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ORIGINAL ARTICLE

Experimental Investigations on Electrospun Mat Production: For Use in High-Performance Air Filters

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ABSTRACT
Electrospun nanofibrous filter media have attracted considerable attention in the last decade. The present study aimed to develop the electrospun PAN (polyacrylonitrile) filter media through experimental investigations for application in high-performance air filters. For this purpose, an experimental design was proposed to assess the effect of electrospinning process conditions including solution concentration, electric voltage and nozzle-collector distance on the structural properties of filter media including the fiber diameter, percent of porosity and bead number. Optimization of electrospinning parameters was conducted through the response surface methodology (RSM) to obtain the desired values for fibrous media variables. The morphology of the mats (including bead number and fiber diameter) were studied using SEM images through, Microstructure Measurement image analyzer. The porosity was determined using image analysis algorithms by MATLAB. The findings indicated that the concentration is the most influencing factor on fiber diameter (r= 0.73, P<0.05) and bead number (r=-0.51, P>0.05), so that the lower concentrations led to lower fiber diameter and more bead number. Among the electrospinning parameters, the highest correlation coefficient was achieved between porosity of PAN media and applied voltage (r=0.39, P>0.05). There was a negative relationship between fiber diameter and both percent of porosity (r=-23; P>0.05) and bead number(r=-0.53; P<0.05). Thus, media with the lower fiber diameter had the higher porosity and more bead number. Since the fibers diameter, bead number and porosity can have different effects on the quality factor of filters, the well-considered selection of electrospinning conditions can be of great importance for obtaining the arbitrary values of filter characteristics.

KEYWORDS: *Electrospinning, Polyacrylonitrile, Nanofiber, Filtration*

INTRODUCTION
As higher air qualities are becoming more concerned, high-efficiency filtration is becoming more important. Effective filter media are increasingly needed in industries applying clean-air technologies. Nowadays, the necessity for, fibrous filters (especially nonwoven ones) are developing the high-performance air filters has been more and more felt [1]. Among the existing filters for removing fine particle from gas stream commonly used [2]. They are economical and easy to operate and they have high collection efficiency with relatively low pressure drop [3].

The structure of nonwoven filter media has a significant role in their performance. The overall

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collection efficiency of the filter is expressed as follows [1]: $\eta = 1\text{-}\exp[(-4\alpha E L)/(\pi d_F (1-\alpha))]$, where α filter solidity (dimensionless), E single fiber collection efficiency (dimensionless), L filter thickness (m) and d_F fiber diameter (m). This formula indicates the performance of the filter strongly depends on the fiber diameter and the filter porosity (1−α) [1]. The high collection efficiency is achieved by application of low fiber diameter, high packing density (filter solidity) and low porosity. Meantime, all of them can increase resistance to flow leading to high-pressure drop across the filter media. In practice, the high collection efficiency and low air resistance are the two main features of an effective filter. Hence, determination of the optimum balance between these two parameters is a major concern. The filter quality factor (the figure of merit), QF, as an indicator of filter media performance, is the fraction of collection efficiency and the pressure drop across the filter; and it can be regarded as a benefit-to-cost ratio as follows: $QF= -\ln(1-\eta)/\Delta P = 4\alpha\eta L/\pi d_F \Delta P$, where, the normalized filtration efficiency $-\ln(1-\eta)$ as the benefit ,and pressure drop ΔP as the cost [4-5]

There are different ways to increase the filter performance such as pleating the filter surface that provides higher collection efficiency and lower pressure drops through increased area [6], and using the nanofibers, which exhibit high performance in the capture of fine particles [7].

Nanofibrous filter media that are mostly fabricated via electrospinning technique have attracted considerable attention in the last decade. Nanofibrous mat and its potential use as filter media for removing fine particles started, particularly from Barhate and Ramakrishna [8]. After that, some of experimental projects have been resulted in commercial products and some are under developing. Electrospun nanofibrous media provide high dust-holding capacity, small pore structures, high specific surface area, and low basis weight [9]. Most Penetrating Particle Size (MPPS) of the electrospun membrane can be significantly reduced [10]. The quality factor (Q_F) of microfiber filter can be improved by covering it with a layer of nanofibers [11].

Electrospinning process utilizes a strong electric field between a needle tip of a syringe containing a polymer solution and a collector plate for fibers. As the polymer solution is exiting the needle tip, it is affected by electric filed and it is drawn toward a grounded collector. In the meantime, solvent is evaporated and the solidified jet is turning into the nanofibers [12]. Morphological properties of electrospun nanofibers can be strongly affected by electrospinning operational parameters (including electric field strength and distance between tip and collector), and polymer solution properties (including concentration and viscosity) [12]. Many researches have been conducted to understand the effects of electrospinning parameters on the morphology of nanofibers, while dealing with different materials for different application. A review article by Subbiah et al. revealed many details about these features [13].

Among the many organic polymers that can be electrospun, polyacrylonitrile (PAN) as a versatile polymer for its good physical characteristics, reasonable price and ease of electrospinning has a potential application in different science fields. PAN, as an activated carbon nanofiber precursor, has also attracted attention of many air purification researchers [14].

The objective of the present study was to develop the electrospun nanofibrous filter media through experimental investigations for application in high-performance air filters. Hence, we aimed to 1) Fabricate PAN nanofibers by electrospinning, 2) To optimize the electrospinning parameters for achieving the desired properties of filter media including fiber diameter, and porosity, 3) Investigate the relationship between the electrospinning parameters and the features of filter media, 4) Provide mathematical model for the variables, and 5) Analyze the direct and interaction effects of electrospinning parameters on the filter properties.

MATERIALS AND METHODS

An experimental design was proposed using Design-Expert software (Version 7, Stat-Ease: Minneapolis). The effect of electrospinning process conditions including solution concentration, electric voltage and nozzle-collector distance on the structural properties of fabricated fibers including the fiber diameter, porosity and bead number were studied. Polyacrylonitrile (PAN) polymer (Mw: 80,000) was purchased from Polyacryle Co. (Iran), and 99% N-N, dimethyl formamide (DMF) solvent was obtained from Merck Co. (Germany). Electrospinning solutions in a concentration range of 8-16 wt % of PAN dissolved in DMF were prepared. Each solution was stirred by an electromagnetically driven magnet at room temperature for 12–24 h to reach the enough homogenization. In total, 15 experimental runs were conducted according to the study design (Table 1) and all the trials were performed via electrospinning process under the mentioned conditions as follow: applied voltages: 10 - 20 kV, nozzle–collector distance: 10-15 cm, temperature: 30°C, flow rate: 1ml/h, collector: covered with aluminum foil, syringe 5 ml, and needle diameter: 1.2 mm. The boundary values for electrospinning parameters were determined through pilot studies to form continuous fibers and based on the operating conditions of the electrospinning set-up.

A scanning electron microscope (SEM,

Hitachi S 4160) was used for morphology studies (like bead number) of the fibers. The mean fiber diameter was measured by an image analyzer (Microstructure Measurement, Ferdowsy University) through 50 measurements of random fibers.

Table 1. Design of experiment

Experiment Standard No	Concentration (wt.%)	Applied Voltage (kV)	Tip to collector distance (cm)
STD1	16.0	20	10.0
STD2	16.0	10	15.0
STD3	8.0	20	15.0
STD4	8.0	10	10.0
STD5	9.6	15	12.5
STD6	14.4	15	12.5
STD7	12.0	12	12.5
STD8	12.0	18	12.5
STD9	12.0	15	11.0
STD10	12.0	15	14.0
STD11	12.0	15	12.5
STD12	12.0	15	12.5
STD13	12.0	15	12.5
STD14	12.0	15	12.5
STD15	12.0	15	12.5

The porosity was determined using image analysis algorithms by MATLAB (Math Works, Version7) based on global thresholding method.

According to the global thresholding, the binary images were created and a single constant threshold was used to segment the images (Figure 1). All pixels up to and equal to the threshold were considered the object and the remaining ones were belonged to the background. Percent of porosity was determined as below:

$$P = (1 - \frac{n}{N}) \times 100 \quad \textit{(Equation. 1)}$$

Where, n is number of white pixels, N is total number of pixels in image and P is percent of porosity. The porosity of thin layer of nanofiber webs (thickness<100µm) was calculated based on image analysis algorithms through conversion of SEM image with the original magnification of 10000 times to binary images. However, this method can provide a two-dimensional (2D) porosity instead of total porosity, and many studies have introduced the image analysis as a reliable technique for characterization of porosity [15-16].

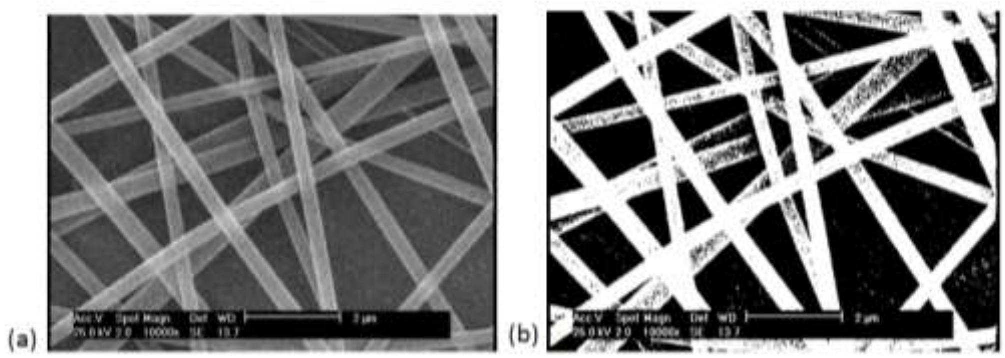


Fig.1. (a) SEM image of an electrospun nanofiber web, (b) Binary image of the web.

Optimization of electrospinning parameters was determined through the response surface methodology (RSM) based on central composite design (CCD) to obtain the desired values for filter variables. RSM is a combination of mathematical and statistical techniques for evaluating the relative significance of factors affecting the output variable with a limited number of planned experiments [17].

RESULTS

The Table 2 is tabulated the results of the filter attributes including average fiber diameter, porosity of media and the number of beads formed on fibers. SEM images obtained from 15 trial runs are shown in Figure 2.

The results reveal that the best morphological properties of fibers (the formation of bead-free and uniform fibers) are obtained in STD 1 and STD 2. For these STDs, the maximum average fiber diameter is also achieved. A very poor formation of fiber array is obtained in STD 5 and STDs 8-10. In STD 5, beads are the main product; thereby it seems that electrospinning has become electrospray. If electrospinning solution has low viscosity, jet will be broken to fine charged particles which are deposited on the collector; it is known as electrospray [18].

The results of the present study indicate that the maximum porosity occurs in STD 1, if STD 5 in which beads are the main product is ignored.

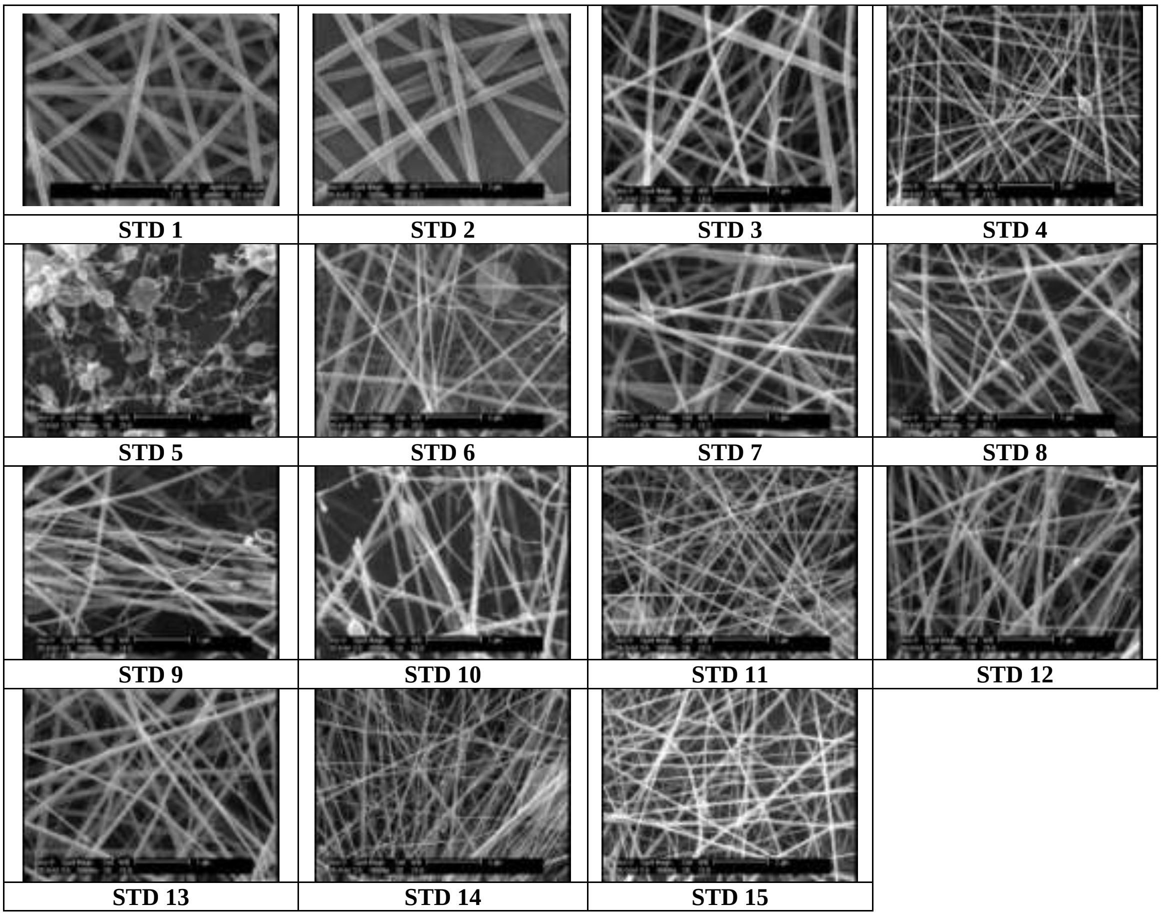


Fig.2. SEM images in 15 trial runs

Table 2. Average fiber diameter (nm), porosity percent of mat and bead number

STD No.	Response variables Average fiber diameter (nm)	porosity of media (%)	Bead (625x magnification)
1	154.99	51.28	2
2	141.77	48.65	7
3	77.3	43.70	250
4	90.36	36.55	420
5	71.6	51.59	>1000
6	131.82	38.96	255
7	105.66	35.01	300
8	63.33	51.13	510
9	116.82	33.65	450
10	68.61	49.34	450
11	82.59	48.98	390
12	104.58	46.29	380
13	83.63	48.98	400
14	103.58	45.29	390
15	85.23	48.98	400

The relationship coefficients between electrospinning parameters and filter variables are presented in Table 3. Solution concentration has the highest correlation with the filter variables including fiber diameter (r=0.73) and bead number (r= -0.51) compared to other independent variables; however, only the relationship between concentration and fiber diameter was statistically significant (P <0.05). As can be seen, the lower concentration gives lower fiber diameter, lower porosity and higher bead number. Applied voltage and electrospinning distance were inversely related to the fiber diameter and the bead number, and positively to porosity. The results revealed a weak negative correlation between average fiber diameter and percent of porosity (r=-0.23, P>0.05; Pearson correlation; IBM SPSS Statistics 22). There was a negative significant correlation between fiber diameter and bead number (r=-0.53, P<0.05).

Table 3. Correlation coefficient between electrospinning parameters and response variables

		Response variable		
		Diameter (nm)	**Porosity (%)**	**Bead no.**
Electrospinning parameters	Solution concentration (wt. %)	0.73*	0.24	–0.51
	Applied voltage(kV)	–0.11	0.39	–0.02
	Electrospinning distance (cm)	–0.25	0.28	–0.06
Response variable	Diameter (nm)	-	–0.23	–0.53*
	Porosity (%)	–0.23	-	–0.27
	Bead number.	–0.53*	–0.27	–

* Correlation is significant at the 0.05 level

The interaction effects of electrospinning variables on filter properties were investigated through the models suggested by ANOVA analysis (Table 4). Insignificant terms which are not included in the models are aliased according to the suggestion of DX software. Model fitting was revealed that a quadratic model for fiber diameter and bead number and a 2FI (two factor interaction) model for porosity gave the best fit, and they were found to have insignificant lack of fit. The obtained data suggest that all of the independent variables including concentration, applied voltage and spinning distance (showing as A, B and C in Table 4, respectively) as well as some of their interactions including AB and AC have a statistically significant effect on average fiber diameter (P <0.05).

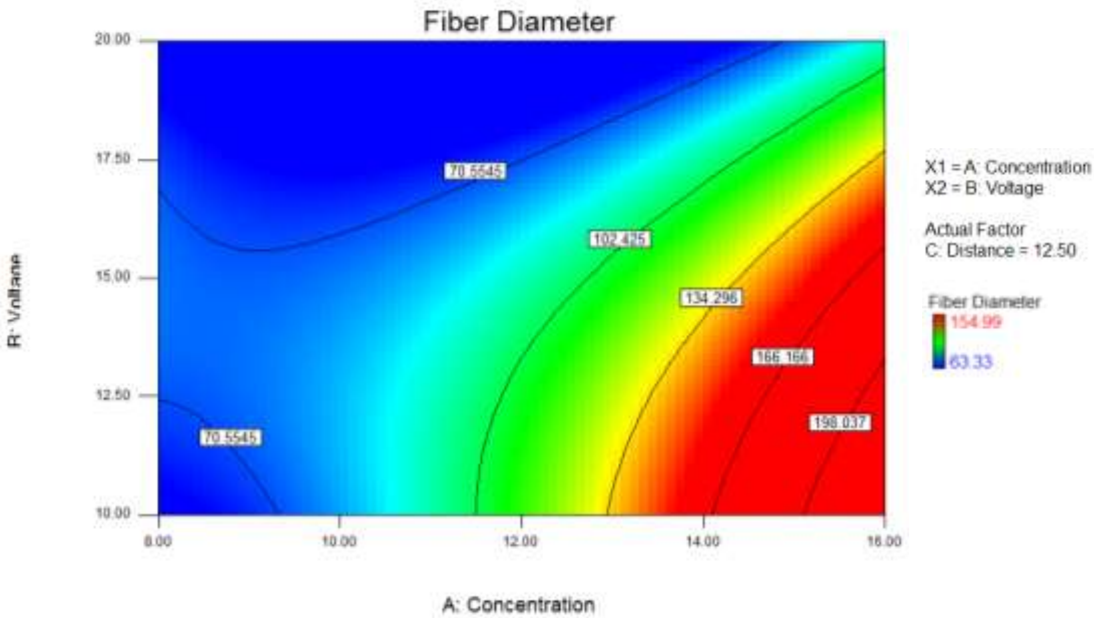
Table 4. Response surface quadratic model of response variables

Response variable	Model	Equation
Diameter (nm)	Quadratic	Diameter= +30.58 +32.98 A^{*} + 13.45 B^{**} - 25.66 C^{***}- 1.68 AB – 3.53 AC
Porosity (%)	2FI	Porosity= +11.7 -25.32 A + 10.96 B + 10.57 C+ 0.59 AB + 1.09 AC – 1.23 BC
Bead number.	Quadratic	Bead number = -16554 -1654.9 A– 69.78 BC+45.47A^2

* *A-Concentration*
** *B-Voltage*
*** *C-Distance*

For the 2 FI model of porosity, all of the independent variables and their interactions including A, B, C, AB, AC, and BC were statistically significant (P <0.05). In quadratic model of bead number, there were only three significant terms including: A, BC, A^2.

Figure 3 presents the contour plots the interaction effects of solution concentration and applied voltage on the filter properties, while spinning distance is keeping fixed. The curvilinear profiles in Fig. 3 is in compliance with the 2 FI and quadratic models fitted. For example, the porosity in higher concentration will be become greater by in creasing the applied voltage.



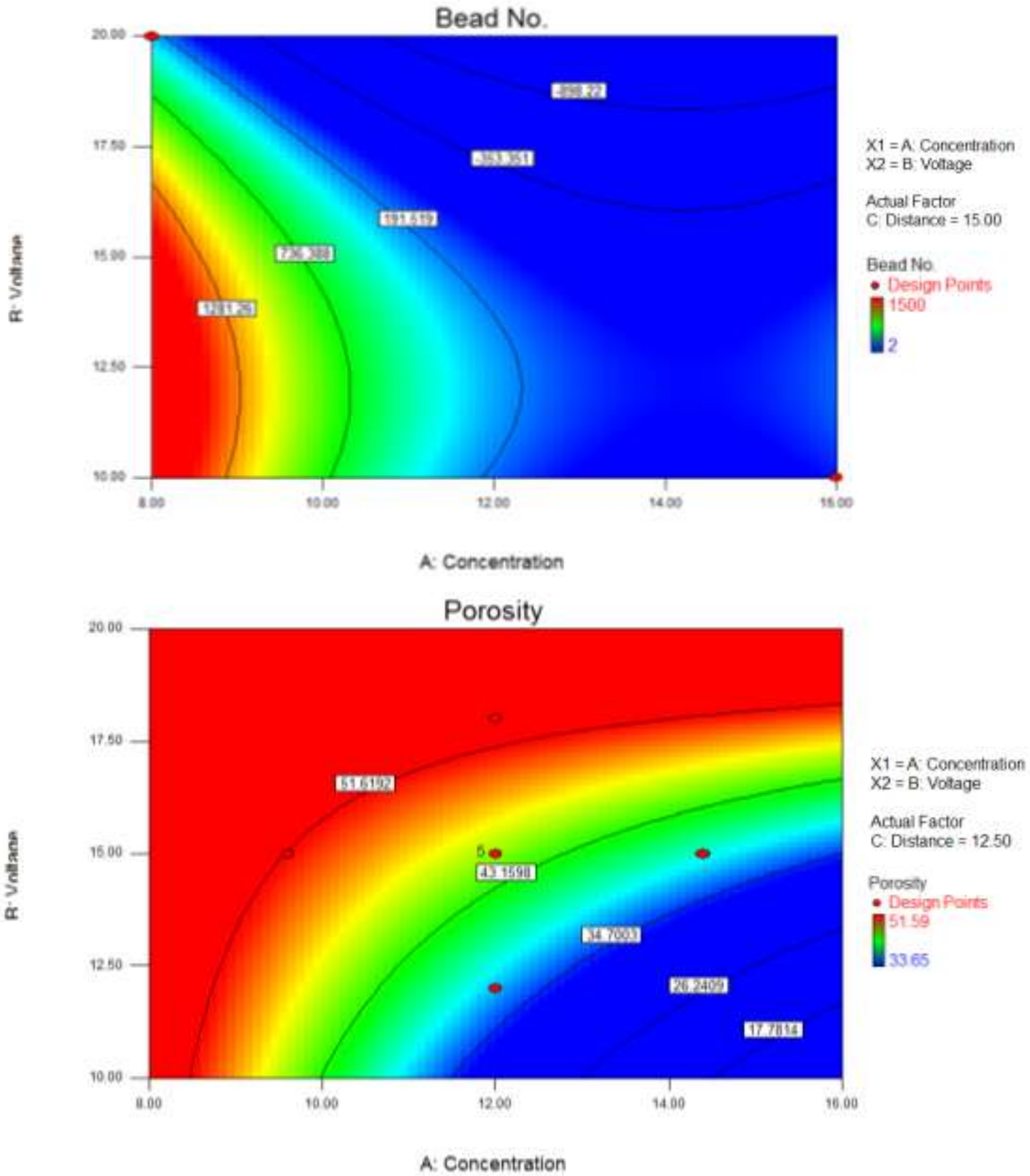


Fig.3. Contour plots of interaction effects of solution concentration and applied voltage on response variables

For obtaining the desired filter properties, the optimization of electrospinning was done using the DX (Table 5). Since the fibers diameter, bead number and porosity can have different effects on the quality factor of filters, in the optimization process, the response variables are assessed “in range”, instead of minimization or maximization. After the optimization, DX suggested some solutions of electrospinning parameters and the predicted values of response variables. The proposed solutions were validated by performing the new experimental runs (three runs) according to the suggested conditions and then by comparing the experimental data with the predicted data. The results indicated that the experimental values are in agreement with the predicted responses (percentage variation <1%).

Table 5. Some of optimum solutions

Constraints Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
Solution Concentration (wt. %)	is in range	8.00	16.00	1	1	3
Applied Voltage(kV)	is in range	10.00	20.00	1	1	3
Electrospinning distance (cm)	is in range	10.00	15.00	1	1	3
Diameter (nm)	is in range	63.35	154.99	1	1	3
Porosity (%)	is in range	33.65	51.59	1	1	3
Bead No.	is in range	2.00	1500.00	1	1	3

Solutions							
Number	**Concentration (wt. %)**	**Voltage (kV)**	**Distance (cm)**	**Diameter (nm)**	**Porosity (%)**	**Bead No.**	**Desirability**
1	8.40	10.20	10.03	95.63	34.36	280.70	1
2	14.42	16.19	11.38	146.5	36.54	100.58	1
3	12.99	14.02	13.54	92.84	47.24	248.98	1

DISCUSSION

According to Table 2 and Figure 1, the average fiber diameter in higher concentrations is much larger than that of fibers spun at lower concentrations. Morphological quality of fibers represented by diameter uniformity, absence of beads and defects and formation of continuous fibers without droplet breakup, was getting worse in lower concentrations as observed in STDs 3-5. The previous studies indicated that the formation of spindle-like beads will be increased in the lower PAN solution concentrations [19] and the higher average fiber diameter will be obtained at the higher concentrations [19], which is in consistency with the results. The relationship between electrospinning parameters and average fiber diameter of electrospun PAN nanofiber was studied earlier [12]. The authors observed the smaller fiber diameter in the lower solution concentration. In higher solution concentration, there are more polymer chain entanglements and less chain mobility that can result in the harder jet extension and the formation of the larger diameter fiber [12]. The bead formation is commonly occurred when polymer chain entanglement due to low solution concentration is insufficient. The higher concentration and so higher viscosity can decrease the formation of beads [20]. Beads are usually formed due to congregating of solvent molecules which at high concentrations, these molecules are distributed among entangled chains and their tendency for aggregation decreases [21].

Among understudy parameters, the highest correlation coefficient was found between concentration and fiber diameter and this relationship was statistically significant ($r=0.73$; $P<0.05$). As discussed before, the higher solution concentration would give the liquid a higher viscosity, so lower jet elongation and thinner fibers [22]. These findings are consistent with the results of many studies and indicate that polymer solution plays the most important role in determining the fiber diameter [12, 23-24]. In this study, a very weak negative relationship has been detected between fiber diameter and applied voltages. The higher applied voltage with constant distance between nozzle and collector can increase electric field strength, and higher electrostatic repulsive force on the solution jet, and in this way it can decrease the fiber diameter [12]. Increasing the tip to collector distance can give more time for the solvent to evaporate, which can result in the narrower fiber diameter [12]. A weak negative correlation between fiber diameter and electrospinning distance was also observed in this study.

The higher porosity of PAN media is achieved at the higher applied voltage ($r=0.39$), higher concentration ($r=0.24$) and larger distance ($r=0.28$). The increase of applied voltage and spinning distance when solution concentration is fixed at given value can be lead to the higher porosity [25]. The higher concentration at constant value of applied voltage and spinning distance will lead to the higher porosity and higher air permeability [26]. Since fiber diameter has a major role in forming the pore structure and porosity of media [27], these effects of electrospinning parameters on porosity would be justifiable.

Polymer concentration is directly related to its viscosity, and so it is considered the most effecting factor on bead number ($r=-0.51$). In lower concentration, the more bead number has been formed and in very low concentration, electrospray will be occurred [28]. The lower solution viscoelasticity and electric charge density and higher solution surface tension are the main factors affecting on the bead formation. The solution concentration is effective agent on the viscoelasticity, type of polymer, solvent are the effective factors on the surface tension and the applied voltage is effective factor on electric charge density [29].

Table 2 shows there is a negative relationship between fiber diameter and both percent of porosity ($r=-23$; $P>0.05$) and bead number($r=-0.53$; $P<0.05$), so the lower fiber diameter was observed in the media with the higher porosity and more bead number; these findings in line with the previous studies [28-30]. According to filtration theories, the porosity can be inferred from fiber diameter, so that the number of pores per area of media is inversely proportional to the quartic root of fiber diameter [30]. It has shown that the decrease in fiber diameter of poly L-lactide-co-ε-caprolactone can reduce the percent of porosity [30].

Interaction effects of the electrospinning conditions on the properties of PAN mat was assessed by ANOVA analysis. According to the quadratic model for fiber diameter, all of the independent variables and some of their interactions (AB and AC) have a statistically significant impact on average diameter (P <0.05). Gu and Ren (2005) performed the process optimization of Poly (d,l-lactide) fibers based on RSM and concluded that concentration, applied voltage, and their interaction had a significant impact on average fiber diameter (at a constant distance of 15 cm) [20]. In a quadratic model , the concentration (A), voltage (B) , distance (C) , AB, A^2 and B^2 are significant terms (P < 0.05) [12]. The higher concentration in constant values of applied voltage and distance may increase the percent of porosity and filter air permeability [26]. The observed differences can be due to the different polymer properties (polymer rheology and solvent thermodynamics properties), different process parameters (flow rate and collection plate) and ambient conditions (temperature and humidity) [20].

DX software has presented the different suggestions through numerical optimization to set the goals for each response (Table 4). The confirmation tests indicated that the experimental values are in agreement with the predicted responses. For air filtration purpose, those combinations which can give thinner fiber, higher morphology quality and higher porosity can be chosen. High performance filters have the high collection efficiency and low pressure drop. Higher porosity and larger fiber diameter can result in lower pressure drop, and also can negatively effect on the collection efficiency. Therefore, determination of the optimum points among the effecting parameters is so important.

CONCLUSION

The present study aimed to develop the electrospun PAN nanofibrous filter media through experimental investigations for application in high-performance air filters. According to these findings, the solution concentration and the applied voltage had the main role in fiber diameter and porosity, respectively. High correlation coefficient between the input and output variables revealed the worthy evaluation of experimental data by polynomial regression model. RSM could satisfyingly determine the relationship between the electrospinning variable and the filter properties. The good agreement between the experimental values and the predicted ones was examined by the confirmation experiment. Since the fibers diameter, bead number and porosity can have different effects on the quality factor of filters, the well-considered selection of electrospinning conditions can be of great importance for obtaining the arbitrary values of filter characteristics. It is hoped that the results of the presented experimental model in this work, will be useful guidance to develop the nanofibrous mats for application in high-performance air filters.

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