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Fabrication of electrospun membranes for air sampling applications: A statistical optimization approach

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ABSTRACT

Introduction: The applicability of Nanofiber (NF) membranes in air sampling of pollutants for the purpose of determining the airborne concentration has received little attention around the world. The present study aims to optimize the fabrication of NF membrane for the of air sampling application.

Materials and methods: The polyvinyl chloride NF membranes were fabricated using needle-based solution electrospinning technique. The experimental design was prepared by Design-Expert v7.0 and data analysis was done by Central Composite Design (CCD) base on Response Surface Methodology (RSM) technique. The ability of the fabricated membranes in air sampling applications was performed by sampling of airborne crystalline silica by them using the National Institute for Occupational Safety and Health (NIOSH7602) method and then comparing with the commercial PVC membranes.

Results: The fabricated NF membranes had a mean porosity of 31.60% compared to a porosity of 25.1% in the case of commercial Polyvinyl Chloride (PVC) membranes. The electrospun NF membranes had mean pressure drop of 194.23 Pa, which is lower than the 204 Pa pressure drop of commercial PVC filters. The mean concentration of silica sampled by the electrospun NF membrane was 0.14 mg/m^3 while this was 0.03 mg/m^3 for commercial PVC membrane. The difference concentration of crystalline silica sampled by NF and commercial PVC membranes had the strongest relationship with the electrospinning solution concentration (r=-0.785, p>0.05).

Conclusion: The NF membrane has high performance in sampling the crystalline silica dust from the air stream compared to commercial PVC membranes.

Introduction

The production of suitable membrane substrates for use in the filtration and sampling of airborne contaminants is one of the scientific fields currently being expanded [1]. The Nanofiber (NF) membrane has high performance in separating particles and their structural properties can easily be controlled via the electrospinning process in order to improve their collection of

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airborne contaminants [2]. Among the common methods used in NF membrane production, electrospinning is quickly advancing and due to its ability to create contiguous membranes with a diameter smaller than 100 nm [3]. The electrospun NF membrane has a high specific surface area, excellent membrane diameter, high porosity, small pore size and high air filtration performance [4]. Air filtration using NF membranes have caught the attention of researchers in recent years due to their large collection surface and low resistance against the movement of air [5]. The release of nanomaterials from uniform electrospun NF filters was low [6].

The sampling and analysis of airborne pollutants are undoubtedly the first step in identifying their health effects, and have always been considered one of the main principles involved in controlling their health risk [7]. One of the common methods available for the sampling of airborne particles is the filtration [8]. The main attributes of the air sampling membrane filter include its pore size, cross-section diameter and the type of material used. The physical and chemical characteristics of the particles, the maximum allowed contaminant load on the membrane, air temperature requirement during sampling, air humidity, pressure drop, method of analysis and costs are among the influential parameters in selecting a suitable membrane [9].

Crystalline silica is the most commonly found mineral on the earth's crust and can be either amorphous or crystalline in form [10]. Recent studies have shown that more than 2.3 million workers in the United States (US), more than 2 million workers in the European Union (EU) and more than 23 million workers in china have had exposure to crystalline silica in their work environment [11, 12]. In October 1996, the International Agency for Research on Cancer (IARC) categorized silica as a human carcinogen [13]. Workers occupied in industries involving casting, stone crushing, grinding, construction, tiles, glass and sandblasting are particularly at risk of exposure to silica dust with severe health implications [14-20]. Numerous studies have shown correlations between exposure to silica dust and pulmonary fibrosis, cancer and shortness of breath [21]. PVC membrane filters are currently being used as recommended filter by NIOSH for the air sampling of crystalline silica in the workplaces [22].

Today, electrospun NF membranes are being used for various filtration applications such as in air filters, respiratory masks and automobile filters [23]. Despite their high performance in collecting airborne particles and air purification, the applicability of NF membranes in the collecting and sampling of airborne contaminants for the purpose of determining the concentration of pollutants has received less attention around the world. Thus, in the present study, the fabrication of the NF membrane via electrospinning and the optimizations of the electrospinning parameters for application of air sampling of crystalline silica was carried out. Moreover, the obtained results for air sampling of crystalline silica have been compared to ones of commercial PVC membrane filters. In the present study, influential electrospinning parameters such as polymer concentration, applied voltage, electrospinning duration time and needle-collector distance were optimized. The purpose of optimization is to achieve membranes with desired porosity and diameter or membranes with suitable morphology such as uniform distribution of fiber, reduced beaded, lower pressure drop and better collection of airborne particles.

Materials and methods

Materials

The PVC-S6532 polymer (Arvand Petrochemical Co, Iran, MW=62.50 g/mol and a density of 1.3-1.45 g/cm^3) was purchased to prepare electrospinning solution. The Tetrahydrofuran (THF) (MERCK Co, Germany, MW=72.11g/mol, purity=99.8%) and Dimethylformamide (DMF) (MERCK Co, Germany, MW=73.09g/

mol, purity>99.8%) were used as solvents at a 1:1 ratio. KBr powder code 104905 (MERCK Co, Germany, MW=119.01g/mol, purity>99.5%) was also used in the making of the pellet needed for the FT-IR device. The PVC membrane filter (Pore size 5 µm, 25 mm -SKC, Inc.; United States) was obtained to be compared with the results of air sampling of the electrospun NF webs.

Measurement method

This experimental study was conducted in 8 phases. The experimental design was made using the Response Surface Methodology (RSM) based on the Central Composite Design (CCD). The Polyvinyl Chloride (PVC) NFs were fabricated using the needle-based solution electrospinning technique. Structural features (fiber diameter, porosity) and performance features (concentration of airborne silica, pressure drop) were also determined. Identifying optimized conditions along with validation experiments were also conducted.

Determining the boundary values of input variables

Pilot studies were done and the related research papers were reviewed in order to determine the range of values for each of the electrospinning parameters such as polymer concentration, applied voltage, electrospinning duration and the needle-collector distance. The aim was to fabricate continuous fibers without them breaking up into droplets and the suitable level of mechanical strength of membrane. The remaining electrospinning parameters such as the solvent ratio and rate of injection were chosen by considering the results of pilot studies and they were also selected based on the results of previous researchers [24, 25].

Experimental design

The experimental design was devised with Design Expert software (version 7; DX7; Stat-Ease, Minneapolis, MN, USA) using the Response Surface Methodology (RSM) and based on the Central Composite Design (CCD). Input (independent) variables included electrospinning polymer concentration (11-13 wt%), applied voltage (15-20 kV), needle-collector distance (12-15 cm) and electrospinning duration (2-4 h). Output (dependent) variables included morphological characteristics (fiber diameter, membrane porosity) and performance characteristics (concentration of airborne silica, pressure drop). A total of 30 experimental runs were suggested by the software for the optimization of the electrospinning parameters used for PVC NF membrane fabrication (Table 1).

Preparing the electrospinning solution

Polyvinylchloride (PVC) polymer, N solvent, Dimethylformamide (DMF) and Tetrahydrofuran (THF) were used to prepare the electrospinning solution. The PVC/DMF/THF solutions was prepared at different concentrations (11, 12 and 13 wt%). In order to achieve homogeneity, the solution was heat stirred at room temperature for 3-6 hours and the PVC solution was dissolved in the THF: DMF solvent at a 1:1 ratio [26].

NF fabrication

The design of the present study is presented in Table 1. PVC NF membranes were fabricated using an ES2000 electrospinning machine (Fanavaran Nano-Meghyas Co., Iran) per the following conditions: a temperature of 25 °C, a collection plate covered with spun-bonded polypropylene substrate, an injection rate of 2 mm/h, a syringe size of 5 mm, an 18-gauge needle (internal diameter of 0.84 mm), a nozzle scanning rate of 300 mm/minute and a drum rotation speed of 700 rpm.

Measurement location

The casting section of an auto parts factory was selected as the air sampling site. The types of materials used in this section included crystalline

Table 1. Study design

Run	Duration (h)	Concentration (wt%)	Distance (cm)	Voltage (kV)
1	2	13	15	15
2	2	12	12	20
3	4	13	12	15
4	2	12	12	20
5	2	13	15	20
6	4	12	13.5	17.5
7	4	12	15	15
8	2	12	13.5	17.5
9	2	12	12	17.5
10	4	12	15	15
11	3	12	13.5	20
12	2	11	12	20
13	3	13	15	15
14	4	13	13.5	17.5
15	3	12	13.5	15
16	4	11	13.5	17.5
17	3	11	13.5	17.5
18	3	12	12	20
19	3	12	13.5	17.5
20	4	13	15	15
21	4	13	15	15
22	3	11	12	20
23	4	13	15	20
24	3	11	13.5	17.5
25	2	11	13.5	20
26	3	12	15	17.5
27	3	11	12	17.5
28	3	11	13.5	17.5
29	2	13	12	15
30	3	11	13.5	17.5

silica, foundry sand, iron alloys, scrap metal and brittle materials. Measurement data related to the annual assessment of workplace pollutants in the industry and the opinion of experts present, showed the difference of pollution level in different parts of the factory in the casting section. Therefore, according to this issue, a location in the sand casting section with medium pollution level (DISAMATIC molding machines (DISA)) comparing to others, was selected for air sampling. DISA use foundry molding horizontal equipment [27].The presence of silica at the selected point was previously confirmed by a pilot study. Air samples for the pilot study were taken in compliance with the National Institute for Occupational Safety and Health (NIOSH) 7602 method [22]. All measurement for each experimental run were done at the same time. The Cast Iron section consists of three main parts including the Smelting part which is responsible for preparing food for Badische Maschinenfabrik Durlach (BMD) and DISA parts which produce various pieces through the Badische Maschinenfabrik Durlach (BMD) machine and DISA machines , respectively, and Finishing work part in which pieces are inspected [28].

Air sampling and analysis

The air sampling of crystalline silica was carried out based on NIOSH 7602 method [22] at the casting unit of an automobile parts manufacturing plant. The air sampling was done simultaneously using both the fabricated NF membranes and commercial PVC NF membranes. The sampling pumps (SKC Air Check Sampler 224-44XR, USA) was calibrated with a rotameter (Platon-NG; Austria) using a soap bubble flow meter. The membranes were kept inside a desiccator for 24 h before air sampling and recording their initial weight. For air sampling, the membranes were placed inside a cyclone (SKC 225-01-02, USA/UK) and connected to the sampling pump. Sampling was done at a flow rate of 2.5 L/min for a fixed duration to obtain the sample volume of air (400-1000 L). Atmospheric parameters such as dry temperature (27.9 OC), relative humidity (~52%) and atmospheric pressure (88654.19 Pa) was measured in each sampling stage using a multipurpose anemometer (BENETE GM8910, China). After the sampling, the NF membranes were placed inside a desiccator for 24 h and then the secondary weight of the membranes was measured using a scale. Assessment of the control samples was conducted under the exact same conditions and methods as the main samples. Since this study is the first phase of a larger study, the results of the determination of accuracy and precision of measurement presented in a study by some researchers [29].

In order to prepare the samples, the membranes were placed inside a crucible and submerged under 200 mg of potassium bromide. Then, the samples were placed inside a muffle furnace (HORST UHLING KG – Type U 24/St, 220v, 1200 w) at a temperature of 600 °C for a duration of 2 hours. Once the sample had cooled, it was placed in a crucible and homogenized. The sample was then transferred to a 13 mm steel cast and pressed under 20 MPa pressure until a pellet is formed. This pellet is then placed inside the Fourier Transform Infrared Spectroscopy (FT-IR) device (Rayleigh WQF 510 A, China) and scanned at 710-825 nm. The surface area below the peak reading for crystalline silica was calculated using the provided FT-IR device software. In order to draw the calibration curve, standardized silica samples were made. The concentration of the collected silica was calculated for each sample using the calibration curve and the corresponding peak surface area. In order to estimate the Limit of Detection (LOD) and the Limit of Quantitation (LOQ) of the determination of silica, the PVC filter was analyzed. LOD of 0.049 μg and LOQ of 0.162 μg were obtained

Determining morphological and performance characteristics

The structural attributes of the NF membrane such as its morphology (fiber diameter, membrane porosity) was determined using

Table 2. The response variables in each experimental run

Run	Fiber diameter (nm)	Porosity (%)	Pressure drop (Pa)	Crystalline silica concentration from nanofiber membranes (mg/m^3)	Crystalline silica concentration from commercial PVC filters (mg/m^3)	Difference in crystalline silica concentration between NF membranes and commercial PVC
1	526	21.5	295	0.14	0.03	0.11
2	290	29.06	225	0.16	0.03	0.14
3	560	29/40	193	0.12	0.02	0.10
4	280	31.06	185	0.18	0.04	0.15
5	510	39.43	239	0.11	0.02	0.09
6	469	35.89	199	0.11	0.02	0.10
7	538	32.28	185	0.14	0.03	0.11
8	450	39.7	190	0.08	0.03	0.05
9	279	15.33	277	0.12	0.02	0.10
10	460	31.26	189	0.12	0.01	0.11
11	415	28.5	166	0.12	0.01	0.11
12	210	29.97	185	0.20	0.03	0.17
13	612	22.57	300	0.06	0.03	0.03
14	368	35.6	190	0.14	0.03	0.11
15	580	39.6	198	0.08	0.03	0.05
16	337	36.34	150	0.17	0.04	0.14
17	200	28.36	145	0.21	0.02	0.19
18	265	35.08	128	0.16	0.03	0.13
19	418	39.89	190	0.15	0.02	0.13
20	546	35.56	188	0.06	0.02	0.03
21	570	38.5	282	0.09	0.06	0.05
22	284	37.76	160	0.18	0.04	0.14
23	578	23.5	255	0.07	0.02	0.05
24	215	31.20	150	0.25	0.07	0.19
25	282	24.57	222	0.17	0.03	0.14
26	410	22.25	175	0.10	0.00	0.10
27	210	30.5	148	0.22	0.04	0.18
28	205	31.4	146	0.25	0.06	0.19
29	312	41.53	125	0.11	0.02	0.10
30	205	30.12	147	0.22	0.03	0.19

SEM photography. The SEM images were evaluated using SEM analysis algorithms and the Image J v1.5 (Wayne Rosband, National institutes of health, USA). Pressure drop tests were performed using an FT150EG filter testing device (Fanavaran Nano Meghyas Co, Iran) per the ISO29463 standard [30].

Statistical Analysis

The determining of optimum conditions for NF membrane fabrication and analysis of the independent and combined effects of the variables was carried out using the Design Expert. This is able to model and optimize the parameters by using statistical and mathematical approaches [5].

Determining Optimum Conditions

Design Expert is able to offer the optimum condition for the experimental variables to maximize or minimize the value of dependent ones. Validation of the proposed regressions equations was done by randomly selecting and performing three different runs from the list of proposed solutions.

Results and discussion

The resulting of response variables obtained for morphological and performance characteristics for each run is presented in Table 2.

Morphological characteristics

The mean diameter of the fabricated NF membranes was 386nm±136.57. The largest diameter achieved belonged to run 13 at 612 nm while the smallest diameter achieved belonged to run 17 at 200 nm. The mean porosity of the fabricated NF membranes was 31.6%. The highest porosity achieved was 41.53% which belonged to run 29 while the lowest porosity achieved was 15.33% and belonged to run 9. The main and interaction effect of the electrospinning parameters on morphological characteristics of PVC NF have been reported in published article of the authors [31]. Therefore, only some of the results will be given in the present study. Table 3 presents the correlation between electrospinning parameters and the morphological characteristics. Among the electrospinning parameters, the highest correlation coefficient was observed between fiber diameter and solution concentration which was statistically significant ($r=0.756$; $p=0.001$). A positive but weak correlation was observed between fiber diameter and needle-collector distance ($r=0.65$; $p=0.021$) and electrospinning duration ($r=0.42$; $p=0.001$). The fiber diameter and applied voltage was inversely related to each other ($r=-0.502$; $p=0.005$).

Table 3. Correlation coefficient between electrospinning parameters and morphological variables

Electrospinning parameters	Fiber diameter (nm)		Porosity (%)	
	r	p-value	r	p-value
Solution concentration (wt%)	0.75	0.001	0.05	0.794
Applied Voltage (kV)	-0.50	0.005	-0.10	0.597
Electrospinning Duration (h)	0.42	0.001	0.19	0.690
Needle-Collector Distance (cm)	0.65	0.021	-0.072	0.321

Numerous studies [32-34] have stated that solution concentration is the most influential factor that effects the diameter of the NFs. Some researchers optimized the electrospinning parameters in order to achieve desirable morphological characteristics in Polyacrylonitrile NFs. The results showed that the maximum solution concentration yielded the most suitable fibers in terms of morphology and maximum fiber diameter [35]. This can be due to the fact that higher solution concentration would have more polymer chain entanglements and less chain mobility, leading to harder jet extension and higher disruption during the electrospinning process and favoring the formation of a large-diameter fiber [5].

Increasing applied voltage has the reverse effect on the diameter of the NF membrane. The increased applied voltage can enhance the electric field strength, therefore creating a higher electrostatic repulsive force on the polymer jet and decreasing the fiber diameter [5].

NF diameter also increases as the electrospinning duration and needle-collector distance are increased. A researcher investigated the effect of solution concentration and needle-collector distance as well as the combined effect of solution concentration and applied voltage on fiber diameter. It was observed that at a constant applied voltage, increasing the solution concentration from 4% to 6% and 8% while increasing the needle-collector distance results in an increased NF diameter [36]. Increasing the needle-collector distance and also electrospinning duration (leading to increased thickness of NF layer) can decrease electric field strength, which can result in the larger fiber diameter [37].

The fabricated NF membranes had a mean porosity of 31.60% compared to the porosity of the commercial PVC membranes which was 25.1%. The highest porosity obtained from the fabricated NF membranes was 41.53%. A weak correlation was observed between the electrospinning parameters and the porosity of the NF membrane. Among these parameters, electrospinning duration and porosity had the strongest positive correlation (r=0.19; P=0.690), while applied voltage and porosity had the strongest inverse correlation (r=-0. 1, p=0.597). The correlation coefficient between NF membrane porosity and solution concentration was 0.05 (p=0.794), while the correlation coefficient between NF membrane porosity and needle-collector distance was r=-0.07(p=0.321). In a study, researchers found that the increase of applied voltage and needle-collector distance when solution concentration was fixed at given value resulted in increased porosity measured by image analysis [38]. Other researchers in a study indicated that there was a positive relationship between solution concentration and percent porosity when keeping applied voltage and spinning distance constant in a way that increase in concentration leads to increase in porosity (r=0.66, P<0.01). There are not enough studies on optimization of electrospinning parameters for porosity to make definite conclusions, so the differences between the results can be due to the types of experimental design and different used methods and material [5].

Performance characteristics

The pressure drop of fabricated NF membranes and crystalline silica concentration were measured and compared to commercial PVC membranes (Table 2). In general, the range of pressure drop of the all studied NF membranes was between 125-300 Pa. Mean pressure drop of all 30 runs was 194.23±47.70 Pa which was less than the mean pressure drop of commercial PVC membranes (204 Pa). The pressure drop of run 13 was highest at 300 Pa while run 29 had the lowest pressure drop at 125 Pa. The mean concentration of crystalline silica collected by the fabricated membranes was 0.14 mg/m^3 while for the commercial PVC membranes, this was around 0.03 mg/m^3. In case of the NF membrane, the least concentration belonged to run 13 while the highest belonged to run 24 and 28. The smallest difference in concentration of crystalline silica sampled by NF and commercial PVC membranes belonged to run 13 and 21.

Correlation between electrospinning parameters and pressure drop

The correlation coefficients between electrospinning parameters and performance variables are presented in Table 4. A positive correlation was observed between pressure drop and solution concentration with a correlation coefficient of 0.554 (p=0.001) and also between pressure drop and needle-collector distance with a correlation coefficient of 0.435 (p=0.595). An inverse correlation was observed between applied voltage and pressure drop with a correlation coefficient of -0.171 (p=0.365) and also between electrospinning duration and pressure drop with a correlation coefficient of -0.101 (p=0.016).

Increasing solution concentration or needle-collector distance led to increased pressure drop. Increasing electrospinning duration also led to reduced pressure drop. The applied voltage had very little influence as increasing it had a negligible effect on pressure drop. It must be noted that pressure drop is dependent on the structural attributes of the NF membranes such as diameter and porosity and not the electrospinning parameters themselves. Thus, one can not only look at the independent effects of the electrospinning variables on pressure drop. Here, only the effects of each parameter have been investigated.

As was mentioned above, pressure drop is dependent on the structural attributes of the NF membranes such as porosity and diameter. The results show a positive association between NF pressure drop and diameter with a correlation coefficient of 0.589 which means that increasing fiber diameter also increases pressure drop (p=0.001). An inverse association was observed between porosity and pressure drop with a correlation coefficient of -0.439 meaning that increasing porosity reduces pressure drop (p=0.015). Numerous studies have confirmed this observation and have investigated the relationship between pressure drop and the porosity and diameter of the NFs. Many researchers analyzed nylons 6 electrospun NFs and found that increasing solution concentration from 30% to 35% increases NF diameter from 103 nm to 144 nm which also increases the number of fibers resulting in thicker membranes and increased pressure drop [39].

Table 4. Correlation coefficient between electrospinning parameters and response variables related to performance characteristics.

Electrospinning parameters	Pressure drop (Pa)		Difference in silica concentration between the two membranes	
	r	p-value	r	p-value
Solution concentration (wt%)	0.55	0.001	-0.78	0.066
Applied voltage (kV)	-0.17	0.365	0.39	0.887
Electrospinning duration (hr)	-0.10	0.016	-0.23	0.667
Needle-Collector distance (cm)	0.43	0.595	-0.47	0.746

Correlation between electrospinning parameters and measured silica concentration

Regarding the difference in measured silica concentration between the two membranes (NF and commercial PVC), The smallest difference in concentration of crystalline silica sampled by NF and commercial PVC membranes belonged to run 13 and 21. The highest inverse correlation was observed between electrospinning parameters and solution concentration (r=0.78, p=0.066) and its correlation coefficient with other electrospinning parameters namely electrospinning duration, needle-collector distance and applied voltage was -0.23 (p=0.667), -0.47(p=0.746), and 0.39 p=0.887), respectively.

The independent effect of each electrospinning parameter on the difference in the measured concentration of silica between the NF and commercial PVC membranes, while the other three parameters were at their mean value was fixed at given value (mean value) was presented in Fig. 1. As shown in the Fig. 1, increasing electrospinning duration, needle-collector distance or solution concentration has had a somewhat inverse effect on difference concentration of solution and the black curves show these changes. Black square at the beginning and the end of each curves are mean concentration of silica at lowest and highest limit of electrospinning parameters. The amount of change is insignificant when increasing needle-collector distance and electrospinning duration while the change is significant when solution concentration is increased. Increasing voltage however, increases the concentration of silica captured by the NF membranes and the commercial PVC membranes. This is a performance feature of the NF membranes and therefore one cannot analyze the independent effect of each electrospinning parameter.

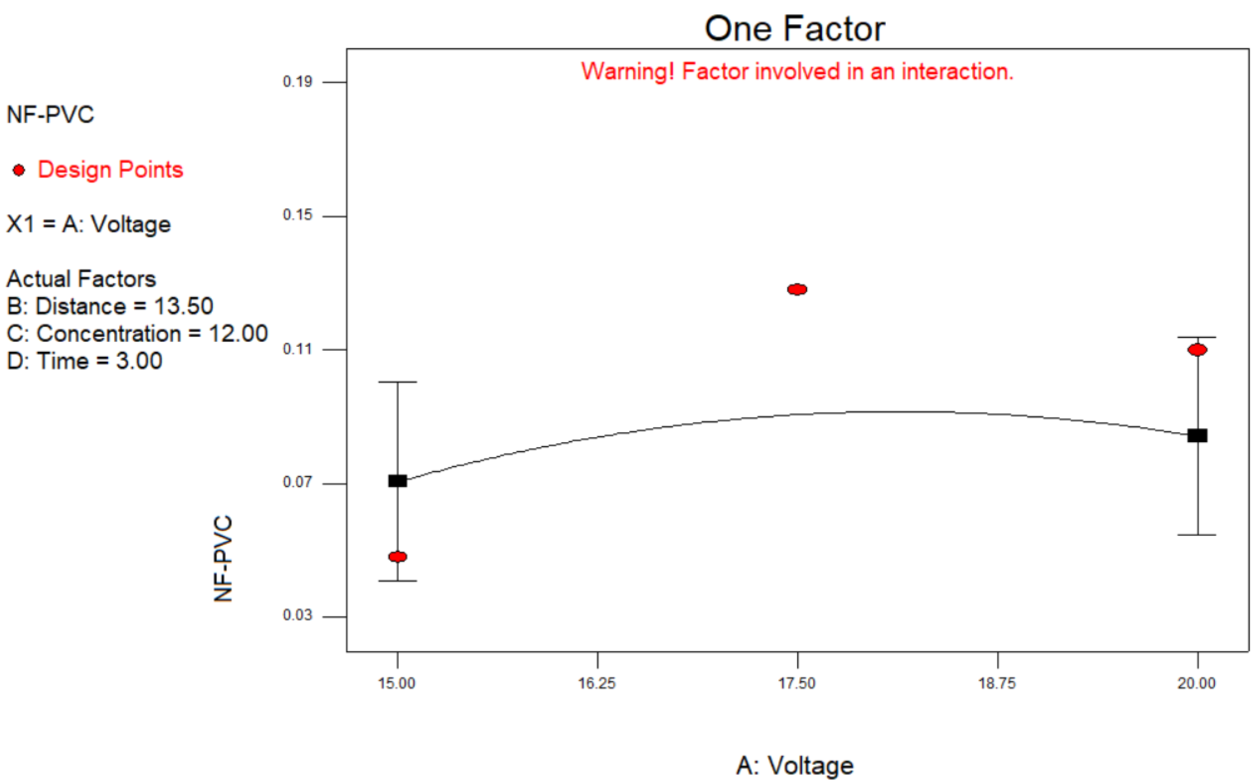
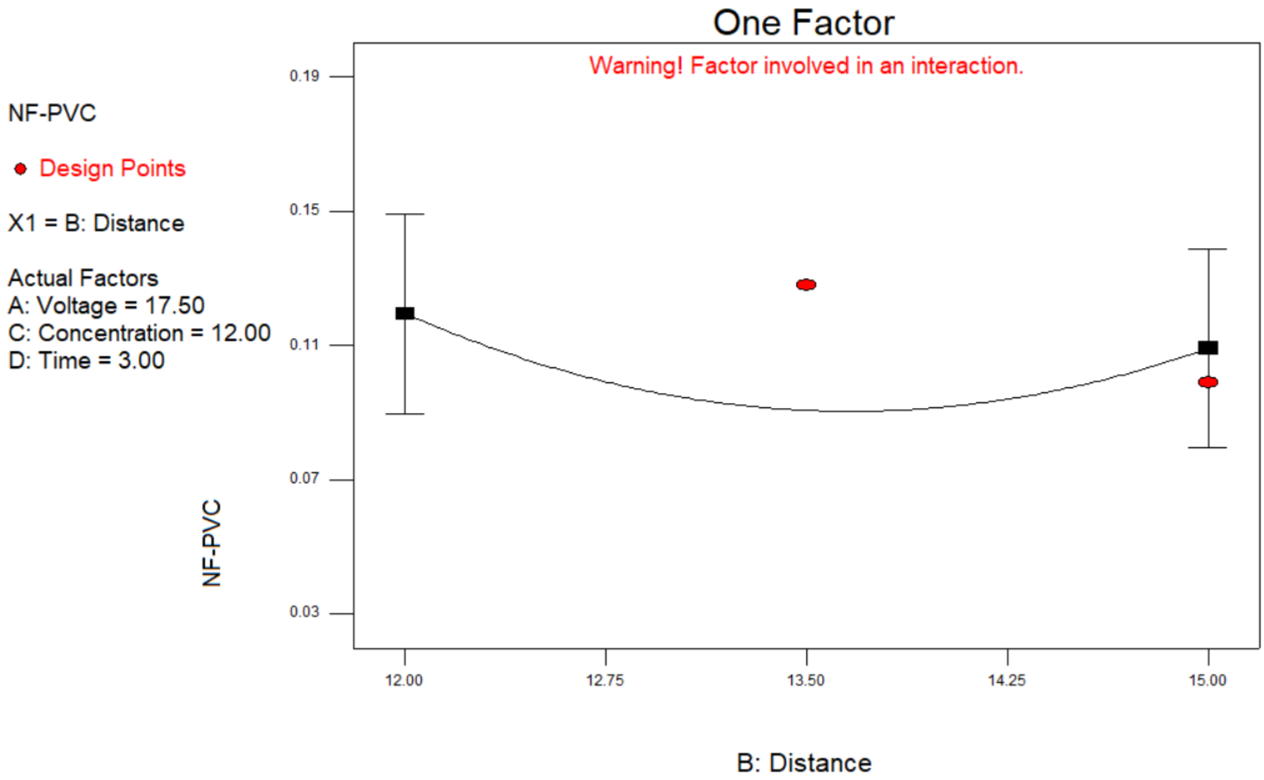


Fig. 1. The independent effect of electrospinning parameters on the difference in the measured concentration of silica by the nanofiber and the commercial PVC membranes



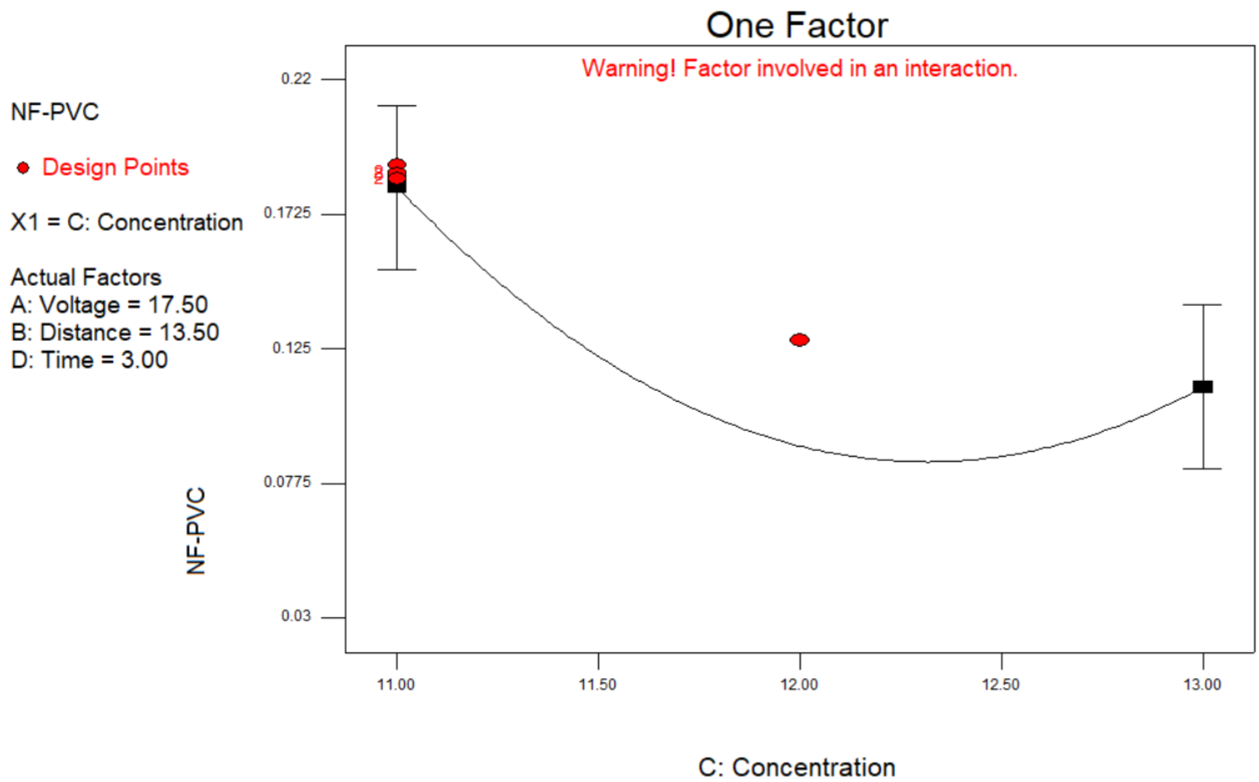


Fig. 1. The independent effect of electrospinning parameters on the difference in the measured concentration of silica by the nanofiber and the commercial PVC membranes

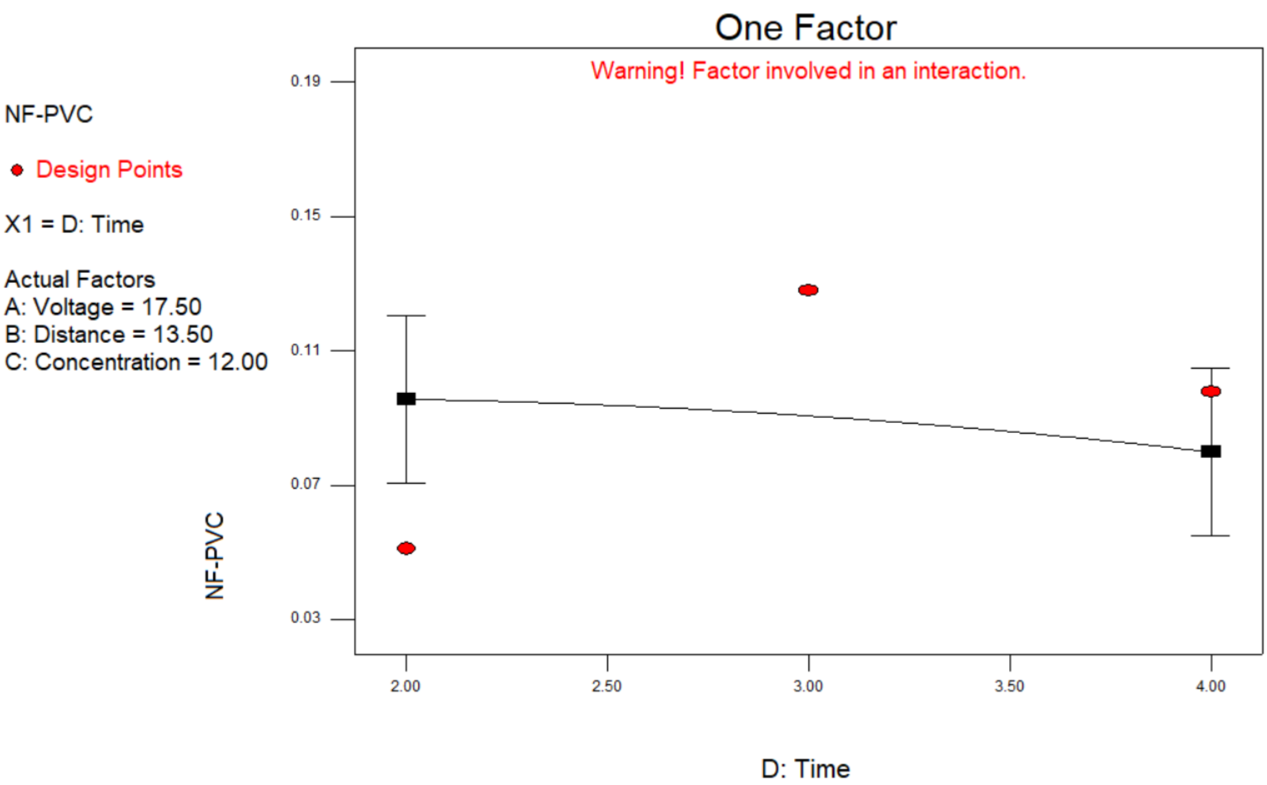


Fig. 1. The independent effect of electrospinning parameters on the difference in the measured concentration of silica by the nanofiber and the commercial PVC membranes

Table 5. Interaction effects of electrospinning parameters on response variables

Response variable	Model	Equation
Diameter (nm)	Quadratic	$-410.2+ 49.39A+49.26C+50.38D+81.14AB+114.38BC+96.53A^2+148.21C^{\prime 2}$
Porosity (%)	Quadratic	$35.38-7.74CD-10.63B^2$
Pressure drop (Pa)	Linear	$194.23+24.04B+28.98C$
Difference in silica concentration between the two membranes	Quadratic	$0.091-0.035C-0.036AB-0.047BC+0.056C^2$

A = Solution concentration, B=Applied voltage, C=Needle-Collector distance, D = Electrospinning duration,

Models of interaction of electrospinning parameters and response variables

Analyzing the combined effect of the electrospinning parameters on the response variables was performed using the multiple regression analysis by Design Expert (Table 5). Insignificant terms not included in the models are aliased according to the suggestion of Design Expert software. Model fitting was done with the help of Design Expert [5].

In the diameter equation, it is clear that increasing solution concentration will increase the diameter of the membranes. In the porosity equation, the highest negative effect size belonged to applied voltage meaning that increasing applied voltage will increase membrane porosity. As for pressure drop, increasing applied voltage will increase membrane pressure drop. The difference in silica concentration captured by the fabricated NF membranes and the commercial PVC membrane increased at lower needle-collector distance. In all equations the variables with p-value was more than 0.05 were removed from the equation.

Determining the optimum conditions

One of the aims of the present study was to determine the optimum electrospinning parameters for fabrication NFs for application of air sampling. After initial analysis and considering the relevant results, optimization was performed using the Design-Expert software with the aim of determining the optimum electrospinning parameters in terms of achieving desired values pertaining to the diameter, porosity and pressure drop of the fabricated NF membrane. For this, diameter was set to minimum, porosity to maximum and pressure drop to minimum, in the optimization instructions of the software. Three of the most desirable electrospinning conditions obtained in the optimization stage have been taken from the list of proposed experiments and presented in Table 6.

Table 6. Some of the optimum solutions

Row	Duration (h)	Concentration (%wt)	Needle distance (cm)	Voltage (kV)	Membrane diameter (nm)	Porosity (%)	Pressure drop (Pa)	Desirability (0-1)	Code
1	4	11	13.83	16.21	298.26	40.80	149.86	0.893	Selected A
2	4	11	13.81	16.09	305.45	41.29	149.34	0.893	Selected B
3	3.99	11	13.85	16.13	297.76	40.76	150.20	0.892	Selected C

Considering the software suggestion, it was determined that in order to achieve the highest optimum degree of desirability, which was 0.893, the electrospinning parameters must be as follows: a solution concentration of 11 wt%, an applied voltage of 16.5 Kv, a needle-collector distance of 13.5cm and an electrospinning duration of 4 hours.

Confirmation experiments

Validation of the equations was tested by performing the first three selected experiments with higher desirability degree under the determined electrospinning conditions. The results indicate that the experimental values are in agreement with the predicted responses, so in case of fiber diameter and Difference in silica concentration between the two membranes (NF and commercial PVC), percentage variation was 9% and 11%, respectively.

Conclusion

In case of relationship between electrospinning parameters and performance characteristics, the highest (negative inverse) correlation was observed between solution concentration and difference in silica concentration between the two membranes (NF and commercial PVC). In case of relationship between electrospinning parameters and structural characteristics, the highest (positive) correlation was observed between solution concentration and fiber diameter. It was determined that a solution concentration of 11 wt%, an applied voltage of 16.5 Kv, a needle-collector distance of 13.5 cm and an electrospinning duration of 4 hours can be one of the optimum electrospinning conditions for fabrication the PVC NFs in application of air sampling. The PVC NF membrane has suitable performance in air sampling of the crystalline silica dust in compared to commercial PVC membrane.

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Competing interests

The authors have no competing interests to declare.

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Ethical considerations

Ethical approval for this study was obtained from the School of Public Health & Neuroscience Research Center at the Shahid Beheshti University of Medical Sciences (IR.SBMU.PHNS.REC.1398.049). Ethical issues (including plagiarism, Informed Consent, misconduct, data fabrication and/or falsification, double publication and/or submission, redundancy, etc) have been completely observed by the authors.

References

1. Lee H, Jeon S. Polyacrylonitrile Nanofiber Membranes Modified with Ni-Based Conductive Metal Organic Frameworks for Air Filtration and Respiration Monitoring. ACS Applied Nano Materials. 2020;3(8):8192-8.

https://pubs.acs.org/doi/10.1021/acsanm.0c0161.

2. Liu H, Zhang S, Liu L, Yu J, Ding B. High-Performance PM0.3 Air Filters Using Self-Polarized Electret Nanofiber/Nets. Advanced Functional Materials. 2020;30(13):1909554. https://onlinelibrary.wiley.com/doi/abs/10.1002/adfm.201909554

3. Kaur GA, Shandilya M, Rana P, Thakur S, Uniyal P. Modification of structural and magnetic properties of Co0. 5Ni0. 5Fe2O4 nanoparticles embedded Polyvinylidene Fluoride nanofiber membrane via electrospinning method. Nano-Structures & Nano-Objects. 2020;22:100428. https://af.booksc.eu/book/81123835/f0cb0b

4. Figoli A, Ursino C, Sanchez Ramirez DO, Carletto RA, Tonetti C, Varesano A, De Santo MP, Cassano A, Vineis C. Fabrication of electrospun keratin nanofiber membranes for air and water treatment. Polymer Engineering & Science. 2019 Jul;59(7):1472-8. https://onlinelibrary.wiley.com/doi/abs/10.1002/pen.25146

5. Dehghan SF, Golbabaei F, Maddah B, Latifi M, Pezeshk H, Hasanzadeh M, et al. Optimization of Electrospinning Parameters for PAN-MgO Nanofibers Applied in Air Filtration. Journal of the Air & Waste Management Association. 2016;66(9):912-21. https://www.sciencedirect.com/science/article/abs/pii/S2352507X20300068

6. Mohraz MH, Yu IJ, Beitollahi A, Dehghan SF, Shin JH, Golbabaei F. Assessment of the potential release of nanomaterials from electrospun nanofiber filter media. NanoImpact. 2020 Jul 1;19:100223. https://www.sciencedirect.com/science/article/abs/pii/S2452074820300173

7. Strandberg B, Julander A, Sjöström M, Lewné M, Akdeva HK, Bigert C. Evaluation of polyurethane foam passive air sampler (PUF) as a tool for occupational PAH measurements. Chemosphere. 2018 Jan 1;190:35-42. https://www.sciencedirect.com/science/article/pii/S004565351731531X

8. Ferguson RM, Garcia-Alcega S, Coulon F, Dumbrell AJ, Whitby C, Colbeck I. Bioaerosol biomonitoring: Sampling optimization for molecular microbial ecology. Molecular ecology resources. 2019;19(3):672-90. https://onlinelibrary.wiley.com/doi/full/10.1111/1755-0998.13002

9. Farhangian M, Dehghan SF, Jafari MJ, Pirposhteh EA, Khalilinejad A, Tavako E. Feasibility Study on the Application of Electrospun Nanofiber Webs for the Air Sampling of Crystalline Silica. Industrial Health 2021;596. https://doi.org/10.2486/indhealth.20

10. Franks CM. Too Little Too Late: The Infeasibility of OSHA's Silica Standards in the Oil Industry. . Mary's LJ. 2017;49:819. https://heinonline.org/HOL/LandingPage?handle=hein.journals/stmlj49&div=28&id=&page=

11. Maciejewska A. Occupational exposure assessment to crystalline silica dust: Approach in Poland and worldwide. International Journal of Occupational Medicine and Environmental Health. 2008 Jan 1;21(1):1. https://pubmed.ncbi.nlm.nih.gov/18482900/

12. Chen W, Liu Y, Wang H, Hnizdo E, Sun Y, Su L, et al. Long-term exposure to silica dust and risk of total and cause-specific mortality in Chinese workers: a cohort study. PLoS medicine.2012;9(4):e1001206. https://journals.plos.org/plosmedicine/article?id=10.1371/journal.pmed.1001206

13. Wilbourn JD, McGregor DB, Partensky C, Rice JMJEhp. IARC reevaluates silica and related substances. Environmental Health Perspectives. 1997 Jul;105(7):756-9. https://

pubmed.ncbi.nlm.nih.gov/9294723/

14. Omidianidost A, Ghasemkhani M, Kakooei H, Shahtaheri SJ, Ghanbari M. Risk assessment of occupational exposure to crystalline silica in small foundries in Pakdasht, Iran. Iranian journal of public health. 2016;45(1):70. https://www.ncbi.nlm.nih.gov/pmc/articles/PMC4822397/

15. Bahrami AR, Golbabai F, Mahjub H, Qorbani F, Aliabadi M, Barqi M. Determination of exposure to respirable quartz in the stone crushing units at Azendarian-West of Iran. Industrial health. 2008;46(4):404-8. https://pubmed.ncbi.nlm.nih.gov/18716390/

16. Akbar-Khanzadeh F, Brillhart RL. Respirable crystalline silica dust exposure during concrete finishing (grinding) using hand-held grinders in the construction industry. Annals of Occupational Hygiene. 2002;46(3):341-6. https://academic.oup.com/annweh/article/46/3/341/271651?login=true

17. Rappaport SM, Goldberg M, Susi PA, Herrick RF. Excessive exposure to silica in the US construction industry. Annals of Occupational Hygiene. 2003;47(2):111-22. https://academic.oup.com/annweh/article/47/2/111/133229?login=true

18. Jaakkola MS, Sripaiboonkij P, Jaakkola JJ. Effects of occupational exposures and smoking on lung function in tile factory workers. International archives of occupational and environmental health. 2011;84(2):151-8. https://link.springer.com/article/10.1007/s00420-010-0603-6

19. Jebelli B, Ghazi I, Mahamoodzadeh A, Ghazanchaei E. Silica exposure in the glass industry and human health risk assessment. International Journal of Health System and Disaster Management. 2015;3(3):151. https://www.ijhsdm.org/article.asp?issn=2347-9019;year=2015;volume=3;issue=3;spage=151;epage=155;aulast=Jebelli

20. Akgun M, Gorguner M, Meral M, Turkyilmaz A, Erdogan F, Saglam L, et al. Silicosis caused by sandblasting of jeans in Turkey: a report of two concomitant cases. Journal of occupational health. 2005;47(4):346-9. https://pubmed.ncbi.nlm.nih.gov/16096363/

21. Chen W, Yang J, Chen J, Bruch JJAjoim. Exposures to silica mixed dust and cohort mortality study in tin mines: Exposure-response analysis and risk assessment of lung cancer. American journal of industrial medicine. 2006 Feb;49(2):67-76.2006;49(2):67-76. https://pubmed.ncbi.nlm.nih.gov/16362950/

22. NIOSH. SILICA, Respirable Crystalline, by IR (KBr pellet)7602:NIOSH Manual of Analytical Methods (NMAM), Fifth Edition. Fifth Edition ed. California NIOSH; 2017. https://manualzz.com/doc/9342194/niosh-method-7602---silica--crystalline--by-ir--kbr-pellet-

23. Liu Y, Hao M, Chen Z, Liu L, Liu Y, Yang W, et al. A review on recent advances in application of electrospun nanofiber materials as biosensors. Current Opinion in Biomedical Engineering. 2020 Mar 1;13:174-89. https://www.mdpi.com/2073-4360/13/21/3746/htm

24. Matulevicius J, Kliucininkas L, Martuzevicius D, Krugly E, Tichonovas M, Baltrusaitis J. Design and characterization of electrospun polyamide nanofiber media for air filtration applications. Journal of nanomaterials. 2014;2014. https://www.hindawi.com/journals/jnm/2014/859656/

25. Bao L, Seki K, Niinuma H, Otani Y, Balgis R, Ogi T, et al. Verification of slip flow in nanofiber filter media through pressure drop measurement at low-pressure conditions. Separation and purification technology. 2016;159:100-7. https://www.sciencedirect.com/science/article/abs/pii/S1383586615304147

26. Tarus B, Fadel N, Al-Oufy A, El-Messiry M. Effect of polymer concentration on the morphology and mechanical characteristics of electrospun cellulose acetate and poly (vinyl chloride) nanofiber mats. Alexandria Engineering Journal. 2016 Sep 1;55(3):2975-84. https://www.sciencedirect.com/science/article/pii/S1110016816300813

27. Mazlomi A, Golbabaei F, Farhang Dehghan S, Abbasinia M, Mahmoud Khani S, Ansari M, et al. The influence of occupational heat exposure on cognitive performance and blood level of stress hormones: A field study report. International journal of occupational safety and ergonomics. 2017;23(3):431-9. https://pubmed.ncbi.nlm.nih.gov/27852154/

28. Mohammadi H, Dehghan SF, Moradi N, Suri S, Pirposhteh EA, Ardakani SK, et al. Assessment of sexual hormones in foundry workers exposed to heat stress and electromagnetic fields. Reproductive Toxicology. 2021;101:115-23. https://doiorg/101016/jreprotox202012015

29. Farhangian M, Dehghan SF, Jafari MJ, Pirposhteh EA, Khalilinejad A, Tavakol E. Feasibility study on the application of electrospun nanofiber webs for the air sampling of crystalline silica. Industrial Health. 2021:2020-0236. https://pubmed.ncbi.nlm.nih.gov/34588378/

30. ISO. ISO 29463: High-efficiency filters and filter media for removing particles in air--Part 3: Testing flat sheet filter media. Geneva: International Organization for Standardization; 2011. https://www.iso.org/standard/51837.html

31. KhaliliNeJad A. Fabrication study of elecrospun polyvinyl chloride nanofiberous filter media for crystalline silica sampling in air. Tehra, Iran: Shhid Beheshti University of Medical Sciences; 2020. http://dlib.sbmu.ac.ir/faces/search/bibliographic/biblioFullView.jspx?_afPfm=3xqg8a7ns

32. Li Y, Huang Z, Lǚ Y. Electrospinning of nylon-6, 66, 1010 terpolymer. 2006;42(7):1696-704. https://www.sciencedirect.com/science/article/abs/pii/S0014305706000553

33. Boland ED, Wnek GE, Simpson DG, Pawlowski KJ, Bowlin GLJJoMS, Part A. Tailoring tissue engineering scaffolds using electrostatic processing techniques: a study of poly (glycolic acid) electrospinning. Journal of Macromolecular Science, Part A. 2001 Nov 30;38(12):1231-43. https://www.tandfonline.com/doi/abs/10.1081/MA-100108380

34. Gu SY, Ren JJMm, Engineering. Process Optimization and empirical modeling for electrospun poly (D, L-lactide) fibers using response surface methodology. Macromolecular materials and Engineering. 2005 Nov 4;290(11):1097-105. https://onlinelibrary.wiley.com/doi/abs/10.1002/mame.200500215

35. Dehghan SF, Golbabaei F, Maddah B, Yarahmadi R, Zadeh AS. Fabrication and optimization of electrospun polyacrylonitrile nanofiber for application in air filtration. Iran Occupational Health. 2016;13(5):11-23. http://jips.ippi.ac.ir/index.php/issst/article_1749.html?lang=en

36. Iqbal T. An investigation on the effect of solution concentration, applied voltage and collection distance on electrospun fibres of PVA solutions (Doctoral dissertation, University of Birmingham). https://www.tandfonline.com/doi/pdf/10.1080/10962247.2016.1162228

37. Dehghan S, Golbabaei F, Mousavi T, Mohammadi H, Kohneshahri M, Bakhtiari R. Production of nanofibers containing magnesium oxide nanoparticles for the purpose of bioaerosol removal. Pollution. 2020;6(1):185-96. https://www.ncbi.nlm.nih.gov/pmc/articles/PMC8655750/

38. Ziabari M, Mottaghitalab V, Haghi AK. A novel approach for analysis of processing parameters in electrospinning of nanofibers. Nanofibers: Fabrication, Performance, and Applications. 2009 Jan 1:153-82. https://dro.deakin.edu.au/view/DU:30125441

39. Danwanichakul P, Danwanichakul D. Two-dimensional simulation of electrospun nanofibrous structures: connection of experimental and simulated results. Journal of Chemistry. 2014 Jan 1;2014. https://www.hindawi.com/journals/jchem/2014/479139/