

Artificial Neural Networks Modeling of Electrospinning of Polyethylene Oxide from Aqueous Acid Acetic Solution

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ABSTRACT: The artificial neural networks (ANNs) were used to provide a model for investigating the relationships of the electrospinning parameters with the diameter of polyethylene oxide (PEO) nanofibers from acid acetic aqueous solution. The effects of four parameters including PEO concentration, acetic acid concentration, applied voltage, and temperature of the electrospinning media on the nanofibers mean diameter were investigated. To train, test, and valid the model, three datasets of the input variables with random values were prepared and the mean diameters obtained were taken as the output for the network. The datasets were analyzed by ANNs software and the correlation coefficient, R-squared (R^2), between the predicted values of the nanofibers mean diameter and actual amount were obtained. The results demonstrate the capability of the ANNs model for predicting the nanofibers diameter. The 3-D plots generated from the model show complex and nonlinear relationships between the param-

eters and nanofibers diameter. From the model, increasing the PEO concentration above a critical point leads to a sharp increase in the nanofibers mean diameter. The effects of applied voltage and temperature are mainly dependent on the PEO concentration. The acetic acid concentration, in general shows a direct relation with the nanofibers mean diameter. The plots also show that to produce nanofibers with the lowest diameter, both the PEO concentration and AcOH concentration should be at lowest values regardless the applied voltage and temperature. In contrast, highest nanofibers diameters are obtained when the PEO concentration and AcOH concentration are at their high values. © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 125: 1910–1921, 2012

Key words: electrospinning; nanofiber; artificial neural networks; PEO

INTRODUCTION

Among nanofibers production techniques, electrospinning is a popular approach because of its simplicity, speed, efficiency, and low preparation cost. In this process, a polymeric jet is driven through a high voltage electric field that renders a typical mesoscale fluid jet into nanoscale fibers. When the electric force of the induced charges on the polymer liquid overcomes the surface tension of polymer solution, a thin polymer jet is ejected. The charged jet, is elongated and accelerated by the electric field, undergoes a variety of instabilities, becomes dry and is deposited on a collector as a mat composed of

randomly oriented nanofibers. In this technique, the diameter of the nanofibers is the most important structural characteristic of the electrospun webs. Understanding how the fibers diameter and their distribution are affected by the processing variables is essential to produce nanofibers with desired properties for certain applications.^{1,2}

Various factors affect electrospun nanofibers diameter and morphology. These factors can be classified into three categories; the solution parameters, processing parameters, and ambient parameters. The main solution parameters are concentration, viscosity, conductivity, molecular weight, and surface tension. The major process parameters include applied electric field, tip to collector distance, and feeding or flow rate. A change in value of each parameter may affect morphology and diameter of the obtained nanofibers, and by proper manipulation of these parameters one can get nanofibers with desired morphologies and diameters. Additionally, ambient parameters such as humidity and temperature of surroundings have been shown to play a significant

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role in determining the morphology and diameter of electrospun nanofibers.¹ Because of the complexity and nonlinear relationships among parameters which affect nanofibers diameter,³ applying a one-factor-at-a-time approach to determine electrospun fibers diameter is inefficient and time consuming. Hence, an efficient approach which can predict fibers diameter with high precision is necessary. A number of investigators have used different ways to determine electrospinning parameters and resulting fibers diameter. Hohman et al. used a “whipping model” for the electrospinning process that mathematically describes the interaction between the electric field and fluid properties to predict “terminal” jet diameter.^{4,5} In another study, McKee et al. applied rheological model which rely on molecular weight and concentration as two process parameters that significantly influence the fibers diameter.⁶ Recently, dimensional analysis was used to predict fibers diameter for the electrospinning process elsewhere.⁷ These models have conflicting requirements such as surface tension or viscosity, inclusion or exclusion of fluid flow rate, allometric, or isotropic relations.³

Recently, artificial neural networks (ANNs) have been used as a new approach for modeling the interaction between nanofibers diameter and electrospinning parameters.⁸ ANNs are computational tools for pattern recognition and use computer technology to model a biologic neural system. By training on a known set of data, ANNs learn complex interaction among inputs and are capable to produce an output for new inputs.⁹ Sarkar et al. investigated the viability of ANNs as a tool for predicting the diameter of fibers formed by electrospinning process. Their results demonstrated the ability of the neural network approach as a promising tool for predicting nanofiber diameter.³

On the other hand, poly(ethylene oxide) (PEO) is a biocompatible polymer¹⁰ has been used in many applications such as wound dressing composites¹¹ and as injectable cartilage.¹² The solution of PEO in water, chloroform, dimethylformamide (DMF), alcohol, or water/alcohol has been electrospun in several works.^{13–18} PEO has been also added to other polymers such as cellulose derivatives¹⁹ and chitosan²⁰ to produce nanofibers in electrospinning technique. PEO has also been used as a matrix in inorganic–organic hybrid or nanocomposite nanofibers produced through electrospinning.²¹ However, electrospinning of PEO in acetic acid solution as solvent has not been considered yet.

In this study, we investigated the electrospinning of PEO in different concentration of aqueous acetic acid solution. We used the artificial neural networks to generate a model for determining the interactions and effects of three other electrospinning param-

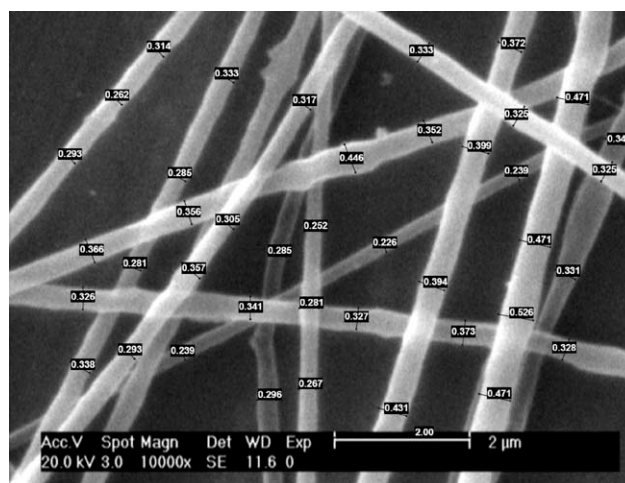


Figure 1 A typical sample of nanofibers mats of PEO that has been analyzed by image analyzer software.

eters, namely, PEO concentration, applied voltage, and temperature on the fibers mean diameter. Applying the response surfaces obtained from the model, the effects of each parameter on the fibers diameter were studied.

EXPERIMENTAL

Materials

Polyethylene oxide (PEO) (MW 900KD, Acros Organics) and glacial acetic acid (AcOH, Merck Chemical) were purchased. The electrospinning process was carried out using Electroris (FNM Ltd., Iran, www.fnm.ir) as an electrospinner device.

Electrospinning and measuring nanofibers diameter

The PEO solutions were prepared by dissolving various amount of PEO powder in different concentration of aqueous AcOH solution as solvent under stirring for 24 h at 37°C. For producing PEO nanofibers, the PEO solutions were placed into a 5 mL plastic syringe with a metallic blunt-ended 18G needle as nozzle for electrospinning. A sheet of aluminum foil was wrapped on the drum of electrospinner device as a collector. The needle was located at a distance of 14 cm from grounded collector. A syringe pump fed solution to the needle tip at injection rate of 1.0 mL/h. A positive high voltage was connected to the metallic needle and the collector was connected to the ground. The speed of drum was 15 rpm and the electrospinning time was about 10 min. A nanofibrous mat was formed on the aluminum foil in each electrospinning condition which was investigated for the diameter measurements.

The size of the produced nanofibers was determined by scanning electron microscopy (SEM)

TABLE I
Training and Testing Data Set for ANNs Modeling (the Last Three Ones Represent the "Test Data")

Sample No.	Input parameters				Output parameter
	PEO concentration (wt %)	AcOH concentration (% v/v)	Applied voltage (kV)	Temperature (°C)	Mean diameter (nm)
1	2	70	15	25	247
2	3.5	50	25	26.6	268
3	5	40	20	27.7	342
4	2.5	60	25	28	238
5	3.5	70	25	27.5	302
6	2	30	25	28	126
7	2	0	15	28.5	97
8	4.5	90	15	28.5	2900
9	4	30	15	28.5	229
10	4	30	10	35	390
11	3.5	20	24.5	30.2	310
12	2.5	100	25	30	579
13	4	80	15	30	315
14	4	80	25	45	695
15	3	30	10	33	330
16	2	90	10	40	509
17	3	0	15.8	42	289
18	2.5	50	25	35	280
19	2	40	25	36	195
20	2.5	40	20	38	261
21	5	10	10	40	361
22	4.5	10	24	30	239
23	3	10	20.5	40	232
24	3	10	24	42	216
25	5	30	12	40	945
26	3.5	50	10	43	405
27	2	10	15	42	193
28	2	60	25	45	268
29	2.2	85	10	28.5	428
30	2.8	75	15.5	28	490
31	4.8	5	11.9	36.5	450
32	3.3	35	17.9	37.5	493
33	4.1	25	22	43	497
34	2.3	95	23.3	26.5	347
35	3.1	45	18.8	39.3	604
36	4.1	25	17	25	361
37	3	0	20	32	161
38	3.7	55	20.8	33.7	459
39	4.3	15	16	35.5	438

TABLE II
Validation Data Set Used to Validate the Generated Model

Sample No.	Input parameters				Output parameter
	PEO concentration (wt %)	AcOH concentration (% v/v)	Applied voltage (kV)	Temperature (°C)	Mean diameter (nm)
40	4	20	25	28	213
41	3.5	10	10	26	269
42	2.5	0	10	30.9	198
43	3	20	15.7	35	361
44	3	70	10	25	786
45	4.5	80	20.5	30	1638
46	3	10	15	35	285
47	5	90	20	45	2472
48	3.2	65	12.6	30	456

TABLE III
The Training Parameters Set with INForm v4.0

Network structure	No. of hidden layers	1
	No. of nodes in hidden layer	6
Backpropagation type		RPROP ^a
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	0.083333
	Iteration overshoot	200
	Auto weight	On
Transfer function	Smart stop enabled	On
	Output	Linear
	Hidden layer	Asymmetric Sigmoid

^a Resilient backpropagation.

TABLE IV
The Observed and Predicted Nanofibers Mean Diameter for Training Data

Sample No.	Observed nanofibers mean diameter (nm)	Predicted nanofibers mean diameter (nm)
1	247	307
2	268	269
3	342	342
4	238	287
5	302	344
6	126	104
7	97	69
8	2900	2882
9	229	348
10	390	356
11	310	268
12	579	479
13	315	392
14	695	675
15	330	396
16	509	506
17	289	277
18	280	310
19	195	164
20	261	342
21	361	367
22	239	238
23	232	285
24	216	221
25	945	943
26	405	434
27	193	154
28	268	269
29	428	396
30	490	383
31	450	402
32	493	491
33	497	524
34	347	387
35	604	505
36	361	263

TABLE V
The Observed and Predicted Nanofibers Mean Diameter for Test Data

Sample No.	Observed nanofibers mean diameter (nm)	Predicted nanofibers mean diameter (nm)
37	161	214
38	459	526
39	438	483

(ZEISS DSM 960A Oberkochen, Germany) of a small section of the nanofibrous mats after sputtering by gold. SemAfore software was then used to process the SEM images of the produced nanofibrous mats. Approximately 50 nanofibers were considered to measure mean size of the obtained nanofibers.

Figure 1 shows a typical SEM image of a sample that has been analysed by this software.

Data mining tool

The ANNs model between input and output data was created by applying INForm V4 (Intelligensys, UK). The INForm software uses neural networks to model the nonlinear and complex relations between inputs and outputs and the response surfaces generated for the model are shown as 3D graphs of two input parameters verses single output.^{22,23} The input parameters were PEO concentration, AcOH concentration, applied voltage, and electrospinning media temperature and the only output parameter was nanofibers mean diameter.

Data set

To train the network of the relations between input/output parameters, 36 samples (Table I) having random values for input parameters were prepared and the mean diameters were taken as the output for the network. Additionally, three samples, (i.e., 10% of the dataset, as recommended by the software) were prepared and measured. The dataset from these samples was taken as the test data (Table I) to

TABLE VI
The Observed and Predicted Nanofibers Mean Diameter for Validation Data

Sample No.	Observed nanofibers mean diameter (nm)	Predicted nanofibers mean diameter (nm)
40	213	216
41	269	222
42	198	289
43	361	372
44	786	235
45	1638	1246
46	285	333
47	2472	3051
48	456	363

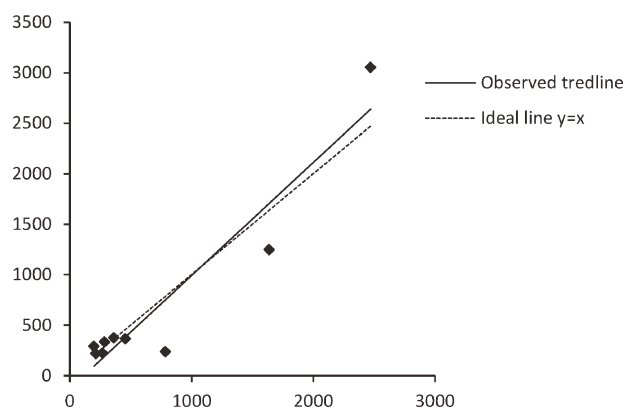


Figure 2 Plot of predicted versus observed nanofibers mean diameter for validation data set.

prevent overtraining as described previously.²³ A further nine samples (Table II) were prepared and the dataset was taken out of training/testing procedure. These “unseen” data were used to validate the predictive ability of model. Subsequent to training the network, using the parameters listed in Table III, the predicted value of mean diameter was determined from the derived model. A properly trained model needs to show acceptable correlation coefficient R -squared (R^2) for training, test, and unseen data [eq (1)].

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

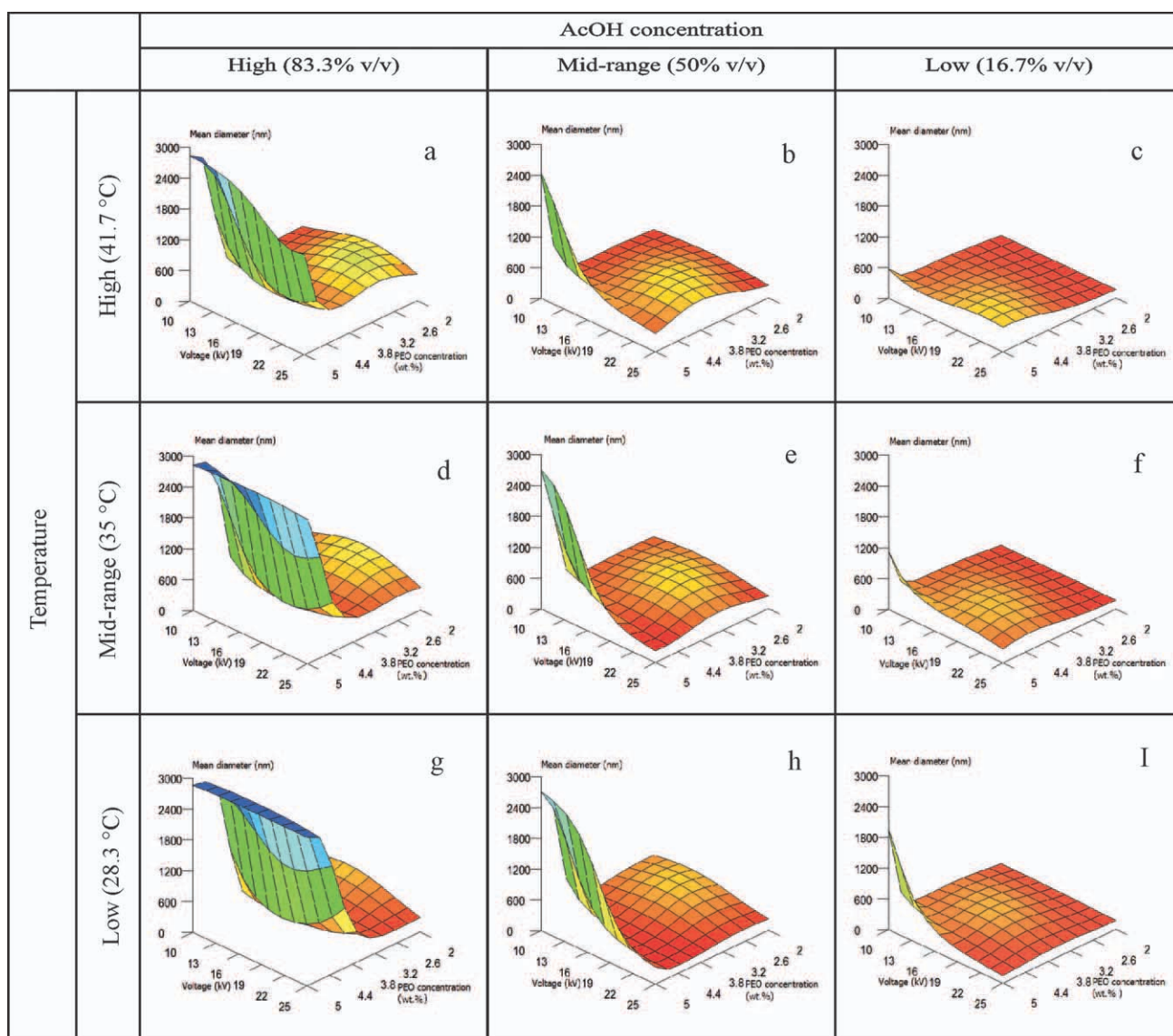


Figure 3 3D plots of nanofibers mean diameter predicted by the ANNs model fixed at low, mid-range, and high values of the temperature and AcOH concentration. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

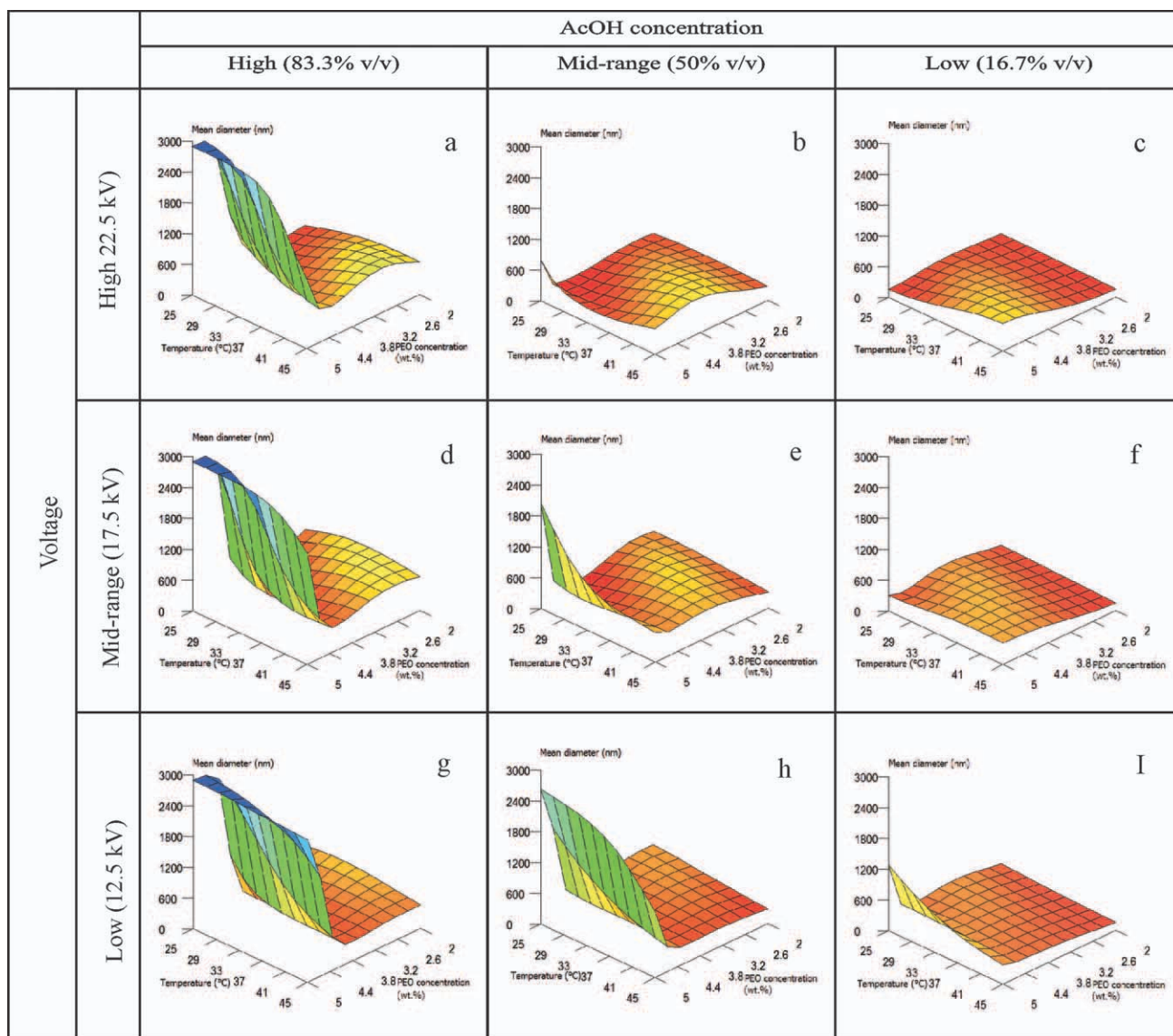


Figure 4 3D plots of nanofibers diameter predicted by the ANNs model fixed at low, mid-range, and high values of the AcOH concentration and voltage. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

where \hat{y} is the value predicted by the model, \bar{y} is the mean of dependent variable, and y_i is actual value of output.

RESULTS AND DISCUSSION

This work reports the electrospinning of PEO in aqueous AcOH solution. The effects of four electrospinning parameters, including PEO concentration (wt %), aqueous AcOH concentration (v/v), applied voltage (kV) and temperature (°C) on the mean diameter of the nanofibers was modeled using ANNs. The obtained SEM images showed that for all samples (training, test, and unseen datasets), continuous and uniform nanofibers have been produced except for sample No. 7 where 2 wt % PEO was dissolved

in pure water. In this case, a few beads have been formed together with nanofibers. The best predictive model from ANNs had R^2 value of 0.99, 0.83, and 0.84 for the training, test, and validation data, respectively. These values indicate a good-quality trained model. The observed and predicted nanofibers mean diameter for the training, test, and validation data are listed in Tables IV, V and VI, respectively.

Figure 2 illustrates the validation agreement between the predicted and observed nanofibers mean diameter for the nine individual sets of the validation experimental data.

This generated model was then used to study the effects of the different input variables on the mean diameter of nanofibers.

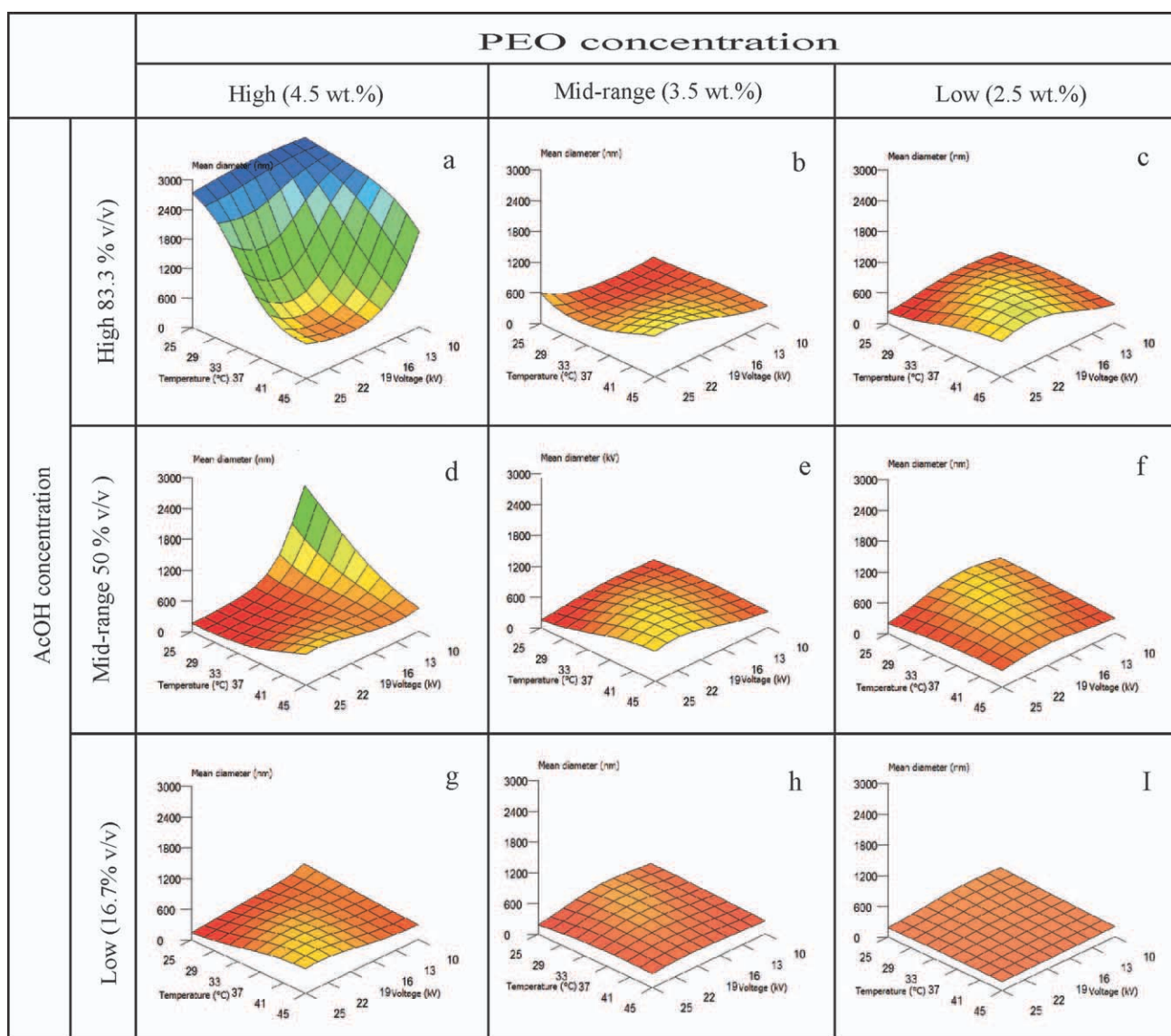


Figure 5 3D plots of nanofibers diameter predicted by the ANNs model fixed at low, mid-range, and high values of the PEO concentration and acetic acid concentration. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figures 3–8 represent the response surfaces obtained from the model to determine the relationships between the input and output parameters. In each 3D-graph, two parameters have been fixed in specific values (i.e., a low, a mid-range, and a high value) and the effects of other two parameters on the nanofibers mean diameter visualized for each set of fixed values.

Figure 3 shows the 3-D plots of PEO concentration and applied voltage against the mean diameter of nanofibers where the AcOH concentration and temperature are fixed at low, mid-range, and high values. From the plots, decreasing the concentration of PEO leads to a sharp decrease in the diameter at a critical point (~ 3.5 – 5 wt %, depending on the AcOH concentration, with lower values observed at

higher AcOH concentrations). Further decreases in the PEO concentration below the critical point do not make the diameter smaller.

The literature shows that nanofibers diameter commonly decrease by decreasing polymer concentration.^{15,24} By decreasing polymer concentration, the viscosity of polymer decreases which decreases the polymer chain entanglement and so causes lesser resistance of polymer solution to be stretched by charges on the jet and thus making smaller fibers diameter.²⁵ Another effect of decreasing viscosity is increasing jet instability which in turn increase jet path from needle to collector. This increased jet path means that there is more stretching on polymer jet which results in smaller fibers diameter.²⁴

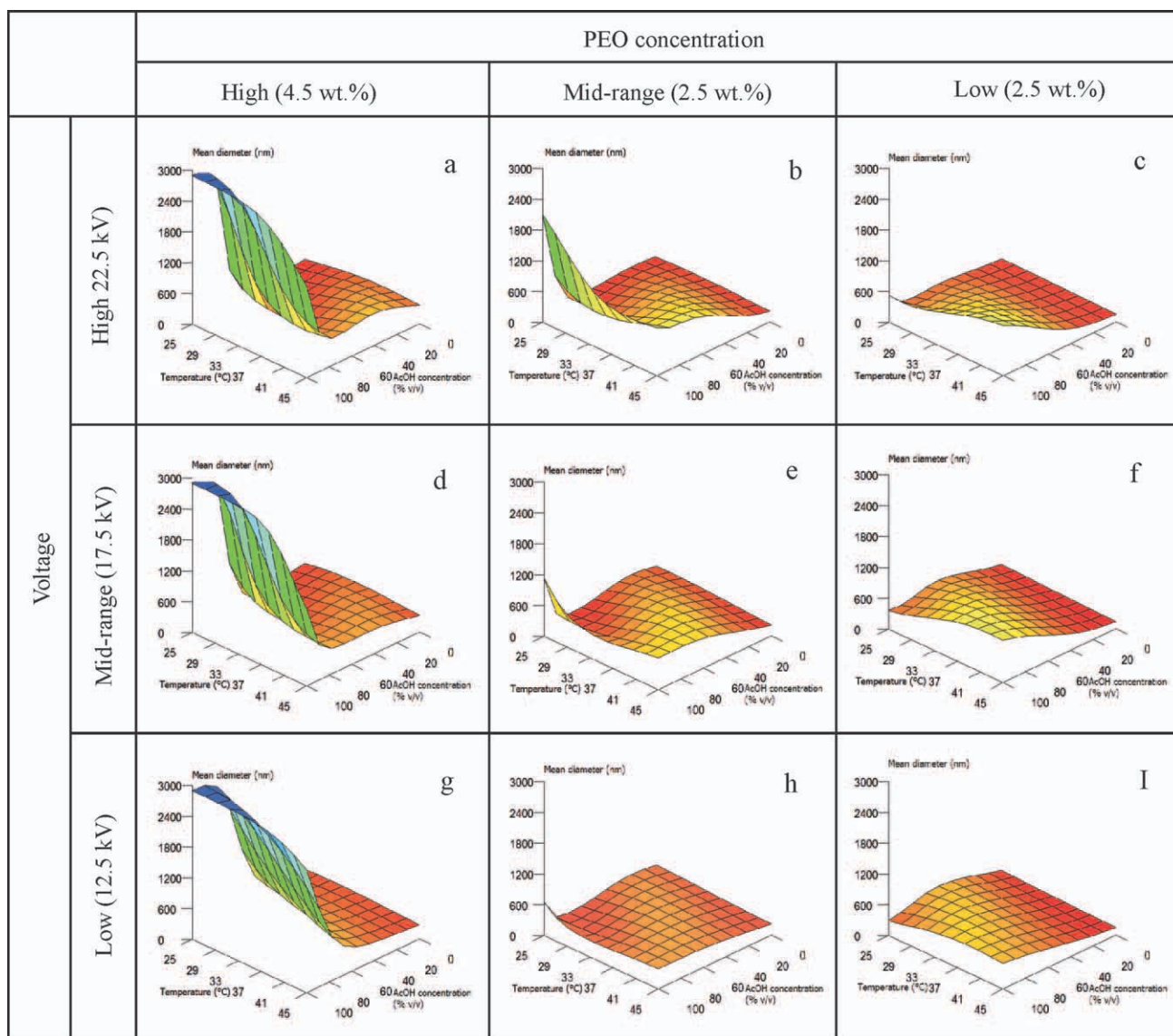


Figure 6 3D plots of nanofibers diameter predicted by the ANNs model fixed at low, mid-range, and high values of the PEO concentration and voltage. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

From Figure 3, it is also observed that in general, voltage has reverse relation with the nanofibers diameter when PEO is above the critical points. According to the literature, increasing applied voltage can decrease or increase fibers diameter by three mechanisms. Higher voltage accelerates stretching the polymer solution because of greater columbic forces in the jet as well as stronger electrostatic field. These then lead to production of smaller fibers diameter.²⁶ Higher voltage also facilitates formation of secondary jets which can reduce fibers diameter.²⁷ In contrast, higher voltage decreases nanofiber diameters by decreasing flight time of electrospinning jet. When flight time of electrospinning jet is short the jet has less time to be stretched thus deposits on collector with larger

diameters.²⁸ It is thus arguable that mechanisms making smaller fibers are involved when the PEO concentration is high. Other parts of the graphs, where only small fluctuations are observed in the mean diameter (i.e., PEO concentration less than the critical point), represent the situation in which each mechanism may temporarily overcome others and has a small effect on decrease/increase in the diameter.

The effects of the temperature and PEO concentration on the mean diameter of nanofibers is showed in 3-D plots of Figure 4, where AcOH concentration and applied voltage are fixed at low, medium, and high values. Details show that a critical point is observed for PEO concentration above which the diameter increases substantially, while at lower values,

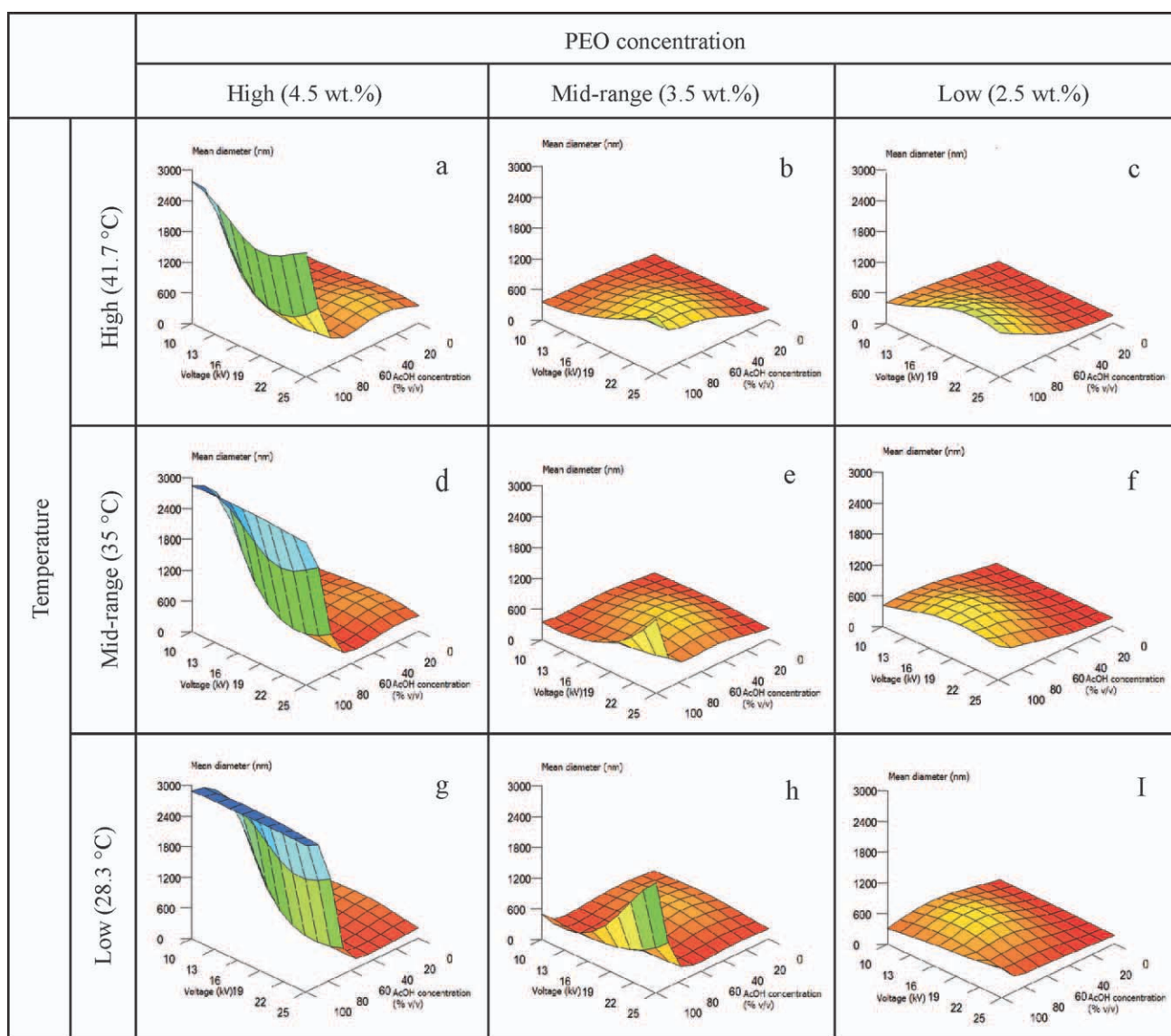


Figure 7 3D plots of nanofibers diameter predicted by the ANNs model fixed at low, mid-range, and high values of the PEO concentration and temperature. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

no important change in diameter may be observed, a finding mentioned above. Additionally, in general, the relation between the temperature and diameter is reverse when the PEO concentration is high. However, when the PEO concentration is at middle or low values, the temperature effects on increasing/decreasing the diameter is less significant. According to literature, reducing solution viscosity by increasing temperature, results in formation of nanofibers with smaller diameter. Reducing viscosity allows more stretching of polymer solution by clombic forces thus resulting fibers with smaller diameter.²⁴ This may be a reason for reducing nanofibers mean diameter by increasing the temperature when PEO concentration is high. Another effect of high temperature is increasing evaporation rate of

solvent from the polymer jet. When the temperature is high, the speed of the solvent volatilization is fast and the charged solution jets have less time to split and elongate during the flight of the jets because of the fast evaporation of the surface solvents. So, the diameter of the fibers becomes larger compare to low and medium temperatures.²⁸ This may be a reason for small increases in nanofibers mean diameter by increasing process temperature in some parts of the plots.

To evaluate the effects of the temperature and applied voltage on the nanofibers mean diameter, the PEO concentration and AcOH concentration have been fixed at low, medium, and high values. The plots of the temperature and applied voltage against nanofibers mean diameter have been given

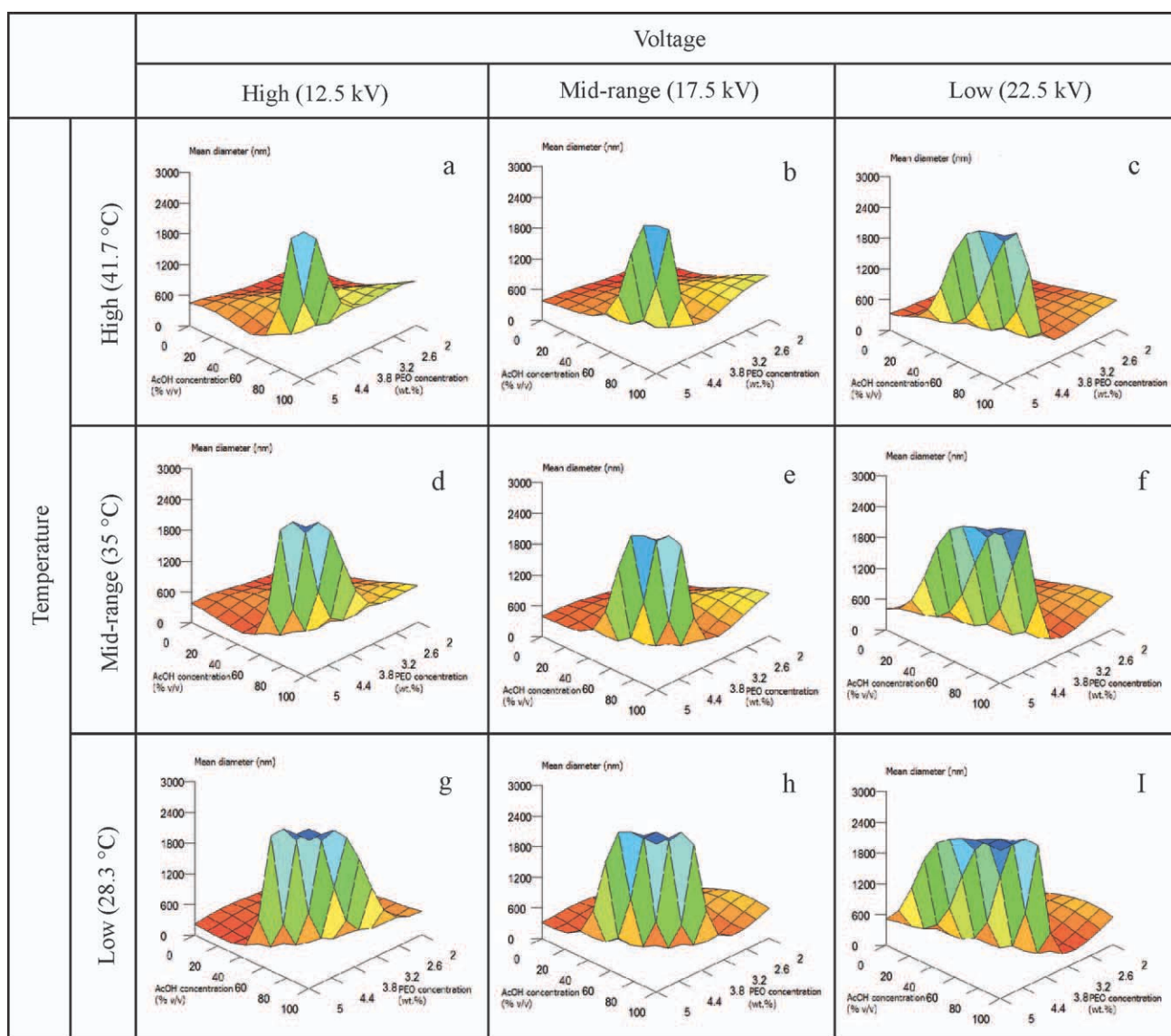


Figure 8 3D plots of nanofibers diameter predicted by the ANNs model fixed at low, mid-range, and high values of the applied voltage and temperature. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

in Figure 5. The main finding from the figure is that when the PEO concentration and AcOH concentration are both at low values [see Fig. 5(i)], the other parameters (i.e., process temperature and applied voltage) have no considerable effect on nanofibers mean diameter and nanofibers mean diameter is at lowest values. If only one of these two parameters is at low values, the effects of the applied voltage, and temperature on nanofibers mean diameter become more considerable. More significantly, when the PEO concentration and AcOH concentration are both at high values, the temperature, and applied voltage have strongest effect on nanofibers mean diameter. Herein, increasing either of the applied voltage or temperature results in reducing nanofibers mean diameter, with

smallest diameter obtained when both parameters are high.

Figure 6 summarizes the 3D plots of the AcOH concentration and temperature against nanofibers mean diameter, when the PEO concentration and applied voltage are fixed. From the data, it is observed that, generally, increasing the AcOH concentration results in larger diameter values. The data also shows that when the PEO concentration is at high values, increasing the concentration of AcOH from the values of approximately 60–90 (% v/v) depending on the voltage, leads to a sharp increase in the diameter. Reviewing the literature, some properties of solvent systems such as conductivity, viscosity, surface tension, and dielectric constant have been shown to influence the quality of

nanofibers.^{16,18,29} Fong et al. showed that addition of ethanol in PEO/water solution leads to production of larger fibers. Addition of ethanol makes the solution viscosity higher, the surface tension lower, and the net charge density lower. They attributed this increase in the fibers diameter to the decreasing in the net charge density of the solution.¹⁶ In another study, Son et al. studied electrospinning of PEO dissolved in four solvents. The result demonstrated that the solvent with higher dielectric constant produces fibers with smaller diameter.¹⁸ In our study, the higher dielectric constant of water than AcOH may be a reason for increasing nanofibers diameter by increasing the AcOH concentration. The dielectric constant for AcOH and water is 6.2 (at 20°C) and 78.5 (at 25°C), respectively.³⁰

From the Figure, the temperature shows reverse relation with the diameter where the AcOH concentration is high and the PEO concentration is high or medium [see Fig. 6(a,b,d,e,g,h)].

The effects of the applied voltage and AcOH concentration on the nanofibers diameter is given in Figure 7, where the polymer concentration and temperature are fixed. The data confirms the finding mentioned above: increasing AcOH concentration results in increasing nanofibers diameter. The data also shows that when the PEO concentration is at high values there is a sharp increase in the nanofibers diameter by increasing AcOH concentration above a critical point (i.e., 60–80, depending on the temperature). In addition, the voltage shows a direct, while relatively considerable effect on the diameter at medium values of the PEO concentration, where AcOH concentration is high [see Fig. 7(b,e,h)].

In Figure 8, the effects of the variation of AcOH concentration and polymer concentration on the diameter have been briefed. As stated above, the highest nanofibers diameters are obtained when the PEO concentration and AcOH concentration are both at high values. The results also show that to get the smallest nanofibers diameter, both the PEO and AcOH concentration need to be fixed at the lowest values.

CONCLUSION

Results of this study demonstrate the ability of ANNs to create a model for investigating the interactions and effects of the electrospinning parameters on the mean diameter of PEO nanofibers from AcOH aqueous solution. The 3D plots generated from this model well demonstrate the complexity and nonlinear relationships between the electrospinning parameters and nanofibers diameter. In general, increasing PEO concentration leads to a sharp increase in diameter where concentration of PEO is

above a critical point depending on the AcOH concentration. The effects of the applied voltage and temperature on the nanofibers diameter are mainly dependent on the PEO concentration. In high PEO concentration, the relations of the voltage and temperature with diameter is reverse, while in the lower PEO concentration there is small increase/decrease in the diameter by increasing these parameters. The AcOH concentration, in general shows a direct relation with the nanofibers diameter. The plots of modeling showed when the PEO concentration and AcOH concentration are at the lowest values, the lowest diameters of nanofibers are obtained regardless the applied voltage and temperature. In contrast, highest nanofibers diameters are obtained when the PEO concentration and AcOH concentration are at their high values.

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