

## RESEARCH ARTICLE

# Evaluation of the effective forspinning parameters controlling polyvinyl alcohol nanofibers diameter using artificial neural network

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## Abstract

In this research, the polyvinyl alcohol (PVA) nanofibers through forspinning process were successfully produced and the effective parameters for predicting nanofibers diameter using artificial neural network (ANN) were investigated. The various parameters of forspinning process including rotational speed, orifice, distance to the collector, and polymer concentration were designed to produce PVA nanofibers. Scanning electron microscopy (SEM) showed that the produced fibers diameter was in the range of 0.56–1.9  $\mu\text{m}$ . The neural network with four input factors, three hidden layers with 5, 10, 1 nodes in each layers, respectively, and one output layer had the best performance in the testing sets. Moreover, the mean squared error (MSE) and linear regression (R) between observed and predicted nanofibers diameter were about 0.1077 and 0.9387, respectively, demonstrating a suitable performance for the prediction of nanofibers diameter using the selected neural network model.

## KEYWORDS

ANN, forspinning, nanofibers, nanomaterials, PVA

## 1 | INTRODUCTION

In recent decades, the demand for nanofibers due to their unique properties such as substantial mechanical flexibility and strength and large surface area to volume ratio is growing in many

medical and engineering industries, including drug delivery, filtration, tissue engineering, food packaging, protective clothing, nanoelectronics, nanobiosensors, nanocatalysis, etc.<sup>[1–9]</sup>

PVA nanofibers, a biodegradable and biocompatible polymer, have the wide application in medical field such as

scaffolding in tissue engineering,<sup>[10]</sup> drug delivery carrier,<sup>[11]</sup> and as anticorrosion coating in industry.<sup>[12]</sup> Therefore, finding a way to mass production of PVA nanofibers is economically affordable.

To date, there are currently used techniques to fabricate ultra-fine fibers such as, phase separation, melt blowing, self-assembly, and electrospinning.<sup>[13]</sup> Among these, electrospinning is the most commonly used technique for creating organic and inorganic ultra-fine fibers in a wide range from micron to nano scales.<sup>[14,15]</sup>

However, the health hazards of high-voltage power source, using toxic solvent, low fiber yield, the inherent low solubility of conjugated polymers, and sensitivity to dielectric constant limit electrospinning application.<sup>[16]</sup> Over the past few years, a novel process, forcespinning, apart from electrospinning restrictions for the fabrication of submicron fibers, has been developed.<sup>[17,18]</sup> The centrifugal power is used to draw jets into fibers in new method rather than electrostatic force in electrospinning. As a result, not only conductive polymers, but also nonconductive substances can be spun in solutions or in melts.<sup>[16]</sup> Furthermore, increased production of nanofibers from 0.1 g/h in laboratory-scale electrospinning to over 1 g/min per nozzle is another advantage of centrifugal spinning.<sup>[18]</sup> During forcespinning, the different parameters significantly influence fiber morphology and diameter such as polymer concentration, spinneret to collector distance, evaporation rate (for solutions), temperature (for melts), and rotational speed. Therefore, changes in mentioned parameters could affect the size and morphology of forcespun fibers.

Fibers morphology is one of the key factors affecting surface-to-volume ratio, porosity, functionality, and performance.<sup>[19]</sup> As the conventional methods are able to fabricate fibers with diameters ranging from 10  $\mu\text{m}$  to 10 nm,<sup>[16]</sup> control and regulation of the process peculiarities is very important to obtain desired fibers.<sup>[19]</sup>

In recent years, the impact of the involved parameters on properties of forcespun fibers was investigated in several systematic studies, and the rotational speed and the solution concentration were considered as the main factors among the aforementioned parameters. The polymer concentration has the significant effects on the fibers morphology, whereas the adjustment in below and above a critical solution concentration leads to the formation of beads to continuous forcespun fibers, respectively.<sup>[16]</sup> Also, a lower polymer concentration leads to a thinner fiber diameter and narrower fibers size distribution, while the rotational speed has a subtle effect on the fiber diameter.<sup>[16]</sup>

The improvement of efficiency of the process for obtaining desired productions via multidisciplinary optimization methods is an important step in modeling a complex process. Using these techniques is essential not only for improving the performance of the systems, but also for increasing

the yield of the processes without high cost. The classical optimization procedure is the one-factor-at-a-time (OFAT) technique, which is a time-consuming approach and does not represent the complete effects of the applied parameters on the process.<sup>[20]</sup> The alternative approaches to overcome disadvantages of the classical OFAT method are Response Surface Methodology (RSM) and Artificial Neural Network (ANN).

RSM is a useful and practical technique to improve and adjust processes via modeling and analyzing the problems using a collection of statistical and mathematical procedures. In RSM, a number of variables influence the response of interest and the aim is the optimization of this response in the matching process. It has an important application for design of new products and development of the existing product formulation. Instead of an OFAT method, RSM defines the effect of the independent variables (alone or in combination) on process modeling.<sup>[20]</sup> In addition to the RSM technique, the other knowledge-based approaches such as ANNs and fuzzy logic (FL) are used for modeling objectives in a wide range of biological and industrial processes.<sup>[21–23]</sup>

ANNs are retrieved from modeling and simulation (M&S) technology with the wide capability for better understanding some of the fundamental aspects of life (i.e. a variety of problems in pattern recognition, optimization, prediction, associative memory, and control). The computer-based algorithms, ANNs, design the simulation of the way, wherein the human brain processes information. The system is composed of several processing elements (neurons), connected with coefficients (weights), and acts in concert to solve a problem and find the logical connection among inputs and outputs. During the training process, ANNs collect the information and learn through practice via detecting the patterns and relations in the data. The collection of neurons in a network with unique properties such as weights, biases, network architecture, and transfer function, induces power for the computation and optimization plan via ANNs. The adjustable weights of the inputs organize the activated neurons and generate the signals to produce the output of the neuron through transfer function. After the optimization using training and testing process, through the inter-unit connections to minimize the network error in the prediction, network reaches the particular levels of accuracy which can predict the output via new input data.

Recently, ANN as a promising modeling technique has been studied for the prediction of electrospun nanofibers diameter in which its viability has been investigated based on the obtained results.<sup>[4,19,24]</sup> To our knowledge, this is the first study on the fabrication of forcespun PVA nanofibers and the prediction of the produced nanofibers diameter via ANN modeling. The various set of selected variables (rotational speed, orifice, distance to the collector, and polymer concentration) were used to prepare PVA nanofibers.

Furthermore, the validity of ANN models in the prediction of PVA nanofibers diameter was evaluated via MSE, correlation coefficient, and regression.

## 2 | EXPERIMENTAL

### 2.1 | Materials

Materials include polyvinyl alcohol (72,000, Merck, Germany), deionized water as solvent and forcespinning process was performed for producing nanofibers using the forcespinning device (Charkhris, Iran)

### 2.2 | Fiber production

Four main variables including polymer concentration, rotor speed, orifice diameter, and nozzle to collector distance were used as forcespinning parameters (as seen in Table 1). The fibers images were taken by SEM to evaluate fibers diameter and morphology. Then, mean diameter of 20 fibers was computed by Image J software (Sun Microsystems, USA) (Figure 1).

### 2.3 | Artificial neural network

Prediction of fiber diameters was carried out by ANN and validity of the designed models was assessed via MSE, regression, and correlation coefficient. Also, *k*-fold cross-validation method was used to attain more appropriate results.

### 2.4 | Network models designed for prediction

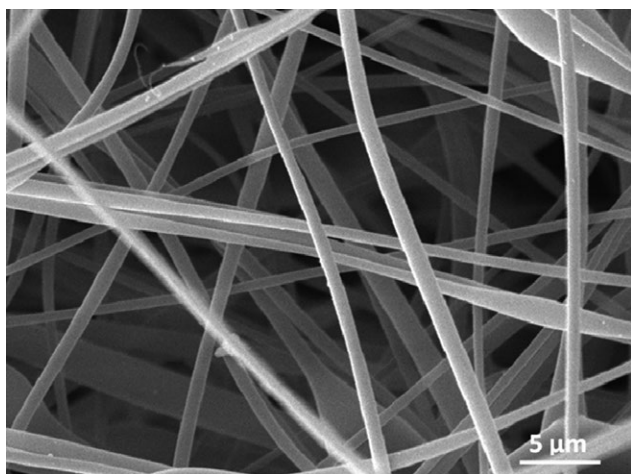
The study was carried out to predict fibers diameter produced via forcespinning method. A literature review shows that several parameters such as rotor speed, orifice radius, polymer concentration, surface tension, evaporation rate, temperature, nozzle to collector distance affect fibers diameter.<sup>[18]</sup> However, for investigating the parameters and their effects on the fibers diameter, ANN seems to be appropriate method to detect relationships of the parameters and to find their impacts on the fibers diameter.

Different settings of four principal parameters on the fibers diameter which include polymer concentration (X1) (w/v), rotor speed (X2) (RPM), orifice diameter (X3) (mm),

**TABLE 1** Values of process variables used in the forcespinning experiments

Sample	Concentration (%)	Rotor speed (RPM)	Orifice diameter (G)	Collector to nozzle distance (cm)	Nanofibers diameter (nm)
1	10	2,400	25	5	859
2	8	2,400	25	5	789
3	8	2,400	27	5	1,904
4	10	2,400	27	5	1472
5	8	2,400	29	5	1,559
6	6	2,400	27	5	1,331
7	6	2,400	25	5	1,647
8	6	3,600	29	5	1,379
9	6	3,600	29	5	1,119
10	10	4,600	25	5	1,830
11	8	2,400	25	15	1,452
12	8	2,400	27	15	1,226
13	6	2,400	25	15	561
14	6	2,400	29	15	1,160
15	6	2,400	29	15	1,075
16	6	4,600	29	15	711
17	6	4,600	27	15	1,165
18	8	4,600	29	15	1,480
19	8	4,600	27	15	1,791
20	8	4,600	27	15	1,639
21	10	4,600	27	15	970
22	8	4,600	23	15	805
23	10	4,600	23	15	1,103

and nozzle to the collector distance ( $X_4$ ) (cm) are chosen (Table 2). Data were classified in two groups: training data are applied to regulate network values, and testing data are used to assess network efficiency. However, when small or large database is intersected by simple training-test, performance or reliability of ANN decreases. Instead of simple-test,  $k$ -fold cross-validation method was used to classify data. The technique of  $k$ -fold cross-validation is a statistically convincing conclusions and more reliable method than simple training-test.<sup>[25]</sup> In this approach, database divided arbitrary into the  $k$  equal subsets (as shown in Table 3) including testing and training set, and function of estimation repeated  $k$  times to fit function using training process. At each phase, one  $k$  subset is allocated for test set and other  $k-1$  subsets



**FIGURE 1** Image of forcespun nanofibers produced via forcespinning method

**TABLE 2** List of input variable for forcespinning

Independent input variable	Description
$X_1$	Polymer Concentration (w/v)
$X_2$	Rotor speed (rpm)
$X_3$	Orifice diameter (mm)
$X_4$	Distance (cm)

**TABLE 3** Training-testing partition pairs using fourfold cross-validation method

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

are put together to form a training set. Then, MSE across all  $k$  trials is calculated, and finally is referred to assess the network validity.

## 2.5 | Network training using $k$ -fold cross-validation procedure

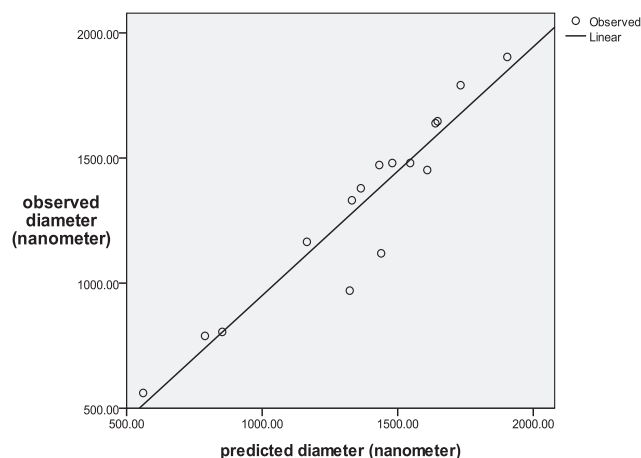
Several samples of fibers (Table 1) were synthesized by forcespinning and applied as ANN model training-testing datasets.

**TABLE 4** ANN training parameters

Algorithm = trainlm
(Levenberg-Marquardt back propagation)
Transfer function in hidden layers = log-sigmoid and purelin
Number of epochs between showing the progress = 50
Learning rate = 0.01
Momentum constant = 0.9
Maximum number of epochs to train = 100;
Performance goal = 1e-5;

**TABLE 5** Mean square error and Regression of test data in the selected ANN network

NETWORK	MSE	$R$
1	0.1069	0.9344
2	0.1235	0.9272
3	0.1763	0.9102
4	0.0240	0.9831
Mean	0.1077	0.9387



**FIGURE 2** Regression plot between observed diameters and predicted diameters

Before Using ANN Process, Data Normalization was done  
The data normalization is given by the equation (1):

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

where  $y_{\text{min}}$  and  $y_{\text{max}}$  are equal to  $-1$  and  $1$ , respectively. The parameter of  $x$  is the data that should be normalized.  $x_{\text{max}}$  and  $x_{\text{min}}$  are the maximum and minimum amounts of  $x$ .

**TABLE 6** Pearson correlation between observed and predicted nanofibers diameter

		Observed diameter (nm)	Predicted diameter (nm)
Observed diameter (nm)	Pearson Correlation	1	0.948*
	Sig. (2-tailed)		0.000
	N	16	16
Predicted diameter (nm)	Pearson Correlation	0.948*	1
	Sig. (2-tailed)	0.000	
	N	16	16

\*Correlation is significant at the 0.01 level (2-tailed).

## 2.6 | ANN models training

First, several neural networks with different features that include four input units, one output and various hidden layers with different nodes were designed for prediction of PVA fibers diameter process. After examining the plans by training and testing dataset, the best network with the lowest error and the highest accuracy was selected as ANN model.

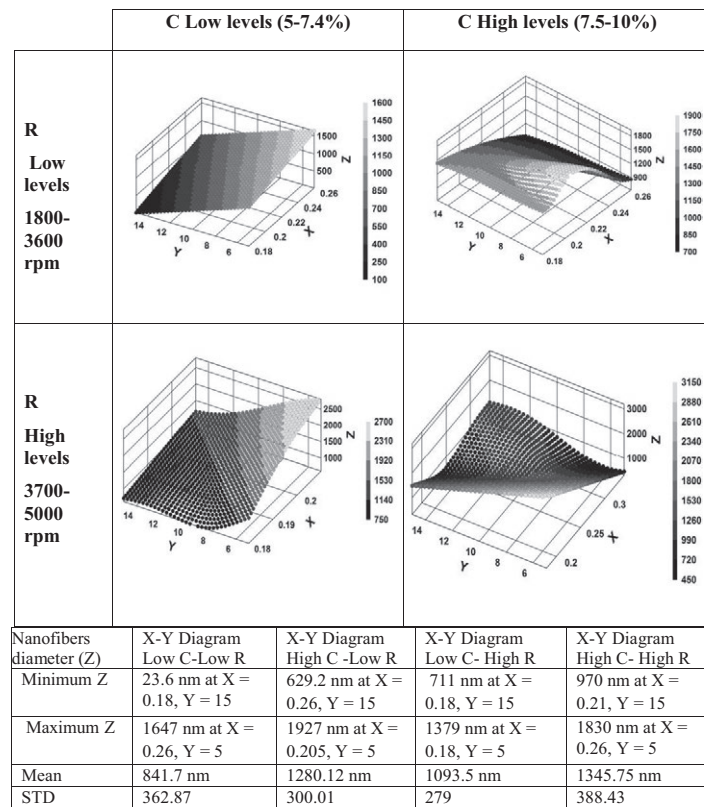
## 3 | RESULTS AND DISCUSSION

The correlation coefficient (R) and mean square error of testing dataset attained from ANN models is demonstrated in Table 4. (Hidden layers = 3) (Number of nodes in the hidden layers = 5, 10 and 1, respectively).

Mean square prediction error (MSPE) is given by equation (2):

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

Where  $d_n$  and  $d_m$  are observed and predicted size of fibers in  $n$  network, respectively. The parameter of  $Nte$  is the numbers



X= Nozzle orifice (mm), Y= Nozzle to collector distance (cm), Z= nanofibers diameter (nm), R=Rotor speed (rpm), C= polymer concentration (%)

**FIGURE 3** The data and 3D plots of nanofibers diameter predicted by ANN fixed in mentioned levels (C-R diagram)

of samples used for network testing, and  $\sigma_{dn}^2$  is the variance of  $d_n$ . The MSPE and correlation coefficient of the test dataset obtained from the ANN are seen in Table 5.

The correlation between observed and predicted fibers diameter was shown via a linear regression (as shown in Figure 2).

The Pearson correlation coefficient between the observed and predicted diameter of the fibers was attained equal to 0.948 that is significant at the 0.01% level (as shown in Table 6).

The Pearson correlation coefficient ( $r$ ) between the observed ( $dn$ ) and predicted ( $dm$ ) fibers diameter is given by equation (3):

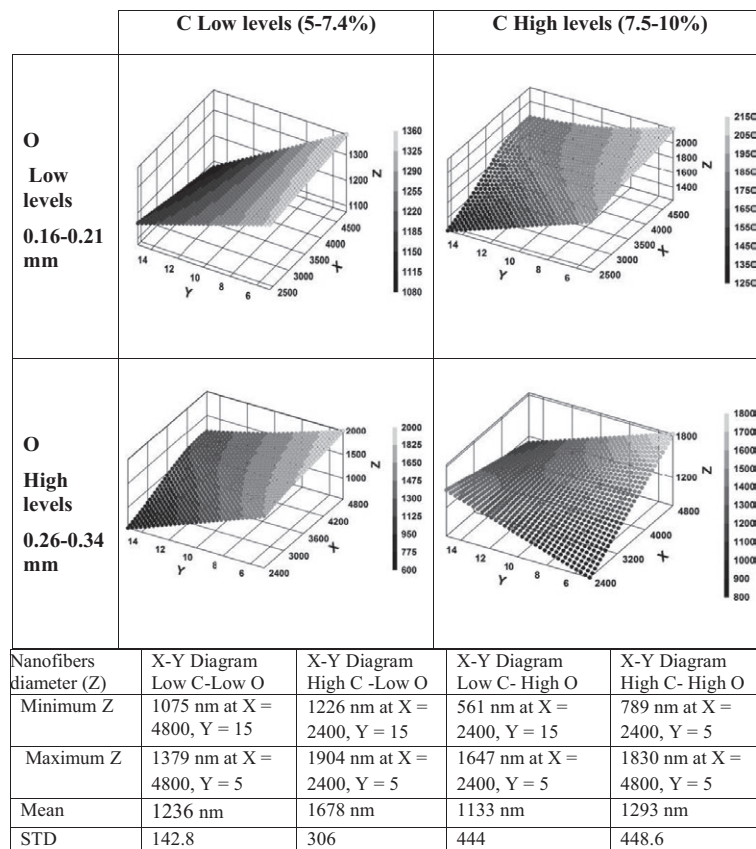
$$r = \frac{n(\sum d_n d_{pn}) - (\sum d_n)(\sum d_{pn})}{\sqrt{[n(\sum d_n^2) - (\sum d_n)^2][n(\sum d_{pn}^2) - (\sum d_{pn})^2]}} \quad (3)$$

In this equation,  $n$  is the number of data.

The 3D plots (figures of 3 to 8) at the defined levels demonstrate the impact of the aforementioned parameters on the diameter of PVA fibers. The results show that the minimum diameter of the fibers (841 nm) is attained in the low PVA concentration and the low rotor speed levels

(Low C-Low R) in Figure 3. Also, the maximum diameter of the fibers (1,678 and 1,538 nm) was obtained in the high range of the polymer concentration and a low level of the nozzle-collector distance (High C-Low D) and orifice diameter (High C-Low O) in figures of 4 and 5, respectively. The results are consistent with our previous studies on the role of polymer solution concentration on the electrospun fibers diameter.<sup>[4,19]</sup> These findings show that polymer concentration plays an important role in the produced fibers diameter via either electrospinning or forspinning.

In all graphs, increasing nozzle-collector distance leads to decrease in the fibers diameter (Figure 3). The graph indicated inverse relationship between the nozzle to the collector distance and the fiber diameter, when the rotor speed and polymer concentration were fixed at a low and the high values, respectively. Besides, it is noticeable that by increasing the orifice diameter, the fibers size enhances in the range of low level of PVA concentration (Figure 3). On the other hand, by increasing the orifice diameter, the fibers diameter decreased when the concentration of the solution is stabilized at the high value. Therefore, it seems that the effect of the orifice diameter on the size of the fibers is insignificant and the result indicated the concentration of the solution is more effective on the fibers diameter. The results of other studies



X= Rotor speed (rpm), Y= Nozzle to collector distance (cm), (nm), Z=Nanofibers diameter, C= polymer concentration (%), O= Nozzle orifice (mm)

**FIGURE 4** The data and 3D plots of nanofibers diameter predicted by ANN fixed in mentioned levels (C-O diagram)

indicated an inverse relationship between the orifice diameter and the fibers size, whereas the concentration of a solution was on a steady state.<sup>[18,26]</sup>

In Figure 4, it can be inferred that the rotor speed has a direct relationship to the fibers diameter. This result is in contrast with some previous studies,<sup>[18,26]</sup> which could be due to the interaction among the forcespinning parameters and the fibers size, although it needs a closer look.

Another important parameter is nozzle to the collector distance which has an inverse relationship to the diameter of the fibers in all groups, except in the high concentration and the high orifice diameter area (High C-High O) in Figure 4. In the High C-High O level, increasing nozzle to the collector distance leads to increasing the fibers diameter. It seems that influence of the polymer concentration and orifice diameter on the fiber diameter is more impressive than the nozzle-collector distance.

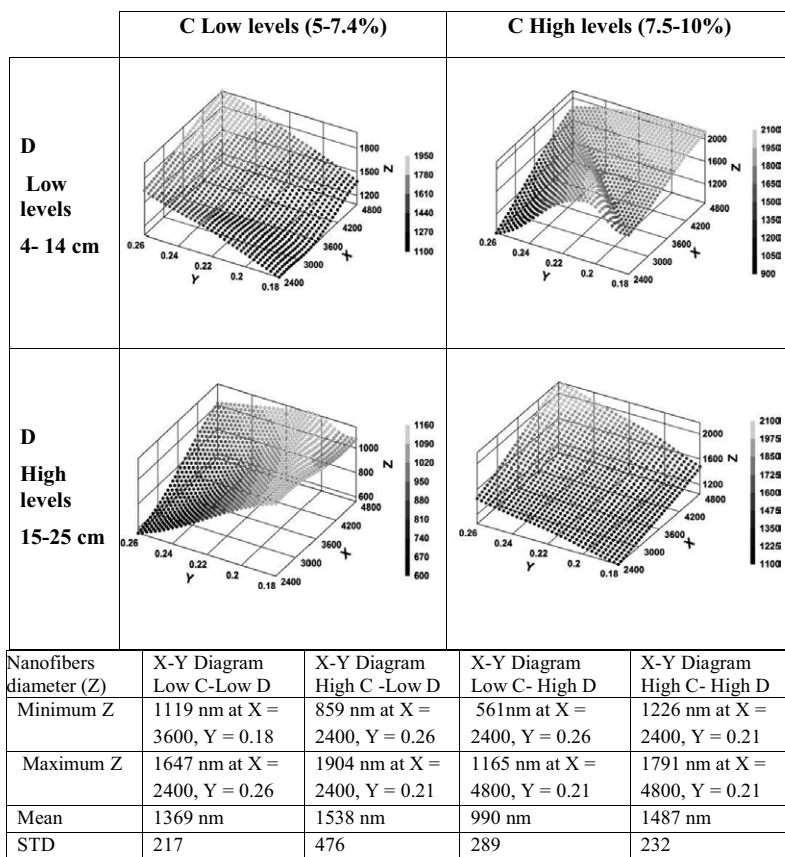
Totally, the movement of polymer solvent in the needle is according to the fluid mechanic laws and the equations such as capillary number, Reynolds number, weber number, and surface tension.

In Figure 5, the concentration of the solution and nozzle-collector distance was fixed at low and high values, respectively, and the effects of the other variables (rotor speed

and orifice diameter) on the fibers diameter were assessed. Similar to the previous figure, in all graphs (Figure 5) by increasing rotor speed, diameter of the fibers increased; however, the orifice diameter had an inverse effect on the size of the fibers.

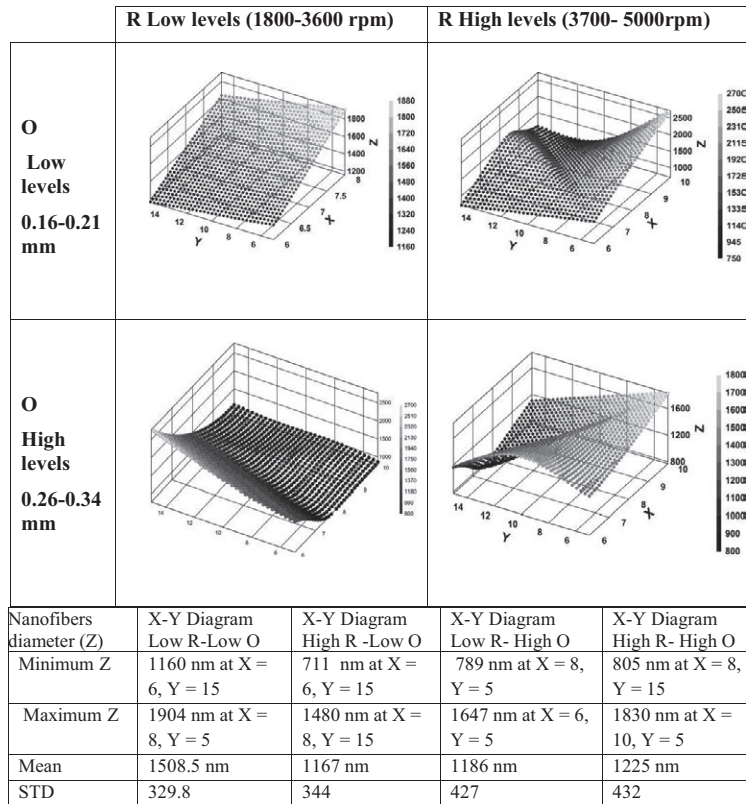
In the High C-High D and Low C-Low D levels, there is a direct relationship between the orifice diameter and the fibers size. However, in the High C-Low D and Low C-High D levels, increasing orifice diameter led to the reduction of the fibers diameter which could be due to the dominant effects of the nozzle-collector distance on the fibers size. It seems that increase in orifice diameter leads to lower flexure of solvent and consequently lower surface and diameter according to Euler-Lagrange equation.

In Figure 6, the rotor speed and orifice diameter were stabilized at the various levels, and the effect of the polymer concentration and nozzle-collector distance on the PVA fibers diameter was evaluated. In all graphs, the polymer concentration and nozzle-collector distance had direct and inverse effects, respectively, on the diameter of the fibers which is consistent with Pardon and colleagues studies.<sup>[18,26]</sup> On the other hand, in the Low O-High R area, both the concentration and the distance variables indicated inverse and direct effects on the fibers diameter, respectively, which exhibit



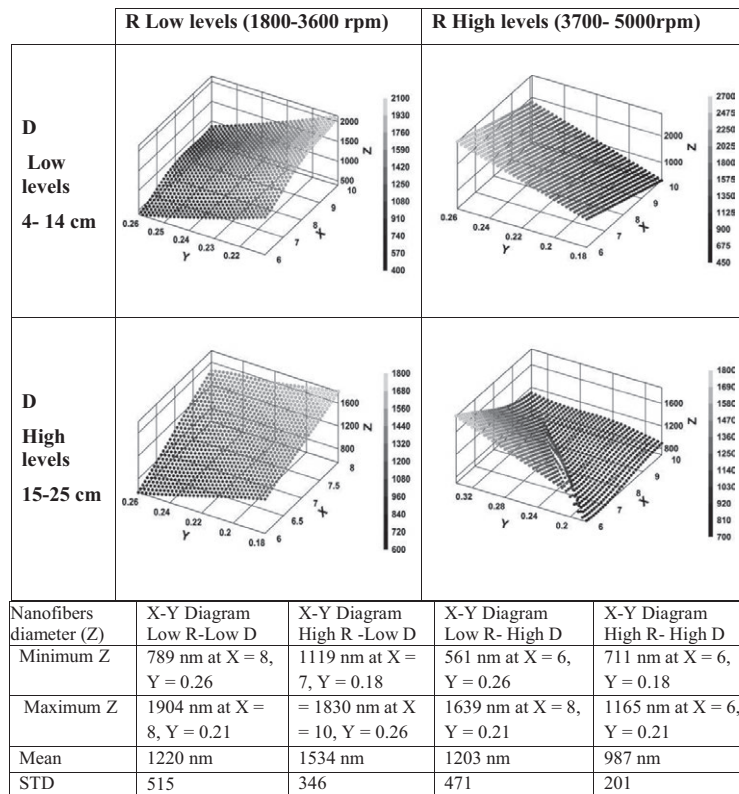
X= Rotor speed (rpm), Y= Nozzle orifice (mm), D=Nozzle to collector distance (cm), Z=Nanofibers diameter (nm), C= polymer concentration (%), O=

**FIGURE 5** The data and 3D plots of nanofibers diameter predicted by ANN fixed in mentioned levels (C-D diagram)



X= polymer concentration (%),Y= Nozzle to collector distance (cm),O=Nozzle orifice (mm), Z=Nanofibers diameter (nm), R= Rotor speed (rpm),

FIGURE 6 The data and 3D plots of nanofibers diameter predicted by ANN fixed in mentioned levels (R-O diagram)



X= polymer concentration (%),Y= Nozzle orifice (mm), D=Nozzle to collector distance (cm), Z=Nanofibers diameter (nm), R= Rotor speed (rpm),

FIGURE 7 The data and 3D plots of nanofibers diameter predicted by ANN fixed in mentioned levels (R-D diagram)



the complexity of relationships between the forspinning parameters. In addition, in High O-Low R area, fiber diameter decreased with increasing polymer concentration, and increased with the nozzle-collector distance. It seems that the cone of polymer do not form in the greater polymer concentration in high orifice diameter, and polymer has less capillary number at the moment of launching which leads to lower nanofibers diameter in the closer distance to the orifice tip.

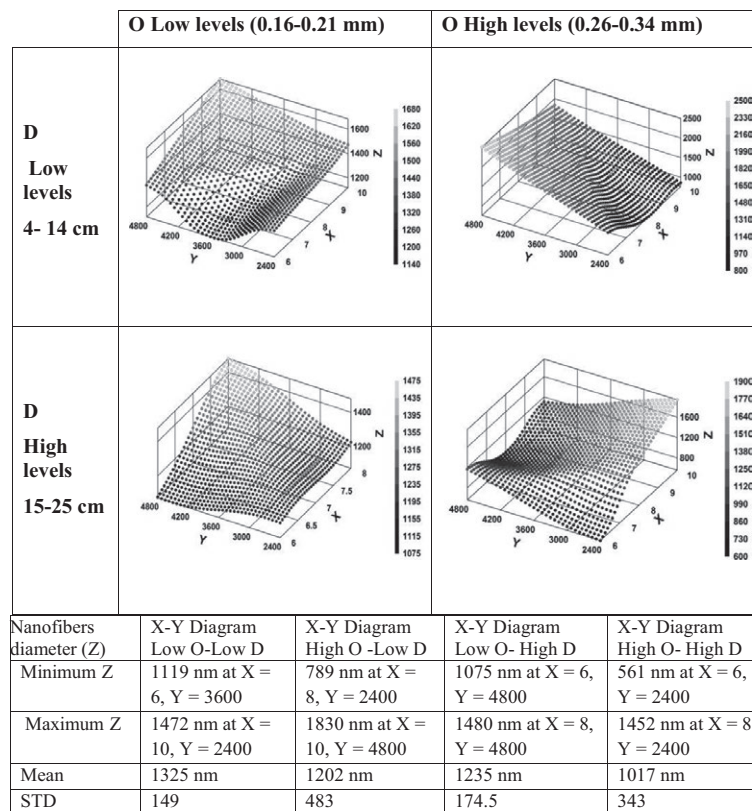
According to Figure 7, by increasing the polymer concentration, size of the fibers depicts the ascending trend, which is consistent with previous results, except in the Low D-High R level, which show the complex interactions between the solution concentration and both nozzle-collector distance and rotor speed parameters on the fibers diameter. Also, it seems that the effect of the orifice diameter on size of the fibers depends on the rotor speed. It means that if the rotor speed is fixed at a low level, increasing orifice diameter leads to increase in fibers diameter. However, in the high level of the rotor speed, increasing orifice diameter leads to drop in the fibers size.

In Figure 8, nozzle to the collector distance and the orifice diameter were stabilized at the high and low level, and the effects of the polymer concentration and rotor speed on the fibers size were depicted. In all graphs, the PVA concentration had the direct effects on the fibers diameter, which is consistent with previous results, except in the High O-Low

D level, in which increasing the polymer concentration leads to decrease in the fibers diameter. Also, the rotor speed had a direct effect on the diameter of the fibers in all groups, except in the High D-Low O level in which thinner fibers were produced via increasing the rotor speed.

## 4 | CONCLUSION

This study indicated the validity of ANN model in determining diameter of forspun nanofibers, despite complex interactions between the parameters involved in the forspinning. The neural network with four input factors, three hidden layers, and one output layer presents the best performance in the testing sets. Besides, MSE and linear regression between observed and predicted nanofibers diameter were about 0.1077 and 0.9387, respectively. The 3D graphs demonstrated that rotor speed and concentration of polymer solution have direct relation to the fibers diameter. Orifice diameter showed an insignificant effect on the diameter of the PVA forspun fibers. Finally, this study indicated complex interactions between the forspinning variables and fibers diameter which require additional research to clarify. However, these process complexities show the importance of modeling techniques to predict the desired outcomes.



X= polymer concentration (%),Y= Rotor speed (rpm),O= Nozzle orifice (mm), D=Nozzle to collector distance (cm), Z=Nanofibers diameter (nm).

**FIGURE 8** The data and 3D plots of nanofibers diameter predicted by ANN fixed in mentioned levels (O-D diagram)

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