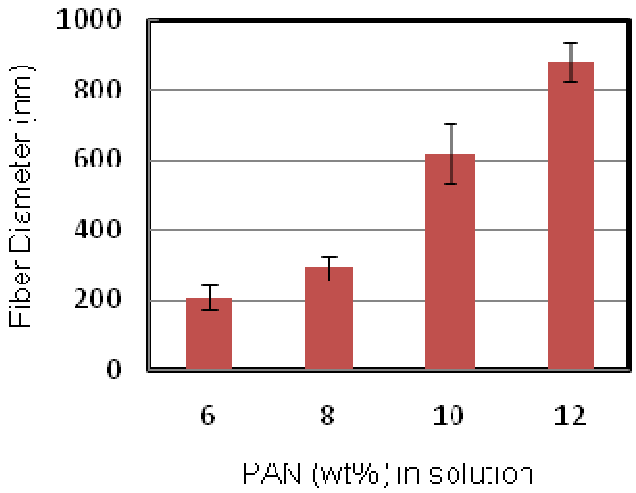

Fig. 3. Average fiber diameter of (6 – 12) wt% PAN/DMF solutions.

B. Process Parameters

After having known the effect of solution concentration and viscosity on the morphological properties of electrospun nanofibers, the next step was to investigate the effect of process parameters (feed rate, spinning voltage, needle collector distance, and needle diameter) on the morphological evolution of electrospun nanofibers. For this purpose, intermediate polymer concentration of 10% PAN was selected which provided a good combination of mechanical and morphological properties. The first step was to acquire an adequate minimum flow rate at which continuous electrospinning is obtained. In order to study the effect of flow rate on the electrospun fibers, several experiments at fixed solution concentration and process parameters (21 kV, 150 mm distance and 21 gauge needle), and only feed rate was varied until the lowest feed rate (0.8 ml/hr) was found at which continuous spinning was observed. At any value of feed rate lower than the 0.8 ml/hr, spinning was discontinuous which resulted in dripping or intermittent droplet formation. Feed rate was then varied form 0.8 ml/hr to 1.4 ml/hr to investigate the effect of increasing feed rate on the morphological appearance of the spun mats. Fig. 4 shows the appearance of all the electrospun mats produced by varying the feed rate. It can be observed that the linear density of fibers (number of fibers per unit area) increases as the feed rate is increased from 0.8 ml/hr to 1.4 ml/hr. This is due to the fact that at lower feed rates inadequate solution flows and slight discontinuity of the spinning process occurs. Increasing the feed rate from 1.2 to 1.4 ml/hr promoted adequate flow of the polymer solution and stability of the spinning which

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resulted in improved morphological appearance (more smoothness and relatively narrow size distribution of electrospun fibers diameter as shown in [Fig. 4(a-d)].

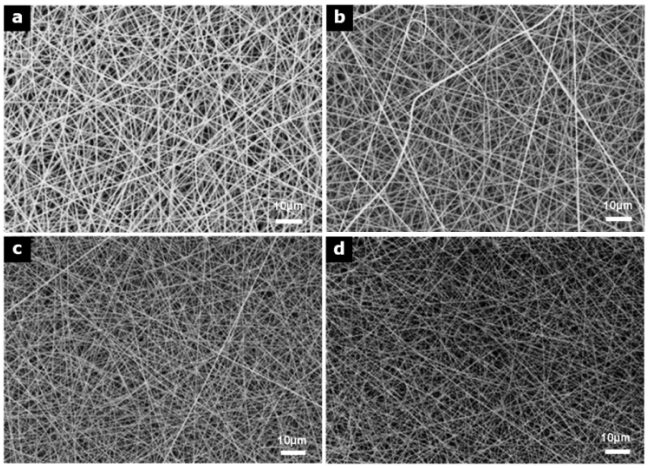

Fig. 4. SEM images of 10wt% PAN taken at 1000X at different feed rates:(a)0.8 ml/hr, (b) 1.0 ml/hr, (c) 1.2 ml/hr, (d)1.4 ml/hr, fixing voltage at 21 kV and distance at 150 mm, (scale bar = 10µm).

More detailed morphology of the electrospun fibers was revealed by FE-SEM images Shown in Fig. 5. The average fiber diameter increased significantly from 398nm to 896 nm when feed rate was increased from 0.8 ml/hr to 1.4 ml/hr, respectively. This increase in fiber diameter is a direct consequence of the higher amount of polymer in the charged polymer jet at higher mass flow rate (feed rate). It can be observed that, not only the average fiber diameter increases with increasing feed rate, the fiber size uniformity increases as well.

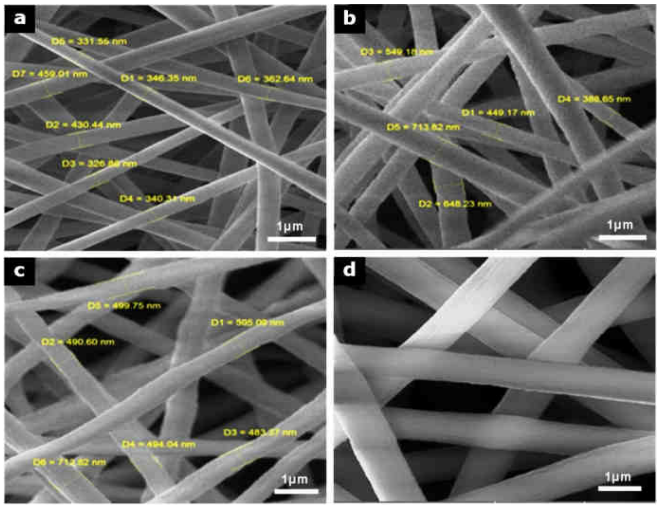

Fig. 5. FE-SEM images of 10%PAN: Effect of feed rate at 21 kV and 150 mm, (a) 0.8 ml/hr, (b) 1.0ml/hr, (c) 1.2 ml/hr and (d) 1.4 ml/hr, (scale bar = 2µm).

Effect of Voltage

In order to investigate the effect of voltage on the morphology of electrospun mats; the spinning voltage was changed from 15 to 21 kV keeping all the other spinning parameters constant. It was found that spinning was not stable for voltage values 15 to 17 kV and resulted in intermittent droplets and dripping of solution. When the spinning voltage was increased from 18 kV to 21 kV the stability of the spinning process improved. Reneker et al.[43], observed no obvious change in fiber diameters with an electric field when they spun polyethylene fibers. But, in our study, an increase in the voltage from 18 to 20 kV, the average fiber diameter reduced from 383 nm to 332 nm, respectively. However, the fiber diameter significantly increased to 750 nm when the voltage was increased to 21kV as shown in Fig. 6. The possible reason for this may be the dual effect of increased voltage. Increasing spinning voltage has competing effects of increasing both whipping instability and mass flow of the polymer solution through the needle, thereby increasing the fiber diameter [25]. In addition, statistical analysis of the electrospun fibers revealed a narrow size distribution at intermediate voltages (e.g. 18 kV and 20kV) but at higher voltage (21 kV) a much wider size distribution was observed. These results suggest that lower voltages yielded smaller and more uniform fiber diameters. Qiang Li et al. [30], and Demir et al. [44], reported similar results when they studied the effect of voltage on electrospinning of PVA and polyurethane, respectively.

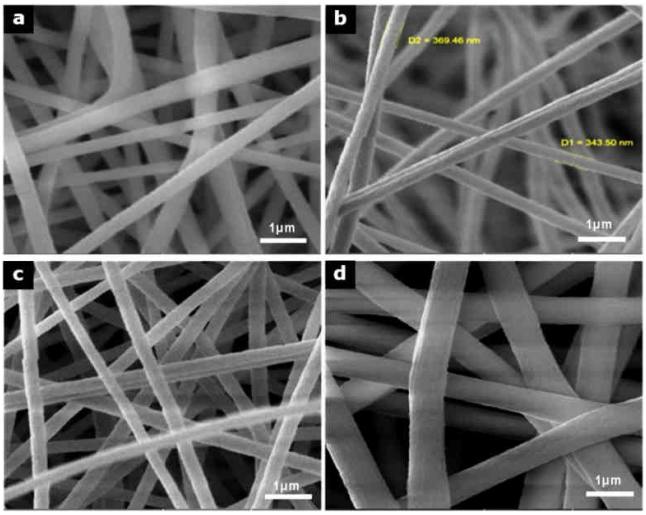

Fig. 6. 10%PAN in DMF: Effect of voltage at 1.5ml/hr and 150mm; (a)18kV, (b) 19kV, (c) 20kV (d) and 21kV,(scale bar = 2µm).

Effect of Needle to Collector Distance

To investigate the effect of distance between the needle tip and collector, the distance was increased from 110mm to 150mm with the other spinning parameters fixed (voltage 21 kV, needle diameter of gauge 18, and feed rate 1.5 ml/hr). P. Heikkilä et al. [42], reported that increasing distance at constant voltage and feed rate can increase the stretching of the solution jet which may result in slight decrease in fiber diameter, but our findings do not show such trends. Little surprisingly, in our study the average fiber diameter was more than tripled (increased from ~200 nm to ~750 nm) when distance was increased from 110 mm to 150 mm as shown in Fig.7. Increasing the distance at constant spinning voltage, have two effects of opposing consequences: firstly, stretching of the fibers is enhanced by increased distance because of larger flight time of the fluid jet and more solvent evaporation. This higher rate of evaporation results in fully dried and stretched fibers leading to thinner fibers [25], [26]. Secondly, with increased distance, electric field strength decreases and this leads to a smaller stretching forces leading to thicker fibers[25]. We suggest the possible reason for the

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much-increased fiber diameter is the decreased electric filed strength with increased distances. This view point is also evidenced by the relatively larger average diameter of spun fibers which is attributed to the decreased field strength at higher distance (150mm). Statistical analysis also showed on increasing distance, fiber size distribution shifts to larger sizes confirming the reason mentioned above.

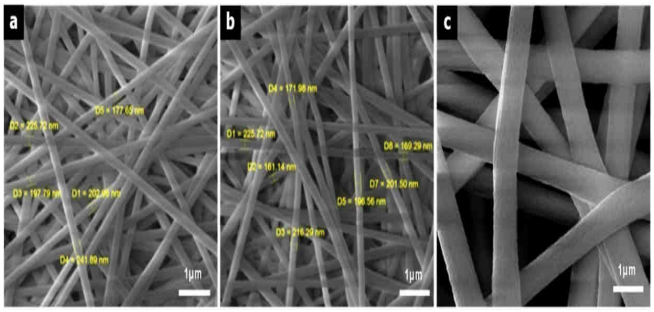

Fig. 7. FE-SEM images of 10 wt% PAN: Effect of distance at 21kV and 1.5ml/hr; (a)110 mm, (b)130mm and (c)150mm, (scale bar = 2µm).

EFFECT OF NEEDLE DIAMETER

Fig. 8 shows the relation between PAN average nanofiber diameters with needle diameter. It is obvious that with increasing needle diameter, the average fiber diameter will decrease. The average fiber diameter for Gauge 14 needle was around 447nm whereas, for example, it was 644nm for Gauge 23 case.

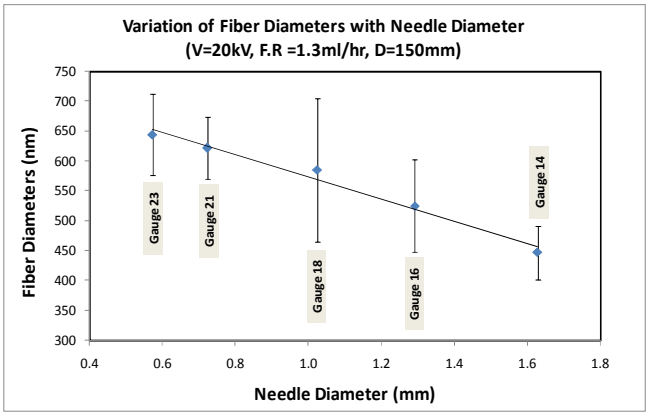

Fig. 8. Variation of average PAN fiber diameter with needle diameter.

In contrast to our results, Sutasinpromprae et al.[45], studied the effect of nozzle diameter on the size of as spun PAN nanofibers. They found that the fiber size increase with increasing nozzle diameters. They suggested that larger nozzle diameter makes ejecting of jet easier, its linear length before bending instability will be higher compared to smaller nozzles, and as a result, solvent will not evaporate completely. Same results were found by Sencadas et al.[46], during the production of chitosan fibers. From other perspectives, some researchers suggested that, for small needle diameter the travel time of jet will be smaller and this will lead to higher stretching action [45], [47], [48], and at the end smaller fiber diameters will be collected[49]. Although linear length of jet before bending instability starting is the case for larger Needle diameters which shorten the time for fibers thinning and as a result larger fiber diameters will be resulted as Sutasinpromprae et al.[45], suggesting and as proven by this work [Fig. 9], the overall time for production of nanofibers in case of smaller needle diameters is shorter than larger ones, this will prevent complete solvent evaporation and will leave larger average fiber diameter, and finally will explain our results.

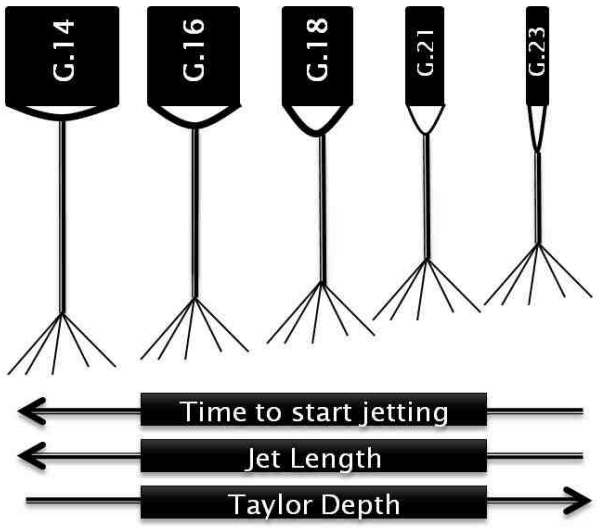

Fig. 9. Effect of the needle diameter on time to start jetting, jet length and Taylor cone shape.

As shown in Fig. 9, increasing needle diameter will affect Taylor cone as well as jet length, in which the time needed to start forming Taylor cone as well as jet length before bending instability region were found to increase in case of larger needle diameters. Taylor cone depth was increased and width was decreased in case of larger needle diameters. Fig. 10 explains this study claim and proves the trend found - decreasing needle diameter will increases fiber diameter- that is coming conversely to some literatures. As shown in Fig. 10(c&d), many fibers found in stick with each other as arrows pointed, this directly indicates that solvent was not completely evaporated because of lesser time to do that, and as well-known that if solvent will not evaporates completely, fiber diameter will be larger. Larger needle diameters –Gauge 16 & 18- as shown in Fig.10(a&b) were not showing the same trend and this was because of enough time required to completely evaporate solvent.

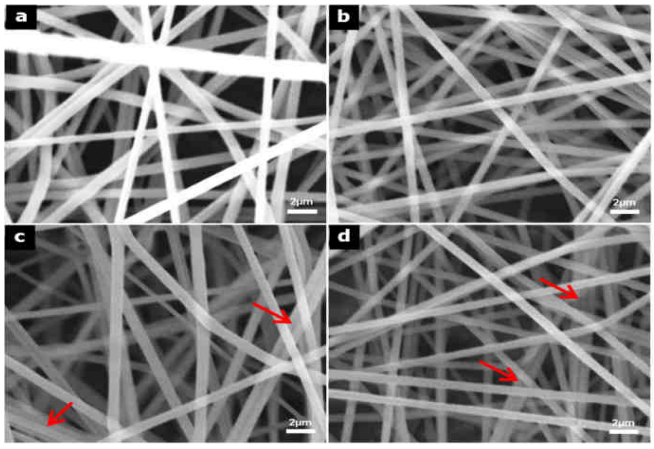

Fig. 10. Nanofiber morphology for different needle diameters, (a) Gauge 16 needle diameter (b) Gauge 18 (c) Gauge 21 and (d) Gauge 23.

To see if the trend found in this study is affected by changing feed rate, different feed rates were tried and its effect on

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resultant fiber morphology at different needle Diameters were discovered as shown in Fig. 11. Increasing feed rate for all needle diameters has the same effect on fiber diameter decrease, in which again there will be lesser time for solvent evaporation in case of higher feed rates.

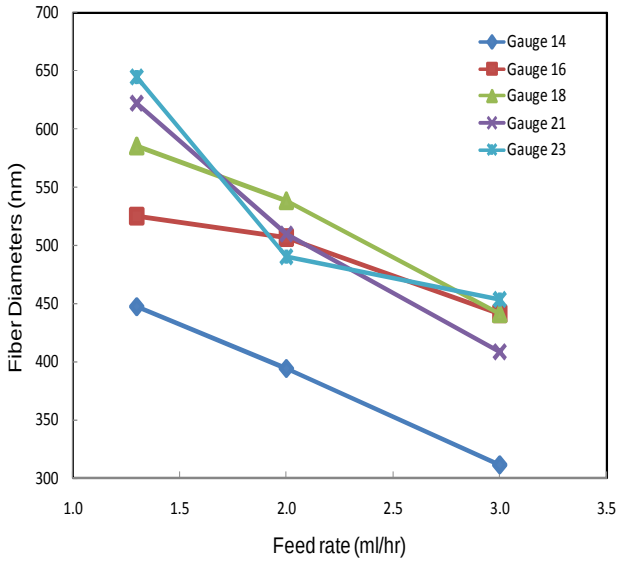

Fig. 11. Variation of average PAN fiber diameter with feed rate for different needle diameters.

Contact Angle Measurement

Contact angle goniometry is a well-recognized method of gauging the wetting/non-wetting behavior of materials ranging from metals, nonmetals to polymers. Higher hydrophilicity (more wetting ability) is characterized by the lower values of contact angle whereas higher values indicate the hydrophobic (non-wetting) surfaces. Wet-ability of solid surfaces is an important property and is governed by both the chemical composition and geometrical microstructure of the surface [50]. About 2 µL of DI water was used to determine the contact angle and was found to be 99.5°, 100.3°, 105° and 115° respectively for 6, 8, 10 and 12 % PAN nanofiber mats as shown in Fig. 12. Surface providing water contact angle lower than 60° are considered to be hydrophilic whereas contact angles higher than 90° are regarded as hydrophobic and super-hydrophobic with the contact angle exceeding 120° [7]. The increase in concentration from 6 to 12% of PAN increases the hydrophobicity of the mats rendering them considerably hydrophobic. The mats are observed to be hydrophobic for the PAN concentrations studied. This increase in hydrophobicity is an attribute of increasing concentration of PAN in the polymer solution.

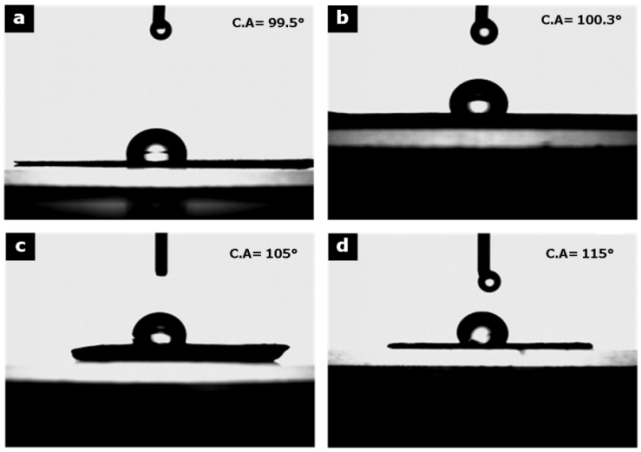

Fig. 12. Contact angle of (a) 6wt%, (b) 8wt%, (c) 10wt% and 12wt % PAN nanofiber mats. Mats were electrospun at: 21 kV spinning voltage, 150 mm needle to collector distance and 1.5 ml/hr feed rate.

Mechanical Properties of Electrospun Mats

The tensile test results for 6-12 wt% PAN concentration nanofiber mats produced at fixed process parameters are listed in Table I. The tensile strength was found to be 3.56MPa and 15.86 MPa respectively for 6% and 12% PAN nanofiber mats. Tensile strength increased by ~346% for an increase in PAN concentration from 6 to 12 wt% as shown in Fig. 13. Similarly, the failure strength of nanofiber mats was observed to be increased from 3.11MPa to 10.22 MPa when PAN concentration was increased from 6 to 12 wt%, indicating an increase in failure strength by 229%. An increase in ductility from 6.9 to 41.69% was observed by increasing PAN concentration from 6 to 12 wt% respectively, which is an overall increment of 504% in ductility. The increase in tensile strength, failure strength and ductility with increase in PAN concentration is attributed to the decrease in bead formation as observed in Fig. 1. The increase in fiber diameter with increase in PAN concentration increases the ductility of the nanofiber mats. The increase in tensile properties co-relates well with the increase in viscosity and fiber diameter of the mats.

Table I. Tensile test results of different PAN nanofiber mats concentration that produced at fixed process parameters: 21 kV spinning voltage, 150 mm needle to collector distance and 1.5 ml/hr feed rate.

PAN (wt%)	Tensile Strength (MPa)	SD	Ductility %	SD	Failure Strength(MPa)	SD
6	3.56	0.18	6.9	1.17	3.11	0.11
8	7.33	0.85	13.46	1.12	6.02	2.26
10	10.89	0.00	39.67	0.00	5.99	0.00
12	15.86	0.27	41.69	0.21	10.22	1.04

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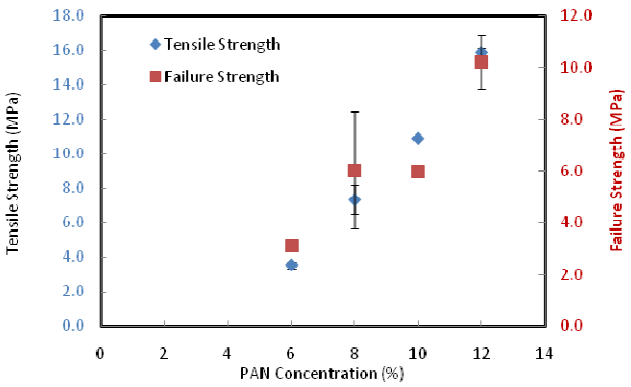

Fig. 13. Nanofiber mats tensile and failure strength of 6-12 wt% PAN concentrations.

IV. Conclusion

Sub-micron sized fiber mats were produced by electrospinning of the 6 to 12 % PAN-DMF solution. Increasing PAN concentration was found to increase fiber diameters. Minimum feed rate required for the adequate mass flow of the polymer solution and hence for the continuous spinning was found to be 0.8 ml/hr. Increasing feed rate up to 1.5 ml/hr. improved the morphology of the individual fibers as well as the spun mats but enlarged the fiber diameters almost four times as compared to their sizes at lower feed rate (0.8 ml/hr). Increasing the applied voltage to some threshold value of decreased fiber diameters and when this threshold was exceeded, fibers increased in diameter. Distance from needle to collector was found to increase fiber diameter when it is increased. Fiber diameters decreased when higher needle diameters were used. A small increase in PAN concentration from 6 to 12 wt% increases the tensile, failure strength and ductility by 346, 229 and 504% respectively. The increase in fiber diameter with increase in concentration increases the ductility of the nanofiber mats. The water contact angle increased with increasing PAN concentration indicating the effect of fiber diameter on hydrophobic nature of the mats.

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