Fabrication and Characterization of Polyurethane Electrospun Nanofiber Membranes for Protective Clothing Applications

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ABSTRACT: Electrospun nanofibrous webs are important in nanotechnology applications due to their high surface area and interconnected porosity. In this study, the effect of electrospinning duration on some physical and mechanical properties of polyurethane (PU) electrospun webs is investigated for potential applications such as protective clothing and membranes. The results show that the thickness and weight of webs and subsequently their tensile strength increase linearly with the electrospinning duration. Air permeability of nanofibrous webs decrease and hydrostatic pressure increases nonlinearly while water vapor permeability remains constant. This work shows that air permeability of PU webs follows Fick's law of diffusion. Some regression models have been proposed to describe electrospun membranes behavior. The results of this investigation indicate that this new generation of nanofibrous materials has a good potential for application as membrane in protective clothing. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 000: 000–000, 2012

Key words: electrospinning; nanofibrous membrane; protective clothing

INTRODUCTION

Attractive features of nanofibrous webs such as soft hand, barrier against liquid penetration, good strength per unit weight, and high surface energy, have opened a wide range of applications for this new generation of materials.[1] Some researchers have focused on production of nanofibers from different polymers via electrospinning process.[2–4] Others have paid attention on modifying the electrospun nanofiber mats.[5–9] Although some works are have been carried out on studying the physical and mechanical properties of the electrospun nanofibers,[10–17] further investigations are necessary for studying the structure-related parameters with respect to the end uses.

Polyurethane (PU) has received considerable attention in electrospinning process due to easy care, resistance to microorganism, and excellent hydrolytic stability.[14–16,18–21] Tensile behavior of thermoplastic polyurethane elastomer (TPUE) is investigated by different researchers.[14–16] These works reported that electrospun TPUE fiber mats showed nonlinear elastic and inelastic characteristics, which may be because of the slippage of crossed nanofibers, i.e., non bonding or physical bonding structure and breakage of the nanofibers at junctions, i.e., point bonded or chemical bonding structure.

Membranes have been used successfully in garments in some special garments. Some examples include PU membrane in polartech[22] and PTFE membrane in Gore-Tex[18] fabric system for making wind proof breathable rain coats. Some researchers have focused on developing light-weight protective apparel using electrospun nanofiber membranes[18,20,23,24] in self-supported or substrate supported forms for protective garments. These ultrathin layers exhibit good degree of tensile strength, breathability (due to excellent moisture vapor transport), elasticity, resistance to liquid penetration and low air permeability which make them good candidates for wind proof breathable rainwear sector.[18,23,24] Gibson et al.[18] stretched porous elastomeric nanofiber membranes under biaxial tension to strain levels of 100% and showed that convective gas flow properties were increased as the inter fiber pores opened up, while water vapor diffusion remained constant. They stated that this interconnected membrane like web presents minimum impedance to moisture vapor diffusion required for protective clothing. Lee and Obendorf[25] proved that the mass of electrospun PU layers per unit area is much smaller than conventional PTFE membrane. They also showed that air permeability of electrospun PU decreases with increasing the web area density, however, no significant difference was observed in moisture vapor transmission. In another research, they showed that barrier/transport properties that may not

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be met in conventional protective garments could be achieved by using electrospun nanofibrous web.[20] Leung et al.[12] studied the effect of packing density and thickness on the filtration performance of a nonwoven substrate coated with nanofibers and reported that layer thickness has a less prominent effect on most penetrating particle size than nanofiber packing density.

This study discusses the merits of PU electrospun nanofibrous webs as wind proof membrane. The effect of electrospinning duration on transfer properties (air and moisture permeability and hydrostatic pressure) and mechanical properties (breaking elongation and tenacity) of nanofiber-based membranes was investigated in this work. For describing electrospun mat behavior, statistical models have been proposed based on regression equations.

EXPERIMENTAL WORKS

Electrospinning process

The electrospinning solution was prepared with 13 % wt/vol of commercial PU (M_w: 65,000, Bayer, Germany) which was dissolved in THF and *N*,*N*-dimethylformamide mixture (60 : 40 v/v). In the electrospinning process, the prepared solution was electrospun simultaneously on the rotating drum from two opposite nozzles (Fig. 1). The optimum distance between nozzles and collector was obtained to be 130 mm. The rotational speed and traverse speed of the drum was, respectively, 150 rpm and 400 mm/min. A voltage of 13 KV was applied to draw the nanofibers from the solution in the electrostatic field. Twelve membrane samples were produced within 1–12 h production times with 1-h interval.

Characterization

The thickness of membranes was measured using a micrometer (Dial Thickness Gage, Mitotoyo, Japan). The average of 20 measurement points was reported

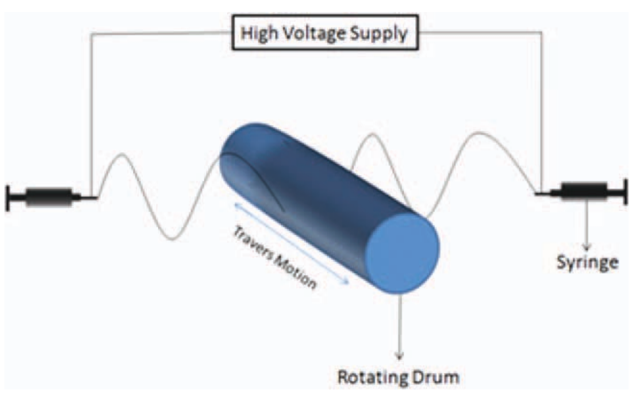

Figure 1 Schematic diagram of double nozzle electrospinning machine with rotating drum. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

as the mat thickness. Morphology of electrospun PU mats was examined using a scanning electron microscope (SEM) (440i, LEO Electron Microscopy, England). The diameters of the nanofibers were determined from SEM images using Image analyzer software (http://rsb.info.nih.gov/ij/). The nanofibers orientation distribution was calculated by a program which was encoded in MATLAB Software (version 7.0.0.1920). For this purpose, coordinates of two end point of a straight part of a fiber were measured as ($x1$, $y1$) and ($x2$, $y2$), then the angle, length, and orientation of fiber were defined according to eqs. (1)–(3):

$$\alpha = \text{Fiber angle} = \tan^{-1}\left|\frac{y_2 - y_1}{x_2 - x_1}\right| \quad (1)$$

$$l = \text{Fibre length} = \sqrt{(x2 - x1)^2 + (y2 - y1)^2} \quad (2)$$

$$\text{Fibers Orientation Frequency} = \frac{100 l_i \ \alpha_i}{\Sigma_{i=1}^{n} l_i} \quad (3)$$

where l_i is the specimen length in the direction α_i. One hundred pieces of fibers in each sample were measured for determining the fiber orientation distribution. The sum of fiber orientation frequency in each 15 degree interval was derived and plotted.

Considering the end use of PU nanofibrous mat as the membranes for protective clothing, air and moisture permeability (the indexes for breathability of protective textiles and membranes), hydrostatic pressure (the important factor in waterproof breathable garments), breaking elongation and tenacity were examined and reported.

The air permeability was determined according to the ASTM D737 using the air permeability tester (Karl Frank GMBH, WEINHEIM-BIRKENAU, Germany). This test determines the resistance of mat to the passage of air (air flow) through a known area under constant pre-set air pressure difference between two surfaces of mat while the sample firmly clamped in the test rig. Electrospun layer in circular shape was clamped into the layer tester and through the use of a vacuum; the air pressure difference was built up on one side of the layer. Air flow passed from the side with higher air pressure, through the layer, to the side with the lower air pressure. From this rate of air flow, the air permeability of the fabric was determined. The arithmetic average of five test results was taken to calculate the mean value of membrane air permeability.

Five specimens from each samples produced in various time intervals were prepared for hydrostatic pressure measurement according to ISO 811 : 1981(Hydrotester III, FX3000, by TEXTEST, Zürich, Switzerland). This test determines the resistance of fibrous material to water penetration under specific pressure. The specimens are clamped on test head facing with water and after starting the test, water pressure is increased linearly with time. The test specimen is observed visually for tracing the evidence of water penetration until the

TABLE I
Characteristic of PU Electrospun Nanofiber Webs (Mean and CV%)

Electrospinning duration (h)	1	2	3	4	5	6	7	8	9	10	11	12
Weight (g/m^2)	17.8	28.5	44.4	55.9	71.2	85.0	92.5	105.0	125.5	140.9	152.2	166.0
	5.3	23.2	17.8	11.7	14.3	5.3	4.4	13.1	8.4	11.3	12.2	13.2
Thickness (μm)	21.9	31.0	70.0	91.7	120.6	142.5	165.6	180.0	210.0	252.1	260.6	280.0
	18.4	7.6	1.82	5.58	8.46	2.50	8.7	10.2	8.8	7.8	5.0	9.8
Air permeability (ml m^{-2} h^{-1})	180.0	115.4	65.2	49.5	27.9	30.5	20.5	20.0	19.3	22.3	18.4	12.8
	0.75	3.22	1.4	1.9	4.3	2.3	2.3	3.3	8.7	3.7	2.2	6.1
WVP (g m^{-2} day^{-1})	39.2	39.0	39.5	39.1	39.0	39.6	38.9	39.2	39.5	40.67	39.928	34.802
	4.3	5.1	12.9	11.32	7.8	2.8	10.4	2.58	3.25	12.2	18.5	15.3
Hydrostatic pressure (cm of water)	12.9	17.8	34. 6	37.9	50.8	50.5	62.0	63.0	65.0	66.0	67.076	75.0
	7.4	1.97	4.5	13.21	9.4	11.22	4.3	2.7	2.8	7.5	2.8	8.7
Tenacity in MD (N)	0.7	1.5	1.6	2.0	3.2	3.4	3.7	4.3	5.5	5.7	8.5	8.6
	7. 53	12.95	7.4	15.8	21.4	14.6	22.1	13.3	6.7	19.0	4.6	15.6
Tenacity in CD (N)	0.8	2.1	2.4	2.7	4.2	4.9	5.3	9.3	8.2	8.1	11.6	11.7
	18. 0	17.4	23.2	6.9	7.7	15.3	5.9	8.3	11.5	5.0	9.9	7.3
Max extension in MD (%)	304.5	147.7	270.1	282.6	286.9	291.3	273.5	382	323.2	291.3	343.9	301.2
	14.7	13.4	5.3	8.2	7.1	9.7	20.0	13.9	7.4	8.7	3.78	1.81
Max extension in CD (%)	289.4	165.8	293.2	291.3	271.4	308.7	261.2	360.6	326.4	291.2	330.2	310.5
	11.4	13.8	14.0	5.1	6.4	12.5	3.2	4.1	8.1	3.9	6.8	5.7

appearance three water droplets. Consequently, the mean of hydrostatic pressure was derived and reported.

Water vapor permeability test was carried out using a tester M261 by Atlas, England, according to ASTM E 96-00. The mats were sealed into the mouth of cups containing pre determined amount of water. After 7 days, the WVP was calculated from the change in cup weight according to eq. (4):

$$\text{WVP}\ (\text{g.m}^{-2}.\text{day}^{-1}) = 10^5(m_2 - m_1)/7S \qquad (4)$$

where $m_2 - \text{m}_1$ is the weight difference (mg) of the cup after 7 days, and S is the area of moisture transport (cm^2).where $\text{m}_2 - \text{m}_1$is the weight difference (mg) of the cup after 7 days, and S is the area of moisture transport (cm^2).

For characterization tensile properties of the mats, dumbbell-shaped specimens were prepared and tested with a crosshead speed of 50 mm/min according to ASTM D-638. This test was carried out on five specimens in machine direction (MD) and five specimens in transverse direction (TD). Minitab package 16 was used for statistical analysis. A summary of various test results is brought in Table 1.

RESULTS AND DISCUSSION

Structural properties of electrospun nanofiber PU webs

The average diameter of electrospun fibers was approximately 480 nm (Fig. 2). The analysis of variance (ANOVA) at 99% confidence level showed that there is no significant difference between mean diameters of nanofibers among the samples with various time intervals of electrospinning.

Figure 3 shows a typical fibers orientation in different directions of one sample. As this figure demonstrates, the fibers are more oriented in cross direction (CD) than the MD. This is attributed to the traverse motion of drum that build up more oriented of nanofibers in this direction. This trend is observed in all the 12 samples.

Studying the effect of the electrospinning duration on thickness and weight of the mats showed that the increase in the process duration, leads to the increase in the thickness and weight of layers (Fig. 4).

Transport properties

One of the important and controlling factors in the clothing systems is gas, vapor, and liquids transport through the layers.[26] The elucidation of the relation between transport properties and macrostructures of electrospun nanofibrous membrane will help to design highly comfortable protective garments.

Air flow resistance of electrospun membranes composed of elastomeric fibers, correlates with the electrospun coating add-on weight,[18,24] but to the knowledge of the authors no model has been developed yet to predict this correlation.

The results of air permeability test show that the relation between electrospinning duration and air permeability is not linear (Fig. 5) but equivalent as follows:

$$\mathit{Air\ Permeability} \propto \frac{1}{\mathit{Electrospinning\ Time}}$$

The regression equation ($R^2 = 97.4\%$) is:

$$\text{Air Permeability}\left(\frac{ml}{m^2.h}\right) = \frac{190.2}{\text{Electrospinning Duration(h)}} \qquad (5)$$

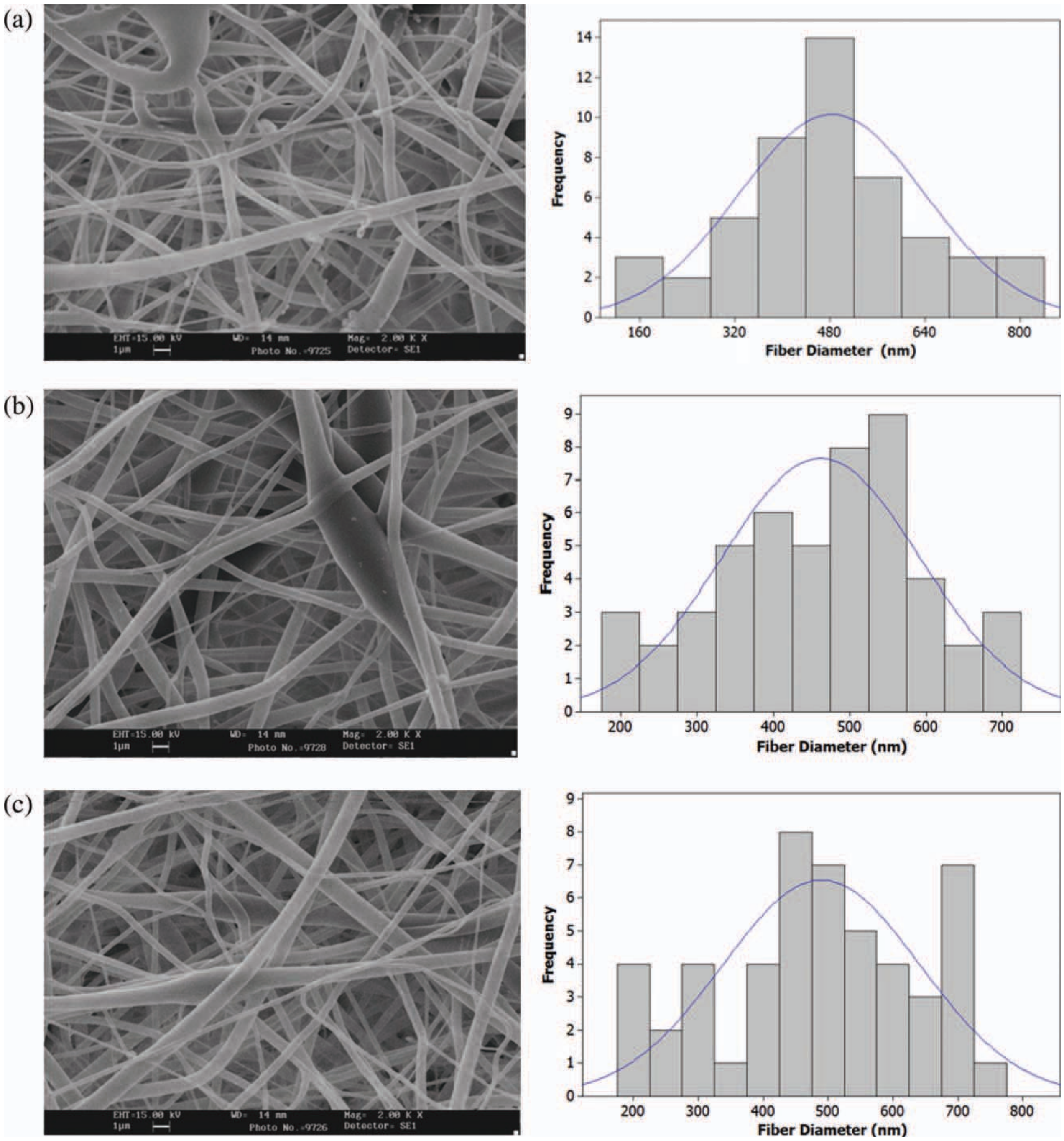

Figure 2 SEM image of electrospun nano web sample after electrospinning: (a) 2 h (483.2 nm), (b) 4 h (462.5 nm), and (c) 6 h (489.5 nm). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Fick's first law of diffusion describes transport process as:

$$J = -D\left(\frac{\partial c}{\partial x}\right) \tag{6}$$

where J is flux in the direction of flow (x), D is the diffusion coefficient and $\frac{\partial c}{\partial x}$ is the concentration gradient.[26] The diffusion coefficient is independent of concentration and diffusion flow is constant in the steady state, though eq. (6) may be integrated to:

$$J = -\frac{n(n_1 - n_2)}{h} \tag{7}$$

where h is the film thickness and $c1$, $c2$ are the concentrations of penetrant at the two faces of the film. For gas transporting according to Henry's law, pressure p is used instead of surface concentration and:

$$c = Sp \tag{8}$$

where S is the solubility coefficient. By combining eqs. (7) and (8), the permeation equation is determined as:

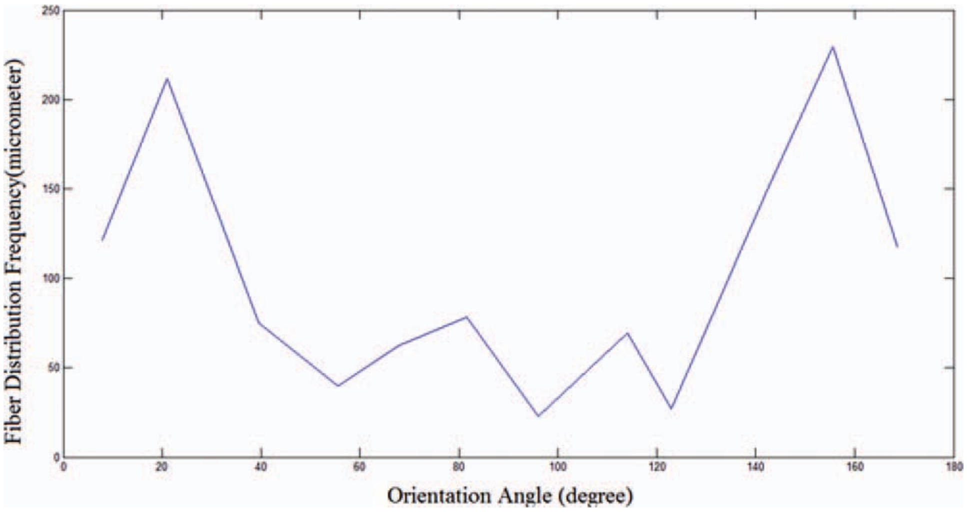

Figure 3 Typical fiber orientation distribution. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

$$J = \frac{P(p_1 - p_2)}{h} \quad (9)$$

where $P = \mathrm{DS}$ is the permeability coefficient, $p1$ and $p2$ are the pressure of the surroundings on two sides of a film with thickness of h. In a steady state, for a certain membrane, P, $p1$, and $p2$ are constant and the eq. (9) can be rewritten as:

$$J = k/h \quad (10)$$

where $k = P(p_1 - p_2)$ and has a constant value.

On the other hand, considering the linear relation between electrospinning duration and thickness, the regression model between air permeability and thickness is:

$$\text{Air Permeability}\left(\frac{ml}{m^2.h}\right) = \frac{3811}{\text{Thickness }(\mu\text{m})} \quad (11)$$

Comparison of eq. (10) with the regression model 11, one can concluded that the air permeability of PU electrospun nanofiber membrane follows the Fick's law of diffusion.

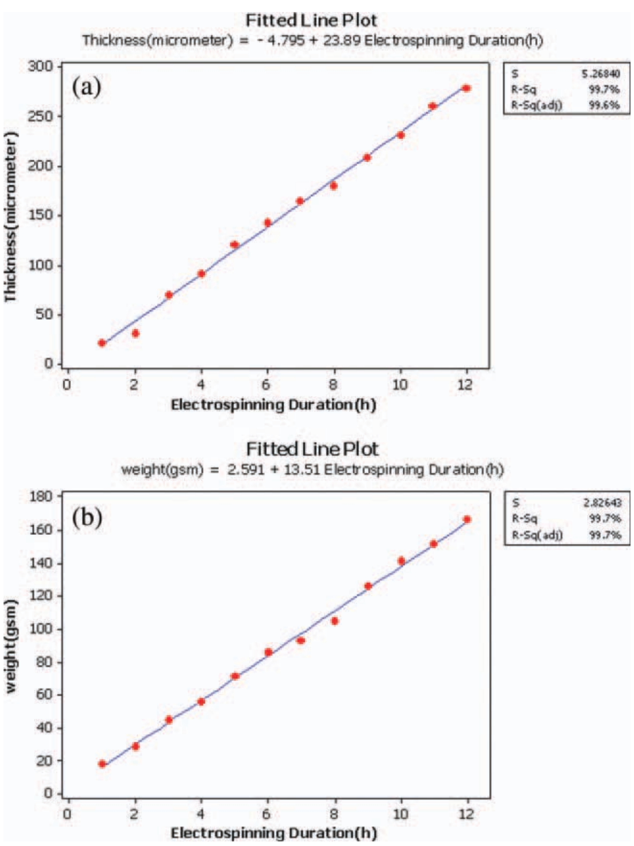

Figure 4 (a) Thickness vs. electrospinning duration, (b) Weight vs. electrospinning duration. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

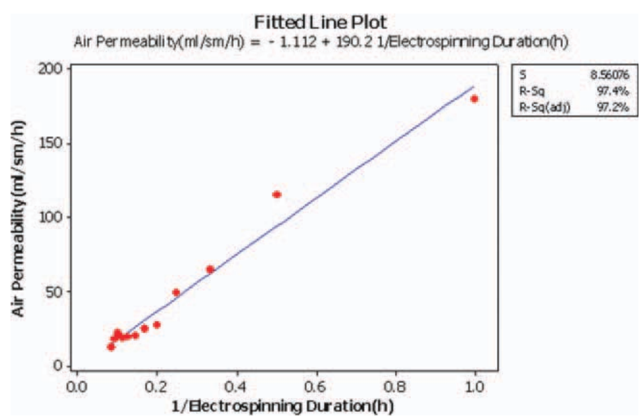

Figure 5 The effect of electrospinning duration (h) on air permeability (ml m^{-2} h^{-1}). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

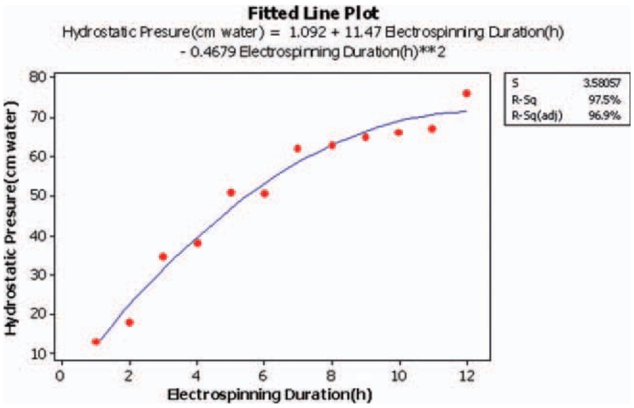

Figure 6 The effect of electro spinning duration on hydrostatic pressure. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

The trend of results shows that increasing the electrospinning duration, which leads to higher web thickness, decreases the air permeability of samples 1–6 considerably. In the other samples, the rate of decrease is low, although it is statistically significant. In other words; >6 h production time has a small effect on resistance of layers against air flow. Hence, 6 h production time is the optimum level of electrospinning duration for air permeability.

Figure 6 shows the hydrostatic pressure of samples vs. electrospinning duration. The result of the test indicates that the relation between electrospinning duration (which results in various thicknesses) and hydrostatic pressure is not linear.

The quadratic regression model can fit the experimental data with $R^2 = 97.5\%$ that is presented in equation:

$$\text{Hydrostatic Pressure (cm water)} = 1.092 + 11.47\ \text{Electrospinning Duration}(h) - 0.4679\ \text{Electrospinning Duration}^2(h) \quad (12)$$

Although the ANOVA test showed significant difference between hydrostatic pressure of different samples, those produced with 7–12 h time intervals, have smaller differences than those with those with 1–7 h. The same as the air permeability test, there is an optimum level for hydrostatic pressure behavior that this optimum level was obtained 7 h electrospinning duration.

Water vapor permeability remained unchanged at different process duration. The nature of the membranes dictates the mode of permeation.[26] In dense membranes, diffusion process takes place through solution–diffusion mechanism. In this mechanism which is indirectly related to the thickness of the membrane, the selective penetrant dissolution in polymer matrix results in mass transport. In porous membranes, permeating species transfer by viscose flow mechanism. This process depends on the ratio of mean free pass of gas molecules to the size of pores. As SEM images (Fig. 2) shows, the produced electrospun mat contains a variety of pores with different sizes by which diffusion process could take place. Therefore, it can be considered that presence of pores makes the transfer through pores the dominant mechanism which is independent of membrane thickness. It seems reasonable to assume that transfer of water vapor molecules (2.7 Å in diameter) through pores ranging from 0.1 to 0.8 micrometer[24] is more probable to occur through pores and not according to solution-diffusion mechanism. Results of this work are in good agreement with some previous researchers.[12,18,20] These results indicate that PU electrospun mats would provide good resistance to the penetration of water, while still allowing significant water vapor transport to promote evaporative cooling of the body.

Mechanical properties

Figure 7 shows a typical stress–strain curve of PU nanofibrous mat samples. As it can be seen the curve is consisted of two regions; a nonlinear part and a quasi linear part. Nonlinear elastic and inelastic behavior of electrospun PU nanofiber mat is reported in different works.[15,16] In this work, stress–strain behavior of produced samples follows the same trend.

The results reveal that these layers are of high strength. This is attributed to the point bonding due to the residual solvent. The correlation between tenacity, breaking elongation, electrospinning duration, and thickness are given in Table 2. As the results demonstrate, there is a good correlation

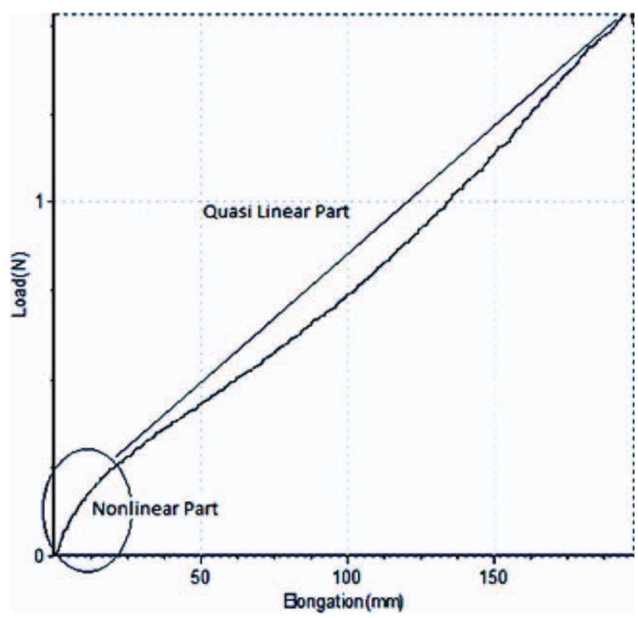

Figure 7 Typical stress–strain behavior of PU nanofiber web. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

TABLE II
Correlation Between Tenacity, Elongation, Electrospinning Duration, and Thickness

Variable	Electrospinning duration (h)	Max extension (%)	Tenacity (N)
Max extension (%)	0.353	1	0.460
Tenacity (N)	0.937	0.460	1
Thickness (μm)	0.989	0.444	0.928

between tenacity (*N*), electrospinning duration (h), and thickness (μm).

One of the most important limitations of most electrospun nanofibrous web is their poor strength.[6] As Table 1 shows, with increasing the electrospinning duration the tenacity of mat increases linearly. The regression equation between tenacity of mats and electrospinning duration is given below as:

$$\text{Tenacity}(N) = -1.12 + 0.782\ \text{Electrospinning Duration}(h) \quad (13)$$

The R^2 of the equation is >93%. There is not any significant relation between elongation and electrospinning duration. The value of tenacity in CD in all samples was more than MD and the relation between them is as following ($R^2 = 98\%$):

$$\text{Tenacity CD}(N) = 1.37\ \text{Tenacity MD}(N) \quad (14)$$

The value of strength in different directions of electrospun nanofiber mats is related to the orientation distribution of the nanofibers in various directions.[27,28] As Figure 3 shows, the fibers are more oriented in CD and subsequently the tenacity in this direction is more than MD. Fracture behavior of layers with process duration more than 9 h (10, 11, 12 h) during strain tests was indicative of the delimitation behavior of this layers.

CONCLUSIONS

The aim of this research is the assessment of some physical and mechanical properties of PU electrospun nanofiber mats that are essential for breathable windproof protective clothing system. The results showed that with increasing process duration, air permeability of electrospun nanofiber web was reduced, hydrostatic pressure increased and water vapor permeability remained unchanged. The results indicate that tenacity of PU mats changes linearly with the mat thickness while transport properties such as air permeability and hydrostatic pressure changes mat nonlinearly with the thickness. It was observed that air permeability behavior of layers follow the first Fick's law of diffusion. Moreover, it was concluded that the time intervals of 6 and 7 are the optimum levels and after this time the changes in air permeability and hydrostatic pressure are not pronounced. Considering this fact and the observation of layer delamination in time intervals of 10, 11, and 12 h, it seems that for improving the mass transfer and mechanical properties of layers, increasing electrospinning duration >6–7 h is not an effective solution. Instead, other methods such as post treatment should be taken into account. The results show that PU electrospun mat would provide good resistance to the penetration of water, while still allowing significant water vapor transport to promote evaporative cooling of the body.

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