

Process optimization and empirical modeling for electrospun polyacrylonitrile (PAN) nanofiber precursor of carbon nanofibers

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Abstract

Ultrafine fibers were spun from polyacrylonitrile (PAN)/*N,N*-dimethyl formamide (DMF) solution as a precursor of carbon nanofibers using a homemade electrospinning set-up. Fibers with diameter ranging from 200 nm to 1200 nm were obtained. Morphology of fibers and distribution of fiber diameter were investigated varying concentration and applied voltage by scanning electric microscopy (SEM). Average fiber diameter and distribution were determined from 100 measurements of the random fibers with an image analyzer (SemAfore 5.0, JEOL). A more systematic understanding of process parameters was obtained and a quantitative relationship between electrospinning parameters and average fiber diameter was established by response surface methodology (RSM). It was concluded that concentration of solution played an important role to the diameter of fibers and standard deviation of fiber diameter. Applied voltage had no significant impact on fiber diameter and standard deviation of fiber diameter.

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Keywords: Electrospinning; Response surface methodology; Polyacrylonitrile nanofibers; Average fiber diameter; Standard deviation of fiber diameter

1. Introduction

1.1. Background

Carbon fibers are now important industrially and have gained a wide range of applications, from sports equipment to the aerospace industry. Carbon nanofibers, like other one-dimensional (1D) nanostructures such as nanowires, nanotubes, and molecular wires, are receiving increasing attention because of their large

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length to diameter ratio. This is due to their potential application in nanocomposites [1], high temperature catalysis, template for nanotubes [2], filters [3], rechargeable batteries [4], supercapacitors [5,6], bottom-up assembly applications in nanoelectronics and photonics [7], etc.

Carbon nanofibers can be produced by traditional vapor growth method [8] or plasma enhanced chemical vapor depositing method which was developed at the beginning of this century [9]. However vapor growth or plasma enhanced chemical vapor depositing method involves a complicated process and high cost. Carbon nanofibers can also be produced by stabilizing, carbonizing, activating electrospun precursors. Chun et al. [3] produced carbon nanofibers with diameter in the range from 100 nm to a few microns from electrospun polyacrylonitrile and mesophase pitch precursor fibers. Wang et al. [10–12] produced carbon nanofibers from carbonizing of electrospun PAN nanofibers and studied their structure and conductivity. Hou et al. [2] reported a method to use the carbonized electrospun PAN nanofibers as substrates for the formation of multi-wall carbon nanotubes. Kim et al. [5,13] produced carbon nanofibers from PAN-based or pitch-based electrospun fibers and studied the electrochemical properties of carbon nanofibers web as an electrode for supercapacitor. Park et al. [14] produced pitch-based carbon fibers by stabilizing, carbonizing, activating electrospun web from petroleum-derived isotropic pitch solution in tetrahydrofuran (THF). The electrical conductivities of the carbonized web were measured to be 6.3 and 8.3 S/cm at 1000 and 1200 °C, respectively, which indicated that the web was suitable to make electrodes of electrical double layer capacitors (EDLCs). But the diameter of fibers from pitch-based electrospun fibers was in the range of several microns.

1.2. Electrospinning

Electrospinning has been recognized as an efficient technique for the fabrication of fibers in nanometer to micron diameter range from polymer solutions or melts. In a typical process, an electrical potential is applied between a droplet of polymer solution or melt held at the end of a capillary and a grounded collector. When the applied electric field overcomes the surface tension of the droplet, a charged jet of polymer solution or melt is ejected. The jet grows longer and thinner due to bending instability or splitting [15] until it solidifies or collects on the collector.

Electrospinning is a process involving polymer science, applied physics, fluid mechanics, electrical, mechanical, chemical, material engineering and rheology. The fiber morphology is controlled by the experimental parameters and is dependent upon solution conductivity, concentration, viscosity, polymer molecu-

lar weight, applied voltage, etc. [16,17]. Much work has been done on the effect of parameters on the electrospinning process and morphology of fibers. Demir et al. [18] reported that the fiber diameter increased with increasing polymer concentration according to a power law relationship and Deitzel et al. [17] reported a bimodal distribution of fiber diameter for fibers spun from higher concentration solution. Boland et al. [19] obtained a strong linear relationship between fiber diameter and concentration in electrospun poly(glycolic acid) (PGA). Mo et al. [20], Ryu et al. [21] and Katti et al. [22] also reported a significant relationship between fiber diameter and concentration in electrospinning process. For the effect of applied voltage, Reneker et al. [23] obtained a result that fiber diameter did not change much with electric field when they studied the electrospinning behavior of polyethylene oxide. Mo et al. and Katti et al. [20,22] reported that fiber diameter tended to decrease with increasing electrospinning voltage, although the influence was not as great as that of polymer concentration. But Demir et al. [18] reported that fiber diameter increased with increasing electrospinning voltage when they electrospun polyurethane fibers. Larrondo and Manley [24–26] determined that doubling the applied electric field decreased the fiber diameter by roughly half. However, Baumgarten [27] showed that the diameter of the jet reached a minimum after an initial increase in field strength and then became much larger with increasing fields. This effect was caused by the feed rate of the polymer solution through the capillary and illustrated one of the complexities of the electrospinning process. That is, increasing the field does increase the electrostatic stress and create smaller diameter fibers, but it also draws more materials out of the syringe. Sukigara [28,29] studied the effect of electrospinning parameters (electric field, tip-to-collector distance and concentration) on the morphology and fiber diameter of regenerated silk from *Bombyx mori* using response surface methodology and concluded that the silk concentration was the most important parameter in producing uniform cylindrical fibers less than 100 nm in diameter.

In order to optimize and predict the morphology and average fiber diameter of electrospun PAN precursor, design of experiment was employed in the present work. Morphology of fibers and distribution of fiber diameter of PAN precursor were investigated varying concentration and applied voltage. Main effect and interaction of factors were studied by two-way analysis of variance. A more systematic understanding of these process conditions was obtained and a quantitative basis for the relationships between average fiber diameter and electrospinning parameters was established using response surface methodology (RSM), which will provide some basis for the preparation of carbon nanofibers.

2. Experimental

2.1. Preparation of polymer solution

PAN (cat 18.131-5) was obtained from Aldrich and used without further purification. 99.5% *N,N*-dimethylformamide (DMF) was purchased from Merck. The concentrations from 6 to 12 wt.% PAN/DMF solution were stirred by an electromagnetically driven magnet at room temperature for 30–50 h in order to obtain homogenous solution.

2.2. Electrospinning set-up

The schematic electrospinning setup used for the electrospinning process is shown in Fig. 1. The spinning solution was placed in a vertically aligned glass pipette with a tip around 1 mm in diameter. A stainless steel electrode was immersed in the solution and connected to a high voltage power supply (Bertan series 230-30R), which can generate DC voltage up to 30 kV. A flat metal plate with aluminum foil was placed below serving as a grounded counter electrode. The voltage between the electrode and the counter electrode could be controlled by the high voltage power supply. The air pressure above the solution was controlled with an air pump so that a stable drop of the solution was suspended at the tip of the capillary before the power was supplied. The solutions were electrospun between 10 and 20 kV vertically to the collector. The grounded collector was located at a distance of 10 cm.

Fibers collected directly on the aluminum foil could be studied by scanning electron microscopy (SEM) after drying under vacuum at room temperature for 24 h.

2.3. Morphology

The morphology of electrospun PAN fibers was observed by scanning electron microscopy (SEM,

JEOL JSM-5600LV) after being gold-coated. The diameter of electrospun fibers was measured with an image analyzer (SemAfore 5.0, JEOL). For each experiment, average fiber diameter and distribution were determined from about 100 measurements of the random fibers.

2.4. Design of experiment

A factorical experiment was designed to investigate and identify the significance of two processing parameters (one is concentration and the other is applied voltage) on average fiber diameter as shown in Table 1.

The experiment was performed for at least three levels of each factor to fit a quadratic model. These levels were chosen equally spaced. Four levels of concentration and three levels of applied voltage resulted in 12 possible combinations of factor setting. A schematic of experimental design is shown in Fig. 2. The values in the brackets are coded values of the factors (χ_1 : concentration, χ_2 : applied voltage) and the values at coordinate points and the brackets behind represent the average fiber diameter and standard deviation of fiber diameter, respectively. For the details of calculation of coded values, please refer to Refs. [30] and [31]. Coded and uncoded values of the factors are listed in Table 2.

Table 1
Design of experiment (factors and levels)

Factor	Factor level
Concentration (wt.%)	6, 8, 10, 12
Applied voltage (kV)	10, 15, 20

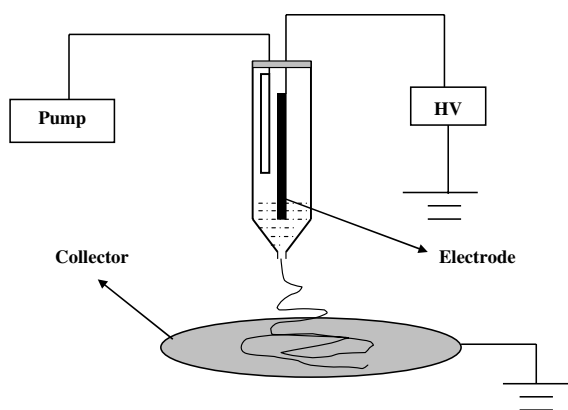


Fig. 1. Schematic set-up of electrospinning.

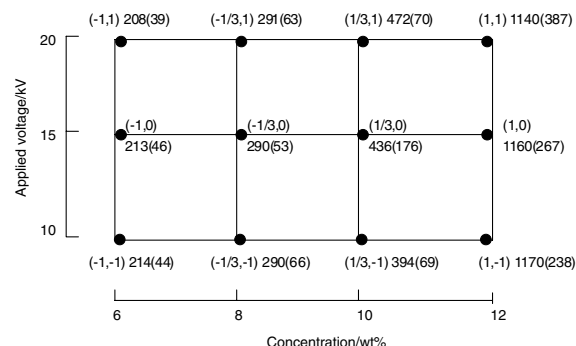


Fig. 2. Design of experimental. Coded values are shown in the brackets (concentration and applied voltage). The values at the coordinate points and the brackets behind show the average fiber diameters (nm) of about 100 measurements and the standard deviation of fiber diameters.

Table 2
Design of experiment (coded and uncoded values)

Experiment no.	Coded value		Uncoded value	
	χ_1	χ_2	Concentration, ζ_1 (wt.%)	Applied voltage, ζ_2 (kV)
1	−1	0	6	15
2	−1	1	6	20
3	1/3	1	10	20
4	−1	−1	6	10
5	−1/3	−1	8	10
6	−1/3	0	8	15
7	1	−1	12	10
8	1	1	12	20
9	1/3	−1	10	10
10	1/3	0	10	15
11	1	0	12	15
12	−1/3	1	8	20

3. Results

3.1. Morphology of fibers

Fig. 3 shows the morphology of fibers obtained from solutions of 6, 8, 10 and 12 wt.% concentration at applied voltage from 10 to 20 kV with a constant tip-to-collector distance of 10 cm. Spindle-like beads formed for fibers obtained from solution of 6 wt.% at lower voltage (for example, at voltage of 10 kV). And when the concentration was over 10 wt.%, for example, at 12 wt.%, the diameter of the fibers was not uniform as shown in Fig. 3. At the concentration of 12 wt.%, the average fiber diameter was much larger than that of fibers spun at lower concentrations. The distribution of fiber diameters at 6, 8, 10 and 12 wt.% concentration is shown in Fig. 4(a)–(d). The fiber distribution became gradually broader with increasing concentration, which is in consistency with the results obtained by Ryu et al. [21]. The average values and standard deviations of fiber diameter were taken for statistics analysis.

3.2. Main effect plots and interaction plot of factors on average fiber diameter

Electrospinning parameters affect the average fiber diameter in different extent. In order to find the extent of the impact on average fiber diameter, the impact of two factors (concentration and applied voltage) on fiber diameter was investigated by two-way analysis of variance. Main effect plots and interaction plot of two factors on average fiber diameter were obtained as shown in Figs. 5 and 6. Apparently concentration has more impact on the average fiber diameter than applied voltage. The results suggest a quadratic relationship between average fiber diameter and concentration which will be

discussed later. And applied voltage has less impact on average fiber diameter. The results are similar to the results obtained by Reneker et al. [23] Fig. 6 shows that the interaction between concentration and applied voltage is not significant, either.

In order to obtain a more systematic understanding of process parameters and establish a quantitative basis for the relationship between electrospinning parameters and fiber diameter, response surface methodology (RSM) was employed in this study.

3.3. Response function

RSM has been used successfully for materials and process optimization in numerous studies including thermoplastic elastomers [32], diamond-like carbon films [33] and poly(vinyl alcohol) hydrogels [34]. This approach has the advantage of taking into account the combined effects of several parameters and it uses statistical methods to fit an empirical model to the experimental data. For more detailed explanation on RSM see Ref. [31]. RSM enables us to obtain an overview of the impact of processing parameters on average fiber diameter. Sukigara et al. [28,29] had used RSM to model and optimize the electrospinning parameters for spinning of regenerated *Bombyx mori* silk. The predicted fiber diameters according to their fitted equation were in agreement with the experimental results. But they have not reported the *P*-value for each factor and have not done analysis for standard deviation of fiber diameter. In this paper, RMS was used to optimize and predict the morphology and fiber diameter of electrospun PAN precursors.

By regression analysis, values for coefficients, *P*-values (a measure of the statistical significance. When *P*-value is less than 0.05, the factor has significant impact on the average fiber diameter. If *P*-value is greater than 0.05, the factor has no significant impact on average fiber diameter.) and *R*² (represents the proportion of the total variability that has been explained by the regression model [30]) for regression models were obtained as shown in Table 3. The fitted second-order equation for average fiber diameter is given by

$$y = 323.50 + 446.80\chi_1 + 5.37\chi_2 + 362.25\chi_1^2 - 2.38\chi_2^2 + 0.38\chi_1\chi_2, \quad (1)$$

where *y* is average fiber diameter, χ_1 and χ_2 are the coded values of concentration and applied voltage, respectively. From the *P*-values listed in Table 3, it is obvious that *P*-values for terms related to applied voltage such as χ_2 , χ_2^2 and $\chi_1\chi_2$ are much greater than significance level of 0.05. That is to say, applied voltage has no significant impact on average fiber diameter and the interaction between concentration and applied voltage is not significant, either. But *P*-values for term χ_1 and χ_1^2 are less

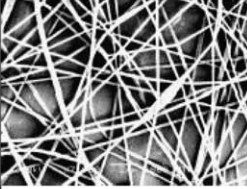
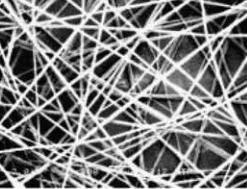
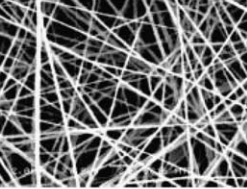
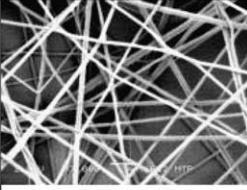

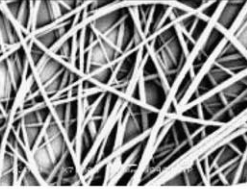
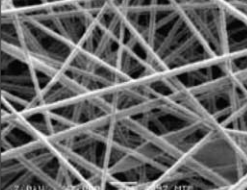
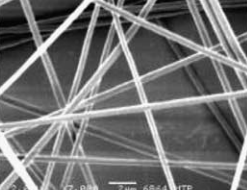
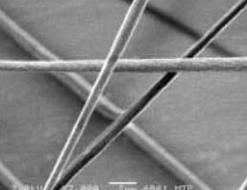
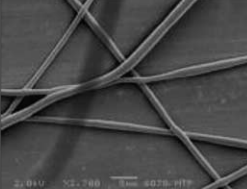
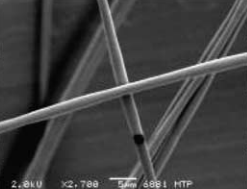
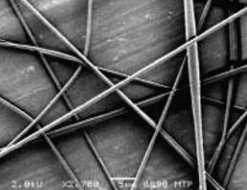
Concentration (wt%)	Applied voltage (kV)		
	10	15	20
6			
	214(44)	213(46)	208(39)
8			
	290(66)	290(53)	291(63)
10			
	394(69)	436(176)	472(70)
12			
	1170(238)	1160(267)	1140(387)

Fig. 3. The morphology of fibers at applied voltage from 10 to 20 kV at concentrations from 6% to 12% with a constant tip-to-collector distance of 10 cm (The values below the images and the brackets show the average fiber diameter (nm) and the standard deviation of fiber diameter. The scale bars for fibers from solution from 6 to 10 wt.% and 12 wt.% are 2 mm and 5 mm, respectively).

than 0.05. Therefore, concentration has significant impact on average fiber diameter. R^2 is 0.975. That is to say, this model explains 98% of the variability in new data.

Because terms related to applied voltage have no significant impact on average fiber diameter, we removed term χ_2 , χ_2^2 , $\chi_1\chi_2$ and fitted the equation by regression analysis again. The model and the results were obtained as shown in Table 4. The fitted equation in coded unit is given by

$$y' = 321.9 + 444.80\chi_1 + 362.30\chi_1^2, \quad (2)$$

where y' is the average fiber diameter. And in uncoded unit,

$$y' = 2241.77 - 575.57\zeta_1 + 40.25\zeta_1^2, \quad (3)$$

where ζ_1 is the concentration of polymer solution. All the P -values are less than the significance level of 0.05. That is to say, average fiber diameter has a quadratic relationship with solution concentration.

In order to find the statistical characteristics of standard deviation of fiber diameter, regression analysis was done for standard deviation at the same time. The results were listed in Table 5. The fitted second-order

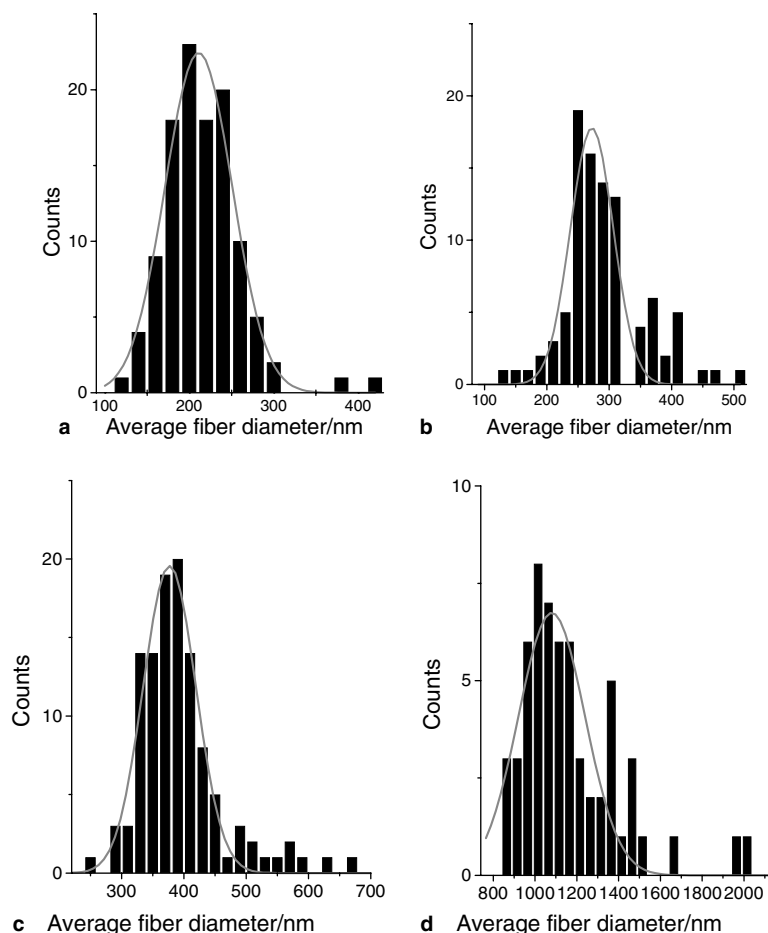


Fig. 4. Distribution of fiber diameters obtained at 10 kV with a tip-to-collector distance of 10 cm. The concentrations for (a), (b), (c) and (d) are 6, 8, 10 and 12 wt.%, respectively.

equation for standard deviation of fiber diameter is given by

$$S_y = 80.92 + 121.00\chi_1 + 17.75\chi_2 + 98.25\chi_1^2 - 13.50\chi_2^2 + 34.95\chi_1\chi_2, \quad (4)$$

where S_y is the standard deviation of fiber diameter. Similar results were obtained. The P -values related to applied voltage are much greater than significance level of 0.05. Therefore applied voltage has no significant effect on the standard deviation of fiber diameter and the interaction between concentration and applied voltage on the standard deviation of fiber diameter is not significant, either. But P -values for term χ_1 and χ_1^2 are less than 0.05. Concentration has significant impact on standard deviation of fiber diameter. R^2 is 0.903. This indicates that this model explains 90% of the variability in new data. After removing term χ_2 , χ_2^2 , $\chi_1\chi_2$, the fitted equation of standard deviation of fiber diameter as shown in Table 6 is given by

$$S'_y = 71.92 + 121.10\chi_1 + 98.25\chi_1^2, \quad (5)$$

where S'_y is the standard deviation of fiber diameter.

In uncoded unit,

$$S'_y = 592.87 - 156.13\zeta_1 + 10.92\zeta_1^2. \quad (6)$$

All the P -values are less than the significance level of 0.05. Therefore, standard deviation of average fiber diameter has a quadratic relationship with solution concentration too.

4. Discussion

During the electrospinning process, the droplet of solution at the capillary tip gradually elongates from a hemispherical shape to a conical shape or Taylor cone as the electric field is increased. A further increase in the electric field results in the ejection of a jet from the apex of the cone. The jet grows longer and thinner until

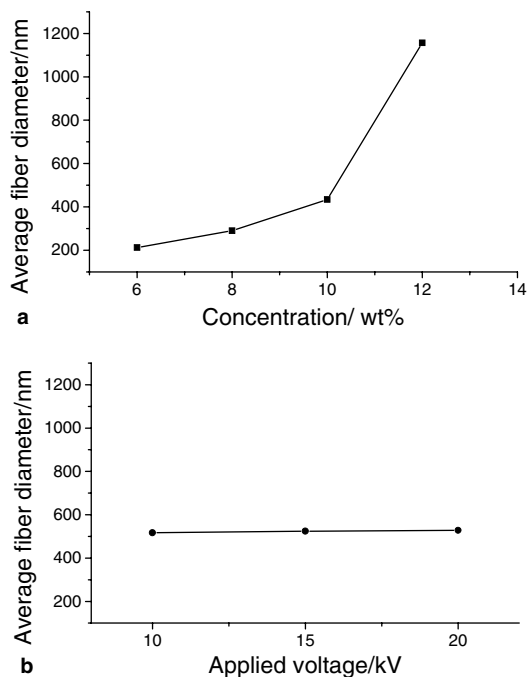


Fig. 5. Main effect plots of concentration (a) and applied voltage (b) on average fiber diameter.

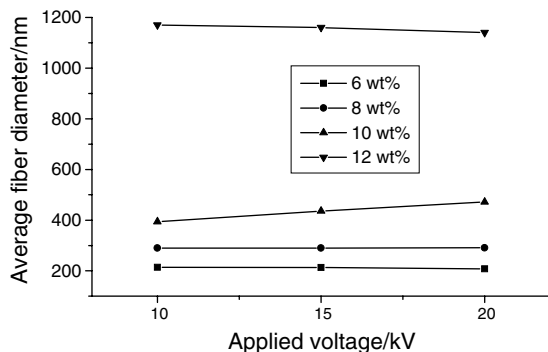


Fig. 6. Interaction plots between concentration and applied voltage on average fiber diameter.

it solidifies and collects on the collector. The fiber morphology is controlled by process parameters. In this study the effect of solution concentration and applied voltage on fiber morphology and average fiber diameter were investigated. Polymer concentration was found to be the most significant factor controlling the fiber diameter in the electrospinning process.

4.1. Polymer concentration

Fig. 3 shows spindle-like bead formation at concentration of 6 wt.% under applied voltage of 10 kV. The

Table 3

Analysis of variance for the two factors (concentration, applied voltage) and coefficients of the model in coded unit* (for average fiber diameter)

Term	Coefficient		P-value
Constant	C_0	323.50	0.035
Concentration: χ_1	C_1	446.80	0.000
Applied voltage: χ_2	C_2	5.37	0.863
χ_1^2	C_{11}	362.25	0.001
χ_2^2	C_{22}	-2.38	0.365
$\chi_1\chi_2$	C_{12}	0.38	0.993
	F	Significance	R^2
	45.91	0.000	0.975

* Model: $y = C_0 + C_1\chi_1 + C_2\chi_2 + C_{11}\chi_1^2 + C_{22}\chi_2^2 + C_{12}\chi_1\chi_2$, where y is average fiber diameter.

Table 4

Analysis of variance for the two factors (concentration, applied voltage) and coefficients of the model in coded unit* (for average fiber diameter, after removing terms related to applied voltage)

Term	Coefficient		P-value
Constant	C'_0	321.90	0.000
Concentration: χ_1	C'_1	444.80	0.000
χ_1^2	C'_{11}	362.30	0.000
	F	Significance	R^2
	171.17	0.000	0.974

In uncoded unit, $y' = 2241.77 - 575.567\zeta_1 + 40.25\zeta_1^2$, where ζ_1 is concentration of polymer solution.

* Model: $y' = C'_0 + C'_1\chi_1 + C'_{11}\chi_1^2$, where y' is fiber diameter.

Table 5

Analysis of variance for the two factors (concentration, applied voltage) and coefficients of the model in coded unit* (for standard deviation of fiber diameter)

Term	Coefficient		P-value
Constant	C_{S0}	80.92	0.035
Concentration: χ_1	C_{S1}	121.0	0.001
Applied voltage: χ_2	C_{S2}	17.75	0.338
χ_1^2	C_{S11}	98.25	0.020
χ_2^2	C_{S22}	-13.5	0.663
$\chi_1\chi_2$	C_{S12}	34.95	0.177
	F	Significance	R^2
	11.12	0.000	0.903

* Model: $S_y = C_{S0} + C_{S1}\chi_1 + C_{S2}\chi_2 + C_{S11}\chi_1^2 + C_{S22}\chi_2^2 + C_{S12}\chi_1\chi_2$, where S_y is the standard deviation of fiber diameter.

formation of beads can be related to the formation of droplets in electrospray. In the case of low-molecular-weight liquid, when a high electrical force is applied,

Table 6

Analysis of variance for the two factors (concentration, applied voltage) and coefficients of the model in coded unit* (for standard deviation of fiber diameter, after removing terms related to applied voltage)

Term	Coefficient		P-value
Constant	C'_{S0}	71.92	0.012
Concentration: χ_1	C'_{S1}	121.1	0.000
χ_1^2	C'_{S11}	98.25	0.014
	F	Significance	R^2
	24.27	0.000	0.844

In uncoded unit $S'_y = 592.87 - 156.13\zeta_1 + 10.92\zeta_1^2$, where ζ_1 is the standard deviation of concentration of polymer solution.

* Model: $S'_y = C'_{S0} + C'_{S1}\chi_1 + C'_{S11}\chi_1^2$, where S'_y is the standard deviation of fiber diameter.

formation of droplets can occur [35]. A theory proposed by Rayleigh explained this phenomenon. As evaporation of a droplet takes places, the droplet decreases in size. Therefore the charge density of its surface is increased. This increase in charge density due to Coulomb repulsion overcomes the surface tension of the droplet and causes the droplet to split into smaller droplets [35,36]. However, in the case of a polymer solution (high molecular weight liquid) the emerging jet does not break up into droplets, but is stabilized and forms a string of beads connected by a fiber. As the concentration is increased, a string of connected beads is seen, and with further increase there is reduced bead formation until only smooth fibers are formed [17]. And sometimes spindle-like beads can form due to the extension causing by the electrostatic stress. In this study, spindle-like beads formed at lower concentration of 6 wt.%. When the concentration was increased, uniform fibers formed. However, when the concentration was increased to 12 wt.%, fibers became rather thick and not uniform. And the fiber diameter distribution became gradually broader with increasing concentration as shown in Fig. 4. The changing of fiber morphology can probably be attributed to a competition between surface tension and viscosity. As concentration was increased, the viscosity of the polymer solution increased. The surface tension attempted to reduce surface area per unit mass, thereby caused the formation of beads/spheres. Viscoelastic forces resisted the formation of beads and allowed for the formation of smooth fibers. Therefore formation of beads at lower polymer solution concentration (low viscosity) occurred where surface tension had a greater influence than the viscoelastic force. However, bead formation was reduced and finally eliminated at higher polymer solution concentration, where viscoelastic forces had a greater influence in comparison with surface tension. But when the concentration was too high, for example 12 wt.%, high viscosity and rapid evaporation of solvent

made the extension of jet more difficult. Thicker and non-uniform fibers were formed.

Besides the effect on fiber morphology, polymer concentration had significant impact on the average fiber diameter from the results obtained by response surface methodology. The fiber diameter increased with increasing polymer concentration because the higher viscosity resisted the extension of the jet. In this study, the average fiber diameter was related to the solution concentration through a quadratic relationship. The relationship between solution concentration and droplet diameter in electrospray followed a power law relationship with exponent of 0.3 from the results of Chen et al. [37]. And Deitzel et al. [17] obtained that the average fiber diameter was related to the solution concentration in a power law relationship with an exponent of about 0.5 while Demir et al. [18] reported a power law relationship with an exponent of 3. However, Boland reported a strong linear relationship between fiber diameter and concentration [19]. The differences in the relationship between fiber diameter and concentration may be attributed to the in-flight splitting or splaying phenomena of electrospinning jet reported by Doshi and Reneker [38]. In our study, splitting of jet was not observed. The quadratic relationship between fiber diameter and concentration indicated that the polymer concentration played an important role in determining the fiber diameter.

4.2. Applied voltage

From the results shown in Figs. 3, 5 and 6, Tables 3, and 5, it is obvious that the diameter of electrospun PAN fibers did not change significantly over the range of applied voltage for the various solution concentrations in the experimental region. The same result was found by Reneker et al. [23] when they studied the variation of nanofiber diameter of polyethylene oxide as process parameters were changed. But it is contradictory with the results obtained by Fennessey and Farris [39], Ding et al. [40] and others [20,22]. In their experiments, the diameter of fibers collected on target decreased as the applied voltage increased because of the increasing of the pulling and stretching force in their experiments. The inconsistency may be caused by the difference of the experimental conditions. The flow rate of solution in Fennessey's experiment was maintained the same with the help of a dual syringe infusion pump, while in our experiment the solution was brought down automatically by the electrostatic force and hydrodynamic force of the fluid. So the flow rate in our experiments was not controlled. The diameter of fiber is a combination result of feed rate and electrostatic force. Increasing the applied voltage does increase the electrostatic force and create smaller diameter fibers. But it also draws more solution out of the capillary. If the increasing of

electrostatic force draws much more solution out of the capillary, the fiber diameter would increase with increasing applied voltage as reported by Demir et al. [18]. In our study, increasing of applied voltage did not affect the flow rate to a large extent. Combination of increasing of applied voltage and flow rate resulted that the fiber diameter did not change significantly with applied voltage.

From the results and analysis above, uniform PAN nanofibers can be obtained from solution with concentration of 8 wt.% and 10 wt.% regardless of applied voltage in our study. Polymer concentration has significant impact on fiber diameter. The fitted model as shown in Eq. (3) could be used to predict the concentration needed to produce a desired fiber diameter within the concentration range that is capable of being spun. The equation is valid only at the experimental conditions listed in this study and would need to be regenerated for any additional polymer solution or electrospinning conditions.

The mechanical properties, structures of the electrospun PAN non-woven precursor and carbon nanofibers as well as carbonization process of the precursors will be carried out in our subsequent studies.

5. Conclusions

The electrospinning of PAN/DMF was processed and fibers with diameter ranging from 200 nm to 1200 nm were obtained depending on the electrospinning condition. Morphology of fibers and distribution of fiber diameter were investigated at various concentrations and applied voltages. Fibers with diameter over 1000 nm were obtained at concentration of 12 wt.%. Uniform PAN nanofibers were obtained at concentration of 8 wt.% and 10 wt.% under applied voltage of 10–20 kV. While for solution with concentration of 6 wt.%, fibers with spindle-like beads were obtained at lower applied voltage and uniform nanofibers were obtained at higher applied voltage.

Two-way analysis of variance was carried out at the significance level of 0.05 to study the impact of concentration and applied voltage on average fiber diameter. It was concluded that concentration of solution was the most significant factor impacting the diameter of fibers and standard deviation of fiber diameter. Applied voltage had no significant impact on average fiber diameter. The average fiber diameter increased with polymer concentration according to a quadratic relationship under the experimental conditions studied in this study. The standard deviation of fiber diameter increased with polymer concentration according to a quadratic relationship too. The model can be used to predict the average fiber diameter and its standard deviation, and to optimize electrospinning conditions. The model is valid only at

the experimental region in this study and would need to be regenerated in other situations.

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