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Using an artificial neural network for the evaluation of the parameters controlling PVA/chitosan electrospun nanofibers diameter

Abstract: The purpose of this study was to investigate the validity of an artificial neural network (ANN) method in the prediction of nanofiber diameter to assess the parameters involved in controlling fiber form and thickness. A mixture of polymers including poly(vinyl alcohol) (PVA) and chitosan (CS) at different ratios was chosen as the nanofiber base material. The various samples of nanofibers were fabricated as training and testing datasets for ANN modeling. Different networks of ANN were designed to achieve the purposes of this study. The best network had three hidden layers with 8, 16 and 5 nodes in each layer, respectively. The mean squared error and correlation coefficient between the observed and the predicted diameter of the fibers in the selected model were equal to 0.09008 and 0.93866, respectively, proving the efficacy of the ANN technique in the prediction process. Finally, three-dimensional graphs of the electrospinning parameters involved and nanofiber diameter were plotted to scrutinize the implications.

Keywords: ANN; electrospinning; modeling; nanofibers; PVA/Chitosan.

DOI 10.1515/epoly-2014-0198

Received October 30, 2014; accepted January 19, 2015; previously published online February 20, 2015

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1 Introduction

Chitosan (CS), a biopolymer derived from chitin, has recently attracted increasing interest in both research and development. Chitin is a basic element of D-glucosamine and can be extracted from shellfish such as shrimp, lobster and crabs. Generally, chitin has excellent biocompatible and biodegradable properties, non-toxicity and ease of solubility in organic acids (1). According to its distinguished properties, chitin can be applied in practically related technologies such as textile materials, starting materials for plant growth enhancer and nanofibers (2). Poly(vinyl alcohol) (PVA) is typically a non-toxic and water-soluble synthetic polymer. It is also biocompatible and degradable, which yields comparably high fiber forming.

Electrospinning is a fiber-forming process by which either polymer solutions or melts are charged by high voltage to form fine jets. It was first reported by Formhals (3) in 1934. Fiber formation by electrospinning of polymer solutions has been extensively studied in terms of voltage, tip-to-collector distance, feeding rate and polymer solution properties (4, 5).

Electrospinning is recognized as an efficient technique for the fabrication of submicron-sized polymer nanofibers ranging from 5 to 500 nm, which are 102–104 times smaller than those prepared by the traditional methods of solution or melt spinning (6). Various macromolecules have been successfully electrospun into ultrafine fibers as thin as several nanometers. In terms of the flexibility of the process, electrospinning is able to fabricate continuous nanofibers from a huge range of materials.

Bringing materials to the nanometer scale not only improves their properties, but also affords them new advanced characteristics beyond bulk materials. Nanofibers, especially polymeric nanofibers, are promising materials with diverse applications (e.g., drug delivery, tissue engineering and wound healing) (7, 8), owing to their large surface area-to-volume ratio, flexibility in surface functionalities, and superior mechanical performance. Among the various approaches for fabricating

nanofibers, electrospinning, also known as electrostatic spinning, is perhaps the most versatile process. Electrospinning is an attractive method in both academic and industrial applications because (i) it is able to fabricate continuous fibers with a few nanometers in diameters; (ii) it works with a wide range of materials, e.g., synthetic and natural polymers, metals, as well as ceramics and composites; (iii) it is able to prepare nanofibers at low cost and high yield (9–11).

However, electrospinning is an ambiguous process, and detecting the relationship between the electrospinning parameters and the size and morphology of the obtained nanofibers is very difficult and time consuming (12, 13). Since nanofiber diameter directly affects the performance (for example, the improvement of the nanofibers diameters similar to extra cellular matrix fibrils diameter), it is better to use a modeling approach to estimate fiber morphology before the electrospinning setup.

Different methods are used for predicting the diameter of the produced nanofibers via electrospinning such as response surface methodology (RSM) (14) and artificial neural network (ANN) modeling (15). However, ANN may be more applicable owing to its greater efficiency. ANNs are an attractive choice in designing smart systems because they are based on the natural neural network of the brain. The basic structure of an ANN consists of different parts, e.g., an input layer, various hidden layers and an output layer (15) (as shown in Figure 1). After multiplying the input (p) weights (w) plus the bias (b), the product as input is applied on the function (f) and the final output (α) is obtained. Indeed, the ANN modeling process starts with the design of the network, followed by the training and the testing process. After learning about ANNs using a training dataset via statistically related algorithms, to

evaluate the efficiency of the network in predicting patterns, the network is tested via a test dataset (15). If the designed network is organized well and the modeling process works properly, the network may be used for future purposes.

To date, ANNs have been developed in several studies, particularly in the prediction of electrospun nanofiber diameter (15–17). For example, RSM and ANN were used for analyzing the morphology of nanofibers synthesized by electrospinning. The results demonstrated that both models are efficient in predicting the diameter of polyurethane nanofibers (16). The prediction of polymethyl methacrylate nanofiber diameter via RSM and ANN indicated that the latter slightly outperformed the RSM in the prediction of results (17). In other studies, the ANN model presented an efficient fitting with experimental results for predicting the diameter of electrospun polyacrylonitrile nanofibers (18) and the production rate of electrospun nanofibers was predicted by both the RSM and the ANN, which were in agreement with the experimental data (19). As the production of electrospun CS nanofibers is difficult owing to its high surface tension and high viscosity in solution, blending CS with other polymers such as PVA can be done. In this paper, the diameter of electrospun PVA/CS blend nanofibers was predicted by mixing two polymers, CS and PVA. As the production of Four parameters involved in the fabrication of CS/PVA nanofibers, namely, CS/PVA concentration ratio, temperature, applied voltage and distance between the nozzle tip and the collector, were considered as variable parameters and the validity of the ANN models for predicting nanofiber diameter was investigated.

2 Experimental

2.1 Materials

Polyvinyl alcohol (PVA) (M_w 72,000) and chitosan (CS) (high molecular weight) were purchased from Merck KGaA, Darmstadt, Germany, and Sigma-Aldrich, ST. Louis, USA, respectively. Neural Network toolbox of MATLAB (Natick, MA, USA) was used for modeling process. Glacial acetic acid (Merck) was used as the solvent. To produce the nanofibers, polymer solutions including PVA and CS in different concentrations (PVA/CS) were prepared.

2.2 Methods

The electrospinning method is composed of four main parts (as shown in Figure 2): a syringe pump, a syringe

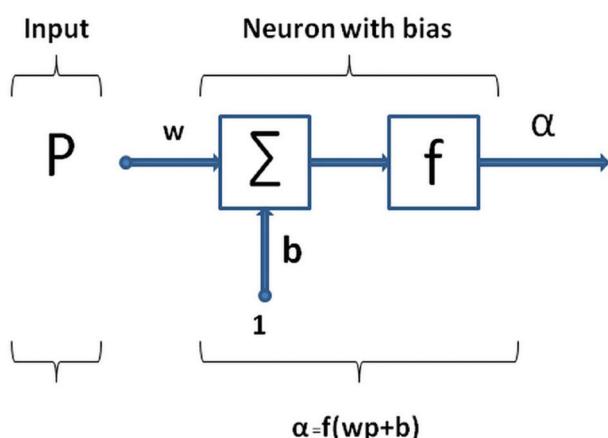


Figure 1: A simple schematic of the performance and architecture of an artificial neural network.

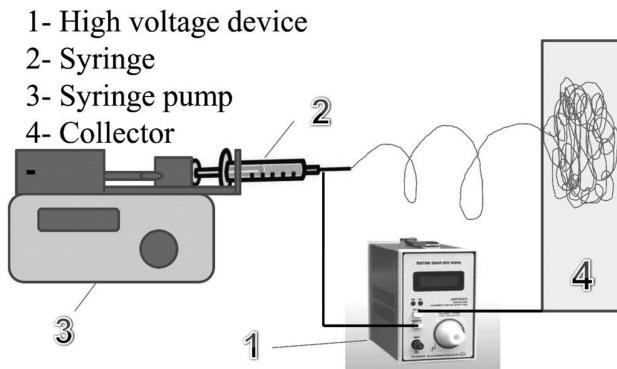


Figure 2: Schematic of an electrospinning device.

containing a polymer solution, a high-voltage device that can make an electrical field of up to 30 kV, and a collector. The Electroris (Fanavaran Nano Meghyas Ltd., Co., Tehran, Iran) was used as the electrospinning device.

With this approach, several conditions could be defined by changing the effective parameters for producing new nanofibers. In this research, four variables (Table 1) were considered: PVA/CS ratio (X_1), applied voltage (X_2), temperature (X_3) and distance between the nozzle tip and the collector (X_4).

The morphology of the nanofibers was assessed via scanning electron microscopy (SEM), and the size of the nanofibers was measured by randomly choosing 50 nanofibers using the ImageJ software (National Institute of Health, USA).

For the modeling of the process, the predictive networks with various hidden layers and nodes were designed. The reliability of the ANN diagram in the prediction of nanofiber diameters was evaluated using the MATLAB software.

2.3 Designed models for diameter prediction

Studies have shown that using a simple dataset for prediction via ANN is not effective when the dataset is small (15, 20). Therefore, to increase modeling validation, a k -fold cross-validation (k -cv) method was used. In this

Table 1: List of input variables for electrospinning.

Independent input variable	Description
X_1	PVA/CS (v/v)
X_2	Applied voltage (kV)
X_3	Temperature (°C)
X_4	Distance (cm)

study, the dataset was divided into fivefolds, the training sets for learning the designed model (k -1 folds) and an evaluation approach using a network in ANN modeling via a testing set (the remaining 1 fold). Thus, the k -cv approach depends on two factors: the training set and the partitioning of data arranged in fivefolds. The five subsets (Table 2) were included in the testing and training sets, and the function of the ANN was replicated k times to fit the function using the training dataset. Totally, the k -1 subsets were put together in each step for the training set and the one remaining subset was used as the test set. Mean squared error (MSE) was measured for all subsets and was used as an index to evaluate network reliability.

2.4 Network training using a k -fold cross-validation procedure

To design the prediction networks using the training and testing subsets, the various samples of polymeric nanofibers were fabricated via electrospinning (as shown in Table 3).

Before using the ANN technique, data normalization was performed.

The data normalization process is given by Equation 1:

$$y_{\text{norm}} = (y_{\max} - y_{\min})(x - x_{\min}) / (x_{\max} - x_{\min}) + (y_{\min}) \quad [1]$$

where $y_{\min} = -1$ and $y_{\max} = 1$

where parameter x is the data that should be normalized, and x_{\max} and x_{\min} are the maximum and minimum values of x , respectively.

Moreover, the training parameter set for the ANN model is shown in Table 4.

2.5 ANN Model training

After designing the seven ANN models with various structures, including four input units, one output unit and various hidden layers with different nodes in each layer, to find a better functioning network, these models were trained via the training dataset and were tested using the testing set.

Table 2: Training-testing partition pairs using the fivefold cross-validation method.

Partition pairs	Training set	Testing set
1	Partition {1, 2, 3, 4}	Partition {5}
2	Partition {1, 2, 3, 5}	Partition {4}
3	Partition {1, 2, 4, 5}	Partition {3}
4	Partition {1, 3, 4, 5}	Partition {2}
5	Partition {2, 3, 4, 5}	Partition {1}

Table 3: List of nanofiber samples produced via electrospinning in various sets of controlling parameters.

Sample	PVA/CS Concentration (v/v)	Applied voltage (kV)	Temperature (°C)	Distance (cm)	Observed diameter (nm)	Predicted diameter (nm)
1	25/75	11	28	10	87±14	80
2	25/75	11	28	15	110±30	146
3	25/75	11	32	10	80±12.2	128
4	30/70	14	30	10	170±73	166
5	60/40	8	30	10	238±48.5	209
6	25/75	11	32	15	90±24.2	113
7	25/75	13	32	10	136±22.9	139
8	60/40	14	28	8	206±56	202
9	25/75	13	32	15	107±46.9	152
10	90/10	13	32	10	225±71	291
11	60/40	11	32	10	327±80	273
12	25/75	13	28	15	191±52.9	266
13	60/40	14	30	15	195±42	206
14	60/40	14	28	15	234±54	207
15	60/40	11	28	15	253±56	232
16	60/40	10	32	15	233±39.7	167
17	75/25	11	32	15	188±45	169
18	85/15	11	26	15	371±52	567
19	90/10	13	32	15	233±36	338
20	25/75	13	32	10	210±35	169

The MSE and correlation coefficient (R) of the test dataset taken from the ANN models are shown in Table 5.

The mean square prediction error (MSPE) is given by Equation 2:

$$\text{MSPE}_n = \frac{100}{\text{Nte} \sigma_{d_n}^2} \sum_{i=1}^{\text{Nte}} (d_n(i) - d_{pn}(i))^2, \quad n=1, \dots, 5 \quad [2]$$

where d_n and d_{pn} are the observed and the predicted size of the nanofibers in n network, respectively, and Nte factor is the number of samples using network testing. Also, $\sigma_{d_n}^2$ is the variance of d_n .

(The number of hidden layers was 3; the number of nodes in the hidden layers was 10, 15 and 5, respectively.)

To determine the correlation between the observed and the predicted diameter of the fibers, a linear regression was calculated (as shown in Figure 3).

Based on the results, the difference between the observed and the predicted mean diameters of the nanofibers was about 17 nm, indicating the reliability of the ANN modeling in the prediction process (as shown in Table 6).

The Pearson correlation coefficient (r) between the observed (d_n) and the predicted (d_{pn}) nanofiber diameter is given by Equation 3:

$$r = \frac{n(\sum d_n d_{pn}) - (\sum d_n)(\sum d_{pn})}{\sqrt{\left[n(\sum d_n^2) - (\sum d_n)^2 \right] \left[n(\sum d_{pn}^2) - (\sum d_{pn})^2 \right]}} \quad [3]$$

In this equation, n is the number of data.

3 Results and discussion

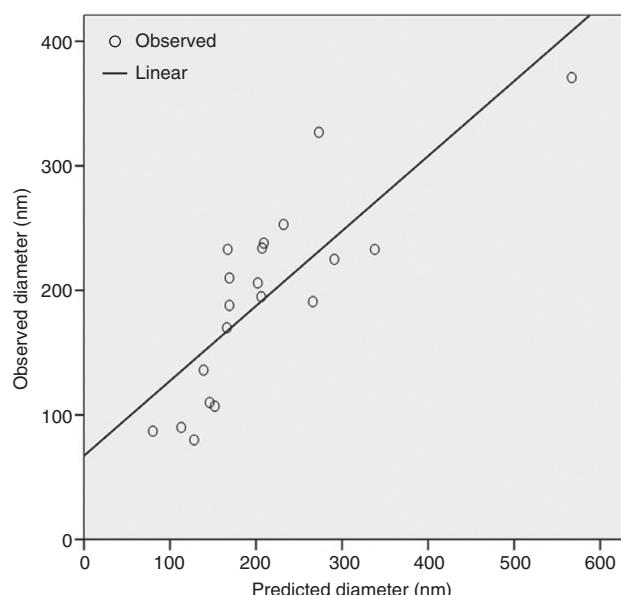
The MSPE and correlation coefficient (R) of the test dataset obtained from the ANN modeling are shown in Table 5.

Table 4: ANN training parameters designed for the prediction process.

Algorithm	Trainlm (Levenberg-Marquardt back propagation)
Transfer function in hidden layers	Log-sigmoid function for the hidden layers and purelin for the output layer
Number of epochs between showing the progress	100
Learning rate	0.09
Momentum constant	0.9
Maximum number of epochs to train	1000
Performance goal	1e-5

Table 5: Results of MSPE and linear regression in the test dataset.

Dataset	Test	
	MSPE	R
1	0.2029	0.8359
2	0.1824	0.885
3	0.013	0.9932
4	0.0183	0.9926
5	0.0338	0.9866
Mean	0.09008	0.93866

**Figure 3:** Regression plot of the observed and predicted diameter of the nanofibers.

The Pearson correlation coefficient between the observed and the predicted nanofiber diameter was equal to 0.817, which is significant at the 0.01% level, indicating the relevant efficacy of the designed ANN for predicting function (as shown in Table 7).

Table 6: Descriptive statistics of the observed and predicted diameter of the nanofibers.

	Number of samples	Nanofiber diameter (nm)			SD
		Minimum	Maximum	Mean	
Observed diameter	20	80	371	194.2	77.56
Predicted diameter	20	80	567	211	105.31
Valid N (list wise)	20				

Table 7: Pearson correlation between the observed and the predicted nanofiber diameter.

	Observed diameter	Predicted diameter
Observed diameter		
Pearson correlation	1	0.817 ^a
Significance (two-tailed)		0.000
N	20	20
Predicted diameter		
Pearson correlation	0.817 ^a	1
Significance (two-tailed)	0.000	
N	20	20

^aCorrelation is significant at the 0.01 level (two-tailed).

3.1 Three-dimensional plots of the predicted patterns of nanofiber diameters

To understand the effects of the different parameters determined (PVA/CS ratio, applied voltage, zone temperature and nozzle-to-collector distance) on electrospun nanofiber diameter (Figures 4–9), various three-dimensional (3D) graphs were plotted at defined levels.

According to the results, the minimum and maximum size of the nanofibers in the low T -low D condition were equal to 80 and 209 nm, respectively. But in the high T -high D level, the minimum and the maximum size of the fibers increased to 113 and 338 nm, respectively (Figure 4). Therefore, a direct relation between the nanofiber thicknesses of T and D can be observed in the graph. It means that the lower or higher range of the T and D variables leads to thinner or thicker fibers, respectively. Also, the results confirm a direct relation between PVA/CS ratio and nanofiber diameter.

The results of the C - T plots indicated that the D and V variables have direct effects on the fiber size (Figure 5). The minimum fiber mean thickness belongs to the low D -low V region, which was about 172 nm. The smallest diameter was shown in the high V -high D region, which was equal to 152 nm. Also, the largest diameter of the fibers was shown in the low V -high D area, which was equal to 567 nm. The largest fiber mean thickness was obtained in the high D -low V area, which was about 339 nm (Figure 5).

Similar to previous data (as shown in Figure 4), PVA/CS ratio indicated a direct relationship with fiber thickness. The results demonstrated that the electrospun zone temperature has an inverse relation with the fiber diameter. In all the conditions, a direct correlation between fiber diameter and PVA/CS ratio was shown.

In the low V -high T level, the minimum mean diameter of the fibers was about 170 nm (Figure 6). In contrast,

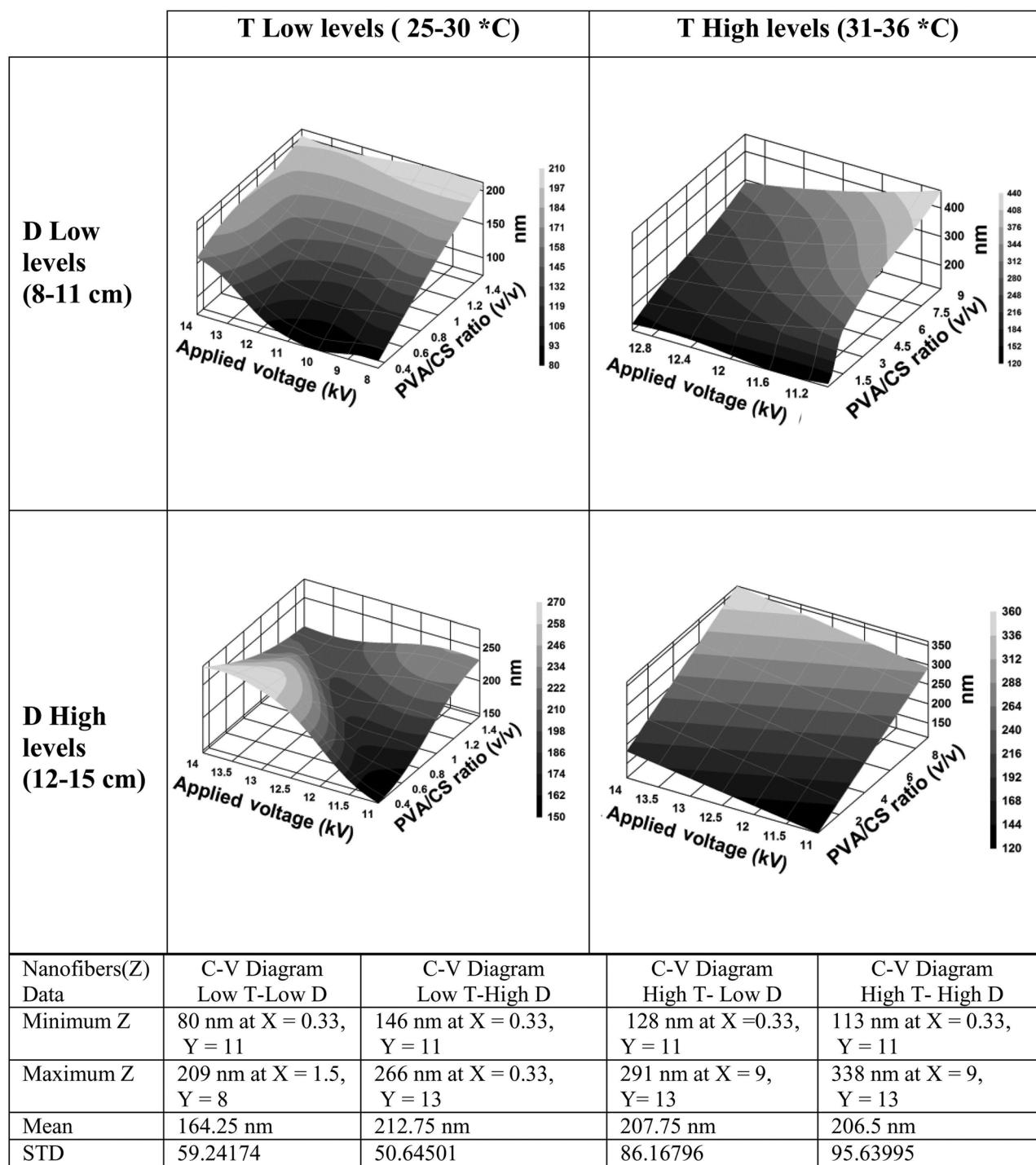


Figure 4: Data and 3D plots of the nanofiber diameters, predicted via ANN, fixed in the mentioned levels (T-D).

the maximum nanofiber mean thickness was obtained in the range of the low V -low T level, which was about 250 nm.

Based on the previous plots (Figure 5), the polymer concentration (PVA/CS ratio) is directly correlated with

nanofiber diameter. The results indicated that, in the high V -low T level, by increasing the nozzle-to-collector distance during the electrospinning process the nanofiber diameter sharply increased, contrary to the results shown in other plots. In the other levels (Figure 6), the distance

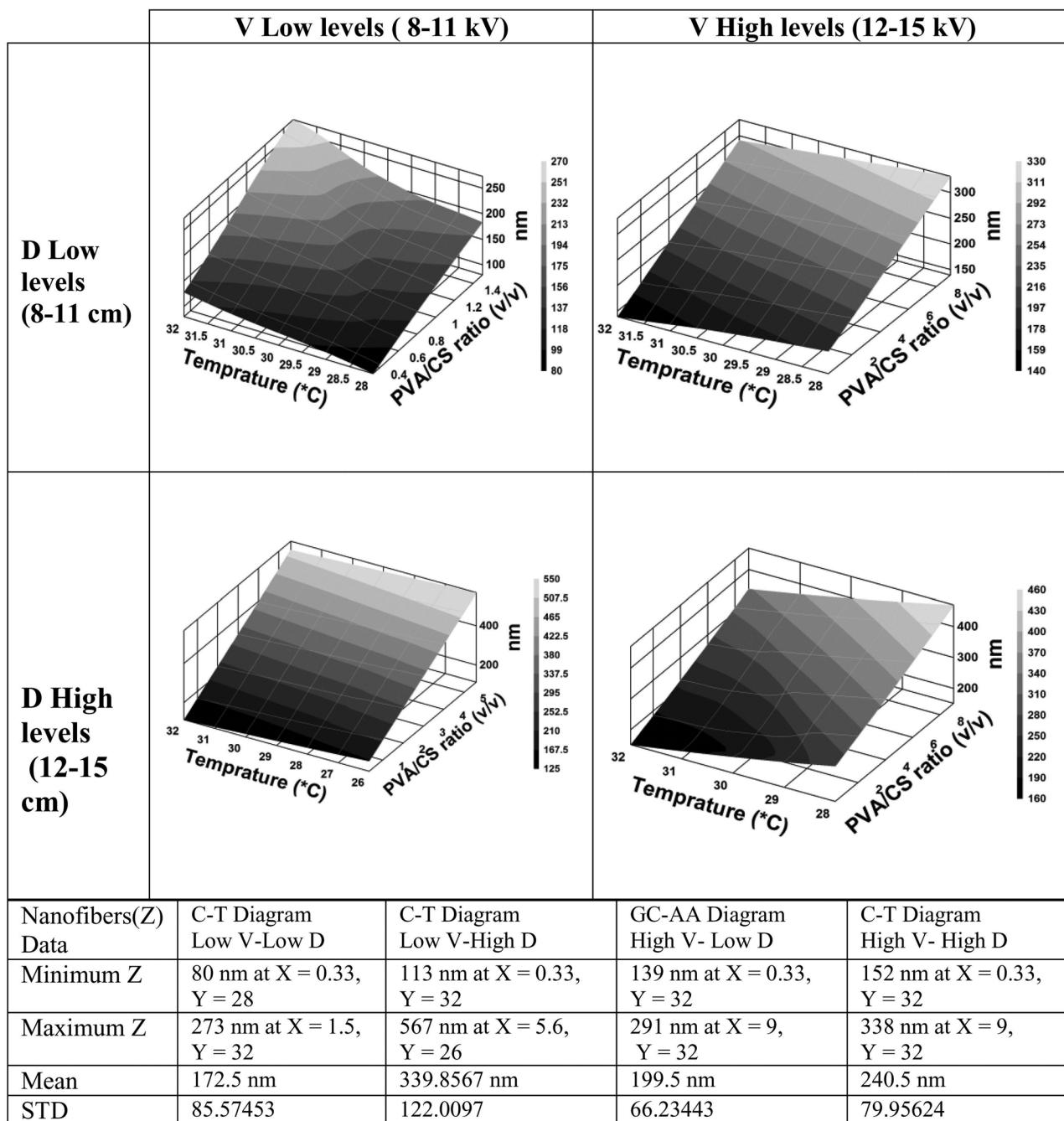
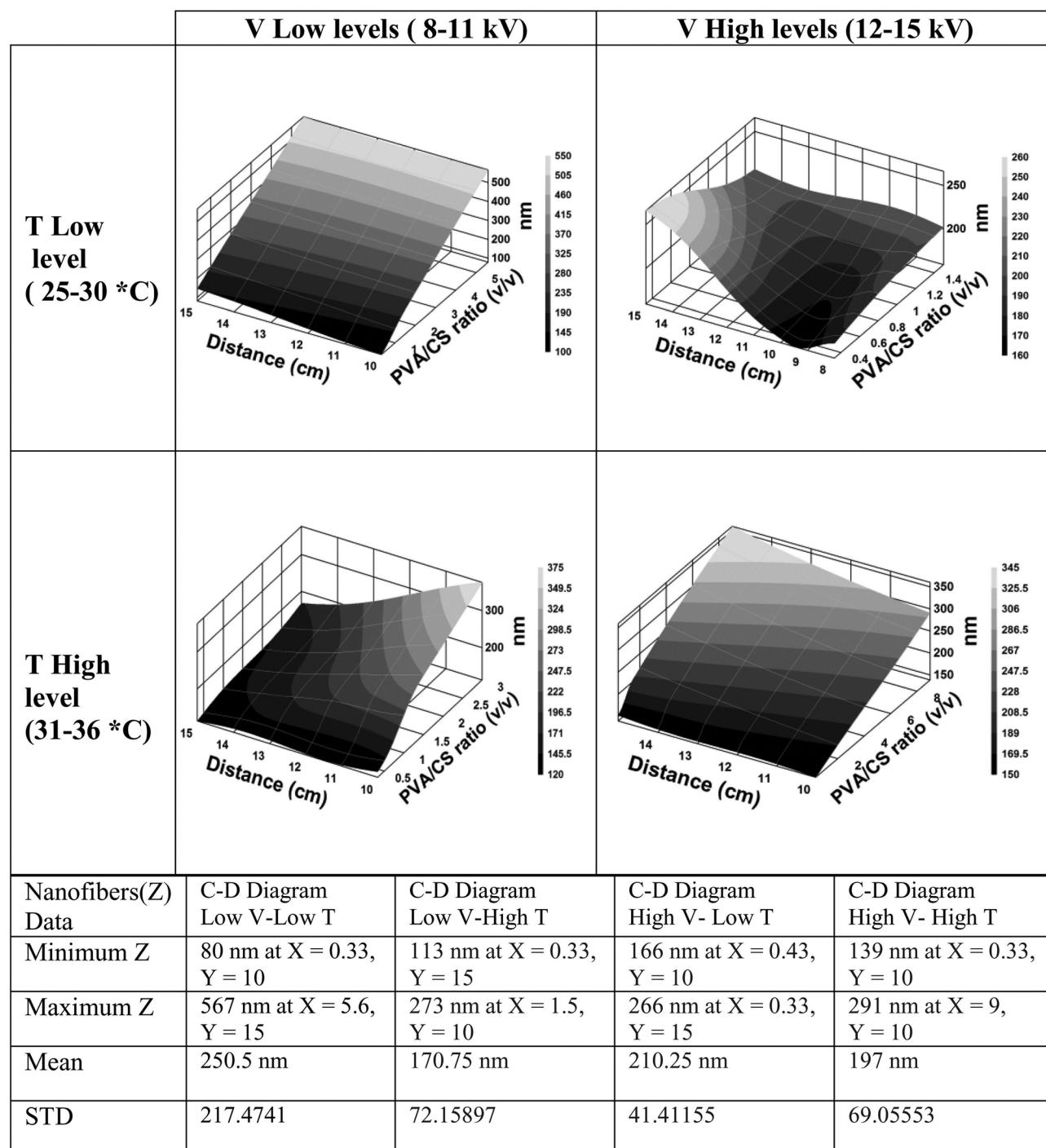


Figure 5: Data and 3D plots of the nanofiber diameters, predicted via ANN, fixed in the mentioned levels (V-D).

had insignificant effects on the size of the electrospun nanofibers.

The data demonstrated that the polymer concentration (C) and distance (D) have direct effects on the fiber diameter. In both levels (high C -high D and high C -low D), the mean thickness was about 230 and 243 nm, respectively (Figure 7). The minimum fiber mean diameter, which was

about 128 nm, belonged to the low C -low D area, proving once again a direct relation between polymer concentration and nanofiber diameter. Also, in the largest diameter in the high C -low D level, the maximum size of the fibers was about 243 nm. Based on the plots, when distance was fixed at the high level, fiber thickness decreased with increasing temperature, in agreement with previous



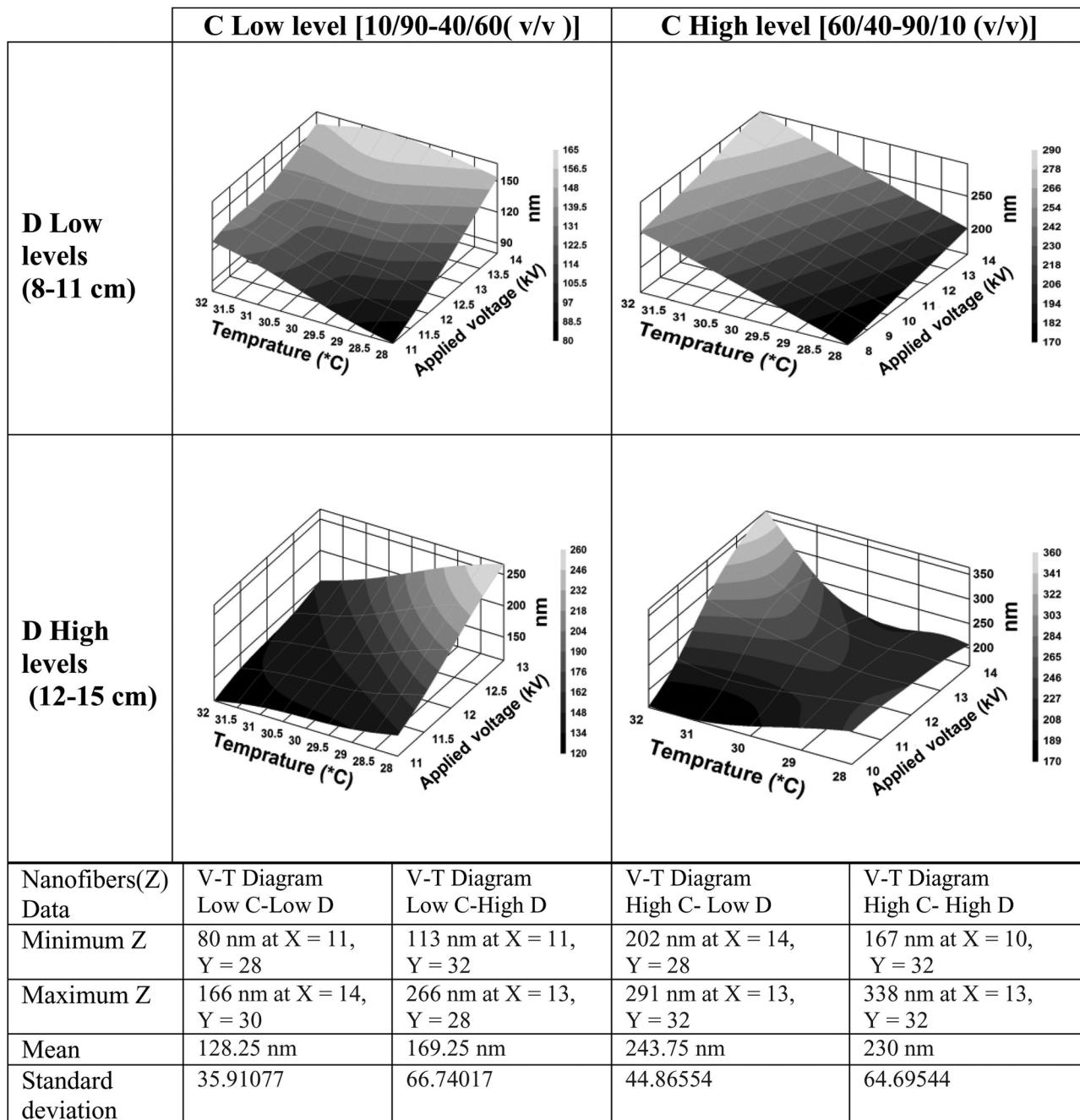
C=PVA/CS ratio (v/v), V=Applied Voltage (kV), T=Temprature (*C), D=Distance (cm), X=C, Y=D

Figure 6: Data and 3D plots of the nanofiber diameters, predicted via ANN, fixed in the mentioned levels (V-T).

data in Figures 4 and 5. But in the low-level distance, the diameter increased with increasing temperature, in contrast to the former plots. Also, with increasing voltage, the increase in fiber thickness indicates a direct relation between V and fiber diameter. But in the high C -high D

condition, fiber thickness remained approximately constant at lower temperature rate, in contrast with the higher rate of temperature in the high C -low D level.

The minimum fiber mean diameter, about 133 nm, was obtained in the low C -high T level. In contrast, the

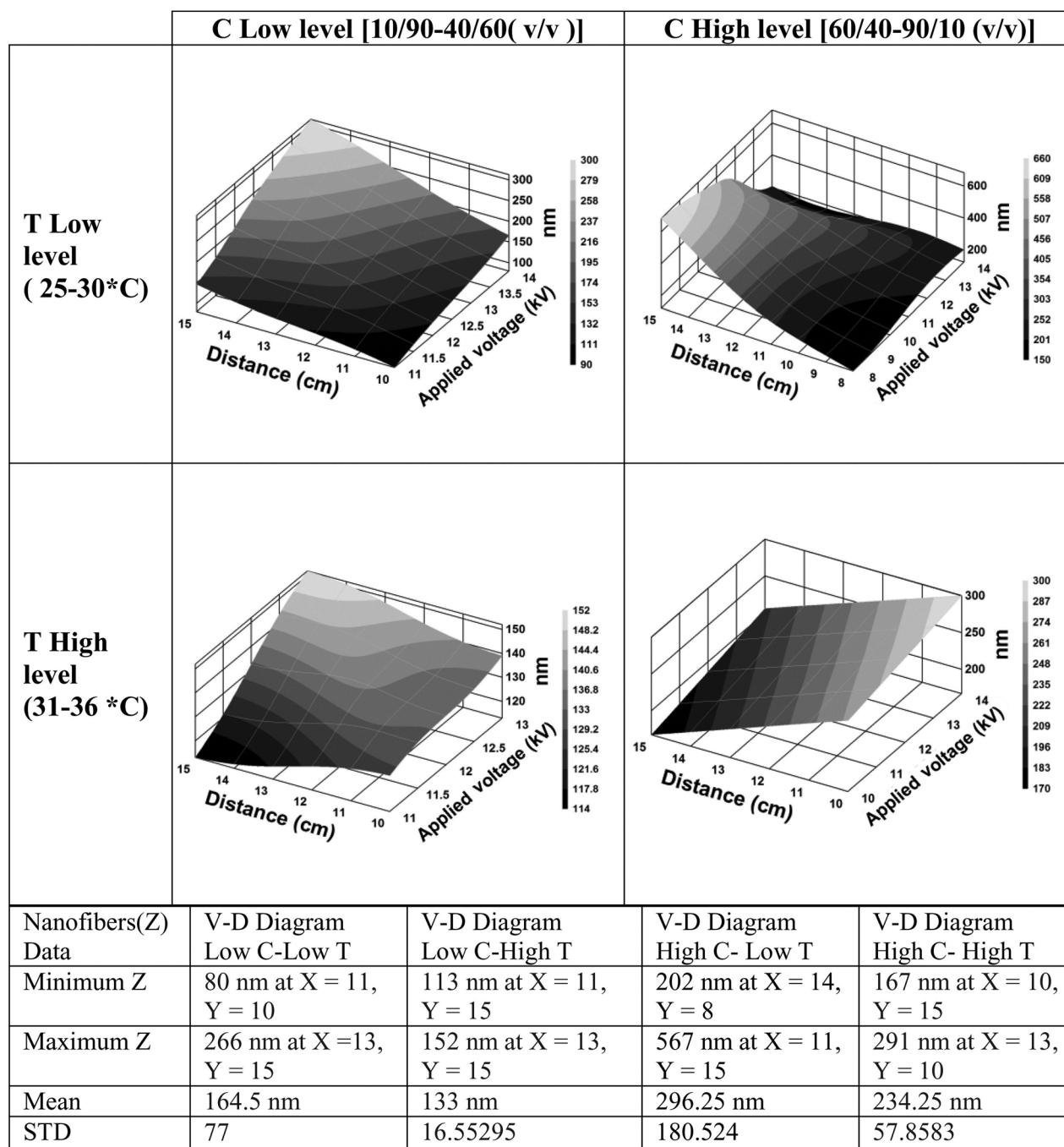


C=PVA/CS ratio (v/v), V=Applied Voltage (kV), T= Temperature (*C), D=Distance (cm), X=V, Y=T

Figure 7: Data and 3D plots of the nanofiber diameters, predicted via ANN, fixed in the mentioned levels (C-D).

maximum fiber mean thickness was about 296 nm, which was obtained in the high C-low T level (Figure 8). These findings clearly indicate a directly proportional relationship between polymer concentration and electrospun nanofiber size, and an inverse correlation between zone temperatures and fiber diameters, which can be used as a basic guideline for producing desired nanofibers. These results are compatible with previous 3D plot outcomes.

Another notable finding indicates an indirect relationship between distance and nanofiber diameter in the high C-high T level. In the low-temperature area (25–30°C), fiber diameter increased with increasing applied voltage. At high level of temperature, the diameter remained approximately constant despite the increased applied voltage. Also, by enhancing the distance in the low-temperature area, the diameter of the fibers increased. This result



C=PVA/CS ratio (v/v), V=Applied Voltage (kV), T= Temprature (*C), D=Distance (cm),X=V, Y=D

Figure 8: Data and 3D plots of the nanofiber diameters, predicted via ANN, fixed in the mentioned levels (C-T).

emphasizes the higher rate of distance in the low *C*-low *T* area. But when the temperature was increased, fiber diameter decreased with increasing distance (in the high *C*-high *T* zone).

In this step, voltage and PVA/CS ratio were fixed at two levels (high and low), and distance and temperature were gradually altered. The smallest fiber mean diameter

(about 116 nm) was obtained in the low *C*-low *V* levels, whereas the largest fiber diameter (about 262 nm) was obtained in the high *C*-low *V* area. It is also noticeable that fiber diameter in both the high *C*-low *V* and the high *C*-high *V* level was very close to 262 and 259 nm, respectively. According to the graph (as shown in Figure 9), the fiber diameter increased in all the plots with increasing

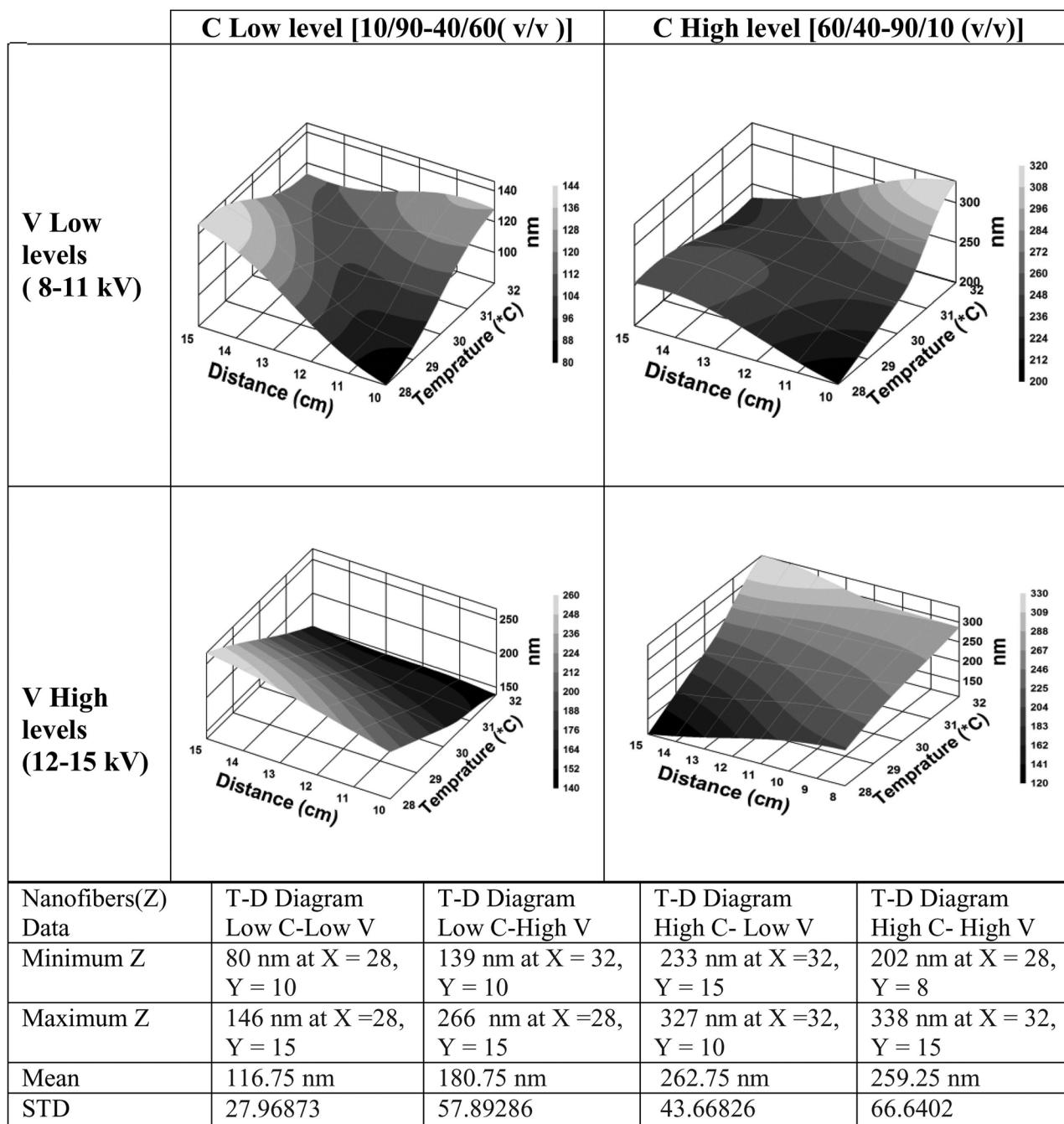


Figure 9: Data and 3D plots of the nanofiber diameters, predicted via ANN, fixed in the mentioned levels (C-V).

temperature, except in the low C -high V condition, where, unlike in the other plots, the diameter of the fibers decreased with increasing temperature. Also, with the increase in distance, fiber diameter increased in all the graphs, except in the high C -high V level.

Finally, the present study clearly shows the complexity of the electrospinning process in producing nanofibers

with a defined form and diameter. The results indicate the advantages of modeling in obtaining the desired nanofiber diameter by regulating the involved parameters based on the prediction outcomes. It seems that experimental design using one at the time methods may not be appropriate in designing an efficient manner for producing electrospun nanofibers.

4 Conclusion

An artificial neural network was used to predict the mean diameter of poly(vinyl alcohol)/chitosan (PVA/CS) nanofibers produced by electrospinning. Twenty different models were designed to fabricate electrospun PVA/CS nanofibers. The PVA/CS concentration ratio, applied voltage, temperature and distance between the nozzle and the collector were considered as variable parameters. The mean squared error and correlation coefficient (R) between the observed and the predicted diameter of the fibers were equal to 0.09008 and 0.93866, respectively, which shows the efficacy of the ANN technique in the prediction process. Moreover, the descriptive statistical results show that the difference between the observed and the predicted nanofiber mean diameter in the selected network was only about 17 nm.

The results indicate a direct relationship between the concentration of chitosan in mixed polymers (PVA/CS) and the electrospun nanofiber size. It shows that the size of nanofibers increases by increasing the ratio of chitosan in mixed polymers. In contrast, there is an inverse relationship between the electrospun nanofiber diameter and the ratio of PVA in mixed PVA/CS polymers. It is suggested that designing and applying the prediction process on various kinds of nanofibers and nanomaterials should be performed via ANN modeling. It might be useful for producing nanomaterials with defined properties by predicting the effects of the involved parameters on their shape, diameters and forms. It could be useful in view of the economic and scientific purposes.

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