Use of Artificial Neural Networks to Determine Parameters Controlling the Nanofibers Diameter in Electrospinning of Nylon-6,6

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ABSTRACT: This study aimed to find out the primary factors influencing the diameter of electrospun nanofibers of nylon-6,6 using artificial neural networks (ANNs). Four variables, namely, polymer concentration, working distance, injection rate, and applied voltage were considered as input parameters and the nanofibers diameter measured by scanning electron microscopy was taken as the output. The data were modeled and validated against a set of unseen data. The generated model was used to study the interactions occurring between the input variables and their effect on the diameter. Results show that the injection rate and the polymer concentration are major factors affecting the nanofibers diameter with inverse and direct relations with the diameter, respectively, while the working distance and the applied voltage have direct but minor effects on nanofibers diameter. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 1589–1597, 2012

Key words: electrospinning; artificial neural networks; nylon-6,6; electrospun nanofibers; nanofibers diameter

INTRODUCTION

In recent years, electrospinning has shown great potential to be employed as a unique technique to prepare polymeric, ceramic, and hybrid nanofibers. This technique involves production of nanofibers from a drop of polymer solution using a high electrostatic voltage. The electrospun nanofibers may be used in several applications such as sensors, membranes, filters, wound dressings, drug delivery, and tissue engineering scaffolds.[1–4] To obtain desired mechanical, electrical, optical, and biomedical properties, control of size and morphology of electrospun nanofibers is an inevitable approach. For instance, Acatay et al.[5] have studied the effect of morphology of the polyacrylonitrile electrospun nanofibers on the resultant hydrophobic behaviors. In other studies, Shin et al.[6] have shown that the size of produced nanofibers affects the filtering properties and Zhang et al. have investigated the effect of solution and process parameters on filtration efficiency in nylon-6,6.[7] Wu and Dzenis[8] have also theoretically showed that behavior of true axial tensile stress of solid nanofibers *versus* the axial tensile stretch may be varied by changing nanofibers radius. He et al.[9] have reported that electrospun nanofibers having less than 100 nm in diameters reveal unusual strength, high surface energy, surface reactivity, high thermal, and electric conductivity due to nano-effect.

Due to high level of complexity in electrospinning process,[10] the one-factor-at-a-time approach to recognize the relations between electrospinning parameters and the size/morphology is not only a time-consuming approach but also probably inefficient way. Thus, many reports have used statistical techniques to investigate the parameters of the process on the size and morphology of the obtained nanofibers.[11–15]

Electrospinning of nylon as a polymer with appropriate mechanical properties and stability was first reported in 1999.[16] Since then, considerable number of reports has detailed the electrospinning of nylons and its composites, studying different properties of the nylon nanofibers,[17] of which, the nanofibers diameter appears to be the dominant factor controlling the transport properties.[18]

Recently, artificial neural networks (ANNs), as brain mimickers in way of processing data have been widely used in dealing with complex and nonlinear

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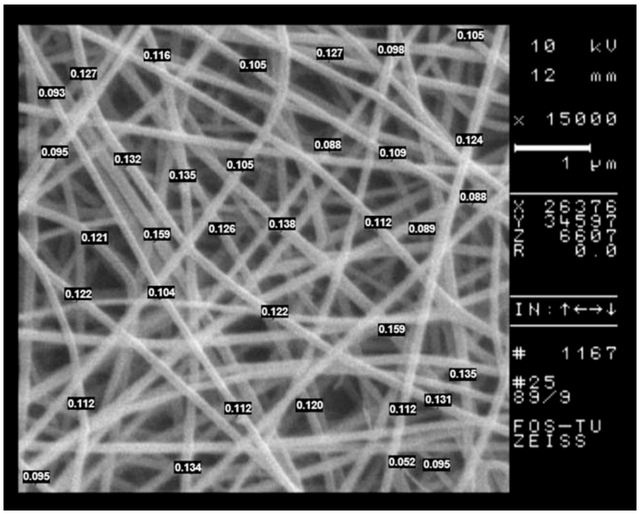


Figure 1 An SEM image obtained in the study.

TABLE I
The Training Parameters Set with INForm v4.0

Network structure	No. of hidden layers	1
	No. of nodes in hidden layer	3
Backpropagation type		RPROP
Backpropagation parameters	Momentum factor	0.8
	Learning rate	0.7
Targets	Maximum iterations	1000
	MS error	0.0001
	Random seed	10000
Smart stop	Minimum iterations	20
	Test error weighting	0.1
	Iteration overshoot	200
	Auto weight	On
	Smart stop enabled	On
Transfer function	Output	Tanh
	Hidden layer	Symmetric Sigmoid

TABLE II
The Training and Tests Data Sets Used in ANNs Modelling

Concentration (% wt/v)	Voltage (kV)	Distance (cm)	Rate (mL/h)	Observed diameter (nm)	Standard deviation	Predicted diameter (nm)
25.0	26.0	18.0	1.0	230.7	68.1	234.9
25.0	26.0	13.0	0.5	288.7	83.3	262.1
25.0	26.0	8.0	1.2	222.4	52.9	226.9
25.0	26.0	8.0	0.6	257.8	80.7	270.8
25.0	21.0	8.0	0.5	209.3	32.3	214.4
25.0	18.8	15.0	1.0	178.7	38.5	174.8
25.0	18.8	15.0	0.6	187.2	30.0	199.4
25.0	18.8	15.0	0.2	282.2	64.0	287.7
25.0	18.8	10.0	0.6	328.1	104.6	312.0
25.0	16.0	8.0	1.0	186.3	27.9	169.8
24.0	22.5	14.0	0.6	121.0	34.7	132.5
24.0	18.8	10.0	0.5	182.1	52.3	185.3
22.0	26.0	18.0	1.5	151.8	29.3	149.2
22.0	15.0	18.0	1.5	144.4	73.3	123.3
21.0	26.0	8.0	1.5	105.0	24.6	105.3
20.5	25.0	13.0	1.0	93.1	14.6	105.1
20.5	21.0	13.0	1.4	90.6	16.3	127.7
20.5	21.0	13.0	0.3	112.0	29.7	105.0
20.5	18.0	13.0	1.0	113.7	37.3	123.5
20.5	21.0	6.0	0.9	115.0	26.4	105.0
20.5	11.0	13.0	0.8	108.6	33.9	123.0
20.0	22.5	20.0	1.0	186.0	47.0	124.1
20.0	22.5	20.0	0.2	164.8	76.5	163.8
20.0	22.5	10.0	1.0	142.1	36.5	104.9
20.0	22.5	10.0	0.8	113.7	25.6	105.0
20.0	20.0	20.0	0.8	112.6	16.3	123.1
20.0	20.0	18.0	0.8	112.6	17.2	123.5
20.0	20.0	16.0	0.8	125.5	22.8	127.2
20.0	18.8	15.0	0.6	143.8	25.1	129.7
20.0	17.5	18.0	0.2	118.0	16.0	123.5
20.0	15.0	10.0	1.0	134.4	20.4	123.1
20.0	15.0	10.0	0.2	159.8	31.9	156.4
19.0	16.0	8.0	0.5	131.6	15.0	131.7
16.0	26.0	18.0	0.9	99.7	7.7	104.7
16.0	26.0	8.0	0.6	78.7	16.2	104.9
16.0	16.0	18.0	0.6	114.3	23.6	123.0
16.0	16.0	12.0	0.5	108.5	31.1	126.0
16.0	16.0	8.0	1.4	114.5	33.0	123.1
25.0	22.5	15.0	0.6	266.7	74.5	263.6[a]
25.0	16.0	11.0	1.5	177.3	24.0	148.2[a]
18.0	16.0	8.0	1.5	118.2	19.9	123.1[a]
16.0	16.0	11.0	1.5	95.0	23.2	123.0[a]

[a] The last 4 data show the test data.

TABLE III
The Validation (unseen) Data Sets Used in ANNs Modelling

Concentration (% wt/v)	Voltage (kV)	Distance (cm)	Rate (mL/h)	Observed diameter (nm)	Predicted diameter (nm)
25.0	26.0	10.0	1.5	217.8	195.9
25.0	21.0	18.0	1.5	165.7	163.9
25.0	20.0	15.0	1.0	179.8	186.2
20.5	21.0	13.0	0.9	97.7	122.4
20.5	21.0	8.0	1.0	104.4	104.8
20.0	17.5	16.0	0.2	115.3	127.5
17.0	18.8	15.0	1.0	146.9	123.4
16.0	26.0	13.0	1.5	70.4	104.8
16.0	26.0	8.0	0.9	85.6	104.9

relations. The massive interconnected structure makes ANNs an exceptional tool which learns through input data while has the ability to model incomplete data without being affected by data noises.[19] Such techniques have proved to be more efficient compared with standard modeling techniques such as response surface methodology (RSM).[20,21] Additionally, the complexities observed in electrospinning process, makes the observed data from this technique considerably noisy, showing a second reason for employing ANNs instead of classical statistical tools when working with electrospinning. Previous works on the use of ANNs in developing models to study the electrospinning parameters and their effects on the size/morphology properties[13,22,23] have also indicated the usefulness of this technique.

In this work, from various parameters affecting the diameter of electrospun nanofibers of nylon-6,6, four main parameters including the polymer concentration, the applied voltage, the working distance (i.e., the distance between the needle and collector), and injection rate of polymer solution were chosen to analyze and model their effects on the diameter size of electrospun nylon-6,6 using ANNs as a systematic approach to determine the interactions among variables of electrospinning. Using this approach, it is possible to simultaneously study the interactions of

TABLE IV
3D Plots of Nanofibers Diameter Predicted by the ANNs Model Fixed at Low, Mid-Range, and High Values of the Voltage and Concentration. [Color table can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

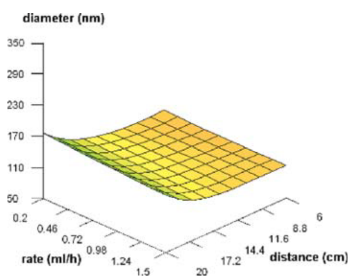
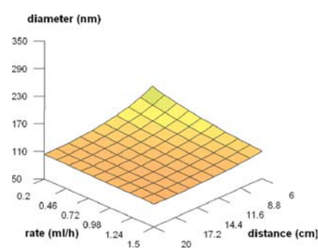
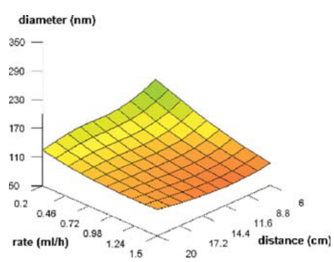
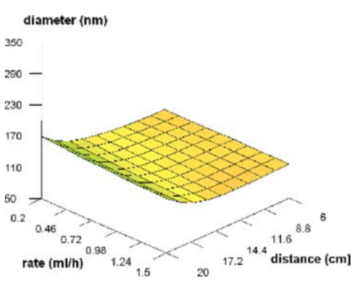
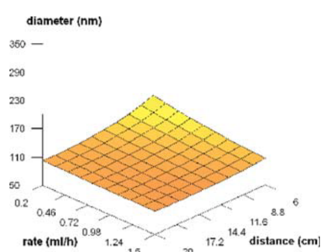
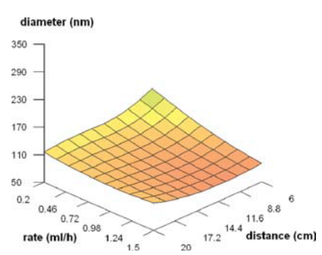
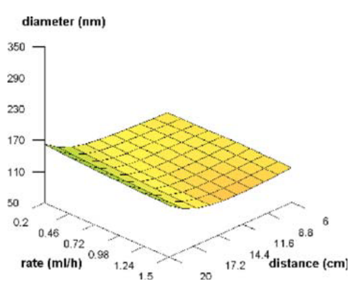
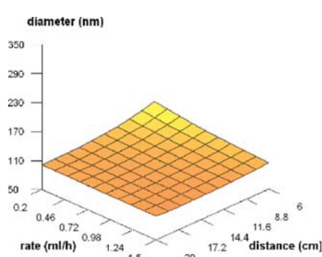
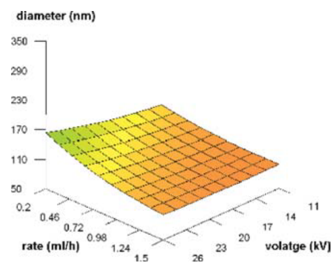
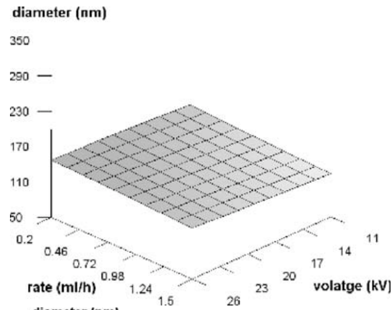
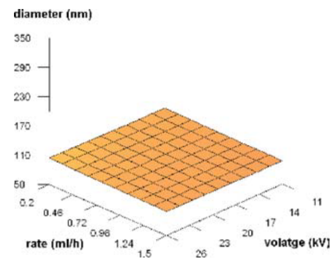
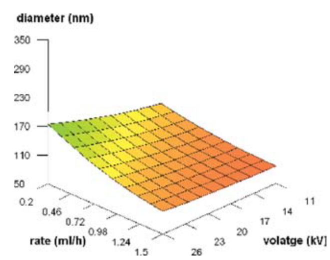
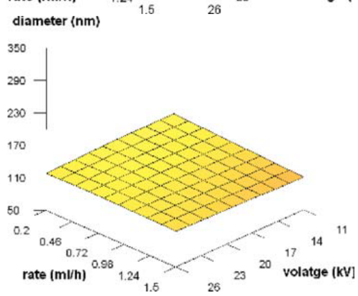
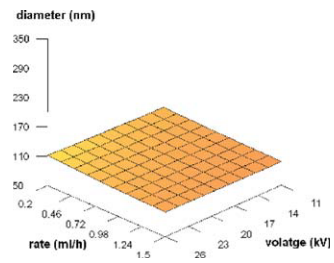
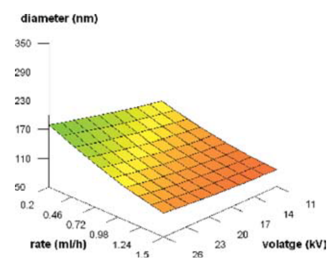
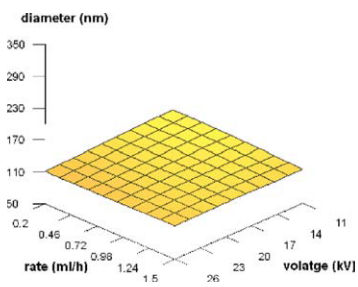
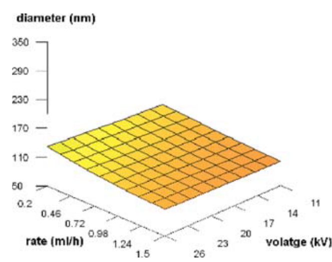
		Concentration		
		High (23.5% wt/v)	Mid-range (20.5% wt/v)	Low (17.5% wt/v)
Voltage	High (23.5 kV)			
	Mid-range (18.5 kV)			
	Low (13.5 kV)			

TABLE V
3D Plots of Nanofibers Diameter Predicted by the ANNs Model Fixed at Low, Mid-Range, and High Values of the Distance and Concentration. [Color table can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

		Concentration		
		High (23.5% wt/v)	Mid-range (20.5% wt/v)	Low (17.5% wt/v)
Distance	High (17.7 cm)			
	Mid-range (13.0 cm)			
	Low (8.3 cm)			

the electrospinning parameters in nylon-6,6 solution and their effects on the nanofibers diameters.

MATERIALS AND METHODS

Materials and apparatus

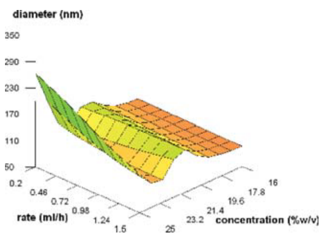
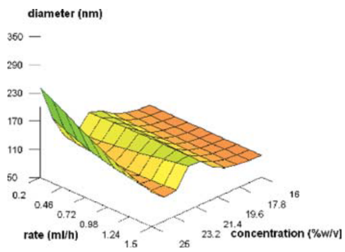
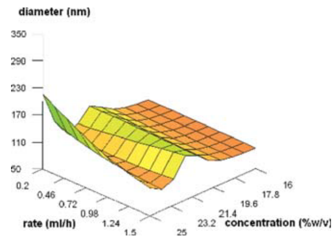
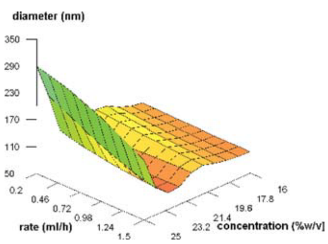
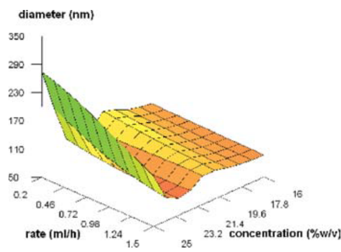
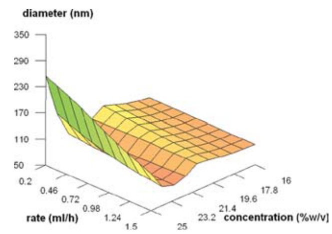
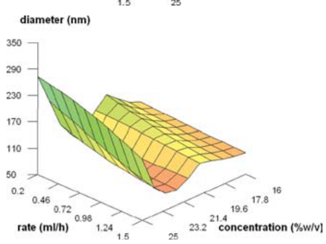
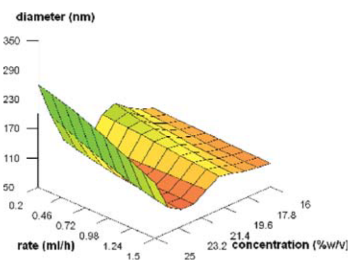
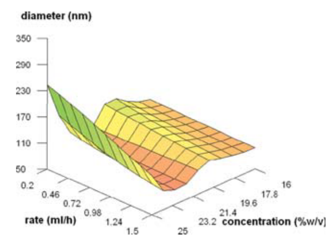
Nylon-6,6 (polyamide-6,6) was of technical grade (medium viscous, DSM Co., The Netherlands). Formic acid was purchased from Merck chemicals co. (Germany). The electrospinning process was carried out using Electroris (FNM Ltd., Iran, www.fnm.ir) as an electrospinner device having a high voltage and a syringe pump controllable in range of 1–35 kV and 0.1–100 mL/h, respectively. This device can control the electrospinning parameters such as the injection rate, the drum speed (i.e., speed of rotating cylindrical collector), the working distance, the needle scanning rate, and the temperature of the electrospinning media.

Methodology

Nanofibers of nylon-6,6 were prepared using the electrospinning method as described previously.[12] Briefly, plastic syringes fitted with metal needles were used as the polymer solution reservoir and nozzle for electrospinning. Positive electrode of the power supply was connected to the metal needle to charge the polymer solution injected by the syringe pump of electrospinner. A grounded aluminum foil was placed as a collector at a fixed distance from the needle during electrospinning. The electrospinning processes were performed at temperature and relative humidity of 30 (±2)°C and 35 (±2)%, respectively, with formic acid (90% wt) as the solvent. The recipe of the experiments is given in Table II. The average diameters of the electrospun nanofibers was determined by measuring and averaging the diameter of approximately 40 random nanofibers in each sample using scanning electron microscopy (ZEISS DSM 960A, Germany) after sputtering by gold. The scanning electron microscopy (SEM) results showed no significant bead in the samples. Figure 1 shows a typical SEM image of the prepared samples.

INForm v4.0 (Intelligensys, UK), as a commercial ANNs software was used in this study to model the

TABLE VI
3D Plots of Nanofibers Diameter Predicted by the ANNs Model Fixed at Low, Mid-Range and High Values of the Distance and Voltage. [Color table can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

		Voltage		
		High (23.5 kV)	Mid-range (18.5 kV)	Low (13.5 kV)
Distance	High (17.7 cm)			
	Mid-range (13.0 cm)			
	Low (8.3 cm)			

relations between inputs and outputs. From our previous study,[12] four factors, namely the polymer concentration (% wt/v), the applied voltage (kV), the working distance (cm), and the rate of injection (mL/h), were considered as input variables of the ANNs and the average of the nanofibers diameter was chosen as the output.

Experimentally, 42 electrospinning experiments were carried out and the obtained nanofibers mats were analyzed using SEM. The obtained data set was then fed to the software. Using INForm v4.0, the data were randomly divided into two groups: the training and the test data sets. The training data set was used to train the network and obtain the relations between the variables/output using the training parameters listed in Table I, while the test data set (10% of the data set, as recommended by the software) is used to stop the learning process before occurrence of overtraining. Upon start of overtraining, the correlation coefficient [see eq.(1)] of the test data decreases and forces the training process to stop. Additionally, as recommended by the software, the maximum number of iterations was set to 1000. Table II lists the training and the test data along with the predicted value by the software.

Subsequent to training, further nine samples were prepared and analyzed using SEM. The obtained data set was used as validation data to assess the ability of the trained network in prediction of "unseen" data (validation data) (see Table III). To validate the quality of the trained model, the predicting ability of the model was confirmed using the correlation coefficient R-square (R^2) for training, test, and validation data from eq. (1):

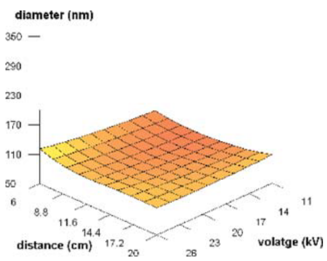
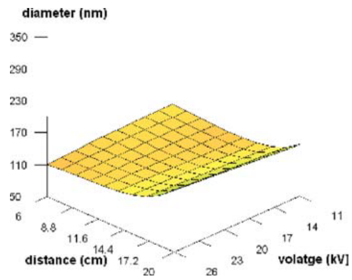
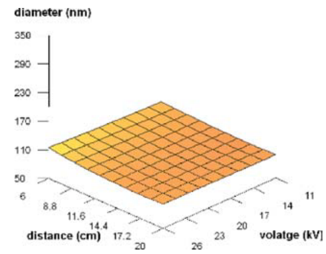
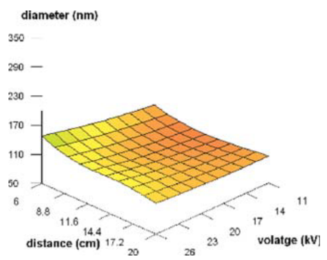
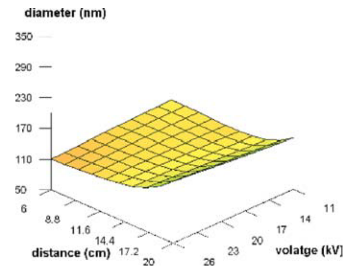
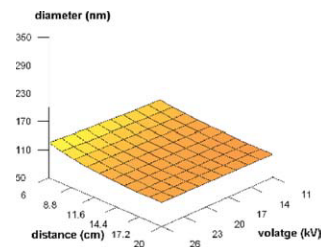
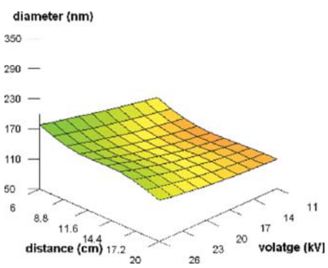
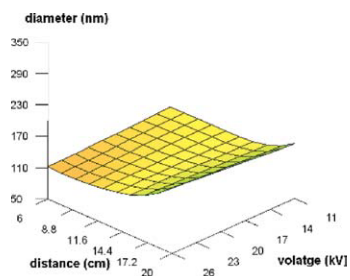
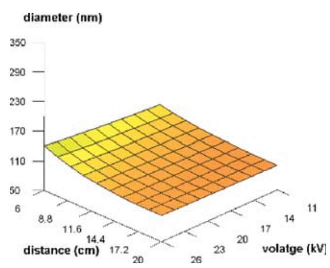
$$R^2 = 1 - \frac{\sum_{i=1}^{n}(y_i - \hat{y}_i)^2}{\sum_{i=1}^{n}(y_i - \bar{y}_i)^2} \quad (1)$$

where $\hat{y}$ is the value predicted by the model and $\bar{y}$ is the mean of dependent variable. An acceptable ANNs model needs to have satisfactory R^2 for all training, test, and validation data. Details of the three types of the data set have been provided elsewhere.[24]

RESULTS AND DISCUSSION

After modeling the data using the ANNs, the best predictive model resulted in R^2 values of 0.92, 0.91,

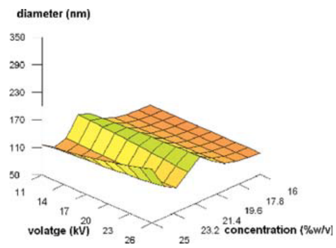
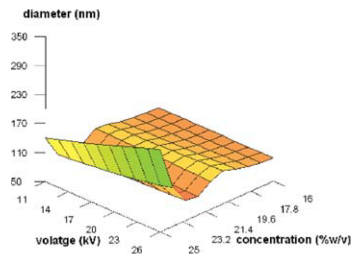
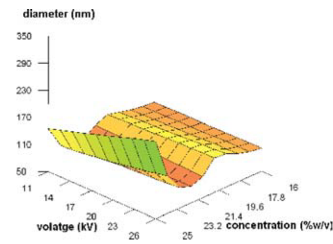
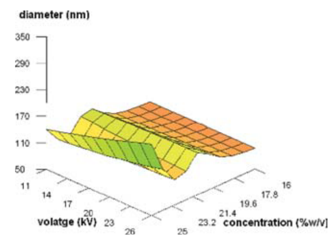
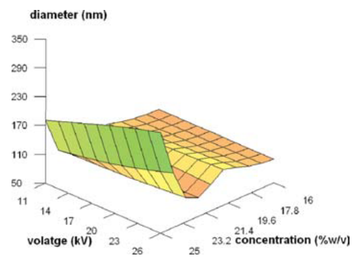
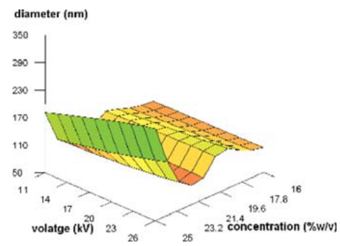
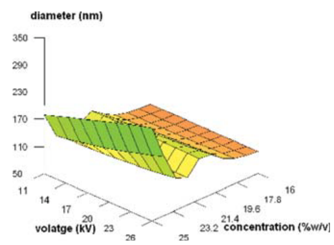
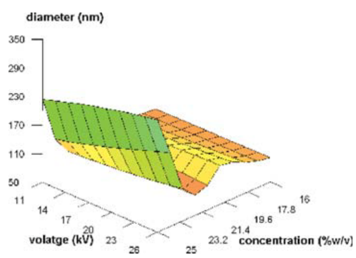
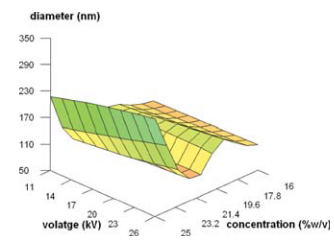
TABLE VII
3D Plots of Nanofibers Diameter Predicted by the ANNs Model Fixed at Low, Mid-Range, and High Values of the Concentration and Rate. [Color table can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

		Concentration		
		High (23.5% wt/v)	Mid-range (20.5% wt/v)	Low (17.5% wt/v)
Rate	High (1.28 mL/h)			
	Mid-range (0.85 mL/h)			
	Low (0.42 mL/h)			

and 0.82 for the training, test, and validation data, respectively. Considering the very high degrees of complexity in relations between the processing conditions and the diameter of the electrospun nanofibers,[9] these values indicate a satisfactory trained model. This model was then employed to study the effect of the different input variables on the diameter of the electrospun nanofibers. To analyze the obtained model, sensitivity analysis approach may be the first option which enables ranking the input variables. However, we used the semiquantitative approach that was reported previously[25–27] to further examine the interactions between the input variables and/or the input/output variables. To brief the method, all but two of the input variables are fixed at three specific values (i.e., a low, a mid-range, and a high value) and for each set of fixed values, the effect of the remaining two variables on the output are visualized using the response surfaces as 3D-graphs produced by the software. Employing this approach, the effect of the two input variables on the output are visualized when the remaining input variables are fixed at low, medium, or high values.

To follow this methodology, in this work to study the effect of working distance and injection rate on the nanofibers diameter, we first fixed the values for applied voltage and polymer concentration at their low, mid-range, and high values (i.e., 13.5, 18.5, and 23.5 kV for voltage and 17.5, 20.5, and 23.5% wt/v for concentration) and reported the generated graphs in Table IV. Details show that in general, increasing the rate would result in reduction of nanofibers diameter. The literature shows that while increase in the flow rate of polymer solution typically increases the nanofibers diameter,[23,24] in some cases the flow rate increase leads to decrease in observed nanofibers diameter.[7,28] The reason for this finding (i.e., reverse relation between flow rate and diameter) could be the fact that by decreasing the injection rates, the solvent on tip of the injection needle will go under further evaporation, compared with high rates of injection. This may increase the

TABLE VIII
3D Plots of Nanofibers Diameter Predicted by the ANNs Model Fixed at Low, Mid-Range, and High Values of the Distance and Rate. [Color table can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

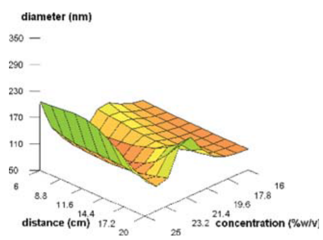
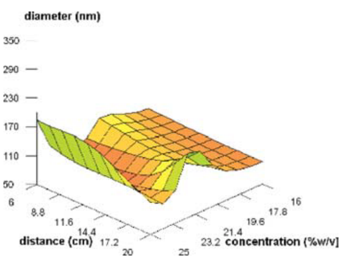
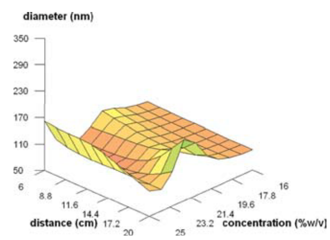
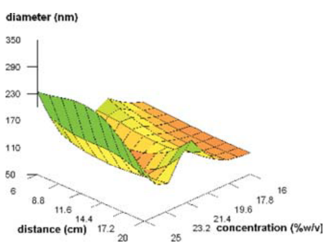
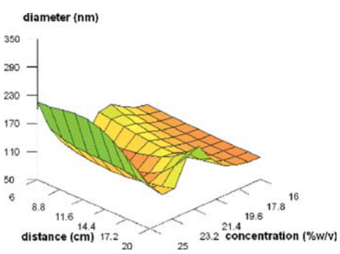
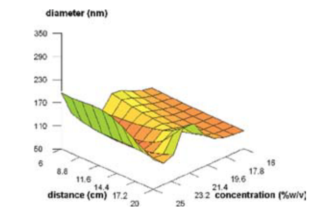
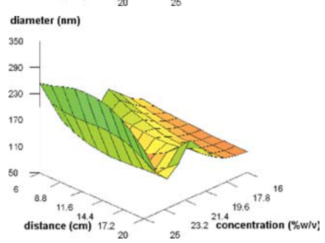
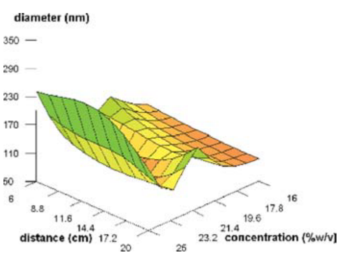
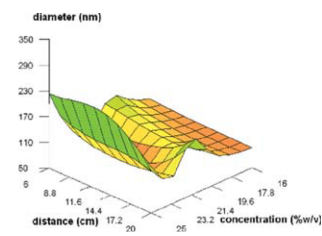
		Distance		
		High (17.7 cm)	Mid-range (13.0 cm)	Low (8.3 cm)
Rate	High (1.28 mL/h)			
	Mid-range (0.85 mL/h)			
	Low (0.42 mL/h)			

concentration of polymer solution on tip of the injection needle and causes an increase in diameter of nanofibers.

The effect of working distance on the nanofibers diameter is however contradictory, while in some cases, it has a direct effect on the diameter (i.e., low and high values for concentration), others (i.e., medium concentration values) show a reverse relation between the working distance and the diameter. Reviewing the literature shows that an increase in the distance between the needle and the collector usually results in a decrease in the nanofibers diameter.[16,27] This is probably due to breaking the formed jet into two or more jets, causing smaller diameter of the fibers. Nevertheless, there are some reports showing that a direct relation may be observed between the nanofibers diameter and the working distance.[23] This is due to the decrease in the electrostatic field strength resulting in less stretching of the fibers.[28]

Considering the details in Table V, where the polymer concentration and working distance are fixed at low, mid-range, and high (i.e., 17.5, 20.5, and 23.5% wt/v for concentration and 8.3, 13.0, and 17.7 cm for distance, respectively) to generate plots of nanofibers diameter against injection rate and applied voltage, confirms the importance of injection rate on the nanofibers diameter. The increase in the injection rate leads to a considerable decrease in the nanofibers diameter. Interestingly, the effect of injection rate on the size becomes more significant at higher concentration values. This could be associated with the increase in viscosity of solution due to synergic effect of increase in the concentration and decrease in the injection rate. Additionally, at low and medium concentration, increase in applied voltage results in a small decrease in the prepared nanofibers. This has been reported previously[16,23] and is thought to be a result of secondary jets during electrospinning at a stronger electric field or more stretches being performed on the polymer chains during electrospining.[28] However, at high concentration values, the increase in applied voltage increases the diameter. We believe, at such conditions due to high concentration values, the polymers stick to each other. Thus, the injected jets (Taylor jets) cannot be easily split to secondary jets while due to higher values of applied voltage, each jet carries more polymer

TABLE IX
3D Plots of Nanofibers Diameter Predicted by the ANNs Model Fixed at Low, Mid-Range, and High Values of the Voltage and Rate. [Color table can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

		Voltage		
		High (23.5 kV)	Mid-range (18.5 kV)	Low (13.5 kV)
Rate	High (1.28 mL/h)			
	Mid-range (0.85 mL/h)			
	Low (0.42 mL/h)			

which results thicker nanofibers compared to lower voltages.

Table VI summarizes the 3D plots of diameter-rate-concentration. From the data it is observed that the increase in the injection rate would result in smaller diameter values when the concentration is high. The effect of polymer concentration is however more complicated. The diameter decreases as the polymer concentration decreases. Decrease in the concentration of polymer in the solution leads to decrease in solution viscosity.[16] Less viscous solutions result in more polymer chain mobility and less polymer chain entanglement, which in turn causes more extension during electrospinning procedure and producing thinner and smaller nanofibers.[10]

The interaction of applied voltage and working distance and their effect on the nanofibers diameter is given in Table VII, where polymer concentration and injection rate are fixed (i.e., 17.5, 20.5, and 23.5% wt/v for polymer concentration and 0.4, 0.9, and 1.3 mL/h for injection rate). From the details, high values of injection rate at high concentrations result in low diameter values regardless of the effect of other variables. Also, voltage shows a direct relation with the diameter, findings that have been mentioned above. The opposing effect of distance on the diameter is clear from the details given in Table VII too.

Tables VIII and IX which illustrate the diameter–voltage–concentration and diameter–distance–concentration may also be used to validate the above findings:

- Injection rate has a key role in decreasing nanofibers diameter.
- The increase in polymer concentration in general results in increase in diameter.
- At high concentrations, applied voltage has a direct influence on the nanofibers diameter.
- Working distance shows only minor effect on the diameter. The outcome of long distance is generally smaller values for diameter of nanofibers at low/high concentrations while it is usually lager values at medium concentrations.

CONCLUSIONS

This study indicated the ability of ANNs in determining the primary factors affecting the fiber diameter in electrospinning. The 3D graphs used in this study showed that the injection rate is the most important factor with high values leading to smaller nanofibers diameter. Concentration of nylon-6,6 also showed a direct relation with the diameter. Applied voltage and working distance were shown not to have considerable effects on the diameter of electrospun nanofibers.

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