

Comparison between Artificial Neural Network and Response Surface Methodology in the Prediction of the Production Rate of Polyacrylonitrile Electrospun Nanofibers

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Abstract: This paper focused on using response surface methodology (RSM) and artificial neural network (ANN) to analyze production rate of electrospun nanofibers. The three important electrospinning factors were studied including polymer concentration (wt %), applied voltage (kV) and the nozzle-collector distance (cm). The predicted production rates were in agreement with the experimental results in both ANN and RSM techniques. High regression coefficient between the variables and the response ($R^2=0.975$) indicates excellent evaluation of experimental data by second-order polynomial regression model. The regression coefficient was 0.988, which indicates that the ANN model was shows good fitting with experimental data. The obtained results indicate that the performance of ANN was better than RSM. It was concluded that applied voltage plays an important role (relative importance of 42.8 %) against production rate of electrospun nanofibers. The RSM model predicted the 2802.3 m/min value of the highest production rate at conditions of 15 wt % polymer concentration, 16 kV of the applied voltage, and 15 cm of nozzle-collector distance. The predicted value showed only 4.4 % difference with experimental results in which 2931.0 m/min at the same setting was observed.

Keywords: RSM, ANN, Nanofiber, Electrospinning, Production rate

Introduction

Fibers with diameters in the range of 1-100 nm are referred to as nanofibers in scientific literature related to fiber science. Structured polymeric nanofibers with diameters in the range of nanometers due to high specific surface area, which enables a high proportion of atoms to be on the nanofibers surface, are considerable interest for various kinds of application fields. Their applications are including: filtration, tissue engineering, drug delivery system, electromagnetic interference (EMI) shielding devices, hydrogen storage, ion adsorption, sensor, protective clothing and etc [1-5].

In recent years, various methods for producing polymeric nanofibers are used such as Drawing [6], Template Synthesis [7], Phase Separation [8], Self-Assembly [9], and electrospinning [10]. Among these methods, electrospinning is the cheapest and the most straightforward technique to produce nanofibers. The electrospinning process into four key stages: launching of the jet, elongation of the straight segment, development of whipping instability and solidification into a nanofibers. The most effective parameters influencing electrospinning have been classified in four groups: polymer properties (molecular weight and solubility), polymer solution parameters (polymer concentration, solution viscosity, conductivity and surface tension), processing conditions (applied voltage, nozzle-collector distance, feed rate and needle diameter), and ambient parameters (temperature, atmosphere

pressure and relative humidity) [11]. According to literature, molecular weight of the polymer, solution concentration, applied voltage, nozzle-collector distance, and feed rate were the main parameters influencing the electrospinning process for many kinds of polymers [1-3].

Response surface methodology (RSM) is a combination of mathematical and statistical techniques useful for empirical modeling and optimizing the effects of several independent variables on the response. RSM reduces the number of experimental runs needed to evaluate multiple parameters and their interactions. Using RSM, it is possible to estimate linear, interaction and quadratic effects of the several factors and a prediction model for the response. Finally, the main objective of RSM is to determine the optimal conditions for the system or to determine a region that satisfies the operating specifications [12,13]. Additionally, artificial neural network (ANN) is an information processing system that roughly replicates the behavior of a human brain by emulating the operations and connectivity of biological neurons. On the other hands, ANN cannot create an equation similar to RSM, but it works as human brain does and it estimates the response based on the trained data in the inquired range. The human brain is composed of 3-4 billions of nerve cells, called *neurons*, and these are interconnected to form the biological neural network. In order to construct a mathematical model of a neuron in ANN, to be called an *artificial neuron* [14,15]. The structure of artificial neuron included *weight*, *bias* and *transfer function*. Parallel connection between artificial neurons generates a layer. The ANN represents a network with a several number of layers consisting of

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parallel elements artificial neuron with different types of connections between layers and transfer function in each layer. In general, ANN is parallel interconnected structure consisting of: input layer of neuron (independent variables), a number of hidden layers, and output layer (response or responses). According to Kolmogorov's theorem, ANN with a single hidden layer should be capable of approximating any function to any degree of accuracy [16].

In the previous papers [17,18], we modified the conventional electrospinning process by replacing the flat collector with a hollow metallic cylinder for achieving highly orienting nanofibers. In this study a systematic statistical approach has been adopted to obtain maximum production rate of the electrospun nanofibers with different process conditions. The influence of process conditions on the production rate of the electrospun nanofibers was carried out using full factorial design (FFD). The RSM was used to develop a mathematical equation between the polymer concentration, applied voltage and nozzle-collector distance on production rate of the electrospinning process. Regression equations were developed for the same and in addition to that the effect of process conditions was also modeled using ANN. Comparison of prediction of production rate using ANN and RSM are discussed in this article.

Experimental

Materials

Polyacrylonitrile (PAN, $M_w=100,000$ g/mol) was supplied with Polyacryl Co. (Isfahan, Iran) and N,N-dimethylformamide (DMF) were obtained from Merck, respectively, as polymer and solvent.

Electrospinning

The solutions of PAN were prepared by dissolving 15, 17, and 19 wt % of sample in DMF separately via magnetic stirrer (Corning Hot Plate Stirrer PC-351) at 40 °C for 24 hours. The experimental set-up used for electrospinning

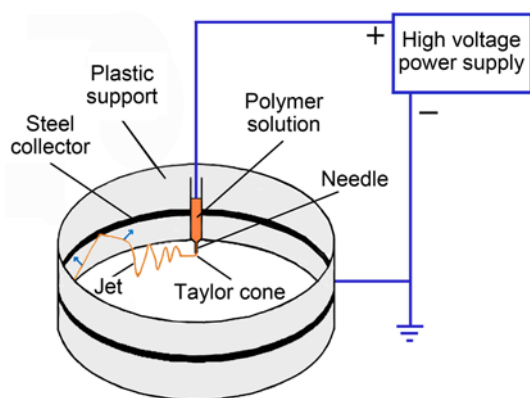
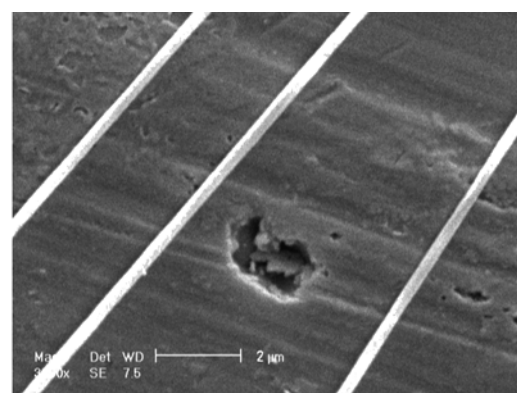


Figure 1. Schematic modified electrospinning setup with hollow cylindrical collector for production uniaxially aligned nanofibers.

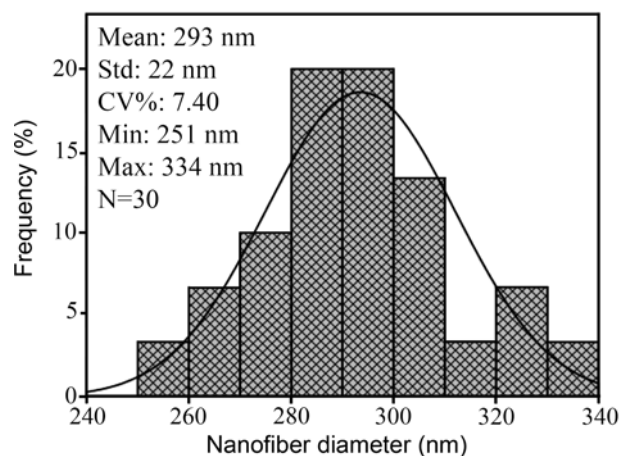
is shown in Figure 1. The key to the success of this technique is the creation of a rotating jet by using a cylindrical collector in which the needle tip is located at its center. The unique advantage of this method among the current methods is the ability of apparatus to weave continuously nanofibers in uniaxially aligned form. Nanofibers produced by this method are well-aligned, with several meters in length, and can be spread over a large area. The prepared PAN solution was added to a glass syringe with a needle tip (22G, L=34 mm, O.D=0.7 mm, and I.D=0.4 mm). The feeding rate of the polymer solutions was 0.25 ml/h and take-up speed 100 RPM was collected electrospun nanofibers. The electrospinning of PAN was performed at 22 ± 2 °C and relative humidity at 40-45 %. After electrospinning the fibrous mats were washed with deionized water and dried at 40 °C for 8 hours.

Measurement and Characterization

The morphology of the electrospun nanofibers was examined



(a)



(b)

Figure 2. (a) a typical SEM photograph of electrospun nanofibers mat, (b) corresponding diameter distribution (polymer concentration: 15 wt %, applied voltage: 12 kV and nozzle-collector distance: 25 cm).

by scanning electron microscope (SEM, Philips, XL-30) at an accelerating voltage of 25 kV under magnification of 35000X and the average diameter of nanofibers was measured with the SEM images using Image J software (National Institute of Health, USA). A typical SEM photograph of the electrospun nanofibers mat and its corresponding diameter distribution are shown in Figure 2. As can be seen, nanofibers produced by this method are well-aligned, with several meters in length, and can be spread over a large area. Therefore, the production rate (S_{exp}) of electrospinning process is calculated by the mathematical equation:

$$S_{exp} \text{ (m/min)} = N \left(\frac{\pi}{50} \right) \frac{D \text{ (cm)}}{T \text{ (min)}} \quad (1)$$

Where, N is the number of nanofibers layers, D is the nozzle-collector distance, and T is the time of electrospinning.

Response Surface Methodology

The effects of the three independent processing parameters namely; polymer concentration (P_1), applied voltage (P_2) and nozzle-collector distance (P_3) on production rate of electrospinning (m/min) were investigated using RSM. FFD are response surface designs, specially employed to require only three levels, coded as -1, 0 and +1, according to equation (2) [19].

$$X_i = \frac{P_i - [P_{Hi} + P_{Li}]/2}{[P_{Hi} - P_{Li}]/2} \quad (2)$$

Where, P_{Hi} and P_{Li} refer to the high and low levels of the variables P_i ($i=1, 2, 3$), respectively.

The total number of experiments ($N=27$) in this study with three factors was obtained from the equation: $N=3^k$, where k is the number of factors ($=3$). The statistical software package, Design-Expert (Version 8.0.3, Stat-Ease, Inc., Minneapolis, MN, USA, 2010) was used for the regression analysis of the experimental data, and to plot the response surface graphs. The corresponding actual values and the coded design experiments for each variable are listed in Tables 1 and 2, respectively.

When there are two levels for each parameter, linear model should be used, but with increasing levels to three or more, quadratic model should be used. According to three levels for each parameter in the FFD, the quadratic model is appropriate. In a system involving three significant independent variables X_1, X_2, X_3 the mathematical relationship between the response and these variables can be approximated by the quadratic polynomial equation [20]:

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

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

Table 1. Actual and coded values of the variables

| Coded values | Actual values | | |
|--------------|------------------------|-----------------|------------------|
| | Concentration (C, wt%) | Voltage (V, kV) | Distance (D, cm) |
| -1 | 15 | 8 | 15 |
| 0 | 17 | 12 | 20 |
| 1 | 19 | 16 | 25 |

Table 2. The experimental design for the three independent variables and response at different factor levels

| No. | Coded values of the variables | | | Response |
|-----|-------------------------------|-------------------|--------------------|---------------------|
| | Concentration (X_1) | Voltage (X_2) | Distance (X_3) | Production rate (S) |
| 1 | -1 | -1 | -1 | 427.8±28.7 |
| 2 | -1 | -1 | 0 | 245.4±19.5 |
| 3 | -1 | -1 | 1 | 159.0±19.5 |
| 4 | -1 | 0 | -1 | 1371.6±78.6 |
| 5 | -1 | 0 | 0 | 787.8±63.9 |
| 6 | -1 | 0 | 1 | 625.2±35.7 |
| 7 | -1 | 1 | -1 | 2931.0±70.3 |
| 8 | -1 | 1 | 0 | 2211.0±80.6 |
| 9 | -1 | 1 | 1 | 1911.0±43.8 |
| 10 | 0 | -1 | -1 | 187.2±20.8 |
| 11 | 0 | -1 | 0 | 117.0±18.1 |
| 12 | 0 | -1 | 1 | 111.0±21.8 |
| 13 | 0 | 0 | -1 | 914.4±69.1 |
| 14 | 0 | 0 | 0 | 648.0±40.7 |
| 15 | 0 | 0 | 1 | 504.6±62.3 |
| 16 | 0 | 1 | -1 | 2214.0±80.7 |
| 17 | 0 | 1 | 0 | 1408.2±64.1 |
| 18 | 0 | 1 | 1 | 788.4±69.8 |
| 19 | 1 | -1 | -1 | 109.2±16.0 |
| 20 | 1 | -1 | 0 | 75.6±7.9 |
| 21 | 1 | -1 | 1 | 52.8±5.8 |
| 22 | 1 | 0 | -1 | 623.4±40.5 |
| 23 | 1 | 0 | 0 | 299.4±15.8 |
| 24 | 1 | 0 | 1 | 196.8±4.7 |
| 25 | 1 | 1 | -1 | 1261.8±29.0 |
| 26 | 1 | 1 | 0 | 615.6±37.3 |
| 27 | 1 | 1 | 1 | 399.6±36.3 |

The equations were validated by the statistical tests called the ANOVA analysis. The quality of the fit of regression model was expressed by the coefficient of determination R^2 and $adj-R^2$ in equations (4) and (5), respectively [21].

$$R^2 = 1 - \frac{SS_{residual}}{SS_{model} + SS_{residual}} \quad (4)$$

$$adj-R^2 = 1 - \frac{SS_{residual}/DF_{residual}}{(SS_{model} + SS_{residual})/(DF_{model} + DF_{residual})} \quad (5)$$

The terms SS and DF corresponds to sum of squares and degrees of freedom, respectively. Response surfaces were drawn to determine the individual and interactive effects of the test variable on the production rate of electrospun nanofibers.

Artificial Neural Network

In this work, multi-layer perceptron ANN with one hidden layer, according to Kolmogorov's theorem was utilized. For all data sets hyperbolic tangent sigmoid transfer function (equation (6)) in the hidden layer and a linear transfer function (equation (7)) in the output node was employed [16].

$$T(x) = \frac{1 - e^{-2x}}{1 + e^{-2x}} \quad (6)$$

$$L(x) = x \quad (7)$$

The experimental data were divided into two groups training and test with 18 and 9 samples, respectively. The ANN was trained using the scaled conjugate gradient backpropagation algorithm (*trainscg*). In order to determine the optimum number of neuron in hidden layer, a series of topologies was used, in which the number of neuron were varied from 2 to 20. Each topology was repeated ten times to avoid random correlation due to random initialization of the weights and bias. The optimal architecture of the ANN model and its parameter variation were determined based on the minimum value of the mean square error (MSE) of the training and testing sets [22]. MSE measures the performance of the network according to the following equation:

$$MSE = \frac{1}{N} \sum_{i=1}^{i=N} [(S_{ANN})_i - (S_{exp})_i]^2 \quad (8)$$

Where N is the number of data point, $(S_{ANN})_i$ is the ANN prediction, $(S_{exp})_i$ is the experimental response, and i is an index of data.

Results and Discussion

Response Surface Methodology

The analysis of variance for the response has been summarized in Table 3. The values of P-values less than 0.05 indicates that the model terms are significant, whereas, the values greater than 0.05 are not significant. The ANOVA analysis of the optimization study indicated that the model terms, X_1 , X_2 , X_3 , X_1X_2 , X_2X_3 and X_2^2 were significant ($P < 0.05$) and X_1X_3 , X_1^2 and X_3^2 were not significant ($P > 0.05$). The final regression model in terms of coded factors is presented as follows.

Table 3. Analysis of variance (ANOVA) for response surface model

| Source | F-value | Prob>F | Remarks |
|----------------------|---------|----------|-----------------|
| Model | 105.68 | < 0.0001 | Significant |
| X_1 -Concentration | 172.90 | < 0.0001 | Significant |
| X_2 -Voltage | 524.65 | < 0.0001 | Significant |
| X_3 -Distance | 97.82 | < 0.0001 | Significant |
| X_1X_2 | 91.61 | < 0.0001 | Significant |
| X_1X_3 | 2.49 | 0.1327 | Not significant |
| X_2X_3 | 44.26 | < 0.0001 | Significant |
| X_1X_1 | 0.31 | 0.5832 | Not significant |
| X_2X_2 | 12.55 | 0.0025 | Significant |
| X_3X_3 | 4.35 | 0.0508 | Not significant |

$$S_{RSM} = 663.5 - 390.9X_1 + 680.9X_2 - 294.0X_3 - 348.4X_1X_2 - 242.2X_2X_3 + 182.4X_2^2 \quad (9)$$

In terms of actual factors the production rate of electrospinning process is expressed by following regression equation:

$$S_{RSM} = -7031.0 + 327.2C + 879.3V + 86.5D - 43.6C.V - 12.1V.D + 11.4V^2 \quad (10)$$

The model P-values (<0.0001) suggested that the obtained experimental data has a good agreement with the model. The regression equation obtained from the ANOVA showed that the R^2 was 0.975. However, since the model equations in our case include additional terms because of the three level independent variables, the adjusted R^2 for the degrees of freedom ($adj-R^2$) was chosen to be examined as well. $Adj-R^2$ is much less sensitive to the degrees of freedom and cannot be affected as seriously by including more terms in the model, while it is always lower than R^2 . Therefore, it is a better criterion of the goodness of the fit. The $adj-R^2$ value for the response was found to be equal to 0.967.

Figure 3 shows the contour plot of the response at different polymer concentration and applied voltage for constant nozzle-collector distance (at minimum and maximum levels). It can be seen, with increase in concentration there is a decrease in production rate of the electrospun nanofibers. As described in the literature, increasing polymer concentration will result in greater polymer chain entanglements and higher viscoelastic force enabling the charged jet to withstand a larger electrostatic stretching force and leading to a lower production rate of electrospinning process [1-3]. Moreover, it can be seen from the figures that at any given concentration the production rate increases with increasing the applied voltage at both lower and higher nozzle-collector distance. Increasing the applied voltage will increase the electric field strength and larger electrostatic stretching force causes the jet to accelerate more in the electric field, thereby

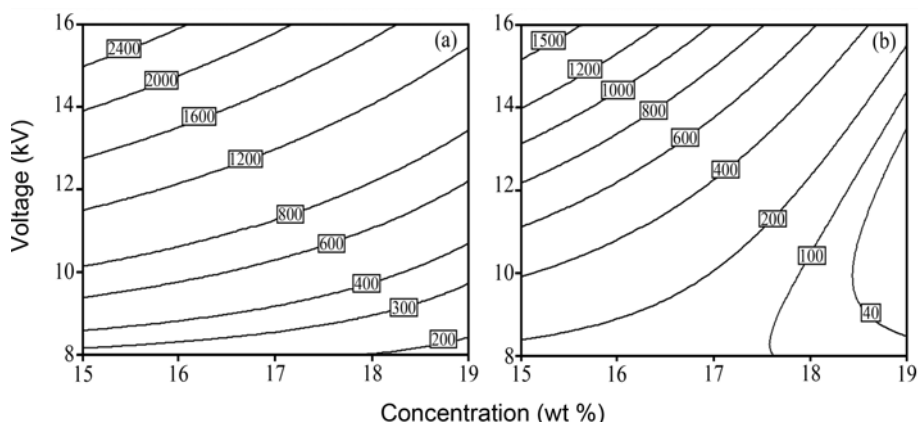


Figure 3. Effect of concentration and voltage on the production rate (m/min) at various levels of the nozzle-collector distances (a) 15 cm and (b) 25 cm.

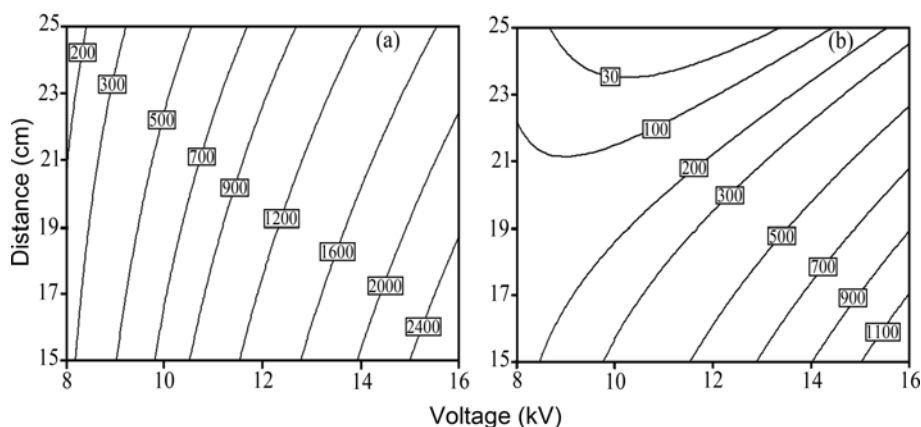


Figure 4. Effect of voltage and distance on the production rate (m/min) at various levels of polymer concentration (a) 15 wt % and (b) 19 wt %.

favoring higher production rate in nanofibers formation. Figure 4 shows the contour plot of production rate of the electrospun nanofibers at different applied voltage and nozzle-collector distance for low and high levels of the polymer concentration. From the figure it can be seen that the response decreases with increase in nozzle-collector distance. Increasing the spinning distance, the electric field strength will decrease resulting in less acceleration hence stretching of the jet which leads to lower production rate in electrospinning process. Moreover, it can be seen from the figures that at any given concentration and distance the production rate increases with increasing the applied voltage.

Artificial Neural Networks

The optimal architecture of the ANN model and its parameter variation were determined based on the minimum value of the MSE of the training and prediction set. In optimization of the neural network, two neurons were used in the hidden layer as an initial estimate. Figure 5 illustrates the relation between MSE (network error) and number of

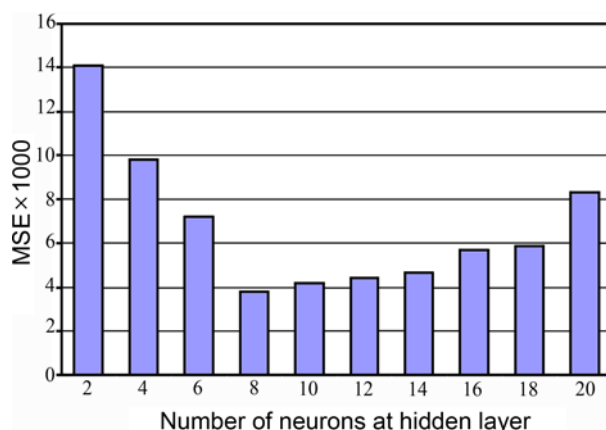


Figure 5. Effect of the number of neurons in hidden layer on the performance of the ANN.

neurons in the hidden layer. As can be seen, the MSE is minimum just about 8 neurons. Hence we used two-layered perceptron neural network (with 8 artificial neuron in hidden

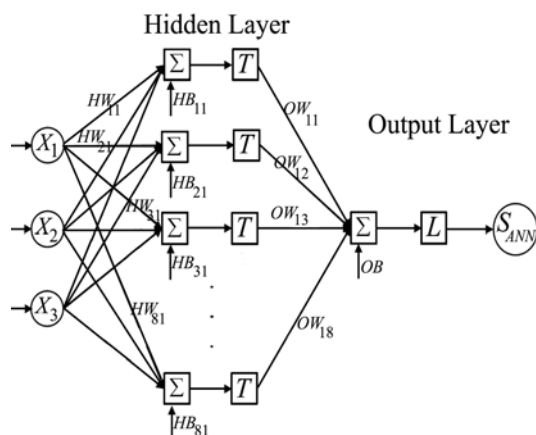


Figure 6. Optimized perceptron neural network structure.

layer) for modeling of production rate of electrospun nanofibers (Figure 6). The ANN was trained up to 1,500 cycles to obtain optimum weights and bias. The weights and bias of ANN for the production rate of electrospun nanofibers are given in Table 4. Figure 7 shows a comparison between experimental production rate values and predicted values using the neural network model. The R^2 value was 0.988, which indicates that the model was shows good fitting with experimental data. Both RSM and ANN model shows a very good relationship between the experimental and predicted response values.

The neural network weight matrix can be used to assess the relative importance (RI) of the various input variables on the output variables. It was proposed an equation based on

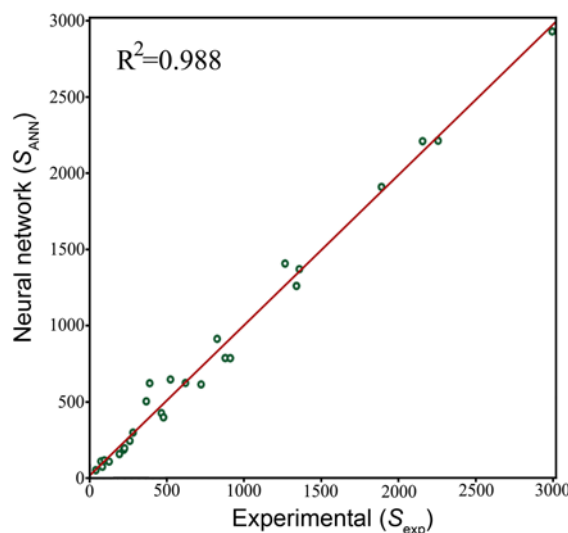


Figure 7. Correlation plot between ANN predicted and experimental values of the output.

the partitioning of connection weights [19,20]:

$$RI_j = \frac{\sum_{m=1}^{N_h} \left(\left(|HW_{jm}| / \sum_{k=1}^{N_i} |HW_{km}| \right) \times |OW_{mj}| \right)}{\sum_{k=1}^{N_i} \left\{ \sum_{m=1}^{N_h} \left(\left(|HW_{km}| / \sum_{k=1}^{N_i} |HW_{km}| \right) \times |OW_{mj}| \right) \right\}} \times 100 \quad (11)$$

where RI_j is the relative importance of the j th input variable on the output variable, N_i and N_h are the numbers of input

Table 4. Weights and bias obtained in training ANN

| Hidden layer | | | Output layer | | |
|--------------------|-----------|-----------|------------------|--------------------|------------------|
| Hidden weight (HW) | | | Hidden bias (HB) | Output weight (OW) | Output bias (OB) |
| HW_{11} | HW_{12} | HW_{13} | HB_{11} | OW_{11} | OB |
| 2.6943 | 0.8222 | -2.4518 | -4.2713 | -0.1706 | 0.2904 |
| HW_{21} | HW_{22} | HW_{23} | HB_{21} | OW_{12} | |
| 0.1972 | 1.7632 | -2.9253 | -2.3166 | 0.2452 | |
| HW_{31} | HW_{32} | HW_{33} | HB_{31} | OW_{13} | |
| -0.4033 | 4.5567 | 4.4331 | -1.6408 | 0.0210 | |
| HW_{41} | HW_{42} | HW_{43} | HB_{41} | OW_{14} | |
| -0.4187 | 0.7464 | -0.3845 | -1.0438 | 1.1241 | |
| HW_{51} | HW_{52} | HW_{53} | HB_{51} | OW_{15} | |
| 0.7014 | -0.4514 | -5.1037 | 0.2800 | 0.0080 | |
| HW_{61} | HW_{62} | HW_{63} | HB_{61} | OW_{16} | |
| -2.4475 | -4.9625 | 1.3645 | -1.5488 | -0.0251 | |
| HW_{71} | HW_{72} | HW_{73} | HB_{71} | OW_{17} | |
| 2.5010 | -2.4792 | -1.3175 | 5.8507 | -0.1658 | |
| HW_{81} | HW_{82} | HW_{83} | HB_{81} | OW_{18} | |
| 0.5225 | 1.6975 | 0.7445 | 3.3729 | 0.0668 | |

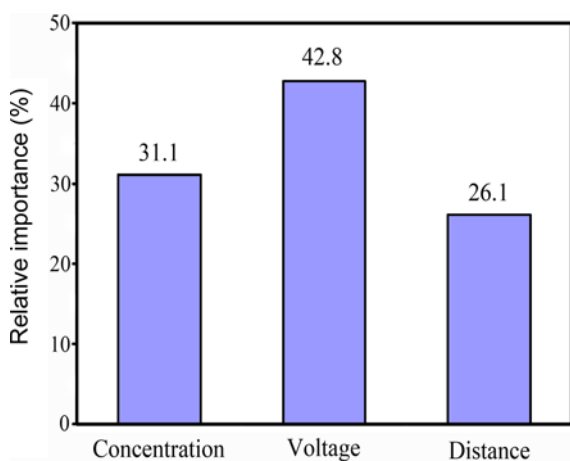


Figure 8. Relative importance of input variables on the value of production rate.

variables and hidden neurons, respectively, HW and OW are connection weights, and subscript ' n ' refer to output response. In this work: $j=1, 2$ and 3 , $N_{\bar{i}}=3$, $N_{\bar{h}}=8$, and $n=1$.

The relative importance of input variables on the value of production rate as calculated by equation (11) was shown in Figure 8. As can be seen, all of the variables (polymer concentration, applied voltage, and nozzle-collector distance) have strong effects on the production rate. Therefore, none of the variables studied in this work could have been neglected from the present analysis. However, the applied voltage and polymer concentration, with relative importance of respectively 42.8 and 31.1 %, appeared to be more influential parameters in the production rate. These results are in good agreement with the ones obtained with RSM.

Optimization

The last step of the study was to find out a desire area in the design space. Numerical optimizations search the design

space, using the model that created in the analysis to find factor settings that meet the defined goal. Desirability is simply a mathematical method to find the optimum and also, it is an objective function that ranges from zero outside of the limits to one at the goal. Figure 9 shows the 3D and contour graph of desirability for the solutions found via numerical optimization. It can be seen, the numerical optimization finds a point that maximizes the desirability function. In this work, our goal is to maximize the value of production rate of electrospinning process. Optimization finds a good set of conditions that will meet the maximum response. Process conditions including: concentration of 15 wt %, voltage of 16 kV and distance of 15 cm create a maximum in production rate with desirability of 0.955. The theoretical production rate under the above conditions was $S_{RSM}=2802.3$ m/min. The experimental results observed (2931.0 m/min) was 4.4 % greater than the predicted value under the same electrospinning settings.

Conclusion

A response surface model based on the FFD technique, and artificial neural network was used for modeling and predicting the production rate of nanofibers produced by electrospinning. The production rate was investigated by various parameters such as concentrations, applied voltages and nozzle-collector distances (each of them adjusted in three levels). The RSM analysis confirmed that applied voltage and polymer concentration were the main significant factors affecting the production rate of electrospun nanofibers. The configuration of the ANN giving the smallest MSE was a two-layer ANN with tangent sigmoid transfer function at hidden layer with 8 neurons, linear transfer function at output layer and scaled conjugate gradient backpropagation training algorithm. The ANN model shows higher regression coefficient than the RSM model. Therefore, the obtained results indicate that the performance of ANN was better than

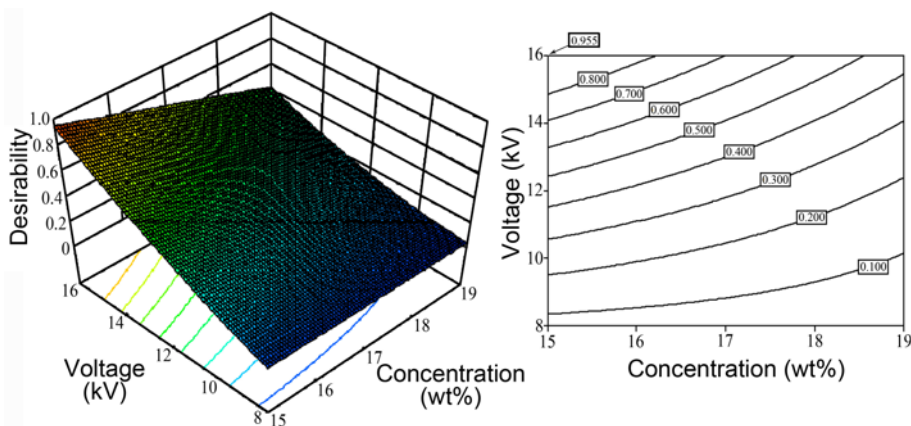


Figure 9. 3D and contour plot of desirability for the conditions found via numerical optimization. Nozzle to collector distance adjusted at constant value of 15 cm.

RSM. Numerical optimization has been performed by considering desirability function to access the region in design space that introduced maximum production rate. Maximum production rate in optimized process conditions (concentration of 15 wt %, voltage of 16 kV and nozzle-collector distance of 15 cm) created with desirability of 0.955. The experimental results observed the production rate (2931.0 m/min) was 4.4 % greater than the predicted value (2802.3 m/min) under the same electrospinning settings.

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