International Journal of Nanoelectronics and Materials
Volume 14 (Special Issue) August 2021 [213-224]

Investigation on Fibre Diameter, Wettability and Tensile Behaviour of Electrospun Polyacrylonitrile Nanofibres

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ABSTRACT

One of the major concerns in membrane distillation technology is membrane wettability. Surface functionalization using superhydrophobic electrospun nanofibre material is thought to be feasible and effective to overcome the issue. However, further understanding on characteristic and mechanical behaviour of electrospun fibres is required. This paper studied the effect of different electrospinning parameters on fibre diameter, wettability, and tensile behaviour of polyacrylonitrile electrospun nanofibres. Polyacrylonitrile in dimethyl-formamide solution of 10 wt.% concentration was electrospun under different applied voltages and electrospinning distances. The characteristic and behaviour of PAN electrospun nanofibres were characterised by using scanning electron microscope, water contact angle method and tensile test. Based on scanning electron micrographs, the average fibre diameters were in the range of nanometre. It was also observed that increasing the applied voltage would increase the fibre diameter, meanwhile, increasing the distance between spinneret and grounded collector would decrease fibre diameter and fibre deposition rate. The average contact angle and the tensile strength of PAN electrospun nanofibres also was determined in this study. The results from this study provide crucial information for the development of new filtration material for membrane distillation.

Keywords: Electrospinning, polyacrylonitrile, fibre diameter, wettability, tensile

1. INTRODUCTION

Nanotechnology plays a significant role in modern manufacturing and pioneering research studies mainly due to its promising capabilities and benefits. Scientific knowledge underpinning the effects of reduced particle size for various applications has been well investigated by researchers [1,2]. For example, researchers found that nanoscale fibrous materials or nanofibres have several prodigious characteristics such as lightweight, small diameter, controllable pore structures, and high surface area to volume ratio [3]. Nanofibre is defined as ultrafine fibre with an average fibre diameter typically between 100 nm to a few microns [4]. In recent years, there has been an increasing interest in nanofibre applications. For instance, electrospun nanofibres have been proposed for the development of rechargeable batteries, renewable energy, conductive polymer, supercapacitor, membrane filters, and water filters [5–11].

Several methods or techniques have been introduced in polymeric nanofibre production such as electrospinning, plasma treatment, sol-gel method, phase separation, and lithography [12,13]. Among these techniques, electrospinning provides a consistent fibre spinning process that can produce fibres with diameters in the nanometre and micrometre range. Furthermore, electrospinning is also known as a simple and economical technique for fabricating nanofibres from a wide range of polymer solutions [14,15]. To date, many types of polymers have been used to produce electrospun nanofibres. Among others are polyurethanes (PU), polyacrylonitrile (PAN), polyvinyl alcohol (PVA), polyvinylidene fluoride (PVDF), and polyethylene oxide (PEO) [16]. In particular, polyacrylonitrile (PAN) is widely used in electrospinning due to its good mechanical properties and good solvent resistance [17]. Previous studies reported that the tensile strength of PAN nanofibre membrane ranges from 10 MPa up to 140 MPa [18–20], making it applicable for various fields of fabrication. Material concentration, thickness and fibre diameter are the main factors that directly affect the tensile strength of electrospun nanofibre membrane [21]. In addition, PAN is the favourable precursor material for developing high-performance nano and micro size carbon fibres [4,22].

A basic electrospinning setup typically consists of four main components, i.e., a high voltage power supply, a tip with a small orifice (also known as spinneret), a polymer solution supply, and a grounded collector electrode. The working principle of electrospinning process is based on the use of a high-voltage supply to charge a polymer droplet with a high number of electrostatic charges. This action will cause a polymer jet formation at the vertex of the conical-shaped droplet, also known as Taylor's cone [23–25]. The charged polymer jet travels towards the opposing grounded collector electrode in a fairly straight trajectory momentarily. After a certain distance, when the longitudinal forces caused by the electric fields become weaker, the jet buckles and journey continue in an unstable motion known as the whipping instability. The whipping instability occurs due to lateral perturbation growth in response to repulsive forces between the adjacent like charges [26]. Throughout the journey, the fibre thinning process takes place as the remaining solvent in the polymer jet continues to evaporate. In addition, enormous mechanical stretching introduced by the electric fields further thinning the fibres before finally deposited onto the grounded collector as solid nanofibres.

Despite the simplicity of the process, electrospinning requires in-depth understanding on processing parameters that will significantly affect the morphology and fibre diameter of the fibres. The processing parameters can be divided into two; (a) intrinsic properties of the solution, i.e. polymer concentration, molecular weight, conductivity, and viscosity; and (b) the operational conditions, i.e. applied voltage, the feed rate of the polymer solution, the distance between spinneret and grounded collector, and the design of the grounded collector [16,27–29]. However, among these parameters, applied voltage is the most critical parameter in electrospinning process. Kumar et al. reported that a higher applied voltage would produce a stronger stretching of the fibres and thus producing smaller fibres [28]. However, contradictory findings have also been reported, which claimed that a higher applied voltage would produce larger fibres [29]. Hence, for each and every electrospinning system, a comprehensive study on processing parameters is required in order to obtain good quality fibres.

In certain applications such as membrane distillation, surface wettability plays an important role in determining the membrane performance. For example, achieving superhydrophobicity surfaces (water contact angle above 150°) can delay the ageing of the membrane surface, enhance the antifouling properties and others [30]. In a previous study, the water contact angle (WCA) of PAN electrospun nanofibres was found to be in the 100° to 130° range [31]. Moreover, PAN electrospun nanofibres also were proposed as surface coating in the development of new filtration membranes [5,31]. Though according to Ebrahimi et al. [5], the as-spun PAN electrospun nanofibre membranes may not be sufficient in preventing declination of vapor flux and wetting problems. Thus, the authors recommended the enhancement of nanofibre membranes through optimization of fibre diameter for improved water repelling behaviour. In achieving this goal, questions arise in particular relating to electrospinning parameters, polymer-solvent concentration, optimized fibre diameter, hydrophobicity, and mechanical behaviour of the nanofibres.

Thus, the aim of this study was to investigate fibre diameter, wettability, and tensile behaviour of PAN electrospun nanofibres. An initial experiment was conducted using different applied voltages and electrospinning distances in order to get optimum electrospinning parameters. Fibre diameter, wettability, and tensile behaviour of the PAN electrospun nanofibre membranes were investigated subsequently. This study was conducted as part of the laboratory's ongoing objective in developing superhydrophobic coating material for membrane distillation system.

2. METHODOLOGY

Polyacrylonitrile (PAN) with an average molecular weight of 150,000 g/mol and N, N-Dimethylformamide (DMF) were purchased from Sigma-Aldrich. The solution was prepared by weighing and mixing 5 g of PAN powder with 45 g of DMF as solvent (PAN/DMF final concentration of 10 wt%) using an analytical balance (Mettler Toledo – ML204) and a magnetic stirrer (IKA – C-MAG HS7). The mixture was stirred at room temperature for approximately 5 – 6 hours.

The PAN/DMF solution was filled inside a 12 ml plastic syringe and mounted onto a syringe pump (NLS 20, Nanolab Instruments). The feed rate of the polymer solution was set at 1.1 ml/h. An electrospinning machine, Electrospinz model ES1a (Electrospinz Ltd., New Zealand) was used throughout this experiment. The electrospinning process was carried out at room temperature. Aluminium foils were attached to the grounded collector to collect deposited fibres. Two types of grounded collectors were used i.e., a flat plate collector and a rotating drum collector. The flat plate collector was used to collect a small amount of fibre for microscopy and wettability examinations, whilst the rotating drum collector was used to collect a wider fibre membrane for tensile test.

Samples for fibre diameter and morphological examination were prepared at an applied voltage between 10 kV – 15 kV and with electrospinning distance between 10 cm – 25 cm. Aluminium foils were placed onto the collector to collect fibres. The duration of electrospinning process was fixed at 5 min. After electrospinning process, samples were left for at least 24 hours to ensure the samples were completely dry. Then, the samples were cut to approximately 1 × 1 cm. The morphology of the PAN electrospun was examined using a scanning electron microscope (SEM) model JSM-6010 PLUS/LV (JEOL Ltd., Japan). The magnification of the SEM was fixed at ×5000. ImageJ version 1.5 software was used to measure the average fibre diameter.

Samples for surface wettability study were prepared by electrospinning directly onto a glass slide. The samples were prepared with a duration of 5 to 15 min. All samples were left for 24 hours before testing. A dedicated contact angle instrument (A-CAM) was used to capture the image of the water contact angle (WCA) in accordance with *ASTM D5725 – 99* standards test method for surface wettability test using an automated contact angle measurement. A distilled water droplet (5 µl) was dripped onto the samples. Three samples were prepared by depositing electrospun fibre directly onto glass slides within 5 min, 10 min, and 15 min of electrospinning durations. The applied voltage and electrospinning distance was fixed at 15 kV and 15 cm, respectively. The WCA of PAN electrospun was measured using ImageJ software, following a similar method that was used by Jasmee et al. [32].

Samples for tensile test were prepared using a rotating drum collector. The rotation speed of the drum collector was set at 120 rpm. The test samples were prepared at different electrospinning times of 60 min, 90 min, and 120 min. The applied voltage and distance between the tip and the grounded collector were kept constant. The samples were weighed using an analytical balance. The tensile mechanical test was carried out using a Universal Testing Machine (UTM - 200 Series Single Column Test Machines, Shimadzu) with a load cell of 50 N. All tests were conducted at room temperature. The test samples were cut to 10 mm (width) by 60 mm (length) in accordance with *ISO 13934* standards for determining the tensile properties of fabrics. Then, the samples were carefully mounted onto the grip of the UTM machine to provide a 40 mm gauge length as shown in Figure 1. The strain rate of the machine was set at 2 mm/min.

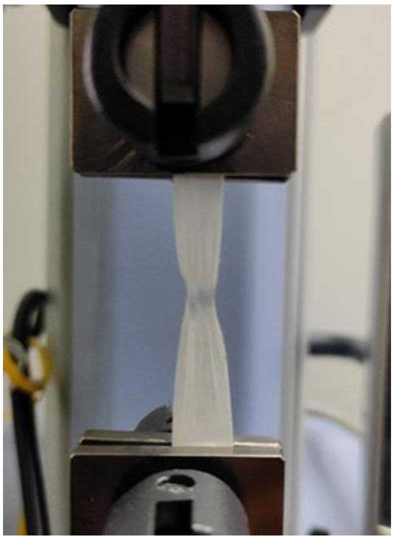


Figure 1. PAN electrospun nanofibre (10 × 60 mm) was mounted onto the grip of the UTM machine with a 40 mm gauge length.

The thickness of electrospun membrane was difficult to measure without damaging the sample. Therefore, the thickness of the fibre membrane was determined using weight difference method. The mass of fibre thin film with and without aluminium foil was measured. The difference will determine the mass of the fibre thin film, m. Then, the area of thin film, A was calculated by multiplying the length, l with the width of the samples, w (10 × 60 mm). The density of the PAN polymer, ρ_ρ of 1.184 g/cm^3 was used. Thus, the thickness of the fibre membrane, t can be calculated using Equation 1.

$$t = \frac{m}{\rho_\rho \times (l \times w)} \tag{1}$$

The cross-sectional area, A_c was calculated by multiplying the thickness, t with the width of the samples, w. The tensile stress, σ and tensile strain, ε were calculated using Equation 2 and Equation 3.

$$\sigma = \frac{F}{A_c} \tag{2}$$

$$\varepsilon = \frac{\Delta l}{l_o} \tag{3}$$

where F is force recorded by the UTM machine, Δl is the elongation of the specimen and l_o is the original length of the specimen (40 mm).

3. RESULTS AND DISCUSSION

Figure 2 shows the SEM micrographs and average fibre diameter of the samples, which were collected at different combinations of applied voltages (10 to 20 kV) and electrospinning distances (10 to 25 cm). Samples that were collected at an applied voltage of 10 kV showed a decreasing trend of average fibre diameters from 613 nm to 514 nm as the electrospinning distance increased from 10 cm to 25 cm, as shown in Table 1. Moreover, Figures 2 (a), (d), (g), and (j) show that the deposition of electrospun nanofibres also decreased as the electrospinning distance increased. This was because as the electrospinning distance increased, the electric field strength between the spinneret and the collector weakened. Thus, the electric forces responsible for driving the fibres towards the collector also reduced [33].

SEM micrographs of samples collected at an applied voltage of 15 kV suggest that the average fibre diameter decreased from 653 nm to 506 nm as the electrospinning distance increased. Based on SEM micrographs in Figure 2 (b), (e), and (h) the deposition of fibre at a distance between 10 cm to 20 cm were more consistent and productive compared to samples when 10 kV of applied voltage was used (Figures 2 (a), (d), (g)). However, as the electrospinning distance was further increased to 25 cm, the deposition of fibres was significantly reduced as evidenced by SEM micrograph in Figure 2 (k).

Table 1 Average fibre diameter of PAN electrospun nanofibre

Voltage (kV)	Distance (cm)	Sample	Average fibre diameter (nm)
10	10	(a)	613 ± 41
	15	(d)	529 ± 22
	20	(g)	587 ± 29
	25	(j)	514 ± 29
15	10	(b)	653 ± 12
	15	(e)	554 ± 25
	20	(h)	506 ± 63
	25	(k)	558 ± 32
20	10	(c)	715 ± 38
	15	(f)	717 ± 45
	20	(i)	626 ± 50
	25	(l)	562 ± 30

Electrospinning at 20 kV of applied voltage produced higher deposition rate of electrospun nanofibres compared to electrospinning distance of 10 cm and 15 cm (Figure 2 (c), (f), (i), and (l)). However, Figure 2 (c) shows that a formation of beaded fibres has occurred. The formation of beaded fibres in Figure 2 (c) suggests that the short electrospinning distance of 10 cm and a high applied voltage of 20 kV was not a good combination. This was because the polymer jet did not have sufficient time to solidify or evaporate while traveling from the spinneret to the grounded collector. Furthermore, the application of high applied voltage at such short electrospinning distance would only create excessive electric drawing forces. Shahreen and Chase [34] suggested that fibres produced under this condition would create bead formation as can be seen in Figure 2 (c). This defect is not favourable because the beads would greatly reduce the surface area to volume ratio of the fibres.

In general, the data in Table 1 suggest that increasing electrospinning distance would produce fibres with smaller fibre diameters. On the contrary, for a given electrospinning distance, the trend shows that an increased applied voltage would increase the average fibre diameter of the fibres. Based on SEM micrographs and average fibre diameter, the results suggest that the best applied voltage for this electrospinning system was 15 kV. Meanwhile, in terms of electrospinning distance, the results suggest that the best electrospinning distance was between

10 cm to 15 cm based on high rate of fibre deposition and no presence of beaded fibres. Thus, it is concluded that the optimal electrospinning parameters for this electrospinning system study were 15 kV applied voltage and 10 cm to 15 cm of electrospinning distance. In addition, the average fibre diameter of 500 nm to 700 nm found in this study was similar to the one reported by Khan et al. [35].

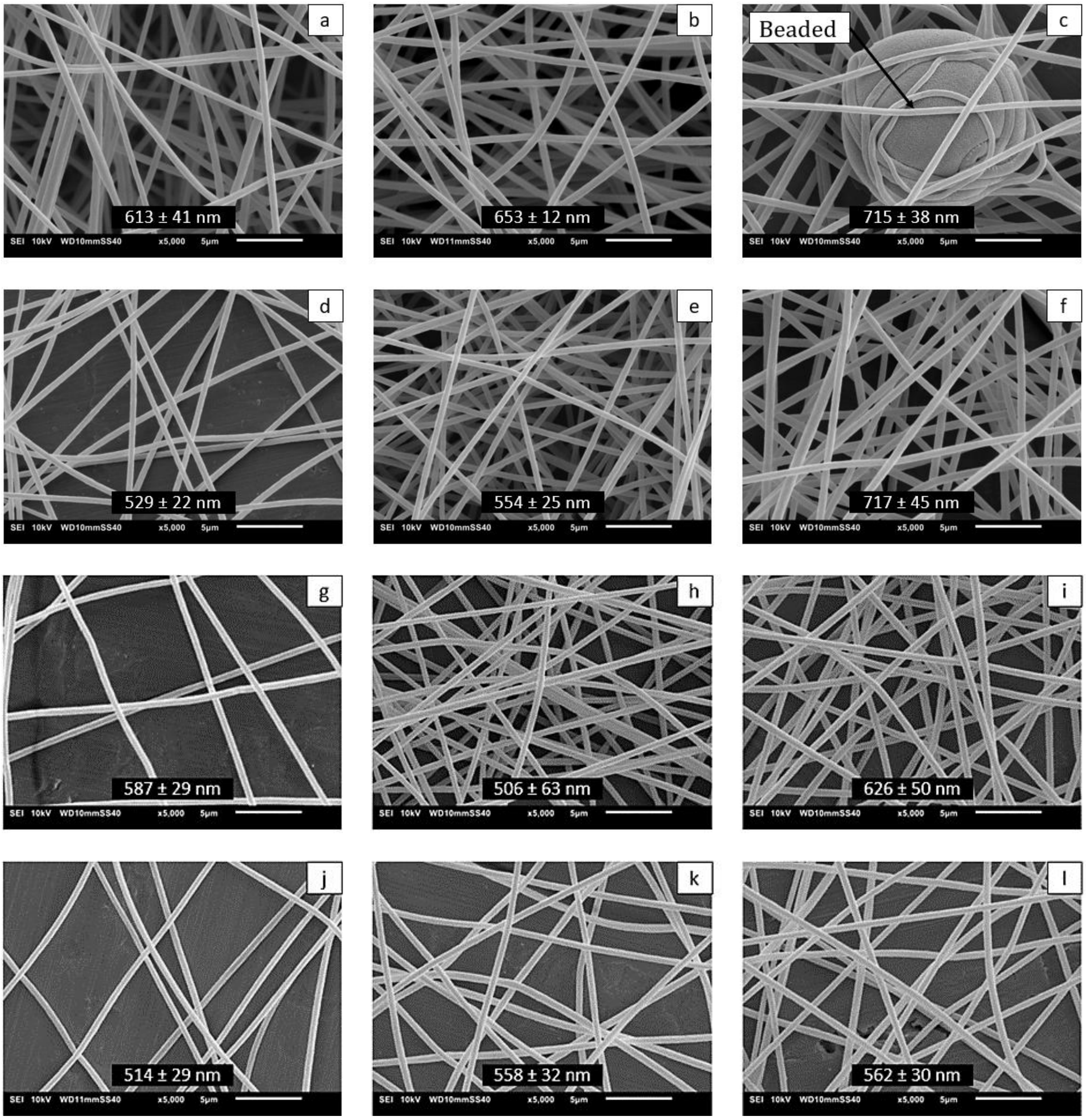


Figure 2. SEM micrographs and average fibre diameters of PAN electrospun nanofibres produced at different electrospinning distances and applied voltages of (a) 10 cm, 10 kV (b) 10 cm, 15 kV, (c) 10 cm, 20 kV, (d) 15 cm, 10 kV, I 15 cm, 15 kV, (f) 15 cm, 20 kV, (g) 20 cm, 10 kV, (h) 20 cm, 15 kV, (i) 20 cm, 20 kV, (j) 25 cm, 10 kV, (k) 25 cm, 15 kV and (l) 25 cm, 20 kV.

Figure 3 shows the test images of average water contact angle (WCA) of PAN electrospun fibres produced at different electrospinning durations of 5, 10, and 15 min. The average value of WCA were 121.34° for 5 min samples, 129.11° for 10 min samples, and 129.79° for 15 min samples. Since the average WCA for PAN electrospun nanofibres were in the range of 120° - 130° (lower than 150°), thus the electrospun nanofibre membrane surfaces can be categorized as hydrophobic. It was also observed that when the distilled water droplet was dripped vertically onto PAN electrospun nanofibres, the water droplet managed to hold its shape momentarily. However, the water droplet could only hold its shape for 5 to 8 seconds before the droplet ruptured. This was because the forces associated with interactions between the nanofibre surface and water droplet were eventually higher than the bulk water, which led to the decrease of surface tensions [36].

Considering the calculated standard deviations of WCA, the average WCA were similar throughout the samples. Hence, it was concluded that the WCA of PAN electrospun nanofibres were not affected by electrospinning duration. This was because the surface roughness of the electrospun fibre membrane remained the same even though the electrospinning duration was increased [37]. The results of this study were similar to the one reported by Szewczyk et al. [37], who reported that the WCA of the electrospun nanofibre membrane was not affected by electrospinning duration and thickness of the membrane.

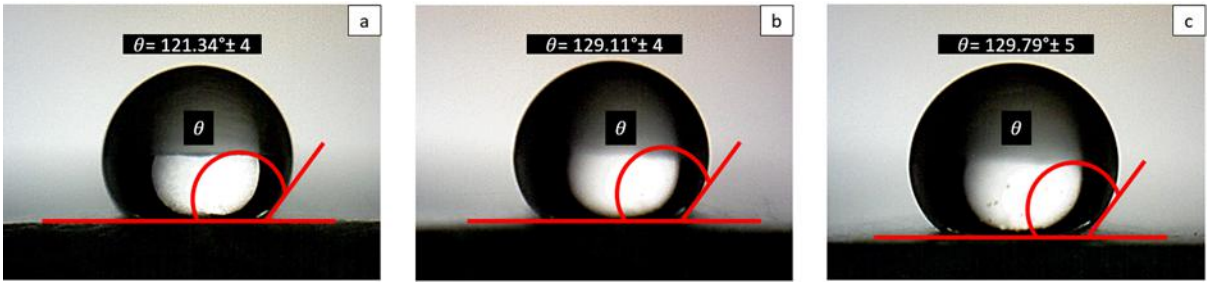


Figure 3. Average water contact angle of PAN electrospun nanofibres produced at different electrospinning duration of (a) 5 min, (b) 10 min and (c) 15 min.

Table 2 compares the findings between previous and current WCA studies on PAN electrospun nanofibre membranes. In general, the results of this study were comparable to previous studies except to the one reported by Hasanzadeh et al. [38]. A slight difference in WCA values was expected as the value of WCA is highly dependable on materials properties, solution concentration, applied voltage, volume flow rate, and electrospinning distance [39].

Table 2 Comparison of WCA studies on PAN electrospun nanofibre membranes

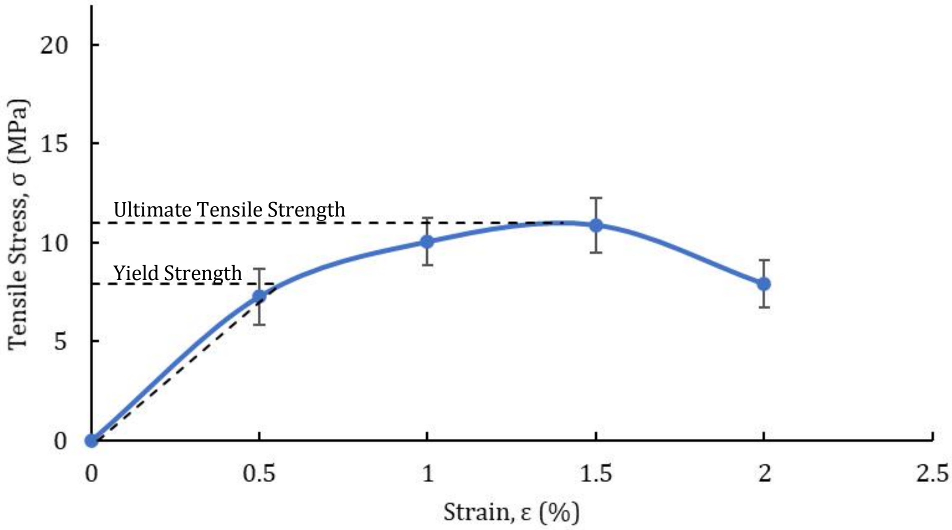
	Electrospinning Parameters				**Results**
Author	**Concentration (wt%)**	**Voltage (kV)**	**Distance (cm)**	**Flow rate (ml/h)**	**Average of WCA (°)**
J. Fang et al., [40]	7	18	15	1.0	109.10
Z. Khan et al., [41]	10	21	15	1.5	105.00
M. Hasanzadeh et al., [38]	10	22	20	2.0	65.00
H. Kahraman et al., [42]	-	16	15	0.6	125.00
Current study	10	15	15	1.1	129.00

The tensile test results for PAN electrospun nanofibre membranes are listed in Table 3. The calculated thickness of PAN electrospun nanofibre increased from 2.70 µm to 7.67 µm as the electrospinning duration increased. From Table 3, the average ultimate tensile strength was 10.97 MPa, 18.69 MPa and 18.04 MPa, for 60 min, 90 min and 120 min samples, respectively. A comparable finding was reported by Khan et al. [41], who reported that the tensile strengths for PAN electrospun nanofibre membranes were in the range of 10 – 16 MPa. Figure 4 shows the stress-strain curves of PAN electrospun nanofibre membranes. The average yield strength increased from 7.91 MPa to 17.83 MPa as the electrospinning duration increased. The elongation break (strain) for 60 min samples was 2 %, while for 90 min and 120 min samples the elongation break was 0.8 %. This suggest that as the electrospinning duration increased, the membrane became stiffer and less ductile as evidenced by the shape of the stress-strain curve in Figure 4(c).

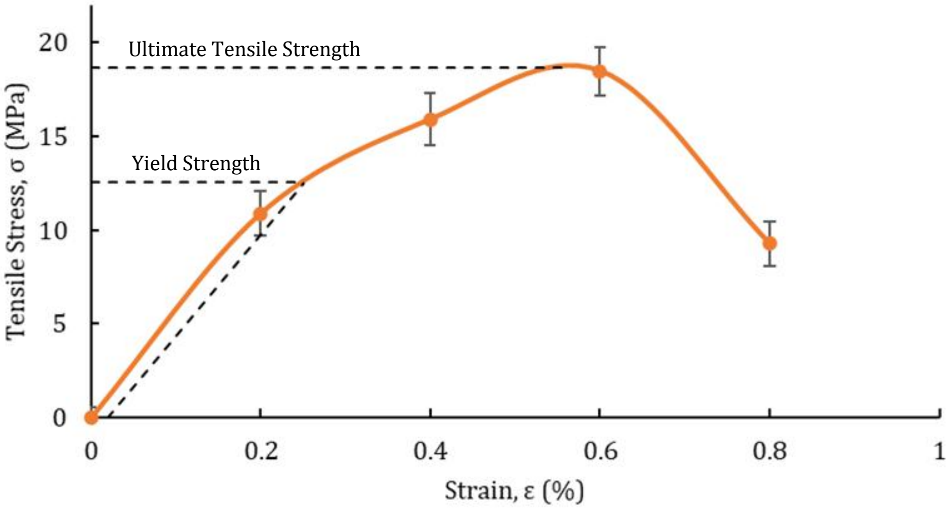
Table 3 Tensile behaviour of PAN electrospun nanofibre membranes

Electrospinning Duration (min)	Sample Mass (g)	Thickness (μm)	Ultimate Tensile Strength (MPa)	Yield Strength, (MPa)
60	0.00192	2.70 ± 0.4	10.97 ± 1.5	7.91 ± 0.3
90	0.00300	4.17 ± 0.5	18.69 ± 2.0	12.53 ± 0.8
120	0.00550	7.67 ± 0.5	18.04 ± 1.8	17.83 ± 0.6

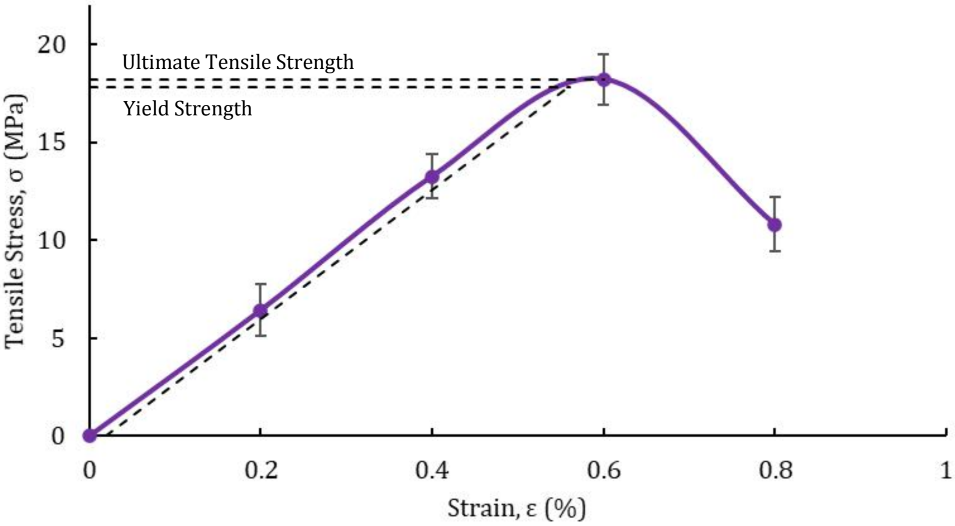
In membrane distillation technology, porous hydrophobic materials are preferable because of the ability to allow vapour transport but at the same time acting as barrier for liquid and other material transfers. In this study, PAN electrospun nanofibre membranes have the potentials to be used as coating materials for membrane distillation due to its small size, good hydrophobicity, and high mechanical strength. However, further investigation on improving the hydrophobicity of the PAN electrospun nanofibres by mean of surface modification is thought required. One of the possible options is by using silica nanoparticles/fluorinated alkyl silane. It is hoped that the combination of topology and surface chemistry of the electrospun nanofibres would help to create highly effective superhydrophobic nanofibre membranes for water desalination system. Currently, the works are ongoing, and the results will be reported elsewhere.



(a)



(b)



(c)

Figure 4. Tensile stress – strain curves of PAN electrospun nanofibre membranes produced at different electrospinning durations of (a) 60 min, (b) 90 min and (c) 120 min.

4. CONCLUSION

In summary, fibre diameter, water contact angle and tensile behaviour of PAN electrospun nanofibres have been investigated. Polyacrylonitrile (PAN) nanofibres were produced using electrospinning technique at different applied voltages and electrospinning distances. Increased applied voltage produced larger fibre diameters, meanwhile, increased electrospinning distance produced smaller fibres. Furthermore, if the electrospinning distance was too far, fibre deposition rate decreased significantly. In this study, the optimal PAN electrospinning parameters were 15 kV of applied voltage and 10 cm to 15 cm of electrospinning distance. At these parameters, the process produces fibres at a high fibre deposition rate without the presence of beaded fibres. The average fibre diameters of PAN electrospun nanofibre were between 500 to 700 nm and the water contact angle was 129°. The maximum tensile strength of the nanofibre membranes was around 18 MPa. Further investigation on surface modification of the nanofibres are underway with the aim of creating superhydrophobic nanofibre membranes.

ACKNOWLEDGEMENTS

The authors would like to thank the Ministry of Higher Education Malaysia and Universiti Teknikal Malaysia Melaka through the funding of Collaborative Research Grant PJP/2019/FKM-CARE/CRG/S01706. Special thanks to the members of Advanced Materials Characterization Laboratory (UTeM), Academia-Industry Collaboration Laboratory, and the members of the Faculty of Mechanical Engineering, UTeM.

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