Modeling and Optimization of Electrospun PAN Nanofiber Diameter Using Response Surface Methodology and Artificial Neural Networks

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ABSTRACT: Response surface methodology (RSM) based on a three-level, three-variable Box-Benkhen design (BBD), and artificial neural network (ANN) techniques were compared for modeling the average diameter of electrospun polyacrylonitrile (PAN) nanofibers. The multilayer perceptron (MLP) neural networks were trained by the sets of input–output patterns using a scaled conjugate gradient backpropagation algorithm. The three important electrospinning factors were studied including polymer concentration (w/v%), applied voltage (kV) and the nozzle-collector distance (cm). The predicted fiber diameters were in agreement with the experimental results in both ANN and RSM techniques. High-regression coefficient between the variables and the response ($R^2 = 0.998$) indicates excellent evaluation of experimental data by second-order polynomial regression

INTRDUCTION

Electrospinning or electrostatic spinning recognized as a simple and inexpensive process for producing continuous polymeric and ceramic nanofibers with diameters ranging from several micrometers down to tens of nanometers. In the electrospinning process a strong electric field is used to create an electrically charged jet of polymer solution out of the capillary tip. Before reaching the metallic collector, the solvent in the jet evaporates, and is collected as a web of small fibers. The electrospun nanofibers have large surface area per mass ratio, high porosity along with small pore sizes, flexibility, and superior mechanical properties, so they are excellent candidates for applications in filter media, tissue engineering, drug delivery, sensor, hydrogen storage, protective clothing, and reinforcement in composite materials.¹⁻⁴

Morphology and the diameter of electrospun nanofibers depend on many parameters which are model. The R^2 value was 0.990, which indicates that the ANN model was shows good fitting with experimental data. Moreover, the RSM model shows much lower absolute percentage error than the ANN model. Therefore, the obtained results indicate that the performance of RSM was better than ANN. The RSM model predicted the 118 nm value of the finest nanofiber diameter at conditions of 10 w/v% polymer concentration, 12 cm of nozzle-collector distance, and 12 kV of the applied voltage. The predicted value (118 nm) showed only 2.5%, difference with experimental results in which 121 nm at the same setting were observed. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 000: 000–000, 2012

Key words: electrospinnig; nanofibers; response surface methodology; artificial neural network; optimizing

mainly divided into four categories: polymer properties (molecular weight and solubility), polymer solution parameters (polymer concentration, solution viscosity, conductivity, surface tension, and etc.), processing conditions (applied voltage, nozzle-collector distance, feed rate, and needle diameter), and ambient parameters (temperature, atmosphere pressure, and relative humidity).^{5–7}

Response surface methodology (RSM) is a combination of mathematical and statistical techniques useful for modeling and optimizing the effects of several independent variables on the response. The main advantage of RSM is the reduced number of experimental runs needed to provide sufficient information for statistically acceptable result.^{8,9} Recently, studies have been carried out to determine the feasibility and to optimize the diameter of electrospun nanofiber with RSM.^{10–14}

Moreover, artificial neural networks (ANN) have been successfully applied to the modeling and the control of electrospinning processes in recent years.^{15,16} ANN cannot create an equation similar to RSM, but it works as human brain does and it estimates the response based on the trained data in the inquired range. The human brain is composed of

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Figure 1 Structure of artificial neuron.

3–4 billions of nerve cells, called neurons, and these are interconnected to form the biological neural network. To construct a mathematical model of a neuron in ANN, to be called a node or an artificial neuron.^{17,18} The structure of artificial neuron included weight, bias and transfer function is shown in Figure 1. Parallel connection between artificial neurons generates a layer. The ANN represents a network with a several number of layers consisting of parallel elements artificial neuron with different types of connections between layers and transfer function in each layer. In general, a neural network is parallel interconnected structure consisting of: input layer of neuron (independent variables), a number of hidden layers, and output layer (response). According to Kolmogorov's theorem, ANN with a single hidden layer should be capable of approximating any function to any degree of accuracy.¹

In this study, a systematic statistical approach has been adopted to obtain optimum diameter of the electrospun nanofibers with different process conditions. The influence of process conditions on the diameter of the electrospun nanofibers was carried out using Box-Behnken design (BBD). The response surface methodology (RSM) was used to develop a mathematical equation between the polymer concentration, applied voltage and nozzle-collector distance on average nanofiber diameter. Regression equations were developed for the same and in addition to that the effect of process conditions was also modeled using artificial neural network (ANN). Comparison of prediction of nanofiber diameter using ANN and RSM are discussed in this article.

EXPERIMENTAL

Materials

Polyacrylonitrile powder (PAN, $\overline{M}_w = 10^5$ g/mol, $\overline{M}_n = 0.7 \times 10^5$ g/mol), consisting of 93.7 wt % acrylonitrile (AN) and 6.3 wt % methylacrylate (MA) was supplied with Polyacryl (Isfahan, Iran) and *N*,*N*-dimethylformamide (DMF) were obtained from Merck, respectively, as polymer and solvent.

Preparation of electrospun nanofibers

The solutions of PAN were prepared by dissolving 10, 12, and 14 w/v% (g/dL) of sample in DMF sepa-

rately via magnetic stirrer (Corning Hot Plate Stirrer PC-351) at 40° C for 24 h.

Electrospinning

The experimental set-up used for electrospinning is shown in Figure 2. The prepared PAN solution was added to a glass syringe with a needle tip (22G, L = 34 mm, O.D. = 0.7 mm, and I.D. = 0.4 mm). The feeding rate of the polymer solutions was 0.25 mL/h and take-up speed 100 RPM was collected electrospun nanofiber. The electrospinning of PAN was performed at 22 ± 2°C and relative humidity at 40–45%. After electrospinning the fibrous mats were washed with deionized water and dried at 40°C for 8 h.

Measurement and characterization

The morphology of the electrospun nanofibers was examined by scanning electron microscope (SEM, AIS-2100, Seron, Korea) at an accelerating voltage of 25 kV under magnification of $35,000 \times$ and the average fiber diameter was measured with the SEM images using Image J software (National Institute of Health, USA) from 200 fibers/sample. A typical SEM photograph of the electrospun nanofiber mat and its corresponding diameter distribution are shown in Figure 3.

EXPERIMENTAL DESIGN

Response surface methodology

The effects of the three independent processing parameters namely; polymer concentration (% w/v), applied voltage (kV) and nozzle-collector distance (cm) on nanofiber diameter (nm) were investigated using response surface methodology. Box–Behnken



DC high voltage supply

Figure 2 Schematic diagram of a general type of electrospinning apparatus in this work.





Figure 3 (a) A typical SEM photograph of electrospun nanofiber mat, (b) corresponding fiber diameter distribution (polymer concentration: 12 w/v%, nozzle-collector distance: 15 cm, applied voltage: 14 kV).

designs are response surface designs, specially employed to require only three levels, coded as -1, 0, and +1, according to eq. (1).

$$X_{i} = \frac{\xi_{i} - [\xi_{\text{Hi}} + \xi_{\text{Li}}]/2}{[\xi_{\text{Hi}} - \xi_{\text{Li}}]/2}$$
(1)

where, ξ_{Hi} and ξ_{Li} refer to the high and low levels of the variables ξ_i (*i* = 1,2,3), respectively.

The total number of experiments (N = 17) in this study with three factors was obtained from the equation: $N = k^2 + k + cp$, where k is the number of factors (=3) and cp is the center of the design (=5) for estimation of a pure error sum of squares. The statistical software package, Design-Expert (Version 8.0.3, Stat-Ease, Minneapolis, MN, 2010) was used for the regression analysis of the experimental data, and to plot the response surface graphs. The corresponding actual values and the coded design experiments for each variable are listed in Tables I and II, respectively.

TABLE IActual and Coded Values of the Variables

	Actual values				
Coded values	Polymer concentration (%w/v) (ξ ₁)	Applied voltage (kV) (ξ ₂)	Nozzle-collector distance (cm) (ξ ₃)		
-1	10	12	12		
0	12	14	15		
1	14	16	18		

In a system involving three significant independent variables X_1 , X_2 , X_3 the mathematical relationship between the response and these variables can be approximated by the quadratic polynomial equation:

$$Y = \beta_0 + \sum_{i=1}^{3} \beta_i X_i + \sum_{i=1}^{3} \beta_{ii} X_i^2 + \sum_{i=1}^{2} \sum_{j=i+1}^{3} \beta_{ij} X_i X_j \quad (2)$$

where, Y is the predicted response, X_i , X_j are independent variables, β_0 is the offset term, β_i is the *i*th linear coefficient, β_{ii} is the *i*th quadratic coefficient, and β_{ij} is the *i*jth interaction coefficient.

The equations were validated by the statistical tests called the ANOVA analysis. The quality of the fitted quadratic model was expressed by the coefficient of determination R^2 and $adj-R^2$. Response surfaces were drawn to determine the individual and interactive effects of the test variable on the nanofiber diameter.

TABLE II The Box-Behnken Experimental Design for the Three Independent Variables and Response at Different Factor Levels

	Coded	Response		
No.	Polymer concentration (X ₁)	Applied voltage (X ₂)	Nozzle-collector distance (X ₃)	Mean diameter ± std (nm)
1	0	0	0	194 ± 17
2	-1	0	1	138 ± 19
3	0	1	1	175 ± 20
4	0	1	-1	$203~\pm~21$
5	1	1	0	236 ± 20
6	1	0	-1	$254~\pm~30$
7	-1	0	-1	153 ± 21
8	0	0	0	197 ± 18
9	-1	-1	0	124 ± 17
10	0	0	0	199 ± 18
11	-1	1	0	161 ± 19
12	0	-1	1	208 ± 16
13	1	-1	0	276 ± 31
14	0	0	0	198 ± 20
15	1	0	1	263 ± 29
16	0	-1	-1	$184~\pm~17$
17	0	0	0	193 ± 21

 TABLE III

 Analysis of Variance (ANOVA) for Response Surface Quadratic Model for Fiber Diameter

Source	SS	DF	MS	<i>F</i> -value	Prob > F	Remarks
Model	28,202.57	9	3,133.73	515.54	< 0.0001	Significant
X ₁ -Concentration	25,651.13	1	25,651.13	4,219.93	< 0.0001	Significant
X ₂ -Voltage	36.13	1	36.13	5.94	0.0449	Significant
X ₃ -Distance	12.50	1	12.50	2.06	0.1947	0
X_1X_2	1,482.25	1	1,482.25	243.85	< 0.0001	Significant
$X_1 X_3$	144.00	1	144.00	23.69	0.0018	Significant
X_2X_3	676.00	1	676.00	111.21	< 0.0001	Significant
$\overline{X_1X_1}$	165.79	1	165.79	27.27	0.0012	Significant
X_2X_2	43.79	1	43.79	7.20	0.0314	Significant
X ₃ X ₃	0.95	1	0.95	0.16	0.7044	0
Residual	42.55	7	6.08			
Lack of fit	15.75	3	5.25	0.78	0.5620	

Artificial neural network

In this work, multilayer perceptron ANN with one hidden layer, according to Kolmogorov's theorem was utilized. For all data sets hyperbolic tangent sigmoid transfer function [eq. (3)] in the hidden layer and a linear transfer function [eq. (4)] in the output node was employed.¹⁹

$$f(x) = \frac{e^x - e^{-x}}{e^x + e^{-x}}$$
(3)

$$g\left(x\right) = x \tag{4}$$

The ANN was trained using the scaled conjugate gradient backpropagation algorithm (*trainscg*). The experimental data were divided into two groups training and test with 11 and 6 samples, respectively. Moreover, any samples (*Y*) (from the training and test sets) were coded to a new value Y_{norm} as eq. (1). All calculations carried out in Matlab mathematical software (version 7.6) with ANN toolbox.

To determine the optimum number of neuron in hidden layer, a series of topologies was used, in which the number of neuron were varied from 2 to 10. Each topology was repeated three times to avoid random correlation due to random initialization of the weights and bias. The optimal architecture of the ANN model and its parameter variation were determined based on the minimum value of the mean square error (MSE) of the training and testing sets.^{20,21}

RESULTS AND DISCUSSION

Response surface methodology

The analysis of variance for the response has been summarized in Table III. The values of *P*-values less than 0.05 indicates that the model terms are significant, whereas, the values greater than 0.05 are not significant. The ANOVA analysis of the optimization study indicated that the model terms, X_1 , X_2 , X_1X_2 , X_1X_3 , X_1X_1 , and X_2X_2 were significant (P < 0.05) and X_3 , X_3X_3 were not significant (P > 0.05).

The experimental results were evaluated and the approximating function of the average nanofiber diameter obtained from eq. (5):

$$Y = 196 + 56.63X_1 - 2.12X_2 - 19.25X_1X_2 + 6X_1X_3 - 13X_2X_3 + 6.25X_1^2 - 3.25X_2^2$$
(5)

The model *P*-values (<0.0001) and lack of fit value (0.5620) suggested that the obtained experimental data has a good agreement with the model. The regression equation obtained from the ANOVA showed that the R^2 was 0.998. However, since the model equations in our case include additional terms because of the three level independent variables, the adjusted R^2 for the degrees of freedom (adj- R^2) was chosen to be examined as well. Adj- R^2 is much less sensitive to the degrees of freedom and cannot be affected as seriously by including more terms in the model, while it is always lower than R^2 . Therefore, it is a better criterion of the goodness of the fit. The adj- R^2 value for the response was found to be equal to 0.996.

Artificial neural networks results

The optimal architecture of the ANN model and its parameter variation were determined based on the minimum value of the MSE of the training and prediction set. In optimization of the neural network, two neurons were used in the hidden layer as an initial estimate. Figure 4 illustrates the relation between network error (MSE) and number of neurons in the hidden layer. As can be seen, the MSE is minimum just about five neurons.

Hence we used two-layered perceptron neural network (with five artificial neuron in hidden layer) for modeling of PAN nanofiber diameter (Fig. 5). The ANN was trained up to 3000 cycles to obtain optimum weights and bias. The weights and bias of ANN for the diameter of electrospun nanofiber are given in Table IV. The R^2 value was 0.990, which





Figure 4 Effect of the number of neurons in hidden layer on the performance of the ANN.

indicates that the model was shows good fitting with experimental data.

Effects of significant parameters on response

Contour surface plots of the PAN nanofiber diameter (nm) for the experimental factors (concentration, voltage, and distance) are shown in Figure 6.

As described in the literature, increasing polymer concentration will result in greater viscoelastic force enabling the charged jet to withstand a larger electrostatic stretching force and leading to a larger diameter of electrospun nanofibers.^{10,11} Figure 6(a) shows the average diameter of the electrospun nanofibers at different polymer concentration and applied voltage for middle nozzle-collector distance level (15 cm). It can be seen that at any given voltage the nanofiber diameter increases with increasing the polymer concentration. Also, in lower concentration (<12 w/v%) increase in applied voltage result in greater fiber diameter, but at the higher concentration



Figure 5 Optimized two-layer perceptron neural network structure.

tion (>12 w/v%) increase in applied voltage result in thinner fiber diameter.

Figure 6(b) shows the contour plot of average diameter of the electrospun nanofibers at different polymer concentration and nozzle-collector distance for middle applied voltage level (14 kV). In lower concentration (i.e., 10 and 12 w/v%) increase in distance result in finest fiber diameter, but in higher concentration (14 w/v%) increase in distance result in thicker fiber diameter. Also, it can be seen that any given distance the nanofiber diameter at increases with increasing the polymer concentration. Figure 6(c) shows the average diameter of the electrospun nanofibers at different applied voltage and nozzle-collector distance for polymer concentration of 12 w/v%. Applied voltage and distance have two major different interaction effects on the electrospun nanofiber diameter. In low range of applied voltage, increase the distance between nozzle tip-collector results in thicker fiber diameter and this behavior is inverted after the critical point (saddle point) on the response surface.

The neural network weight matrix can be used to assess the relative importance (RI) of the various input variables on the output variables. It was proposed an equation based on the partitioning of connection weights^{20,22}:

$$\operatorname{RI}_{j} = \frac{\sum_{m=1}^{N_{h}} \left(\left(\left| \operatorname{IW}_{jm} \right| / \sum_{k=1}^{N_{i}} \left| \operatorname{IW}_{km} \right| \right) \times \left| \operatorname{LW}_{mn} \right| \right)}{\sum_{k=1}^{N_{i}} \left\{ \sum_{m=1}^{N_{h}} \left(\left(\left| \operatorname{IW}_{km} \right| / \sum_{k=1}^{N_{i}} \left| \operatorname{IW}_{km} \right| \right) \times \left| \operatorname{LW}_{mn} \right| \right) \right\}} \times 100 \quad (6)$$

where RI_j is the relative importance of the *j*th input variable on the output variable, N_i and N_h are the numbers of input variables and hidden neurons, respectively, IW and LW are connection weights, and subscript "*n*" refer to output response. In this work: *j* = 1, 2 and 3, $N_i = 3$, $N_h = 5$, and n = 1.

TABLE IV Weights and Bias Obtained in Training ANN

Layer			Weight			Bias
Hidden layer	IW ₁₁	IW ₁₂	IW ₁₃			<i>b</i> ₁₁
5	-0.643	0.688	2.311			2.249
	IW ₂₁	IW ₂₂	IW ₂₃			b_{21}
	0.790	-0.066	-0.125			-0.055
	IW ₃₁	IW ₃₂	IW33			b_{31}
	-0.906	1.016	-0.670			1.459
	IW_{41}	IW_{42}	IW_{43}			b_{41}
	-1.024	-1.078	-1.379			-1.248
	IW_{51}	IW_{52}	IW_{53}			b_{51}
	-1.155	-2.266	-0.956			-1.882
Output laver	LW ₁₁	LW_{12}	LW ₁₃	LW_{14}	LW ₁₅	Ь
-	0.035	0.834	-0.376	-0.348	-0.480	0.037

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Figure 6 Interaction contour surface plots of the response variables: (a) effect of concentration and voltage at middle level of distance (15 cm), (b) effect of concentration and distance at middle level of voltage (14 kV), and (c) effect of voltage and distance at middle level of concentration (12 w/v%).

The relative importance of input variables on the value of average nanofiber diameter as calculated by eq. (6) was shown in Figure 7. As can be seen, all of



Figure 7 Relative importance of input variables on the value of electrospun fiber diameter.

the variables (polymer concentration, applied voltage, and nozzle-collector distance) have strong effects on the average of nanofibers diameter. Therefore, none of the variables studied in this work could



Figure 8 (a) SEM photograph of electrospun nanofiber mat, (b) corresponding fiber diameter distribution.

TABLE V Experimental and Predicted Values of Electrospun Fiber Diameter by RSM and ANN Models

		Drod	istad	Absolute	
		11eu	error (70)		
No	Experimental	RSM	ANN	RSM	ANN
1	194	196	197	1.03	1.55
2	138	140	143	1.45	3.62
3	175	178	180	1.71	2.86
4	203	203	202	0.00	0.49
5	236	234	240	0.85	1.69
6	254	253	253	0.39	0.39
7	153	152	154	0.65	0.65
8	197	196	197	0.51	0.00
9	124	125	124	0.80	0.00
10	199	196	197	1.51	1.01
11	161	160	148	0.62	8.07
12	208	208	209	0.00	0.48
13	276	277	279	0.36	1.09
14	198	196	197	1.01	0.51
15	263	265	270	0.76	2.66
16	184	182	184	1.09	0.00
17	193	196	197	1.55	2.07
R ² -values		0.998	0.990		
Mean absolute error (%)				0.84	1.60

have been neglected from the present analysis. However, the polymer concentration and applied voltage, with relative importance of respectively 50.1 and 28.5%, appeared to be more influential parameters in the average of nanofibers diameter. These results are in good agreement with the ones obtained with RSM.

Optimizing the nanofiber diameter

In this work, our goal is to minimize the average of nanofibers diameter. Optimization finds a good set of conditions that will meet the minimum diameter. The conditions for finest diameter estimated by the RSM equation were polymer concentration $(X_1) = 10 \text{ w/v}\%$, applied voltage $(X_2) = 12 \text{ kV}$, and nozzle-collector distance $(X_3) = 12 \text{ cm}$. Figure 8 shows the

nanofiber morphology observed by SEM together with the corresponding histogram of the nanofiber diameter distribution. The average nanofiber diameter was estimated to be 121 \pm 16 nm with diameter ranging from 80 to 162 nm.

The theoretical fiber diameter under the above conditions was $Y_{\text{RSM}} = 118$ nm. The experimental results observed (121 nm) was 2.5% greater than the predicted value under the same electrospinning settings.

Comparison between ANN and RSM

The experimental and predicted values using RSM and ANN model were given in Table V. Both RSM and ANN model shows a very good relationship between the experimental and predicted response values. The RSM model shows much lower absolute percentage error than the ANN model. However, it should be noted that both the models have an error percentage less than 2% indicating the reliability of the model developed.

The last step of the RSM was validation study that used for showing how close the new offered conditions by software and experimented conditions. A validation study was performed (by conducting additional experiments) for each of the three factors (polymer concentration, applied voltage, and nozzlecollector distance) to confirm the validity and accuracy of the RSM and ANN models. The result of new offered and experimented conditions was shown in Table VI. There are no significant differences between predicted data by models and experimented data.

Figure 9 shows the nanofiber morphology observed by SEM together with the corresponding histogram of the nanofiber diameter distribution. According to SEM micrographs of nanofiber (Fig. 9), as can be seen RSM and ANN have proper prediction in new experimental conditions. Considering to RSM and ANN results in new experimental conditions that follow the initial data, it can be concluded

 TABLE VI

 Validation of RSM and ANN Using Different Levels of Polymer Concentration, Applied Voltage, and Nozzle-Collector Distance

No	Actu	Nanofiber diameter (nm)				
	Polymer concentration (%w/v)	Applied voltage (kV)	Nozzle-collector distance (cm)	Experimental	RSM	ANN
A	13	12	13.5	227	226	223
В	14	16	12	239	241	241
С	11	13	13.5	161	163	162
D	10	15	18	145	141	142



Figure 9 SEM photographs of electrospun nanofibers and corresponding fiber diameter distribution. (a–d) are nanofibers from corresponding experiments in Table VI.

that the models mentioned above are acceptable in all space design.

CONCLUSIONS

This article presented a study on the effects of processing variables, including polymer concentration (w/v%), applied voltage (kV), and nozzlecollector distance (cm), on the average diameter of electrospun PAN nanofibers. Response surface methodology (RSM) and artificial neural network (ANN) were used to modeling and optimizing of the average nanofiber diameters. The RSM analysis confirmed that polymer concentration and applied voltage were the main significant factors affecting the average nanofiber diameters. Also, all interaction effect terms were found to be significant. The configuration of the artificial neural network giving the smallest MSE was a two-layer ANN with tangent sigmoid transfer function at hidden layer with five neurons, linear transfer function at output layer, and scaled conjugate gradient backpropagation training algorithm. High regression coefficient between the variables and the response $(R^2 = 0.998)$ indicates excellent evaluation of experimental data by quadratic polynomial regression model. The R^2 value was 0.990, which indicates that the ANN model is superior. Moreover, the RSM model shows much lower absolute percentage error than the ANN model. Therefore, the obtained results indicate that the performance of RSM was better than ANN. On the basis of the function, the finest value for each process variable was also determined for PAN concentration $(X_1 =$ 10 w/v%), applied voltage ($X_2 = 12$ kV), and nozzle-collector distance ($X_3 = 12$ cm). The experimental results observed average diameter of nanofibers (121 nm) was 2.5% greater than the predicted value (118 nm) under the same electrospinning settings.

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