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Effects of processing parameters on morphology of electrospun polystyrene nanofibers

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Abstract−This study focused on the preparation of electrospun polystyrene (PS) nanofibers. PS solutions were prepared in single (dimethylformamide; DMF, dimethylacetamide; DMAc or tetrahydrofuran; THF) and mixed solvent (DMF/THF and DMAc/THF) systems with and without tetrabutylammonium bromide (TBAB) salt. The effects of PS concentration, solvent system, the addition of salt, appearance and diameter of PS fibers were examined. The average diameter of the as-spun fibers increased upon increasing PS concentration. The morphology of the fibers significantly depended on the properties of the solvents. The obtained fibers were smooth without any beads and their diameters were affected by the amount of THF in the solvent and PS concentration. The beads in the fibers disappeared and the fiber diameter significantly decreased after the addition of TBAB. The smallest diameter and the narrowest diameter distribution of PS nanofibers (376±36 nm) were obtained from 15% PS solution in DMAc with 0.025% w/v TBAB.

Key words: Electrospinning, Nanofibers, Polystyrene, Salt Concentration

INTRODUCTION

Nanofibers are interesting and used for many applications in various fields such as drug delivery carrier, tissue engineering scaffolding, wound dressing, filter media, nanosensors, cosmetic skin masks and protective clothing. [1] The development of electrospinning techniques has been increasingly investigated. Electrospinning is a simple and easy way to control the morphology of ultrafine fibers. The fibers produced by this method have shown amazing characteristics, such as a very large surface-to-volume ratio and a high porosity with a small pore size. In the electrospinning process, high voltage is applied to a capillary containing a polymer solution or the molten polymer precursor. A droplet of the polymer solution then forms at the tip of the capillary, creating a point known as the “Taylor cone.” As electrostatic forces overcome the surface tension of the polymer solution, the solution is ejected from the apex of Taylor cone. The charged jets of polymer solution move towards a collector, solvent rapidly evaporates and non-woven fiber mat is collected on the collector [1-3]. Many parameters affect the nanofiber morphology such as the polymer parameters (type, molecular weight), the solution parameters (polymer concentration, viscosity, conductivity, surface tension), the processing parameters (applied voltage, distance between the capillary tip and collector, flow rate of the polymer solution), and the ambient parameters (temperature and humidity). In the electrospinning process, all parameters have to be carefully considered for optimizing nanofiber morphology [1-4].

Polystyrene (PS) is one of the most widely used polymers in thermoplastic materials because it has stiffness, gloss, hardness and brilliance and is a long chain hydrocarbon with every other carbon connected to a phenyl group. The major applications of polystyrene are beakers, transparent food packaging, audio/video cassette packs, lamp covers, etc [5]. Polystyrene nanofibers are successfully produced by the electrospinning method [6-10] and it has been demonstrated that electrospun PS fibers have interesting applications in many areas such as tissue engineering [11], filtration [12], enzyme immobilization [13], sensors [14], catalysis immobilization [15], composite materials [16] and prepared ion exchangers on PS fibers for fast water treatment in the field of filtration and separation science and for drug delivery [17]. The morphology of the electrospun PS fibers plays an important role in the physical/mechanical properties of the final products to be used in certain applications. Therefore, reproducible electrospinning of uniform PS fibers is essential.

In electrospinning, unexpected phenomenon and experimental results such as the formation of beads, necklace, ribbon-like and branched jet, etc. have been reported. The formation of beads within electrospun fibers has been observed often. Entov et al. [18] reported that bead formation occurs as a result of instability of the polymer solution jet. Reneker et al. [19], on the other hand, indicated that solution viscosity, surface tension and net charge density induced by the electrospinning are the main factors in the formation of beads during the formation of electrospun fibers. Bead generation is preferred at low concentration polymer solution, while higher concentrations lead to fiber formation. Recently, the effects of solvents and their properties on electrospinnability of the as-prepared PS solutions and morphological appearance of the obtained PS fibers were investigated and it was found that the PS solutions in 1,2-dichloroethane, *N,N*-dimethylformamide (DMF), ethylacetate, methylethylketone (MEK), and tetrahydrofuran (THF) could produce fibers with high enough productivity, while the PS solutions in benzene, cyclohexane, decalin, ethylbenzene, nitrobenzene, and tetralin were not spinnable [6]. Based on qualitative observation of the results obtained, the important factors determining the electro-spinnability of the as-prepared PS solutions are high enough values of both the

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dipole moment of the solvent and the conductivity of both the solvent and the resulting solutions, high enough boiling point of the solvent, and not-so-high values of both the viscosity and the surface tension of the resulting solutions. Kim et al. [7], using mixture of solvents THF and DMF, reported that the bead concentration was controlled by DMF content. The aspect ratio of the formed beads and the diameter of fibers were increased with increasing solution concentration. When PS was dissolved in only THF, an unexpected half hollow sphere (HHS) structure appeared. Uyar and Besenbacher [9] report that among the solvents used--THF, chloroform and DMF, for electrospinning of PS--DMF was found to be the optimal solvent producing bead-free uniform electrospun PS fibers. The conductivity of the solvent was the key factor for the reproducible electrospinning of uniform PS fibers from various sources of DMF solutions. It was shown that even slight changes in the conductivity of the DMF solutions can greatly affect the morphology of the resulting electrospun PS fibers. Polymer solutions are prepared using solvents that have a higher conductivity or salts are added to solution to increase the conductivity. Tetrabutylammonium bromide (TBAB) [9], Hexadecyltrimethyl ammonium bromide (CTAB) [10], benzyltrialkylammonium chlorides [20], pyridine [21], sodium chloride (NaCl) [22-24], octadecyltrimetylammonium bromide (OTAB) [23], sodium phosphate (NaH_2PO_4) and potassium phosphate (NaH_2PO_4) [24] are used to add in the PS solution to increase the conductivity of the solvent. Although bead-free uniform electrospun PS fibers have been extensively reported when various solvent systems and type of salts added were tested, those experiments were separately performed by different investigators. No study has been conducted to demonstrate the effect of single and mixed solvents, the amount of salt as well as the diameter of capillary tip on the morphology of PS fibers in the same experiment. Therefore, the aim of this study was to optimize the parameters of the electrospinning process, including concentration of PS solution, single (DMF, DMAc or THF) and mixed solvent (DMF/THF and DMAc/THF) systems, inner diameter of capillary tip (0.5 mm and 0.9 mm) and the addition of salt (TBAB) in the PS solvent, for receiving bead-free uniform PS fibers with the smallest diameter and narrow fiber diameter distribution.

EXPERIMENT

1. Materials

The polystyrene (PS) resin used in this work was a general purpose grade (Mw=2.99×10^5 Da, Mn=1.19×10^5 Da, 685D, Dow Plastics, USA). Tetrabutylammonium bromide (TBAB) was purchased from Bio Basic INC, Canada. All of these solvents (*N,N* dimethylformamide (DMF, 99.8%, Brightchem Sdn Bhd, Malaysia), dimethylacetamide (DMAc, 99.5%, Sigma-Aldrich Chemical Company, USA) and tetrahydrofuran (THF, 99.5% VWR International, UK) were analytical research grade and used without further purification.

2. Preparation and Properties of Spinning Solutions

The PS solutions (10, 15, 20% w/v) were prepared by dissolving PS in single solvents (DMF, DMAc or THF) or mixed solvents (DMF/THF or DMAc/THF) at the ratio of 100/0, 75/25, 67/33, 50/50, 33/67, 25/75 and 0/100% v/v with and without the addition of salt (TBAB; 0.010, 0.025, 0.050, 0.10, 0.5 and 1.0% w/v). The conductivity of these PS solutions was measured by EUTECH ECtestr11$^+$ conductivity meter (Eutech Instruments Pte Ltd, Singapore). The

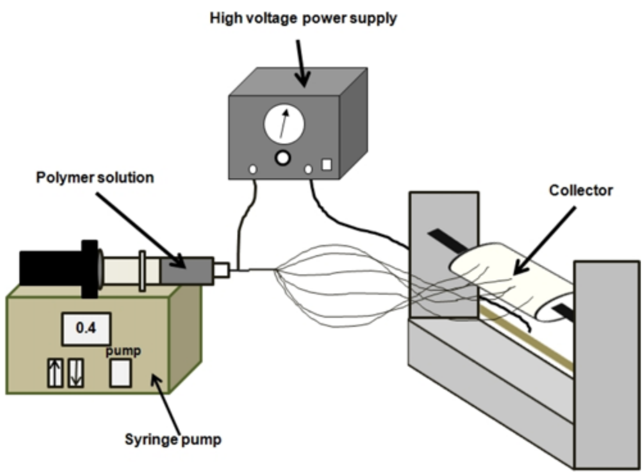


Fig. 1. Schematic representation of the electrospinning process setup.

Table 1. Conductivity and viscosity of 10, 15 and 20% w/v polystyrene solution in various ratios of DMF/THF and DMAc/THF

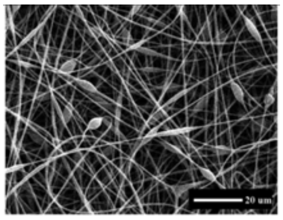
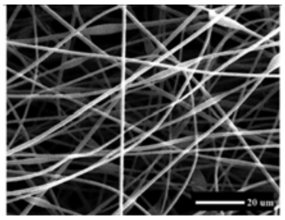
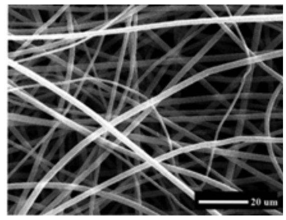
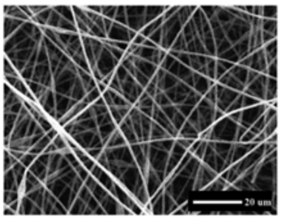
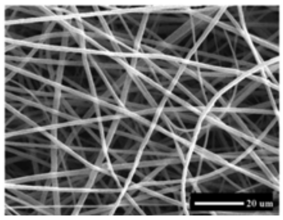
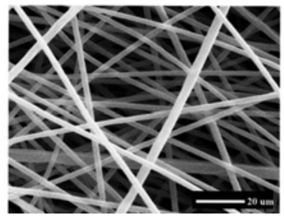
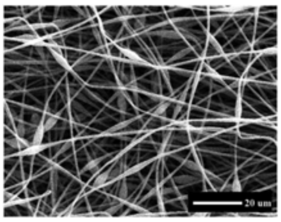
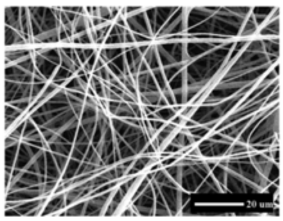
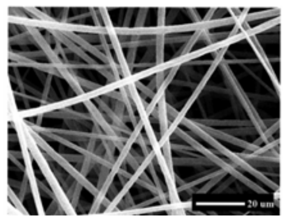
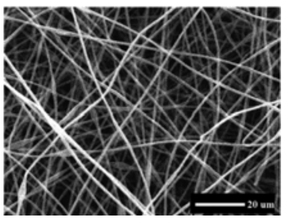
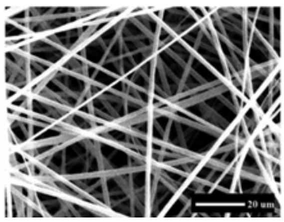
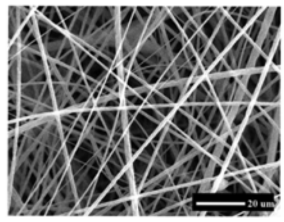
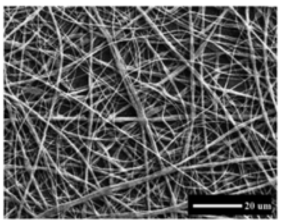
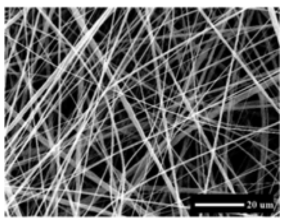
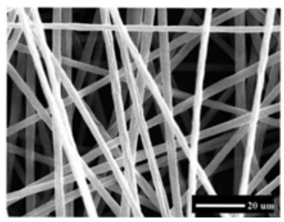
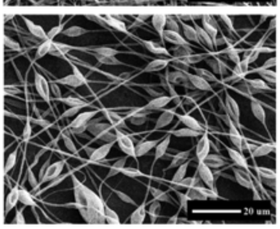
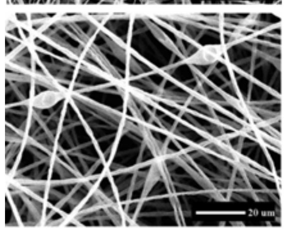
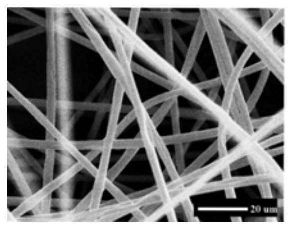
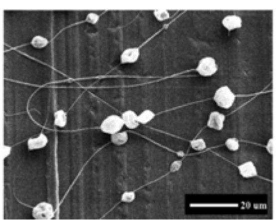
Solvent	Conductivity (μs/cm)			Viscosity (cp)		
	10 (%w/v)	15 (%w/v)	20 (%w/v)	10 (%w/v)	15 (%w/v)	20 (%w/v)
DMF/THF (100/0)	4.3±0.1	1.8±0.1	1.2±0.1	24.7±0.1	77.6±0.2	195.8±0.2
DMF/THF (75/25)	3.5±0.1	1.0±0.1	0.4±0.1	27.6±0.1	82.8±0.2	2004±0.2
DMF/THF (67/33)	3.3±0.1	0.6±0.1	0.2±0.1	27.5±0.3	88.2±0.2	212.3±0.4
DMF/THF (50/50)	3.0±0.1	0	0	25.3±0.1	88.0±0.3	214.4±0.6
DMF/THF (33/67)	2.8±0.1	0	0	28.3±0.2	91.0±0.3	209.4±0.5
DMF/THF (25/75)	2.3±0.1	0	0	30.4±0.1	93.3±0.2	320.8±0.7
DMF/THF (0/100)	0	0	0	33.0±0.1	113.3±0.3	264.7±0.3
DMAc/THF (100/0)	0.8±0.1	0	0	27.6±0.3	108.5±0.2	266.9±0.5
DMAc/THF (75/25)	0.7±0.1	0	0	30.5±0.2	105.1±0.2	252.1±0.4
DMAc/THF (67/33)	0.6±0.1	0	0	33.6±0.2	109.1±0.1	258.6±0.2
DMAc/THF (50/50)	0.7±0.1	0	0	28.6±0.3	104.8±0.4	263.1±0.5
DMAc/THF (33/67)	0.5±0.1	0	0	32.9±0.1	103.9±0.1	278.2±0.2
DMAc/THF (25/75)	0.3±0.1	0	0	34.1±0.1	100.9±0.5	331.2±0.7
DMAc/THF (0/100)	0	0	0	33.0±0.2	113.3±0.3	264.7±0.3

viscosity was determined with a Brookfield viscometer (DV-III ultra, Brookfield Engineering Laboratories, USA).

3. Electrospinning Process

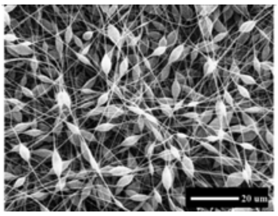
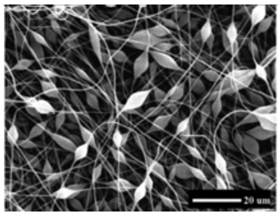
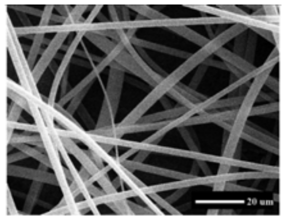
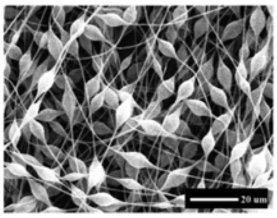
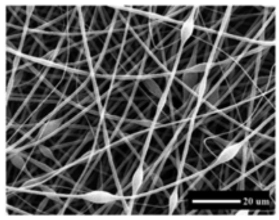
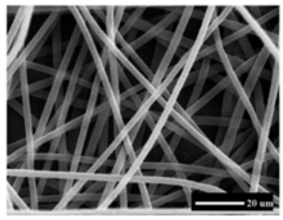
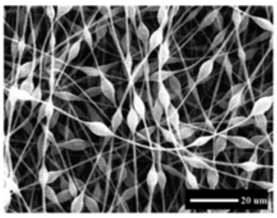
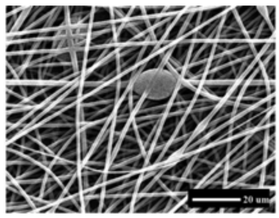
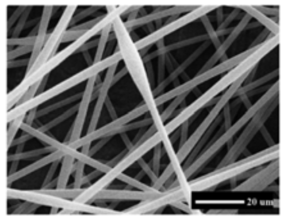
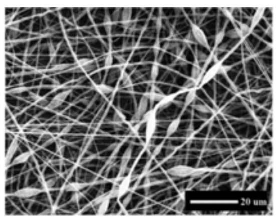
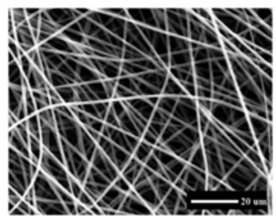
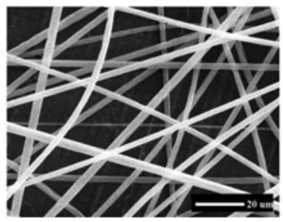
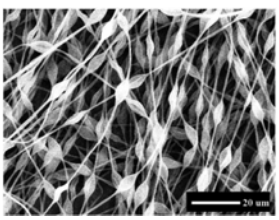
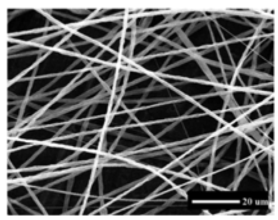
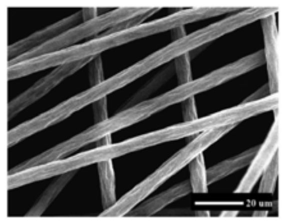
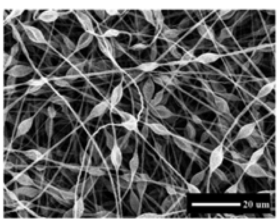
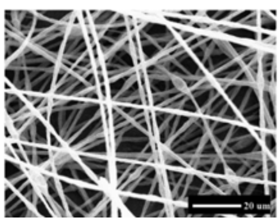
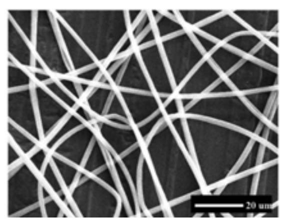
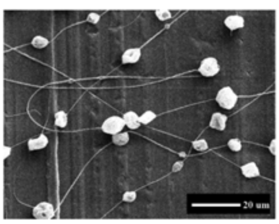
The electrospinning setup is shown in Fig. 1. The PS solution

Table 2. The scanning electron microscopy (SEM) images (500×) of electrospun polystyrene fibers obtained from 10, 15 and 20% PS solutions in the mixed solvents of DMF/THF

DMF/THF ratio	Concentration of polystyrent (PS) solution (% w/v)		
	10	15	20
100/0			
75/25			
67/33			
50/50			
33/67			
25/75			
0/100		-	-

was contained in a glass syringe with a plane tip of stainless steel needle with an inner diameter of 0.5 and 0.9 mm. The feeding rate of the PS solution into the tip was controlled at 0.4 ml/hr using the syringe pump (Model:NE-300, New Era Pump Systems Inc.). The

Table 3. The scanning electron microscopy (SEM) images (500×) of electrospun polystyrene fibers at 10, 15 and 20% PS solutions in the mixed solvents of DMAc/THF

DMAc/THF ratio	Concentration of polystyrent (PS) solution (% w/v)		
	10	15	20
100/0			
75/25			
67/33			
50/50			
33/67			
25/75			
0/100		-	-

applied voltage and the distance between the tip and the collector were fixed at 15 kV and 15 cm, respectively. The electrospun PS fibers were collected on aluminium foil that covered a rotating collector. The electrospinning process was conducted at room temperature (25 °C).

4. Characterization of the Polystyrene Fibers

The morphology and diameter of nanofiber mats were determined by using a scanning electron microscope (SEM, Camscan Mx2000, England). For this process, a small section of the electrospun fiber mats was sputtered with a thin layer of gold prior to SEM observation. The average diameter of the PS fibers was determined by using image analysis software (JMicroVision V.1.2.7, Switzerland).

The chemical structure of electrospun PS fibers was characterized by Fourier transform infrared spectrophotometer (FTIR, Nicolet 4700, USA) with a wave number range of 400-4,000 cm^{-1}.

5. Statistical Analysis

Data were collected from triplicate samples and are depicted as the mean±standard deviation (SD). The different fiber diameter with and without TBAB was analyzed using the independent samples t-test. The significance level was set at $p<0.05$.

RESULTS AND DISCUSSION

Many parameters affect the morphology of nanofibers such as the polymer parameters (type, molecular weight), the solution parameters (polymer concentration, viscosity, conductivity, surface tension), the processing parameters (applied voltage, distance between the capillary tip and collector, flow rate of the polymer solution, inner diameter of capillary tip), and the ambient parameters (temperature and humidity). Usually, three typical morphologies such as bead structure, bead-on-string structure and fiber structure can be obtained by adjusting electrospinning conditions. In this study, the concentration of PS solution, single (DMF, DMAc or THF) and mixed solvent (DMF/THF and DMAc/THF) systems, inner diameter of capillary tip (0.5 mm and 0.9 mm) and the addition of salt (TBAB) in the PS solvent were evaluated as follows.

1. Solution Parameters

PS solutions at the concentrations of 10, 15 and 20% w/v were prepared in single solvents (DMF, DMAc or THF) and mixed solvents (DMF/THF or DMAc/THF) with different volume ratios. The viscosity and conductivity of the PS solutions in these solvents were measured and are listed in Table 1. With an increasing PS concentration, the conductivity in all solvents decreased, whereas the viscosity increased. Among the single solvents used (DMF, DMAc, THF), DMF showed the highest conductivity but the lowest viscosity. For electrospinning of PS, DMF and DMAc were found to be the optimal solvent producing bead-free uniform electrospun PS fibers. PS in THF was clogged at the tip of the syringe during the electrospinning process. The clogging of the droplet solution at the tip was due to the fast evaporation of THF (low boiling point) [6]. SEM images showed that the DMF and DMAc single solvent yielded

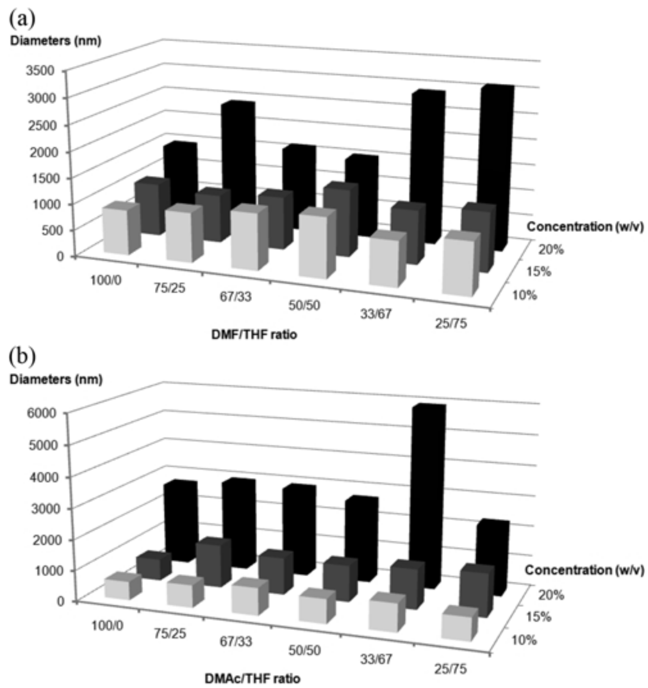


Fig. 2. Diameters of PS nanofiber mats with 10, 15 and 20% PS solution in different mixed solvent ratios (a) DMF/THF (b) DMAc/THF.

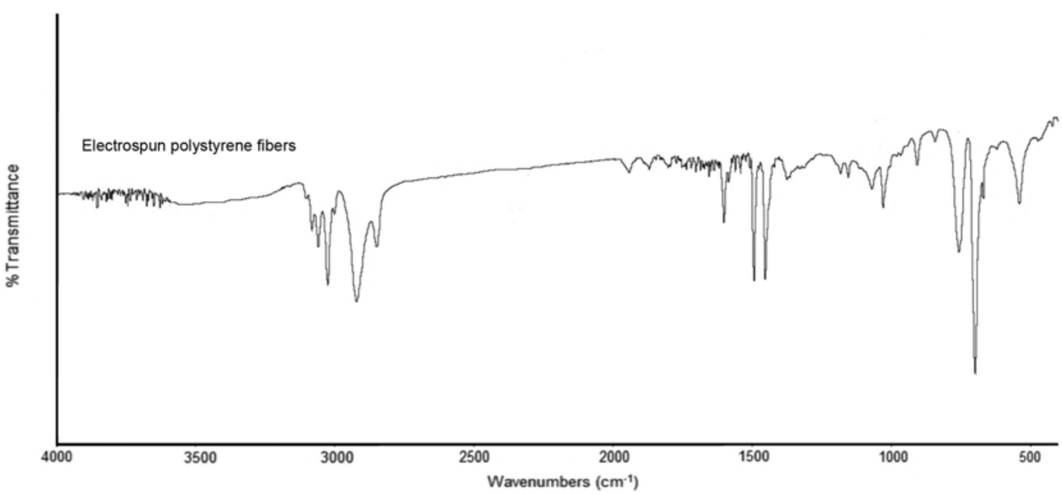


Fig. 3. FT-IR spectra of electrospun polystyrene fibers.

bead-free PS fibers when PS concentration was 20% w/v, but fibers with irregular structures were obtained when THF was used as solvent (Table 2, 3). These results are in good accordance with the previous reports where DMF was the most favorable solvent yielding uniform PS fibers due to its solution conductivity, high dielectric constant and high boiling point [6,7,9,25-27]. PS in THF solutions have no conductivity. At 10% w/v PS solution, bead-on-string structure was obtained from all solvents, probably due to the high content of the solvent which was incompletely evaporated before the fibers were collected at the collector. In comparison, the fiber fabricated from the mixed solvent of DMAc/THF showed bead-on-string structure in a higher extent than those from DMF/THF. This might be from the different boiling point among DMAc (165 °C), DMF (153 °C) and THF (66 °C) which affected the evaporation rate. During the electrospinning process, DMAc evaporated slower than DMF. Increasing PS concentration from 10 to 20% w/v decreased the amount of bead-on-string structure. The bead-free fibers were observed at 20% w/v PS solutions in all solvents. The diameters of the fibers are shown in Fig. 2. The average diameter of fibers increased when PS concentration increased from 10 to 20% w/v. This result was in accordance with a previous study which revealed that the average diameter of the fibers was increased due to a higher resistance of the solution to be stretched with electrostatic force in the electrospinning process when the concentration was increased [6].

In the mixed solvents, when the ratio of THF was more than 67% (33/67, 25/75 and 0/100), the half hollow beads (10% w/v PS solution) and the wrinkled fibers (15 and 20% w/v PS solution) were obtained. The half hollow structure occurred in many cases such as the mixed solvent, applied electric force. It might be that THF can absorb oxygen in air into beads during the electrospinning process [7]. The wrinkled fiber structure was observed due to the fast evaporation of THF. Therefore, the greater the ratio of THF, the more the wrinkled fibers structure obtained. The half hollow structure and the wrinkled fibers were decreased and changed to the smooth fibers when the portion of DMF or DMAc was increased in the mixed solvent due to the increase in the conductivity of the solution and high dielectric constant of both DMF and DMAc improving the electrospinning process [7]. Therefore, the optimized ratio of the mixed solvents of DMF or DMAc/THF was 100/0, 75/25 and 67/33. These ratios could produce the fiber structure without the clogging of the solution at the tip.

The FT-IR spectra of electrospun PS fibers (Fig. 3) present the principal peaks, i.e., C-H stretching of benzene ring at 3,100-3,000 cm^{-1}, overtone and combination bands at 1,945-1,745 cm^{-1}, ring

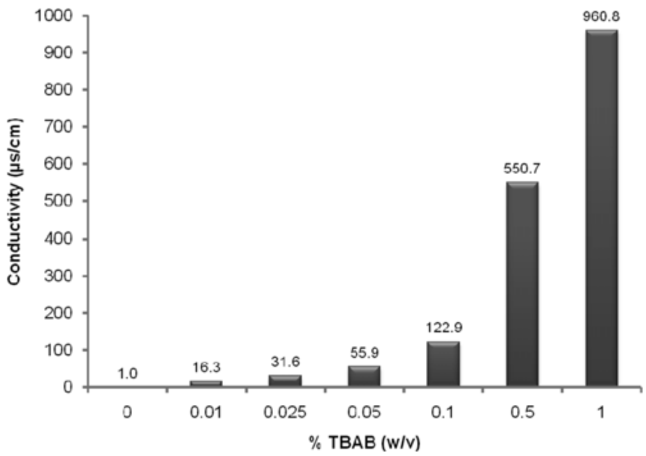


Fig. 4. Conductivity of 15% PS solution in DMF/THF (75/25) with various amount of TBAB.

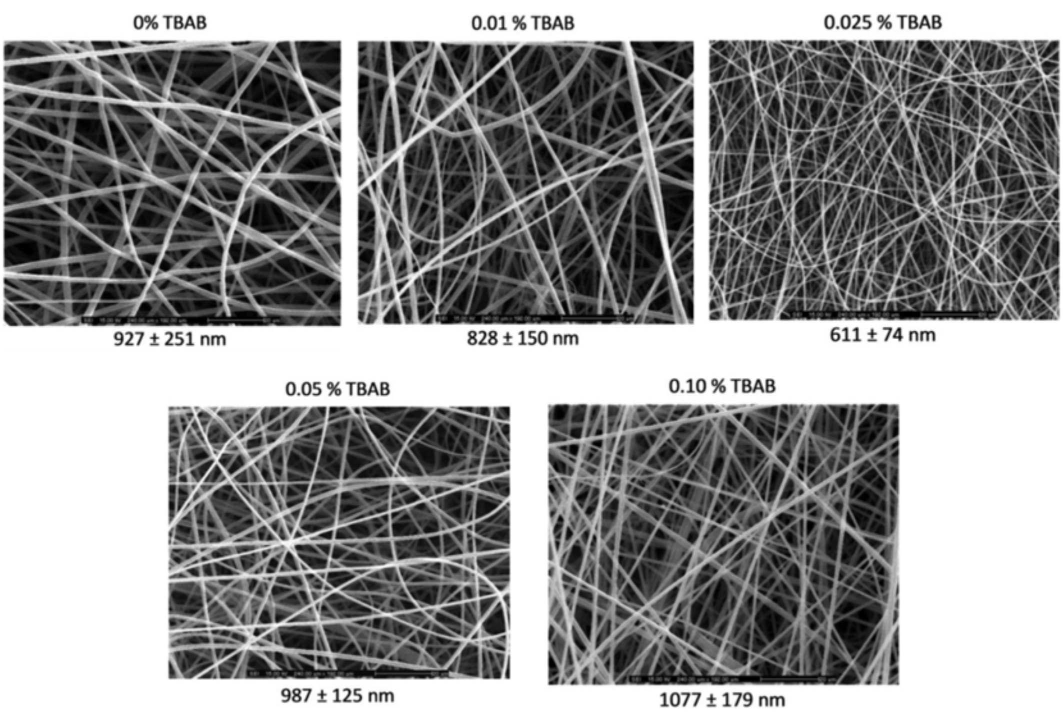


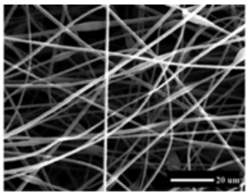
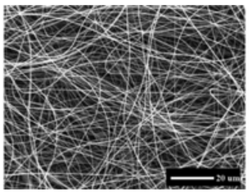
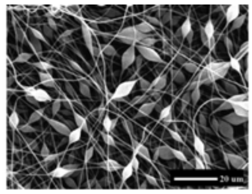
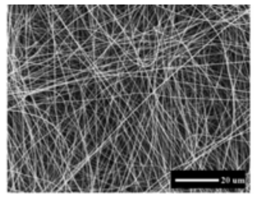
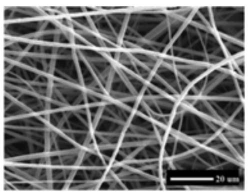
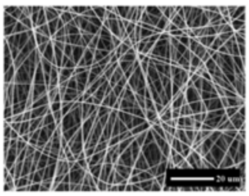
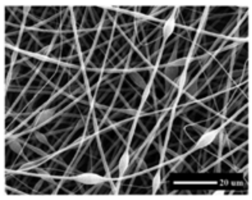
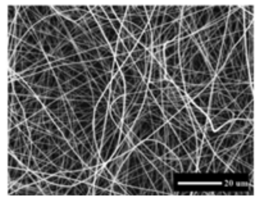
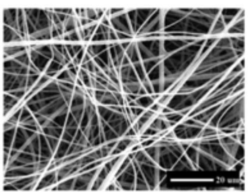
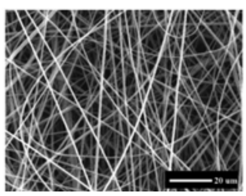
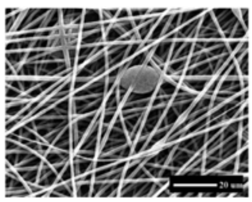
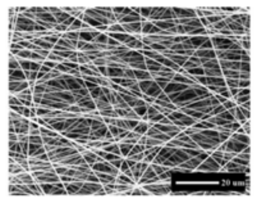
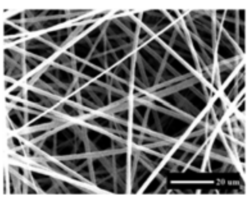
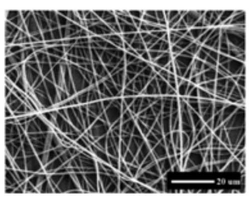
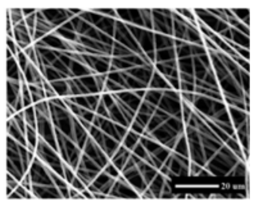
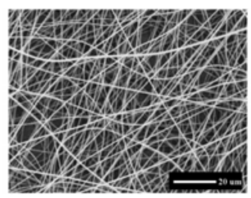
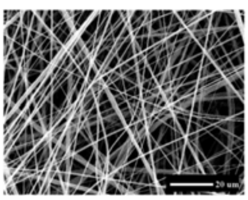
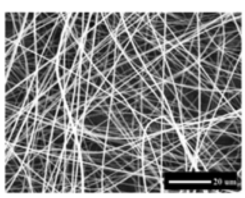
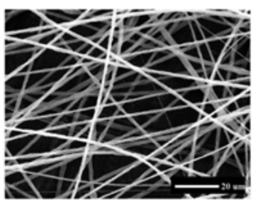
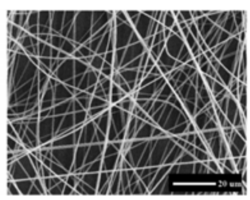
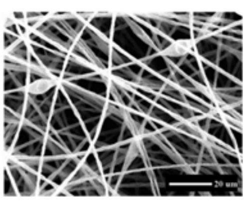
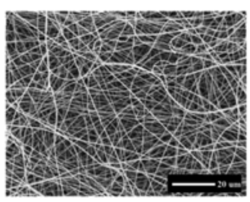
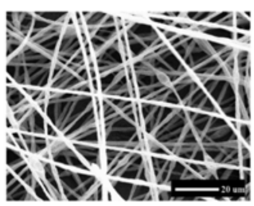
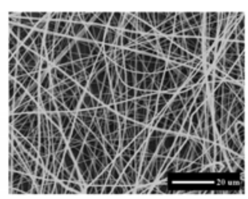
Fig. 5. The scanning electron microscopy (SEM) images (500×) and diameter of PS nanofiber mats obtained from 15% PS solution in DMF/THF (75/25) with various amount of TBAB.

stretching at 1,600-1,580 and 1,500-1,450 cm^{-1}, C-H in-plane bending at 1,300-1,000 cm^{-1}, C-H out-of-plane bending at 900-600 cm^{-1}, mono-substituted ring at 700 and 780 cm^{-1}.

2. The Processing Parameters

In the electrospinning process, the processing parameters such as applied voltage, distance between the capillary tip and collector, flow rate of the polymer solution and inner diameter of capillary tip affected the nanofibers morphology. Therefore, in this study the feeding rate of the PS solution into the tip, applied voltage, and the distance between the tip and the collector were fixed at 0.4 ml/hr, 15 kV and 15 cm, respectively. The inner diameters of capillary tip used in this experiment were 0.5 and 0.9 mm. We found that the diameter of PS fibers decreased from 2.55 μm to 1.10 μm and 3.30 μm to 1.40 μm for 15% and 20% w/v PS solution, respectively. In

Table 4. The scanning electron microscopy (SEM) images (500×) and diameter of electrospun PS fibers obtained from 15% PS solution with and without 0.025% TBAB in the mixed solvents of DMF/THF and DMAc/THF

Rations	DMF/THF		DMAc/THF	
	0% TBAB	0.025% TBAB	0% TBAB	0.025% TBAB
100/0	 1024±227 nm	 457±41 nm	 713±98 nm	 376±36 nm
75/25	 927±251 nm	 611±74 nm	 1410±184 nm	 516±72 nm
67/33	 1017±138 nm	 786±95 nm	 1206±152 nm	 606±52 nm
50/50	 1288±306 nm	 802±154 nm	 1187±108 nm	 643±55 nm
33/67	 1031±185 nm	 705±100 nm	 1303±196 nm	 941±156 nm
25/75	 1137±178 nm	 487±78 nm	 1406±217 nm	 666±128 nm

10% w/v PS solution, the bead size was decreased when the inner diameter of the capillary tip was decreased from 0.9 to 0.5 mm but the beads were still existed in the fibers. At 15% w/v PS solution, the bead-on-string structure was changed to fiber structure and the diameter of the fibers was decreased. At 20% w/v PS solution, the diameter of fibers was decreased. These results are similar to the previous study by Mo et al. [28] revealing that decreasing the needle diameter led to bead-free electrospun Poly(l-lactide-co-e-caprolactone) fibers and no clogging at the tip. Katti et al. [29] also reported that the diameter of poly(lactide-*co*-glycolide) nanofibers decreased with an increase in needle gauge from 16 to 20. In this study, we found that the clogging of the solution at the tip depended not only on needle diameter but also on the evaporation of the solvent used and concentration of polymer solution. Therefore, an inner diameter of capillary tip of 0.5 mm was selected to further experiment.

3. The Addition of Salt (TBAB) in the PS Solvents

The conductivity of polymer solution was increased when salts were added in the solvents resulting to morphological change (bead-on-string structure to fiber structure), decreased fiber diameter and narrow fiber diameter distribution. Therefore, in this study, TBAB (0.01, 0.025, 0.05, 0.1, 0.5 and 1.0% w/v) was added in the 15% PS solvent of DMF/THF (75/25). The conductivity of the solvents is shown in Fig. 4. The results revealed that with increasing the amount of TBAB, the conductivity values of the corresponding solutions was also increased. At high amount of TBAB (0.5 and 1.0% w/v), it could not be electrospun because of excessively high conductivity resulting in the clogging of PS solution at capillary tip during the electrospinning process. Whereas at low amount of TBAB (0.01, 0.025, 0.05, 0.10% w/v), it could be electrospun without clogging. This result indicated that the optimal conductivity of PS solution for fibers structure was less than 550 µs/cm. This result is similar to the previous study by Uyar and Besenbacher [9] which revealed that bead-free fibers are obtained when the conductivity of PS solution is 150-180 µs/cm. The morphology and diameter of electrospun PS fibers added various amount of TBAB are shown in Fig. 5 By increasing the amount of TBAB, the fiber diameters reached the smallest diameter at 0.025% w/v TBAB with an increase in fiber diameter by further increment of the amount of TBAB. Therefore, 0.025% w/v TBAB was selected to further experiment on the other mixed solvents.

The morphology and the average fiber diameter of PS fibers prepared with 15% PS solution with and without 0.025% TBAB in various DMF/THF and DMAc/THF ratios are shown in Table 4. The bead-free and uniform fibers were obtained when 0.025% w/v TBAB was added in all of the mixed solvent ratios. In comparison, the average diameter and the narrow diameter distribution of the fibers obtained from all solvents with 0.025% w/v TBAB was significantly smaller ($p<0.05$) than without TBAB. The change in morphology of obtained fiber prepared with PS solution with TBAB was: (a) the case of bead-on-string structure, the amount of beads in the fibers was decreased and changed to fiber structure, (b) in case the fibers structure, the fiber diameter was decreased. This might be due to the fact that addition of salt increases the charge density in ejected jet which causes a stronger elongation force of the jet due to self-repulsion of the excess charges under the electrical field following a greater repulsion and a greater bending instability during the electrospinning process, resulting in smaller fiber diameter, eliminating of beads and narrow fiber diameter distribution [9,10,20,22-24]. The smallest diameter (376±36 nm) with narrow distribution was obtained from 15% PS in DMAc solvent with 0.025% w/v TBAB.

CONCLUSION

Electrospun PS nanofiber mats have been successfully prepared by optimization of the parameters in the electrospinning process including PS concentration, ratio of mixed solvent, inner diameter of capillary tip and the addition of TBAB in the mixed solvent. The smallest diameter (376±36 nm) and the narrowest fiber diameter distribution of PS nanofibers were obtained from 15% PS solution in DMAc with 0.025% w/v TBAB. This PS nanofiber has a potential for application in the field of drug delivery, tissue engineering, filtration and prepared ion exchangers on PS fibers.

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