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On the contact angle of electrospun polyacrylonitrile nanofiber mat

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In this work, effects of four electrospinning parameters, including solution concentration (wt.%), applied voltage (kV), tip to collector distance (cm), and volume flow rate (ml/h), on contact angle (CA) of nanofiber mat are studied. To optimize and predict the CA of electrospun fiber mat, response surface methodology (RSM) and artificial neural network (ANN) are employed and a quantitative relationship between processing variables and CA of electrospun fibers is established. It is found that the solution concentration is the most important factor impacting the CA of electrospun fiber mat. The obtained results demonstrated that both the proposed models are highly effective in estimating CA of electrospun fiber mat. However, more accurate results are obtained by ANN model as compared to the RSM model. In ANN model the determination coefficient (R^2) and absolute percentage error between actual and predicted response are obtained as 0.965 and 1.97, respectively.

Keywords: Electrospinning, Contact angle, Response surface methodology, Artificial neural network

1. INTRODUCTION

The wettability of solid surfaces is a very important property of surface chemistry, which is controlled by both the chemical composition and the geometrical microstructure of a rough surface [1-3]. When a liquid droplet contacts a rough surface, it will spread or remain as droplet with the formation of angle between the liquid and solid phases. Contact angle (CA) measurements are widely used to characterize the wettability of rough surface [3–5]. There are various methods to make a rough surface, such as electrospinning, electrochemical deposition, evaporation, chemical vapor deposition (CVD), plasma, and so on.

Electrospinning as a simple and effective method for preparation of nanofibrous materials have attracted increasing attention during the last two decade [6]. Electrospinning process, unlike the conventional fiber spinning systems (melt spinning, wet spinning, etc.), uses electric field force instead of mechanical force to draw and stretch a polymer jet [7]. This process involves three main components including syringe filled with a polymer solution, a high voltage supplier to provide the required electric force for stretching the liquid jet, and a grounded collection plate to hold the nanofiber mat. The charged polymer solution forms a liquid jet that is drawn towards a grounded collection plate. During the jet movement to the collector, the solvent evaporates and dry fibers deposited as randomly oriented structure on the surface of a collector [8–13]. The electrospun nanofiber mat possesses high specific surface area, high porosity, and small pore size. Therefore, they have been suggested as excellent candidate for many applications including filtration [14], multifunctional membranes [15], biomedical agents [16], tissue engineering scaffolds [17–18], wound dressings [19], full cell [20] and protective clothing [21].

The morphology and the CA of the electrospun nanofibers can be affected by many electrospinning parameters including solution properties (the concentration, liquid viscosity, surface tension, and dielectric properties of the polymer solution), processing conditions (applied voltage, volume flow rate, tip to collector distance, and the strength of the applied electric field), and ambient conditions (temperature, atmospheric pressure and humidity) [9–12].

In this work, the influence of four electrospinning parameters, comprising solution concentration, applied voltage, tip to collector distance, and volume flow rate, on the CA of the electrospun PAN nanofiber mat was carried out using response surface methodology (RSM) and artificial neural network (ANN). First, a central composite design (CCD) was used to evaluate main and combined effects of above parameters. Then, these independent parameters were fed as inputs to

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an ANN while the output of the network was the CA of electrospun fiber mat. Finally, the importance of each electrospinning parameters on the variation of CA of electrospun fiber mat was determined and comparison of predicted CA value using RSM and ANN are discussed.

2. EXPERIMENTAL

2.1. Materials

PAN powder was purchased from Polyacryle Co. (Iran). The weight average molecular weight (M_w) of PAN was approximately 100,000 g/mol. *N-N*, dimethylformamide (DMF) was obtained from Merck Co. (Germany) and was used as a solvent. These chemicals were used as received.

2.2. Electrospinning

The PAN powder was dissolved in DMF and gently stirred for 24 h at 50ºC. Therefore, homogenous PAN/DMF solution was prepared in different concentration ranged from 10 wt.% to 14 wt.%. Electrospinning was set up in a horizontal configuration as shown in Figure 1. The electrospinning apparatus consisted of 5 ml plastic syringe connected to a syringe pump and a rectangular grounded collector (aluminum sheet). A high voltage power supply (capable to produce 0–40 kV) was used to apply a proper potential to the metal needle. It should be noted that all electrospinnings were carried out at room temperature.

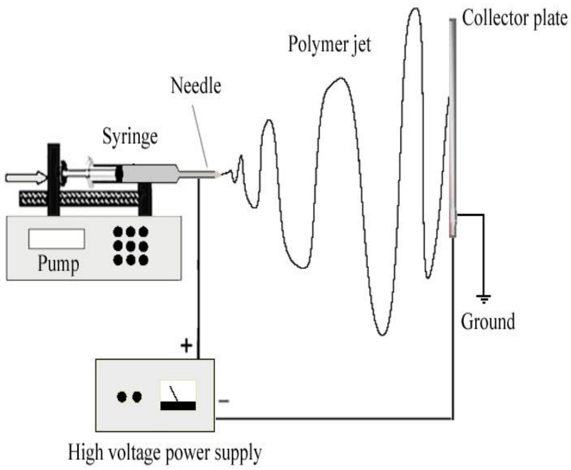

Fig. 1. Schematic of electrospinning set up.

2.3. Measurement and characterization

The morphology of the gold-sputtered electrospun fibers were observed by scanning electron microscope (SEM, Philips XL-30). The average fiber diameter and distribution was determined from selected SEM image by measuring at least 50 random fibers. The wettability of electrospun fiber mat was determined by CA measurement. The CA measurements were carried out using specially arranged microscope equipped with camera and PCTV vision software as shown in Figure 2. The droplet used was distilled water and was 1 µl in volume. The CA experiments were carried out at room temperature and were repeated five times. All contact angles measured within 20 s of placement of the water droplet on the electrospun fiber mat. A typical SEM image of electrospun fiber mat, its corresponding diameter distribution and CA image are shown in Figure 3.

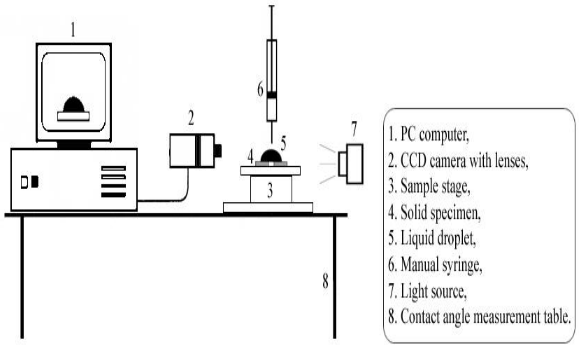

Fig. 2. Schematic of CA measurement set up.

2.4. Experimental design

2.4.1. Response surface methodology

Response surface methodology (RSM) is a combination of mathematical and statistical techniques used to evaluate the relationship between a set of controllable experimental factors and observed results. This optimization process is used in situations where several input variables influence some output variables (responses) of the system [22–23].

In the present study, central composite design (CCD) was employed to establish relationships between four electrospinning parameters and the CA of electrospun fiber mat. The experiment was performed for at least three levels of each factor to fit a quadratic model. Based on preliminary experiments, polymer solution concentration (wt.%), applied voltage (kV), tip to collector

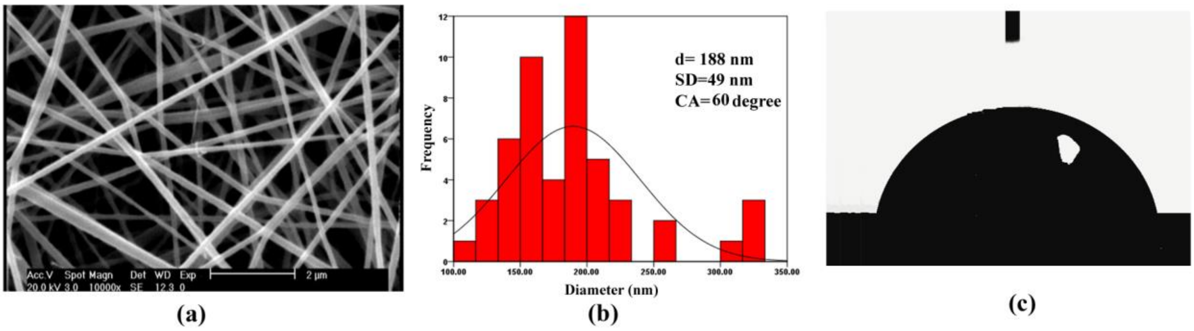

Fig. 3. A typical (a) SEM image, (b) fiber diameter distribution, and (c) CA of electrospun fiber mat.

distance (cm), and volume flow rate (ml/h) were determined as critical factors with significance effect on CA of electrospun fiber mat. These factors were four independent variables and chosen equally spaced, while the CA of electrospun fiber mat was dependent variable. The values of –1, 0, and 1 are coded variables corresponding to low, intermediate and high levels of each factor respectively. The experimental parameters and their levels for four independent variables are shown in Table 1. The regression analysis of the experimental data was carried out to obtain an empirical model between processing variables. The contour surface plots were obtained using Design-Expert® software.

Table 1. Design of experiment (factors and levels)

Factor	Variable	Unit	Factor level		
			-1	0	1
X_1	Solution concentration	(wt.%)	10	12	14
X_2	Applied voltage	(kV)	14	18	22
X_3	Tip to collector distance	(cm)	10	15	20
X_4	Volume flow rate	(ml/h)	2	2.5	3

The quadratic model, Equation (1) including the linear terms, was fitted to the data.

$$Y = \beta_0 + \sum_{i=1}^{4}\beta_i x_i + \sum_{i=1}^{4}\beta_{ii} x_i^2 + \sum_{i=1}^{3}\sum_{j=2}^{4}\beta_{ij} x_i x_j \quad (1)$$

where, Y is the predicted response, x_i and x_j are the independent variables, β_o is a constant, $\beta_i$is the linear coefficient, β_{ii} is the squared coefficient, and β_{ij} is the second-order interaction coefficients [22, 23].

The quality of the fitted polynomial model was expressed by the determination coefficient (R^2) and its statistical significance was performed with the Fisher's statistical test for analysis of variance (ANOVA).

2.4.2. Artificial neural network

Artificial neural network (ANN) is an information processing technique, which is inspired by biological nervous system, composed of simple unit (neurons) operating in parallel. A typical ANN consists of three or more layers, comprising an input layer, one or more hidden layers and an output layer. Every neuron has connections with every neuron in both the previous and the following layer. The connections between neurons consist of weights and biases. The weights between the neurons play an important role during the training process. Each neuron in hidden layer and output layer has a transfer function to produce an estimate as target. The interconnection weights are adjusted, based on a comparison of the network output (predicted data) and the actual output (target), to minimize the error between the network output and the target [6,24–25].

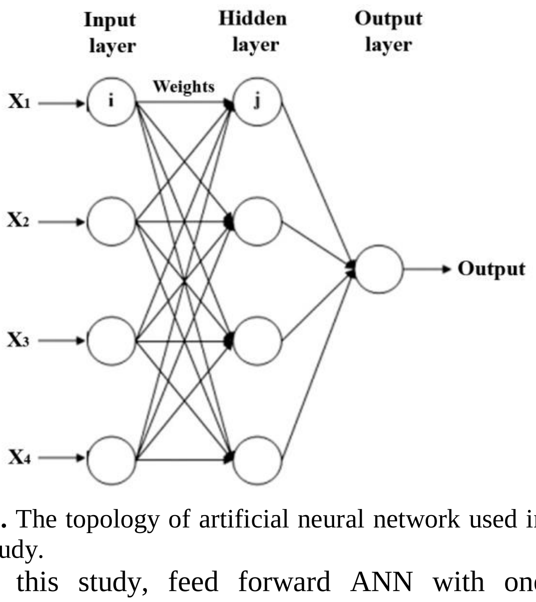

Fig. 4. The topology of artificial neural network used in this study.

In this study, feed forward ANN with one hidden layer composed of four neurons was selected. The ANN was trained using back-propagation algorithm. The same experimental data used for each RSM designs were also used as the input variables of the ANN. There are four neurons in the input layer corresponding to four electrospinning parameters and one neuron in the output layer corresponding to CA of electrospun fiber mat. Figure 4 illustrates the topology of ANN used in this investigation.

3. RESULTS AND DISCUSSION

This section discusses in details the wettability behavior of electrospun fiber mat concluded from CA measurements. The results of the proposed RSM and ANN models are also presented followed by a comparison between those models.

3.1. The analysis of variance (ANOVA)

All 30 experimental runs of CCD were performed according to Table 2. A significance level of 5% was selected; that is, statistical conclusions may be assessed with 95% confidence. In this significance level, the factor has significant effect on CA if the p-value is less than 0.05. On the other hand, when p-value is greater than 0.05, it is concluded the factor has no significant effect on CA.

The results of analysis of variance (ANOVA) for the CA of electrospun fibers are shown in Table 3. Equation (2) is the calculated regression equation.

$$\begin{aligned}CA = {} & 25.80 - 9.89X_1 + 2.17X_2 + 4.33X_3 - 2.33X_4 \\ & - 1.63X_1X_2 - 1.63X_1X_3 + 1.63X_1X_4 - 0.88X_2X_3 - 0.63X_2X_4 + 0.37X_3X_4 \\ & + 7.90X_1^2 + 6.40X_2^2 - 0.096X_3^2 + 2.90X_4^2\end{aligned} \quad (2)$$

From the p-values presented in Table 3, it can be concluded that the p-values of terms X_3^2, X_4^2, X_2X_3, X_2X_4 and X_3X_4 is greater than the

Table 2. The actual design of experiments and response

No.	Electrospinning parameters X_1 Concentration	X_2 Voltage	X_3 Distance	X_4 Flow rate	Response CA (°)
1	10	14	10	2	44±6
2	10	22	10	2	54±7
3	10	14	20	2	61±6
4	10	22	20	2	65±4
5	10	14	10	3	38±5
6	10	22	10	3	49±4
7	10	14	20	3	51±5
8	10	22	20	3	56±5
9	10	18	15	2.5	48±3
10	12	14	15	2.5	30±3
11	12	22	15	2.5	35±5
12	12	18	10	2.5	22±3
13	12	18	20	2.5	30±4
14	12	18	15	2	33±4
15	12	18	15	3	25±3
16	12	18	15	2.5	26±4
17	12	18	15	2.5	29±3
18	12	18	15	2.5	28±5
19	12	18	15	2.5	25±4
20	12	18	15	2.5	24±3
21	12	18	15	2.5	21±3
22	14	14	10	2	31±4
23	14	22	10	2	35±5
24	14	14	20	2	33±6
25	14	22	20	2	37±4
26	14	14	10	3	19±3
27	14	22	10	3	28±3
28	14	14	20	3	39±5
29	14	22	20	3	36±4
30	14	18	15	2.5	20±3

Table 3. Analysis of variance for the CA of electrospun fiber mat

Source	SS	DF	MS	F-value	Probe > F	Remarks
Model	4175.07	14	298.22	32.70	<0.0001	Significant
X_1-Concentration	1760.22	1	1760.22	193.01	<0.0001	Significant
X_2-Voltage	84.50	1	84.50	9.27	0.0082	Significant
X_3-Distance	338.00	1	338.00	37.06	<0.0001	Significant
X_4-Flow rate	98.00	1	98.00	10.75	0.0051	Significant
X_1X_2	42.25	1	42.25	4.63	0.0481	Significant
X_1X_3	42.25	1	42.25	4.63	0.0481	Significant
X_1X_4	42.25	1	42.25	4.63	0.0481	Significant
X_2X_3	12.25	1	12.25	1.34	0.2646	
X_2X_4	6.25	1	6.25	0.69	0.4207	Significant
X_3X_4	2.25	1	2.25	0.25	0.6266	
X_1^2	161.84	1	161.84	17.75	0.0008	Significant
X_2^2	106.24	1	106.24	11.65	0.0039	Significant
X_3^2	0.024	1	0.024	0.0026	0.9597	
X_4^2	21.84	1	21.84	2.40	0.1426	
Residual	136.80	15	9.12			
Lack of Fit	95.30	10	9.53	1.15	0.4668	

Table 4. Weights and bias obtained in training ANN

Hidden layer	Weights	IW_{11}	IW_{12}	IW_{13}	IW_{14}
		1.0610	1.1064	21.4500	3.0700
		IW_{21}	IW_{22}	IW_{23}	IW_{24}
		-0.3346	2.0508	0.2210	-0.2224
		IW_{31}	IW_{32}	IW_{33}	IW_{34}
		-0.6369	-1.1086	-41.5559	0.0030
		IW_{41}	IW_{42}	IW_{43}	IW_{44}
		-0.5038	-0.0354	0.0521	0.9560
	Bias	b_{11}	b_{21}	b_{31}	b_{41}
		-2.5521	-2.0885	-0.0949	1.5478
Output layer	Weights	LW_{11}			
		0.5658			
		LW_{21}			
		0.2580			
		LW_{31}			
		-0.2759			
		LW_{41}			
		-0.6657			
	Bias	b			
		0.7104			

significance level of 0.05, therefore they have no significant effect on the CA of electrospun fiber mat. Since the above terms had no significant effect on CA of electrospun fiber mat, these terms were removed. The fitted equations in coded unit are given in Equation (3).

$$\begin{aligned} CA = {} & 26.07 - 9.89X_1 + 2.17X_2 + 4.33X_3 - 2.33X_4 \\ & - 1.63X_1X_2 - 1.63X_1X_3 + 1.63X_1X_4 \\ & + 9.08X_1^2 + 7.58X_2^2 \end{aligned} \tag{3}$$

Now, all the p-values are less than the significance level of 0.05. Analysis of variance showed that the RSM model was significant ($p<0.0001$), which indicated that the model has a good agreement with experimental data. The determination coefficient (R^2) obtained from regression equation was 0.958.

3.2. Artificial neural network

In this study, the best prediction, based on minimum error, was obtained by ANN with one hidden layer. The suitable number of neurons in the hidden layer was determined by changing the number of neurons. The good prediction and minimum error value were obtained with four neurons in the hidden layer. The weights and bias of ANN for CA of

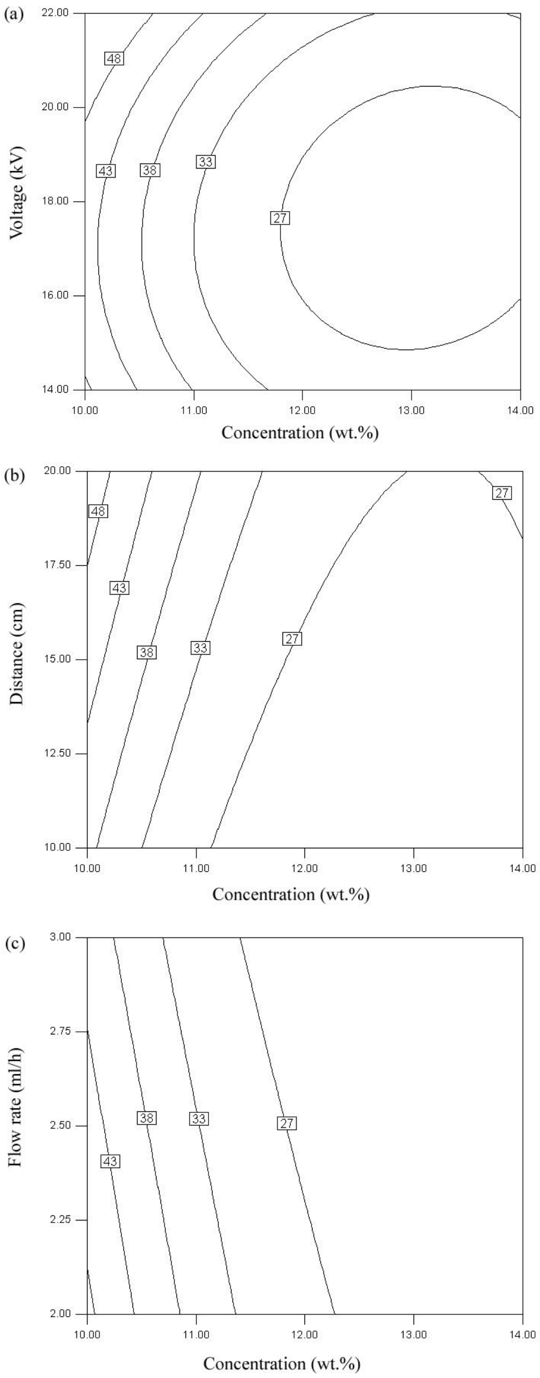

Fig. 5. Contour plots for contact angle of electrospun fiber mat showing the effect of: (a) solution concentration and applied voltage, (b) solution concentration and spinning distance, (c) solution concentration and volume flow rate.

electrospun fiber mat are given in Table 4. The R^2 and mean absolute percentage error were 0.965 and 5.94% respectively, which indicates that the model was shows good fitting with experimental data.

3.3. Effects of significant parameters on response

The morphology and structure of electrospun fiber mat, such as the nanoscale fibers and interfibrillar distance, increases the surface roughness as well as the fraction of contact area of droplet with the air trapped between fibers. It is proved that the CA decrease with increasing the fiber diameter [26], therefore the thinner fibers, due to their high surface roughness, have higher CA than the thicker fibers. Hence, we used this fact for comparing CA of electrospun fiber mat. The interaction contour plot for CA of electrospun PAN fiber mat are shown in Figure 5.

As mentioned in the literature, a minimum solution concentration is required to obtain uniform fibers from electrospinning. Below this concentration, polymer chain entanglements are insufficient and a mixture of beads and fibers is obtained. On the other hand, the higher solution concentration would have more polymer chain entanglements and less chain mobility. This causes the hard jet extension and disruption during electrospinning process and producing thicker fibers [27]. Figure 5 (a) show the effect of solution concentration and applied voltage at middle level of distance (15 cm) and flow rate (2.5 ml/h) on CA of electrospun fiber mat. It is obvious that at any given voltage, the CA of electrospun fiber mat decrease with increasing the solution concentration.

Figure 5 (b) shows the response contour plot of interaction between solution concentration and spinning distance at fixed voltage (18 kV) and flow rate (2.5 ml/h). Increasing the spinning distance causes the CA of electrospun fiber mat to increase. Because of the longer spinning distance could give more time for the solvent to evaporate, increasing the spinning distance will decrease the nanofiber diameter and increase the CA of electrospun fiber mat [28,29]. As demonstrated in Figure 5 (b), low solution concentration cause the increase in CA of electrospun fiber mat at large spinning distance.

The response contour plot in Figure 5 (c) represented the CA of electrospun fiber mat at different solution concentration and volume flow rate. Ideally, the volume flow rate must be compatible with the amount of solution removed from the tip of the needle. At low volume flow rates, solvent would have sufficient time to evaporate and thinner fibers were produced, but at high volume flow rate, excess amount of solution fed to the tip of needle and thicker fibers were resulted [28-30]. Therefore the CA of electrospun fiber mat will be decreased.

As shown by Equation (4), the relative importance (RI) of the various input variables on the output variable can be determined using ANN weight matrix [31].

$$RI_j = \frac{\sum_{m=1}^{N_h}((|IW_{jm}| / \sum_{k=1}^{N_i}|IW_{km}|)\times|LW_{mn}|)}{\sum_{k=1}^{N_i}\left\{\sum_{m=1}^{N_h}((|IW_{km}| / \sum_{k=1}^{N_i}|IW_{km}|)\times|LW_{mn}|)\right\}}\times 100 \quad (4)$$

where RI_j is the relative importance of the jth input variable on the output variable, N_i and N_h are the number of input variables and neurons in hidden layer, respectively (N_i =4, N_h =4 in this study), IW and LW are the connection weights, and subscript “n” refer to output response (n=1) [31]. The relative importance of electrospinning parameters on the value of CA calculated by Equation (4) and is shown in Figure 6. It can be seen seen that, all of the input variables have considerable effects on the CA of electrospun fiber mat. Nevertheless, the solution concentration with relative importance of 49.69% is found to be most important factor affecting the CA of electrospun nanofibers. These results are in close agreement with those obtained with RSM.

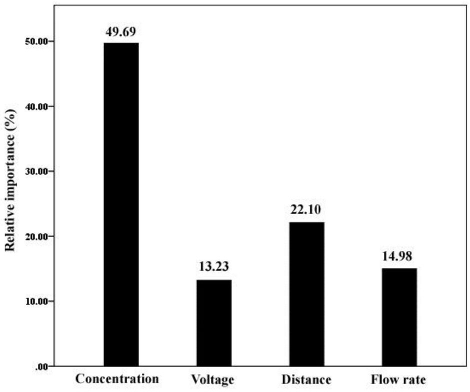

Fig. 6. Relative importance of electrospinning parameters on the CA of electrospun fiber mat.

3.4. Optimizing the CA of electrospun fiber mat

The optimal values of the electrospinning parameters were established from the quadratic form of the RSM. Independent variables (solution concentration, applied voltage, spinning distance, and volume flow rate) were set in range and dependent variable (CA) was fixed at minimum. The optimal conditions in the tested range for minimum CA of electrospun fiber mat are shown in Table 5. This optimum condition was a predicted value, thus to confirm the predictive ability of the RSM model for response, a further electrospinning and CA measurement was carried out according to the optimized conditions and the agreement between predicted and measured responses was verified. Figure 7 shows the SEM, average fiber diameter distribution and corresponding CA image of electrospun fiber mat prepared at optimized conditions.

3.5. Comparison between RSM and ANN model

Table 6 gives the experimental and predicted values for the CA of electrospun fiber mat obtained from RSM as well as ANN model. It is demonstrated that both models performed well and a good determination coefficient was obtained for both RSM and ANN. However, the ANN model shows higher determination coefficient (R^2=0.965) than the RSM model (R^2=0.958). Moreover, the absolute percentage error in the ANN prediction of CA was found to be around 5.94%, while for the RSM model, it was around 7.83%. Therefore, it can be suggested that the ANN model shows more accurately result than the RSM model. The plot of actual and predicted CA of electrospun fiber mat for RSM and ANN is shown in Figure 8.

4. CONCLUSIONS

The morphology and properties of electrospun nanofibers depends on many processing parameters. In this work, the effects of four electrospinning parameters namely; solution

Table 5. Optimum values of the process parameters for minimum CA of electrospun fiber mat

Solution concentration (wt.%)	Applied voltage (kV)	Spinning distance (cm)	Volume flow rate (ml/h)	Predicted CA (°)	Observed CA (°)
13.2	16.5	10.6	2.5	20	21

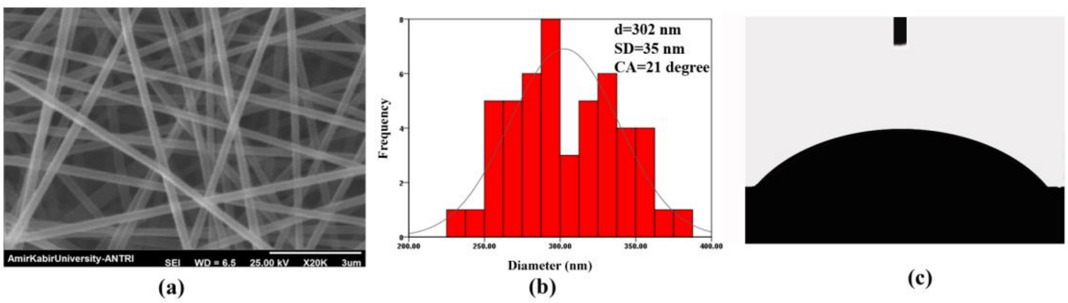

Fig. 7. (a) SEM image, (b) fiber diameter distribution, and (c) CA of electrospun fiber mat prepared at optimized conditions.

Table 6. Experimental and predicted values by RSM and ANN models

No.	Experimental	Predicted		Absolute error (%)	
		RSM	ANN	RSM	ANN
1	44	47	48	6.41	9.97
2	54	54	54	0.78	0.46
3	61	59	61	3.70	0.42
4	65	66	61	2.06	6.06
5	38	39	38	2.37	0.54
6	49	47	49	5.10	0.68
7	51	51	51	0.35	0.45
8	56	58	56	4.32	0.17
9	48	45	60	6.17	24.37
10	30	31	27	4.93	9.35
11	35	36	31	2.34	11.15
12	22	22	21	1.18	4.15
13	30	30	32	1.33	6.04
14	33	28	33	13.94	0.60
15	25	24	25	5.04	0.87
16	26	26	26	0.27	1.33
17	29	26	26	10.10	9.16
18	28	26	26	6.89	5.91
19	25	26	26	4.28	5.38
20	24	26	26	8.63	9.77
21	21	26	26	24.14	25.45
22	31	30	31	2.26	0.57
23	35	31	35	10.34	0.66
24	33	36	32	8.18	2.18
25	37	37	37	0.59	0.34
26	19	29	21	52.11	10.23
27	28	30	30	7.07	8.20
28	39	34	31	12.05	21.30
29	36	35	36	1.72	0.04
30	20	25	20	26.30	2.27
R^2		0.958	0.965		
Mean absolute error (%)				7.83	5.94

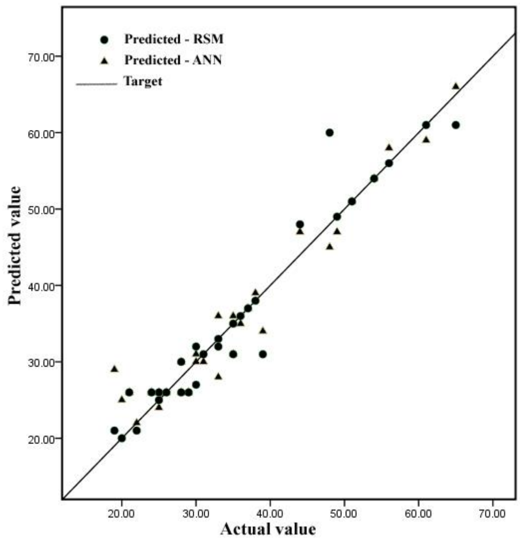

Fig. 8. Comparison between the actual and predicted contact angle of electrospun nanofiber for RSM and ANN model

concentration (wt.%), applied voltage (kV), tip to collector distance (cm), and volume flow rate (ml/h) on CA of PAN nanofiber mat were investigated using two different quantitative models, comprising RSM and ANN. The RSM model confirmed that solution concentration was the most significant parameter in the CA of electrospun fiber mat. Comparison of predicted CA using RSM and ANN were also studied. The obtained results indicated that both RSM and ANN model shows a very good relationship between the experimental and predicted CA values. The ANN model shows higher determination coefficient (R^2=0.965) than the RSM model. Moreover, the absolute percentage error of prediction for the ANN model was much lower than that for RSM model, indicating that ANN model had higher modeling performance than RSM model. The minimum CA of electrospun fiber mat estimated by RSM equation obtained at conditions of 13.2 wt.% solution concentration, 16.5 kV of the applied voltage, 10.6 cm of tip to collector distance, and 2.5 ml/h of volume flow rate.

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ОТНОСНО КОНТАКТИЯ ЪГЪЛ С ПОДЛОЖКАТА НА ЕЛЕКТРОПРЕДЕНИ ВЛАКНА ОТ ПОЛИАКРИЛОНИТРИЛ

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(Резюме)

Изследван е ефектът на четири параметъра на електропреденето: концентрация на разтвора, приложено напрежение, разстояние между дюзата и приемника и обемния дебит върху контактния ъгъл (CA) с подложката на нановлакна от полиакрилонитрил. Приложени са методология на повърхността на отклика (RSM) и изкуствена невронна мрежа (ANN) за оптимизирането и предсказването на контактия ъгъл с подложката на електропредени влакна от полиакрилонитрил. Намерени са количествени зависимости между контактния ъгъл и работните параметри. Намерено е, че концентрацията на разтвора най-важния фактор за контактния ъгъл с подложката. Получените резултати показват, че и двата модела са много ефективни при оценяване на контактния ъгъл. По-точни са резултатите, получени с изкуствена невронна мрежа. При този модел коефициентът на корелация (R^2) и процентната грешка между действителния и предсказания отговор са съответно 0,965 и 1,97.