(REFEREED RESEARCH)

EFFECT OF AMBIENT PARAMETERS ON MORPHOLOGY OF ELECTROSPUN POLY (TRIMETHYLENE TEREPHTHALATE) (PTT) FIBERS

ÇEVRESEL PARAMETRELERİN ELEKTROSPİN YÖNTEMİYLE ÜRETİLMİŞ POLİTRİMETİLEN TEREFTALAT LİFLERİNİN MORFOLOJİSİ ÜZERİNDEKİ ETKİLERİ

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Received: 27.07.2016 Accepted: 03.02. 2017

ABSTRACT

In this study, the effect of relative humidity and temperature on the morphology of electrospun Poly(trimethylene terephthalate) (PTT) fibers is investigated. It is also examined the interaction between these ambient parameters and those of some important parameters such as charge density, tip to collector distance and flow rate. Dichloromethane (DCM) and trifluoroacetic acid (TFA) solvent mixture is used. Field emission scanning electron microscopy (FESEM) is used for determination of surface morphologies and average diameters of electrospun PTT fibers. The results show that average diameter of electrospun PTT fibers decreases with increasing of relative humidity and temperature values for all of the charge density, tip to collector distance and flow rate values. The highest rate of decrease is seen at 0.9 ml/h for increasing of RH from 30% to 70%. All of the obtained electrospun PTT fibers are circular shaped with smooth surface without bead formation for all RH and temperature values.

Keywords: Electrospinning, Polytrimethylene terephthalate, Morphology, Ambient temperature, Relative humidity, Process parameters

ÖZET

Bu çalışmada bağıl nemin ve ortam sıcaklığının elektrospin tekniğiyle üretilen politrimetilen tereftalat (PTT) liflerinin morfolojileri üzerindeki etkileri incelenmiştir. Bununla birlikte bu çevresel parametrelerin; birim yük, toplayıcı-iğne ucu arası mesafe ve debi gibi önemli proses parametreleriyle etkileşimleri de incelenmiştir. Çözücü olarak diklormetan (DCM) ve triflor asetikasit karışımı kullanılmıştır. Elektrospin yöntemiyle üretilen PTT liflerinin yüzey morfolojileri ve ortalama çaplarının analizi için alan emisyonlu taramalı elektron mikroskobu (FESEM) kullanılmıştır. Elde edilen sonuçlara göre incelenen her üç proses parametresi (birim yük, toplayıcı-iğne ucu arası mesafe ve debi) için hem bağıl nemin hem de sıcaklığın artması elektrospin tekniğiyle üretilen PTT liflerinin ortalama çaplarını azaltmaktadır. En yüksek azalma oranı 0.9 ml/sa debide bağıl nemin 30%'dan 70%'e çıkmasında görülmüştür. Tüm bağıl nem ve tüm sıcaklık değerleri için elektrospin tekniğiyle üretilen PTT liflerinin hepsinin dairesel yapıda, düzgün yüzeyli ve boncuksuz olduğu görülmüştür.

Anahtar Kelimeler: Elektrospin, Politrimetilen tereftalat, Morfoloji, Sıcaklık, Bağıl Nem, Proses parametreleri.

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1. INTRODUCTION

Poly(trimethylene terephthalate) (PTT), belonging to the linear aromatic polyester family and having a bio-based origin, attracts commercial interests in engineering and textile applications [1, 2]. PTT is a polymer with a melting temperature of about 228 °C and a glass transition temperature of about 50 °C. The tenacity, moisture regain and elastic recovery (5% strain) of PTT fibers are 4-5 g/denier, 0.2 %-0.3 % and 100%, respectively [3, 4]. PTT, having odd-numbered methylenes in the polymer chains, has higher resilience and elastic recovery than those of poly(ethylene terephthalate) (PET) and poly(butylene terephthalate) (PBT). Therefore PTT is used widely in fabric requiring good resilience especially in carpets [3, 4]. Many studies have been performed about intrinsic properties, fiber properties, spinning techniques of PTT [4-6]. Also there is a rising academic interest on PTT nanofibers produced by electrospinning and other techniques [7-11].

Nanofibers can be used in different application areas such as filtration, biomedical fields, etc. due to their advantageous properties such as small diameters, high-specific surface area and high porosity [12-14]. Among all of the nanofiber production methods, electrospinning is the most prominent method due to its simplicity, repeatability, low cost etc [15, 16].

In electrospinning, three groups of parameters such as polymer parameters, process parameters and ambient parameters affect morphology and diameter of nanofibers [17]. The limited number of studies on ambient parameters in electrospinning method has been performed. In these studies, relative humidity (RH) [18-22], ambient temperature [23,24], and temperature and RH together [25-27] are focused on. It is observed that RH and temperature have important effects on both diameter and shape of electrospun nanofibers.

The diameter of polyamide 4.6, polyamide 6.9 [18] polyethylene oxide (PEO) [19,22] and poly(vinylpyrrolidone) (PVP) [25] decreases, the diameter of cellulose acetate (CA) [25,26] and polyetherimide (PEI) [27] increases with increasing relative humidity. Increasing of ambient temperature from 25 ºC to 75 ºC changes morphology of electrospun silk nanofibers from circular to flat [23]. Also increasing of ambient temperature decreases average diameter of poly (vinylidene fluoride) (PVDF) nanofibers [24].

There is no study on ambient parameters for electrospun PTT fibers. In this study, therefore, the effects of relative humidity and ambient temperature by varying some important parameters such as charge density (CD), tip to collector distance (TCD) and flow rate on average diameter and morphology of electrospun PTT fibers are investigated by using six different temperatures and relative humidity values. Charge density, tip to collector distance and flow rate according to ambient parameters are also investigated. Field emission scanning electron microscopy (FE-SEM) method is used for morphological investigation of electrospun PTT fibers.

2. MATERIALS AND METHODS

2.1. Materials

Poly(trimethylene terephthalate) (PTT, density:1.3 g/cm^3, melting point:228 ºC) is supplied from DuPont Co. Ltd., USA. Trifluoroacetic acid (TFA) and dichloromethane (DCM) (purchased from Sigma Aldrich Co.) with 50/50 volume ratio are used to prepare the polymer solution at a concentration of 16 wt%. The PTT solution is prepared at room temperature by stirring magnetically overnight.

2.2. Electrospinning Set-up and Fabrication

The electrospinning apparatus developed for controlling ambient temperature and relative humidity is described in detail our previous studies [27, 32]. The fan which is used for obtaining homogeneous relative humidity and temperature in the cabin turned off during the electrospinning. The cabin and all of the other equipment are in a temperature controlled room by an air conditioner. Nanofibers are collected on the aluminum foil. All air bubbles are purged prior to electrospinning. The needle diameter is selected as 0.7 mm (22G needle). The other electrospinning parameters are given in Table 1. After investigation of the related studies, the values of concentration, applied voltage and TCD are selected for the steady state electrospinning of PTT judged by visually checking the Taylor cone's stability [7, 10]. The deposition time is selected as 90 s due to the fact that RH and temperature in the cabin is not changed significantly.

2.3. Characterization

Electrical conductivity, surface tension and viscosity of the PTT solution are determined by Orion 4 Star Plus meter, Attention Theta optical tensiometer and Brookfield DV-III Ultra rheometer, respectively. The morphological appearances of the electrospun PTT fibers are investigated by using a field emission scanning electron microscope (FE-SEM, Zeiss Supra 55) after platinum coating with an ion sputter. Image-Pro Plus 6.0 program is used for measurement of diameters of electrospun PTT fibers. Average diameters of nanofibers are calculated by taking the average of sixty measurements.

2.4. Statistical Analyses

The data obtained is subjected to statistical analyses of variance (ANOVA) and Pearson Correlation Test to assess the effects of ambient temperature and RH on morphology of electrospun PTT fibers using SPSS statistical software (IBM SPSS Statistics 22.0.0 trial version; IBM, Chicago, USA). For all statistical tests, the results are considered significant at $p \leq 0.01$.

3. RESULTS AND DISCUSSION

Viscosity, conductivity and surface tension values (at 25ºC) of the PTT solution (16wt%) are determined as 1340 cP, 6.72 µS/cm and 18.14mN/m, respectively.

3.1. Effect of ambient temperature and relative humidity (Group 1)

FESEM images of electrospun PTT fibers produced at three different RH values (20%, 50% and 70%) and six different temperature values (10 ºC, 15 ºC, 20 ºC, 25 ºC, 30 ºC, 35 ºC) were shown (Figure 1). The obtained electrospun PTT fibers are circular shaped with smooth surface for all RH and temperature values. Also no bead formation is seen for all of the electrospun PTT fibers. Those morphological properties can be explained by surface tension and high viscosity of the solution. It is known that high surface tension and low viscosity cause bead formation [24]. At 10 ºC and 20% RH, PTT nanofibers are obtained closely adherent in helical form. It is also observed that adherence level decreases by increasing of temperature especially for low RH values.

Table 1. The investigated parameters in electrospinning of PTT

Group Number	CD (kV/cm)	TCD (cm)	Flow rate (ml/h)	Temperature (ºC)	RH (%)
1	1.5	13	0.6	10-15-20-25-30-35	20-30-40-50-60-70
2	1.0-1.5-2.0	13	0.6	15-25-35	30-50-70
3	1.5	10-13-16	0.6	15-25-35	30-50-70
4	1.5	13	0.3-0.6-0.9	15-25-35	30-50-70

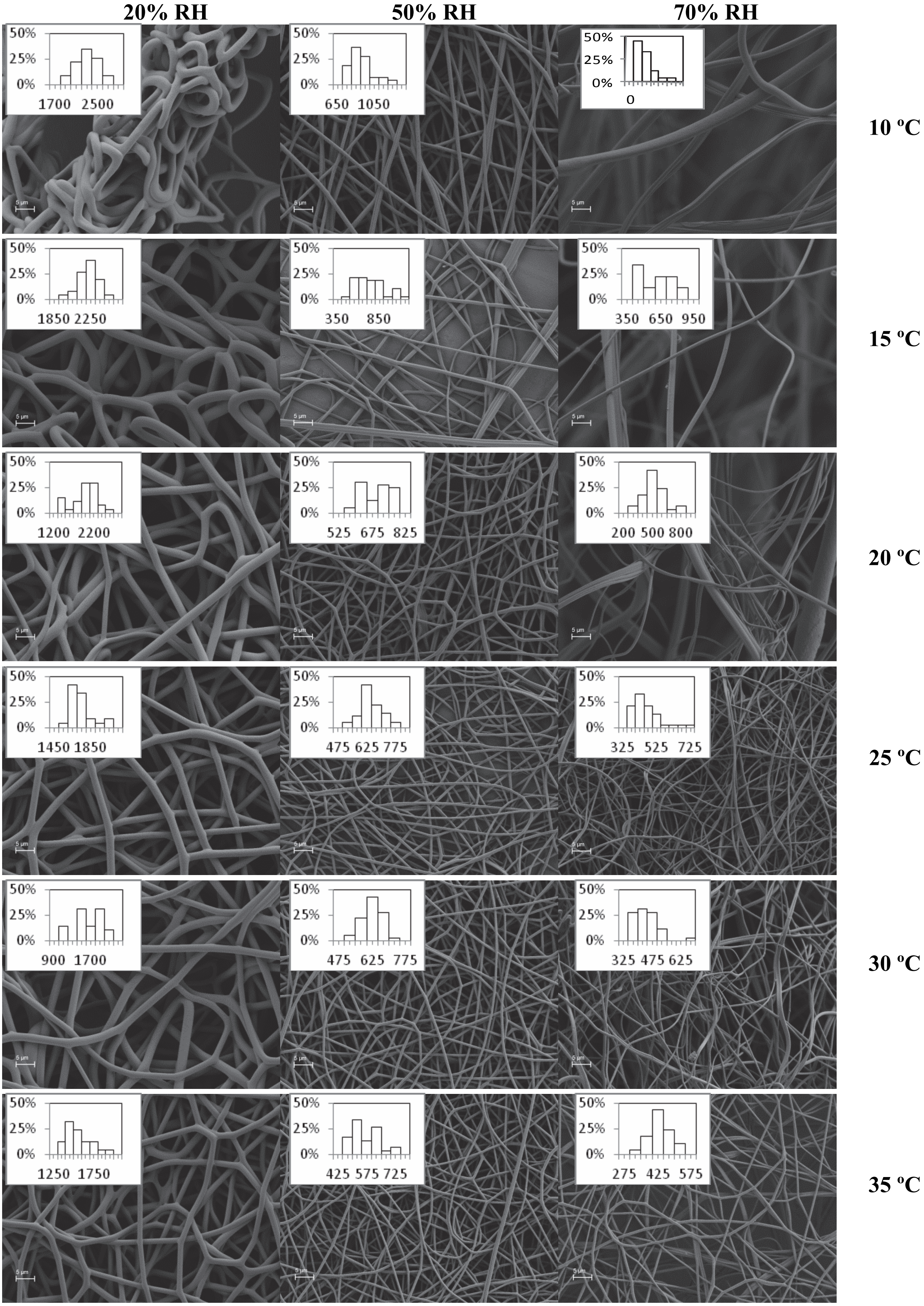

Figure 1.FESEM images of electrospun PTT fibers

Figure 2 shows the relationship between the average fiber diameter and RH for all of the ambient temperature values. Also Table 2 shows the average diameter results of the fibers according to RH and temperature.

Average diameter of electrospun PTT fibers decreases with increasing relative humidity (Figure 2). At higher RH, water amount in the air is high and that slows the solidification process so flight time and elongation of the jet get longer, resulting in thinner fiber formation. These results are similar to PEO, PVP, PA6, PA4.6, PA6.9 polymers [22, 25, 18, 28, 29]. It is also clearly seen that the average diameter of electrospun PTT fibers decreases with increasing temperature (Table 2). Solution viscosity and surface tension decrease with the increase of temperature [24, 30]. Lower surface tension and viscosity reduce the resistance to electric force applied; hence stretching of the polymer solution jet increases, resulting in thinner fibers. The similar results are observed for electrospun PVDF, CA and PEI fibers [24, 26, 27]. According to the statistical analysis of variance (ANOVA), temperature and RH are significantly effective (p ≤ 0.01) on the diameter of the electrospun PTT fibers. Also it is obtained that increasing of RH increases the diameter significantly, while increasing of temperature decreases the diameter significantly according to Pearson Correlation Test (p ≤ 0.01).

3.2. Effect of charge density (CD) at different ambient conditions(Group 2)

The PTT solution (16 wt%) is electrospun under CD of 1 kV/cm, 1.5 kV/cm, 2 kV/cm at ambient temperature of 15 ºC, 25 ºC, 35 ºC and RH of 30%, 50%, 70%. The TCD is constant at 13 cm. Figure 3, which is given as an example at 15 ºC, shows FESEM images of electrospun PTT fibers under different relative humidity and CD values. The obtained electrospun PTT fibers are circular shaped with smooth surface for all CD values. Also no bead formation is seen for all of the electrospun PTT fibers.

Table 3 shows the average diameter results of electrospun PTT fibers as a function of the CD and the ambient parameters. Firstly it is observed that the average diameter of the fibers decreases with increase in CD. Increase in CD means higher applied voltage for the same TCD on polymer jet. So elongation of the jet increases due to more stretching and thinner fibers are obtained [31]. The average diameter of the electrospun PTT fibers decreases with increasing of RH and temperature for the different CD values. Also the rates of decrease are similar for all CD values. According to the statistical analysis of variance (ANOVA), temperature-CD and RH-CD together are significantly effective (p ≤ 0.01) on the diameter of the electrospun PTT fibers.

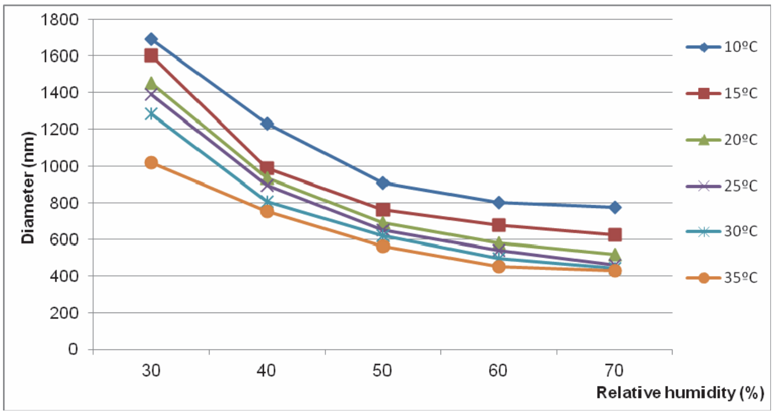

Figure 2. Average diameter of electrospun PTT fibers according to relative humidity

Table 2. The average diameters (nm) of electrospun PTT fibers according to temperature and relative humidity (16 wt%, 1.5 kV/cm and 0.6 ml/h)

Temperature (ºC)	Relative Humidity					
	20%	30%	40%	50%	60%	70%
10	2291±209	1692±106	1230±93	909±131	801±448	773±427
15	2225±112	1601± 60	988±86	761±209	676± 90	626±124
20	1964±311	1454±216	936±91	692± 62	582± 65	520±118
25	1756±122	1392±111	894±96	651± 61	541± 66	461± 86
30	1666±306	1284±397	804±61	621± 47	496± 33	441± 69
35	1553±156	1020±160	753±70	564± 70	451± 50	429± 48

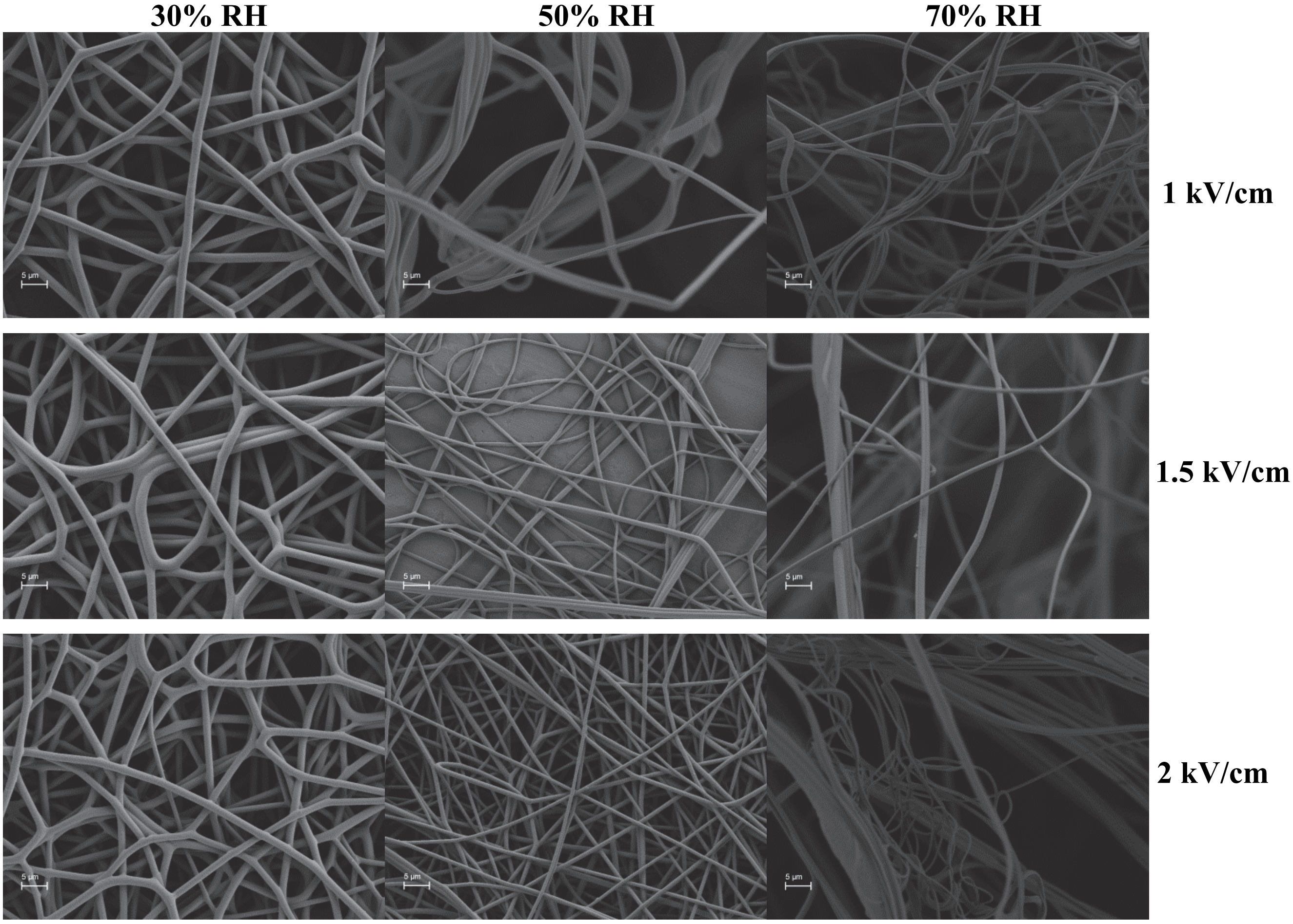

Figure 3. FESEM images of electrospun PTT fibers under different relative humidity and CDvalues (Flow rate: 0.6 ml/h, TCD: 13 cm, Temperature: 15ºC).

Table 3. The average diameters (nm) of electrospun PTT fibers as a function of the CD and the ambient parameters (16 wt%, 0.6mL/h and 13 cm)

Temperature (ºC)	CD (kV/cm)	Relative Humidity				
		30%		50%		70%
15	1.0	1690 ± 132		984 ± 362		703 ± 167
	1.5	1601 ± 60		761 ± 209		626 ± 124
	2.0	1438 ± 158		692 ± 76		503 ± 116
25	1.0	1490 ± 202		833 ± 116		539 ± 133
	1.5	1392 ± 111		651 ± 61		461 ± 86
	2.0	1308 ± 89		568 ± 46		416 ± 54
35	1.0	1378 ± 124		780 ± 71		449 ± 104
	1.5	1020 ± 160		564 ± 70		429 ± 48
	2.0	832 ± 137		501 ± 47		408 ± 64

3.3. Effect of tip-collector distance (TCD) at different ambient conditions (Group 3)

The PTT solution (16 wt%) is electrospun at TCD of 10 cm, 13 cm and 16 cm under ambient temperature of 15 ºC, 25 ºC, 35 ºC and RH of 30%, 50%, 70%. The solution concentration, flow rate and CD are kept constant at 16 wt%, 0.6 ml/h and 1.5 kV/cm, respectively. Figure 4, which is given as an example at 25 ºC, shows FESEM images of electrospun PTT fibers under different relative humidity and TCD values. The obtained electrospun PTT fibers are circular shaped with smooth surface for all TCD values. Also no bead formation is seen for all of the electrospun PTT fibers.

Table 4 shows the average diameters of the electrospun PTT fibers as a function of the TCD and the ambient parameters. Firstly it is observed that the average diameter of the fibers decreases with increase in TCD. Increase in TCD means higher flight time for the same CD on polymer jet. So elongation of the jet increases and thinner fibers are obtained. The average diameter of the electrospun PTT fibers decreases with increasing of RH and temperature for the different TCD values. Also the rates of decrease are similar for all TCD values. According to the statistical analysis of variance (ANOVA), temperature-TCD and RH-TCD together are significantly effective ($p \leq 0.01$) on the diameter of the electrospun PTT fibers.

3.4. Effect of flow rate at different ambient conditions (Group 4)

The PTT solution (16 wt%) is electrospun at flow rate of 0.3 mL/h, 0.6 mL/h and 0.9 mL/h under ambient temperature of 15 ºC, 25 ºC, 35 ºC and RH of 30%, 50%, 70%. The solution concentration, CD and TCD are kept constant at 16 wt%, 1.5 kV/cm and 13 cm, respectively. Figure 5, which is given as an example at 35 ºC, shows FESEM images of electrospun PTT fibers under different relative humidity and flow rate values. The obtained electrospun PTT fibers are circular shaped with smooth surface for all flow rate values. Also no bead formation is seen for all of the electrospun PTT fibers.

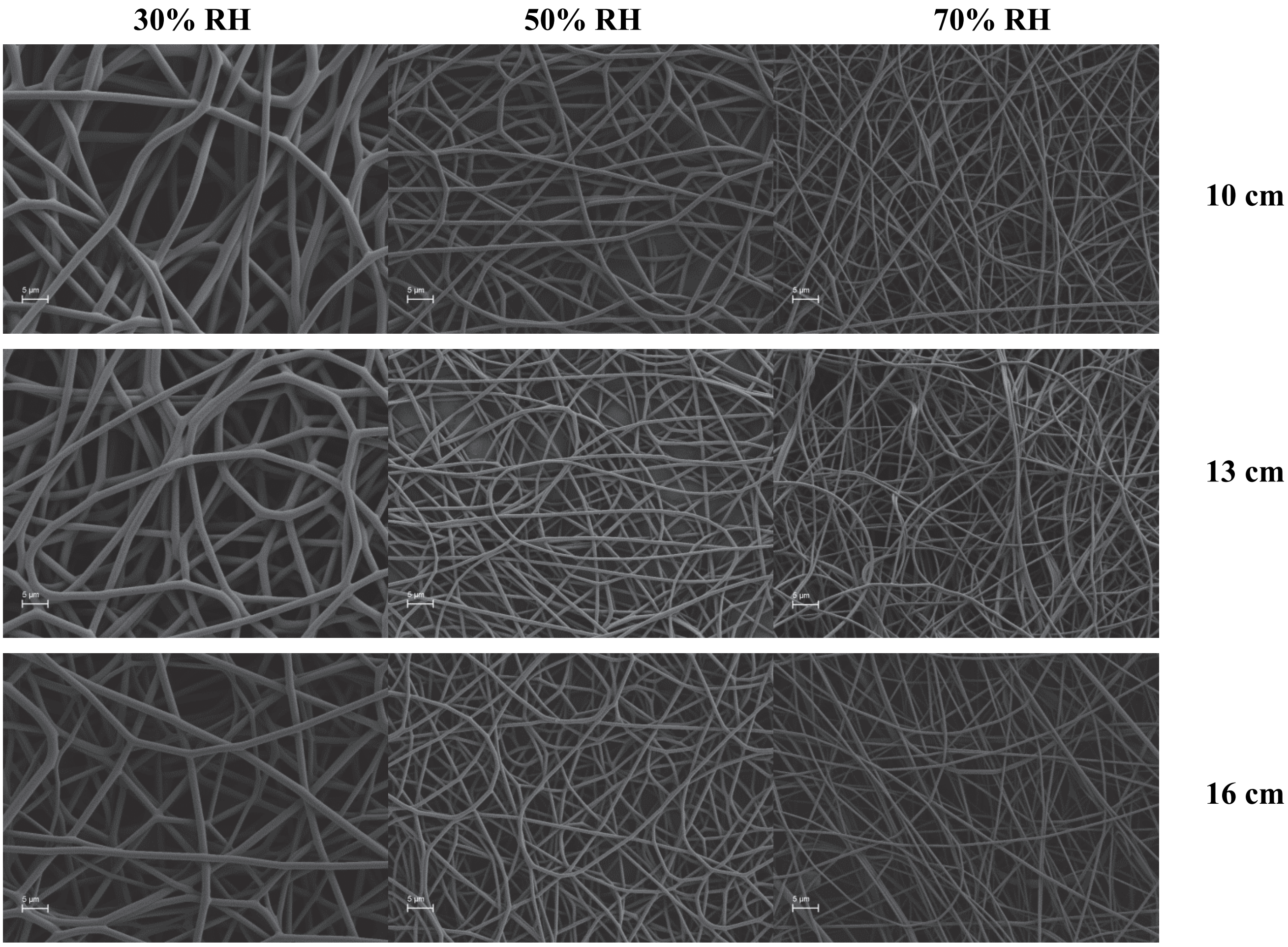

Figure 4. FESEM images of electrospun PTT fibers under different relative humidity and TCD values (Flow rate: 0.6 mL/h, CD: 1.5 kV/cm, Temperature: 25ºC).

Table 4. The average diameters (nm) of electrospun PTT fibers as a function of the TCD and the ambient parameters (16 wt%, 0.6ml/h and 1.5 kV/cm)

Temperature (ºC)	TCD (cm)	Relative Humidity				
		30%		50%		70%
15	10	1722 ± 162		856 ± 114		683 ± 275
	13	1601 ± 60		761 ± 209		626 ± 124
	16	1511 ± 110		706 ± 71		507 ± 82
25	10	1548 ± 138		770 ± 33		498 ± 146
	13	1392 ± 111		651 ± 61		461 ± 86
	16	1231 ± 253		566 ± 40		414 ± 56
35	10	1189 ± 222		673 ± 94		464 ± 94
	13	1020 ± 160		564 ± 70		429 ± 48
	16	947 ± 65		500 ± 105		398 ± 59

Table 5 shows the average diameters of the electrospun PTT fibers as a function of the flow rate and the ambient parameters. Firstly it is observed that the average diameter of the fibers increases with increase in flow rate. This is related to amount of polymer solution given in a defined time. Increase in flow rate means higher amount of solution is given for electrospinning. The effect of applied voltage for per unit of polymer jet decreases and less stretching of polymer jet is seen. So, thicker fibers are obtained. The average diameter of the electrospun PTT fibers decreases with increasing of RH and temperature for the different flow rate values. The rates of decrease are different for TCD values. The highest rate of decrease is seen at 0.9 ml/h for increasing of RH from 30% to 70%. For instance, from 30% RH to 70% RH at 35 ºC, about 69% decrease in average diameter is seen for 0.9 ml/h, while the decrease rates for 0.3 ml/h and 0.6 ml/h are 51% and 58%, respectively. According to the statistical analysis of variance (ANOVA), temperature-flow rate and RH-flow rate together are significantly effective (p ≤ 0.01) on the diameter of the electrospun PTT fibers.

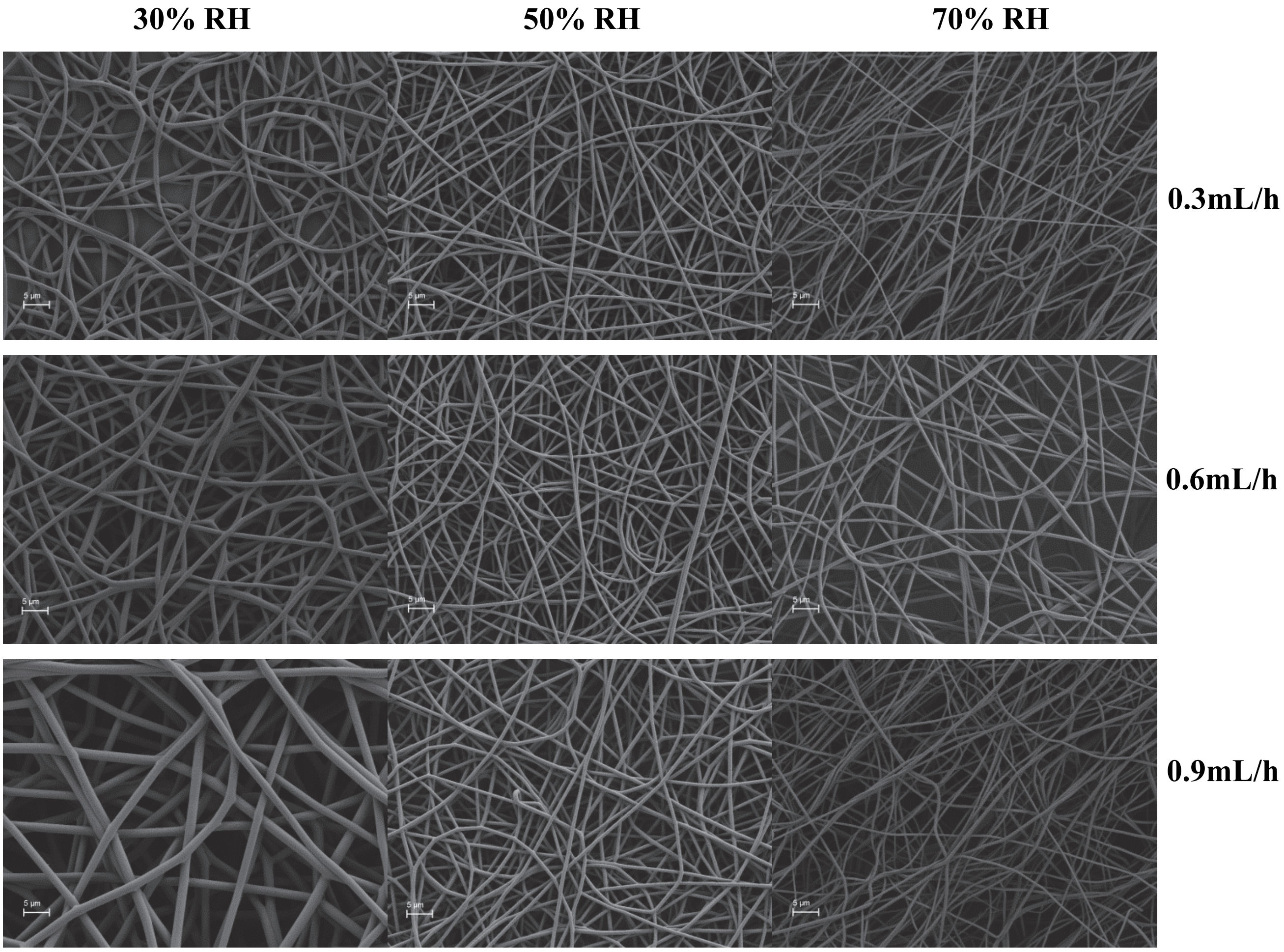

Figure 5. FESEM images of electrospun PTT fibers under different relative humidity and flow rate values (CD: 1.5 kV/cm, TCD: 13 cm, Temperature: 35ºC).

Table 5. The average diameters (nm) of electrospun PTT fibers as a function of the flow rate and the ambient parameters (16 wt%, 1.5 kV/cm and 13 cm)

Temperature (ºC)	Flow Rate (mL/h)	Relative Humidity				
		30%		50%		70%
15	0.3	1074 ± 137		652 ± 84		445 ± 85
	0.6	1601 ± 60		761 ± 209		626 ± 124
	0.9	1805 ± 182		925 ± 129		651 ± 236
25	0.3	790 ± 69		518 ± 38		402 ± 49
	0.6	1392 ± 111		651 ± 61		461 ± 86
	0.9	1602 ± 141		749 ± 65		534 ± 154
35	0.3	756 ± 92		494 ± 40		373 ± 43
	0.6	1020 ± 160		564 ± 70		429 ± 48
	0.9	1535 ± 116		610 ± 33		481 ± 102

CONCLUSION

In this study, effects of ambient temperature and RH by varying some important parameters such as charge density, tip to collector distance and flow rate on morphology of electrospun PTT fibers are investigated. Average diameter of the electrospun fibers decreases with increasing of RH and temperature. Slowing of solidification process by increasing of RH and decreasing of surface tension and viscosity by increasing of temperature can be related to the decrease in diameter of the electrospun PTT fibers. The average diameter of the electrospun PTT fibers decreases with increasing of RH and temperature for the different CD, TCD and flow rate values. The rates of decrease are similar for CD, TCD values, while different for flow rate values. For the flow rate of 0.9 ml/h, the highest rate of decrease is seen from 30% RH to 70% RH. All of the obtained electrospun PTT fibers are circular shaped with smooth surface without bead formation for all RH and temperature values.

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