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Determination of Optimal Electrospinning Distance and Applied Voltage for Polyacrylonitrile Electrospun Fibre Production

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ABSTRACT

Electrospinning process is highly dictated by electric field strength. Thus, two key parameters i.e., electrospinning distance and applied voltage, determine the quality of electrospun fibres. Incorrect selection of these parameters will result in poor fibre qualities. There ought to be an optimal combination of electrospinning distance and applied voltage to produce best quality fibres from a given material. In this study, the optimal combination of electrospinning distance and applied voltage was assessed based on consistency of electrospinning process, amount of fibre, fibre morphology, and average fibre diameter. Polyacrylonitrile (PAN) electrospun fibre samples were prepared at different combinations of electrospinning distance and applied voltage. Scanning electron microscopy and image analysis were conducted to assess the quality and average diameter of the fibres. The results indicate that for electrospinning of PAN, the distance should be between 10 and 20 cm with a 15 to 20 kV of applied voltages. Findings from this study is crucial for producing optimal fibre quality in PAN electrospun nanofibre synthesis.

Keywords: electrospinning; electrospun; nanofibre; polyacrylonitrile

1. INTRODUCTION

Fibre is defined as natural or engineered substances with large length to diameter ratio [1]. Electrospinning is the most widely used technique for producing polymeric nanoscale fibres with an average fibre diameter typically between 50 to 1000 nm [2]. Due to their small scale, such fine fibres exhibit outstanding large surface to volume ratio which is advantageous for many applications including filtrations, sensors, membranes, composites, biomedicine, energy storage, and protective clothing [3], [4]. A wide range of synthetic polymers have been electrospun into fibres such as polyvinyl alcohol, polyethylene oxide, polyvinyl butyral, nylon, polyvinylidene fluoride, polystyrene, and polyacrylonitrile[4]. Aside from synthetic materials, natural polymers such as cellulose, chitin, lignin, protein, and synthetic bio-based polymers such as polylactic acid and polyhydroxyalkanoate have also been used mainly for applications that require biocompatibility and biodegradability of these materials [5], [6]. For applications that require high temperature resistance or good electrical conductivity, polyacrylonitrile (PAN) is commonly used as precursor material for producing carbon nanofibres [7]. For example, Munajat et. al[3] proposed the use of nanocarbon-based sensors from PAN electrospun fibre precursors for high frequency dielectric application. Recently, new and promising applications of this carbon based materials were proposed including in water treatment and clean energy harvesting [8]–[10].

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In a electrospinning technique, high electric voltage typically between 8 to 30 kV is applied to a polymer solution or melt to initiate the process [11]. The electrified end is called the source electrode, whereas the area where the fibres are collected and electrically grounded is called the collector electrode (Figure 1). The distance between these two electrodes is known as the electrospinning distance. Repulsive forces within the charged polymer causes the polymer to fly towards the grounded collector electrode [12]. The polymer exits the spinneret in the form of wet ultrafine fibre streams. The streams initially travel in a straight trajectory before continuing its journey in a spiral looping motion, also known as the whipping instability [13]. Throughout the journey, the fibre thinning process continues by means of axial stretching from acting electrostatic forces, and solvent drying before landing on the collector electrode as dried randomly orientated fibres [14].

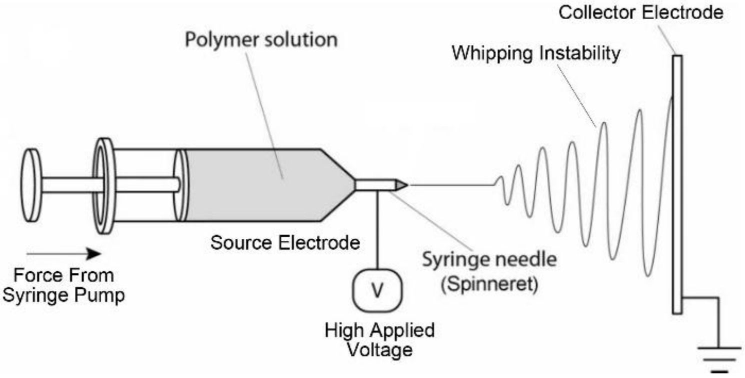

Figure 1. Schematic of electrospinning process.

Since electrospinning is solely driven by electric charges, selection of electrospinning distance and applied voltage is crucial. Electrospinning distance and applied voltage influence the electrospinning system in the following reasons. Electrospinning distance must be adequately spaced to ensure fibre drying and stretching to occur without interruption [15]. Studies have shown that electrospinning at a very short distance will produce thick, wet, and sometimes with other irregularities such ribbon-shaped fibres or beaded fibres due to incomplete solvent evaporation [16]. However, for a given voltage, an overextended electrospinning distance would only weaken the electric fields causing less fibre production. Therefore, in order to maintain a consistent and productive electrospinning operation, the electrospinning distance and voltage must be optimally chosen to allow fibre thinning and drying to occur, whilst asserting a certain level of electric field strength for fibre deposition process [17].

The objective of the study was to determine the optimal electrospinning distance and applied voltage for the production of PAN electrospun fibres. Experiments were performed using several variations of electrospinning distances and voltages, and the quality of the fibres was assessed using scanning electron microscopy and fibre diameter analysis.

2. MATERIALS AND METHODS

Polyacrylonitrile (PAN) with an average molecular weight of 124,000-130,000 g/mol was purchased from Sigma Aldrich (181315). N, N-Dimethylformamide (DMF) with an average molecular weight of 73.09 was purchased from Merck (1030532500). A mixture containing 10 wt.% of PAN in DMF was stirred overnight at room temperature using a magnetic stirrer (IKA C-MAG HS 7). The collection of fibre samples was carried out using a laboratory scale electrospinning unit. The PAN-DMF solution was fed into the electrospinning machine using a syringe pump (NLS20, Progenelink) at a flow rate of 1.1 ml/hr. Cut outs of aluminium foil were used as substrate materials and fixed onto the collector electrode. Several combinations of electrospinning distances (5, 10, 15, 20, 25, and 30 cm) and applied voltages (5, 10, 15, and 20 kV) were used, and triplicate samples of each combination were prepared.

The morphology of the electrospun fibres was inspected using a scanning electron microscope (SEM) (JEOL JSM-6010PLUS/LV). SEM accelerating voltage was set between 10 to 15 kV. Prior to SEM, samples were platinum coated for 240 seconds using an auto fine coater (JEOL JEC-3000FC). For image analysis, magnification of SEM was fixed at ×10,000 to maintain consistency. Image analysis was performed using ImageJ version 1.50 software for average fibre diameter measurement (NIH, USA) (Figure 2). Fibre diameter was averaged from 100 measurements taken from each SEM micrograph.

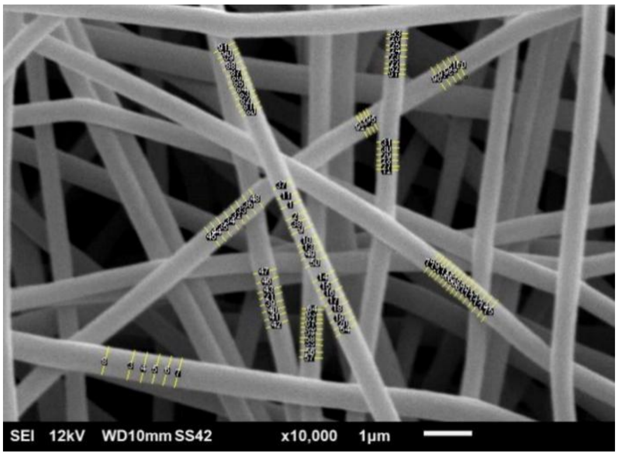

Figure 2. Average fibre diameter measurement on an SEM micrograph of PAN electrospun fibres (×10,000) using ImageJ software.

3. RESULTS AND DISCUSSION

3.1 Visual Observation

The behaviour of the electrospinning jet and fibre formation at the collector electrode were observed when different electrospinning distances and applied voltages were used. When the electrospinning distance was too short, the electrospinning process became chaotic producing strings of wet fibres hanging between the spinneret and the collector as illustrated in Figure 3. This was because at a short electrospinning distance, there was less time for the solvent to evaporate. In addition, the whipping instability region was ended prematurely due to the short distance [18]. At times especially when a low applied voltage was used, beads or drips of polymer solution can also be observed (Figure 3). On the other hand, when the electrospinning distance was too far, the electrospinning process became inconsistent. The situation occurred because the overextended distance weakened the electric field strength between the spinneret and the collector causing less drawing of fibres. It was observed that at an overextended distance, fewer fibres were deposited at the collector and some of the fibres were seen drifting away from its normal trajectory as illustrated in Figure 3. Less deposition of fibres was also observed when conducting electrospinning process at low applied voltages.

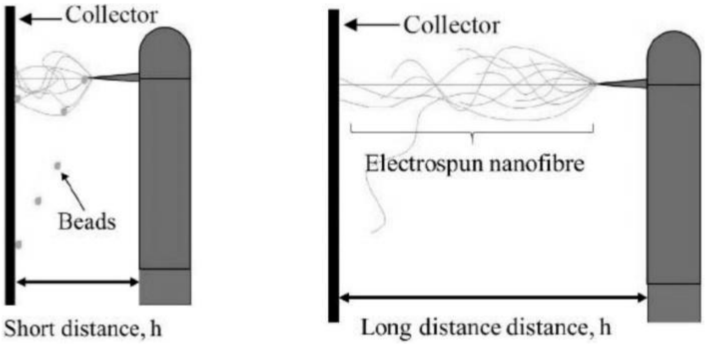

Figure 3. Electrospinning at short and long distances.

3.2 Fibre Morphology

As mentioned earlier, the electrospinning process became chaotic when it was carried out at a short distances. From SEM micrographs of fibres produced at electrospinning distance of 5 cm, very thin and crinkled fibres were produced when low applied voltage was used at 5 kV (Figure 4 (a)). When the applied voltage was increased to 10 kV, mostly thicker and uniform fibres were produced (Figure 4 (b)). However, small fibres similar to the one observed in Figure 4 (a) were also found suggesting that the process was inconsistent. Electrospinning at a higher applied voltage of 15 kV produced fibres with more consistent fibres as shown in Figure 4 (c). However, when electrospinning was carried out at 20 kV, non-uniformed fibres or fibres with beads were found as shown in Figure 4 (d).

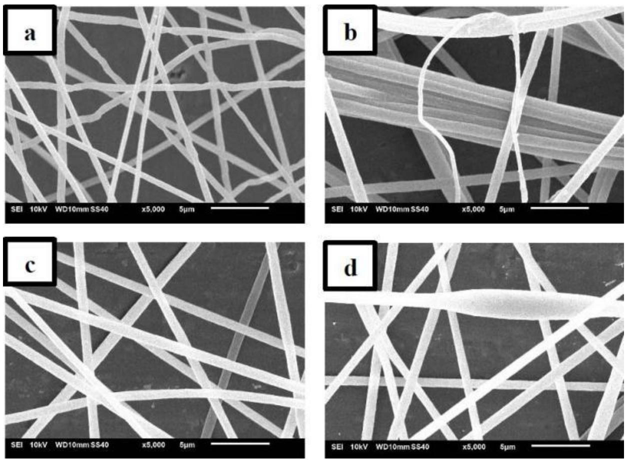

Figure 4. SEM micrographs (×5,000) of PAN electrospun nanofibres at electrospinning distance of 5 cm and applied voltage of (a) 5 kV, (b) 10 kV, (c) 15 kV, and (d) 20 kV.

At electrospinning distance of 10 cm, inconsistent electrospinning similar to the one happened at 5 cm was observed. From the SEM micrographs, very thin fibres were produced when the applied voltage was 5 kV (Figure 5(a)) whilst thicker fibres were produced when the applied voltages were increased to 10 kV (Figure 5 (b)), 15 kV (Figure 5 (c)) and 20 kV (Figure 5 (d)). Due to the inconsistency of the process at 5 kV, less fibres were collected as can be seen in Figure 5 (a). At electrospinning distance of 15 cm, less fibres were produced at lower applied voltages of 5 kV and 10 kV as shown in Figure 6 (a) and Figure 6 (b). In addition, fibres with beads were observed in Figure 6 (b) which suggest that the combination of applied voltage and electrospinning distance was not suitable. More fibres were produced at 15 kV and 20 kV as shown in Figure 6 (c) and Figure 6 (d). Furthermore, the fibres produced at 15 kV and 20 kV were uniform with no beads found on the surface of the fibres.

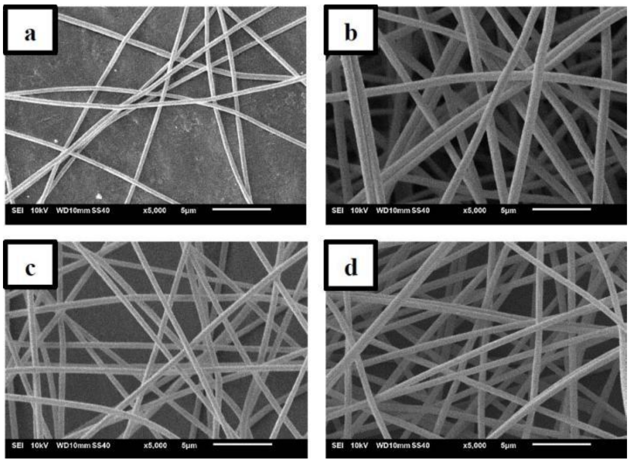

Figure 5. SEM micrographs (×5,000) of PAN electrospun nanofibres at electrospinning distance of 10 cm and applied voltage of (a) 5 kV, (b) 10 kV, (c) 15 kV, and (d) 20 kV.

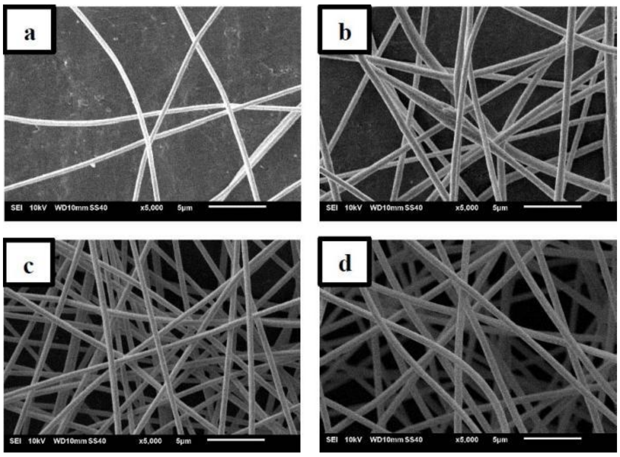

Figure 6. SEM micrographs (×5,000) of PAN electrospun nanofibres at electrospinning distance of 15 cm and applied voltage of (a) 5 kV, (b) 10 kV, (c) 15 kV, and (d) 20 kV.

The fibres produced at electrospinning distance of 20 cm were smooth without any formation of defects. However, electrospinning process at low voltage of 5 kV (Figure 7 (a)) and 10 kV (Figure 7 (b)) produced less fibres on the collector. As the electrospinning distance increased, a low applied voltage was not suitable due to weak electric field strength between the spinneret and the collector. The SEM micrographs in Figure 8 and Figure 9 show that significantly less fibres were produced at 25 cm and 30 cm electrospinning distances regardless of applied voltages. However, there was no defect found on the appearance of the nanofibres. Despite the good appearance of the fibres, the results suggest that electrospinning distance of 25 cm and 30 cm were not suitable because it would require a much longer deposition time to collect an appropriate amount of fibres. Therefore, the low production of fibres made the process impractical to be carried out at electrospinning distance of 25 cm and 30 cm.

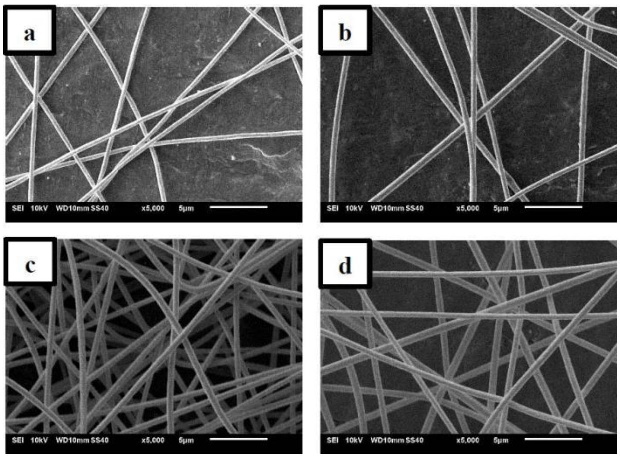

Figure 7. SEM micrographs (×5,000) of PAN electrospun nanofibres at electrospinning distance of 20 cm and applied voltage of (a) 5 kV, (b) 10 kV, (c) 15 kV, and (d) 20 kV.

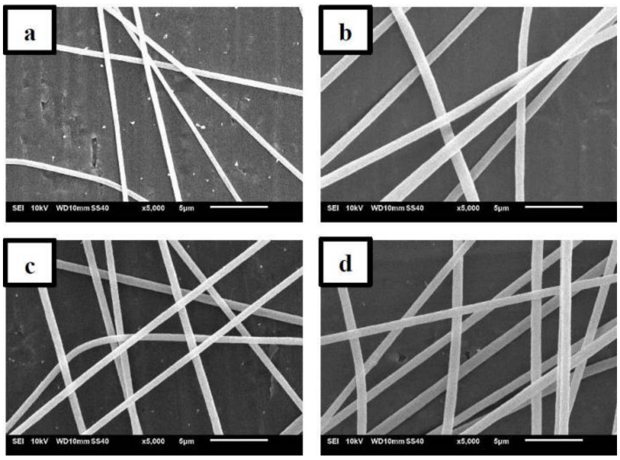

Figure 8. SEM micrographs (×5,000) of PAN electrospun nanofibres at electrospinning distance of 25 cm and applied voltage of (a) 5 kV, (b) 10 kV, (c) 15 kV, and (d) 20 kV.

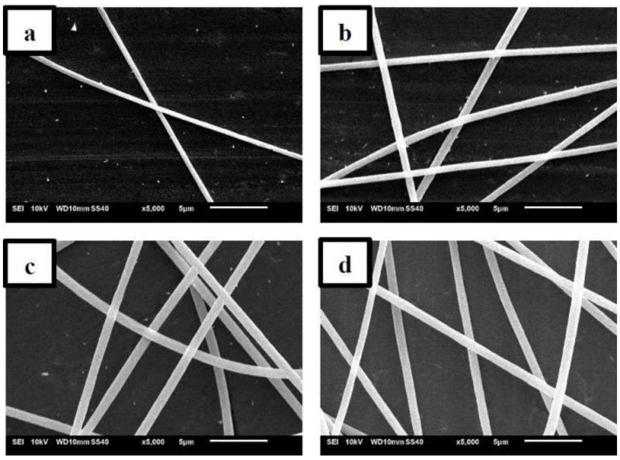

Figure 9. SEM micrographs (×5,000) of PAN electrospun nanofibres at electrospinning distance of 30 cm and applied voltage of (a) 5 kV, (b) 10 kV, (c) 15 kV, and (d) 20 kV.

3.3 Average Fibre Diameter

Figure 10 shows plots of average fibre diameter as a function of applied voltage. At electrospinning distance of 5 cm, the smallest average fibre diameter was found when the applied voltage was 5 kV (609 ± 80 nm) (Figure 10 (a)). However, the average fibre diameter increased significantly to around 900 nm when the applied voltage was increased to 10 kV, 15 kV, and 20 kV. At electrospinning distance of 10 cm, thicker fibres were produced at 10 kV (846 ± 50 nm) (Figure 10 (b)) while thinner fibres were obtained at 5 kV (414 ± 26 nm). Furthermore, as the applied voltage increased to 15 kV and 20 kV the average fibre diameter decreased. The finding was consistent with a finding by Kumar et al. [19] who claimed that a higher applied voltage would impose a stronger stretching of the fibre producing thinner fibres.

At electrospinning distance of 15 cm, the largest average fibre diameter was found when the applied voltage was 10 kV (666 ± 45 nm) while the smallest was 5 kV (461 ± 25 nm) (Figure 10 (c)). The average fibre diameters were around 500 nm when the applied voltage was increased to 15 kV and 20 kV. At electrospinning distance of 20 cm, the smallest fibre diameter was obtained at an applied voltage of 5 kV (412 ± 19 nm) (Figure 10 (d)). The fibre diameter was consistent at round 500 nm when the applied voltage was at 10 kV, 15 kV, and 20 kV. At electrospinning distance of 25 cm, thickest fibre diameter was found when the applied voltage was at 10 kV (920 ± 90 nm) and smallest fibre diameter was obtained at 5 kV (501 ± 50 nm) (Figure 10 (e)). After that, the fibre diameter seemed to increase when electrospinning at 15 kV (665 ± 27 nm) and 20 kV (819 ± 82 nm). Finally, when electrospinning distance was at 30 cm, the thickest fibre diameter was obtained at an applied voltage of 15 kV (828 ± 49 nm) and the smallest fibre diameter was obtained at 5 kV (452 ± 38 nm) (Figure 10 (f)).

3.4 Discussion

In general, Figure 10 shows a common trend on the effect of applied voltage on average fibre diameter. For a given electrospinning distance, the trend initially suggests that an increase in applied voltage would increase the average fibre diameter of the fibres. The finding was consistent with studies by Huang et al. [20] and Zhu et al. [21] who claimed that a higher applied voltage would produce thicker fibres due to the formation of bigger polymer droplet at the spinneret. However, the trend of the figures later suggests that if the electrospinning distance were fixed, further increment of applied voltage would eventually produce smaller fibres. This finding was similar with a remark made by Ramesh Kumar et al. [19] who suggested that an increase of applied voltage would increase the stretching of the fibres and therefore produces fibres with smaller diameters. It is also worth noting that when electrospinning at a distance of 5 cm, smaller fibres were not produced even at a higher applied voltage of 15 to 20 kV. The average fibre diameters seemed to be consistent at around 900 nm because the thinning of the fibres was impeded prematurely by the short distance between the spinneret and the collector [22].

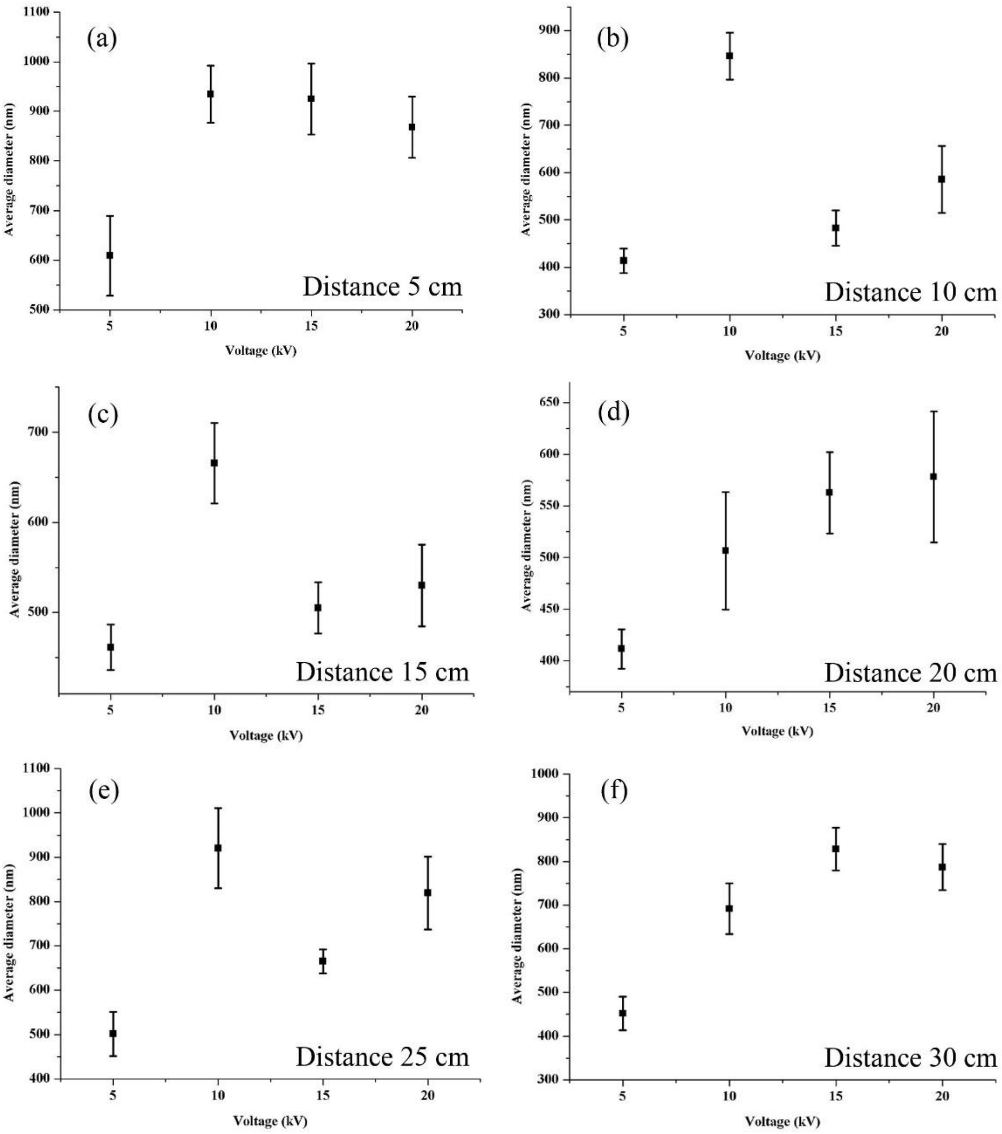

Figure 10. Average fibre diameter of PAN electrospun nanofibres produced at electrospinning distance of (a) 5 cm, (b) 10 cm, (c) 15 cm, (d) 20 cm, (e) 25 cm, and (f) 30 cm.

Summary of findings from earlier sections are presented in Table 1. The results suggest that electrospinning distance of 5 cm was not suitable for PAN polymer solution used in this study. Although small fibres were produced at 5 kV, however the fibres were crinkled and non-uniformed. Smaller fibres were produced at such a low applied voltage of 5 kV because less electrostatic forces were developed causing a smaller electrospinning jet to form at the vertex of the polymer droplet. A similar finding was also reported by Reneker & Yarin [23] who claimed that a lower applied voltage would produce smaller fibres. A smaller electrospinning jet also means that the process was using less polymer than the amount that was being supplied by the syringe pump. This caused the formation of polymer beads or drippings as illustrated in Figure 3. On the other hand, when a higher applied voltage was used, a higher polymer drawing rate happened. However, because of the short electrospinning distance, the thinning and drying process of the fibres was shortened before arriving on the collector electrode [20]. Therefore, thicker fibres with beads along the fibres were produced as can be seen in Figure 4 (c) and Figure 4 (d).

Table 1. Summary of findings

Electrospinning Distance/Voltage	5 kV	10 kV	15 kV	20 kV
5 cm	Crinkled Fibres	Inconsistent Process	Thick Fibres	Thick & Beaded
10 cm	Inconsistent Process	Thick Fibres	✓	✓
15 cm	Inconsistent Process	Thick Fibres	✓	✓
20 cm	Low Output	Low Output	✓	✓
25 cm	Very Low Output	Very Low Output	Low Output	Low Output
30 cm	Very Low Output	Very Low Output	Very Low Output	Very Low Output

The results also suggest that electrospinning distance of 10 cm was not suitable for certain applied voltages. When applied voltage was at 5 kV, fibre production rate was too low due to weak electric field strength developed at such low applied voltage. At an applied voltage of 10 kV there were more electrostatic forces introduced and therefore more fibres were produced as can be seen in Figure 5 (b). However, the fibres were thick due to lack of fibre stretching at 10 kV. Therefore, based on production rate, appearance of the fibre, and average fibre diameter, it can be concluded that 15 kV and 20 kV were the most suitable applied voltages at electrospinning of 10 cm.

At 15 cm electrospinning distance, it was clear that electrospinning at an applied voltage of 5 kV and 10 kV did not produce enough fibres as can be seen in Figure 6 (a) and Figure 6 (b). The most suitable applied voltages at electrospinning distance of 15 cm were 15 kV and 20 kV based on the smaller fibre diameter and higher fibre production rates. Moreover, the electrospun nanofibres produced at 15 kV and 20 kV were smooth without any formation of beads or uneven fibre diameter. Meanwhile the results show that the best applied voltage for 20 cm electrospinning distance was 15 kV and 20 kV (Figure 7) due to smaller fibre diameter and higher production rates. At any applied voltage, the fibre produced were smooth with no presence of defects.

The results indicate that electrospinning distance of 25 cm and 30 cm were not suitable for the material used in this study. This was based on the obtained average fibre diameter and low production rate of the fibres. The reason was due to the inconsistency of electrospinning process when the electric field strength was weakened, thus producing less fibres [24]. It was also observed that during electrospinning process some of the fibres were drifting away from its normal trajectory. This happened because electrospinning at an overextended distance made the collector less attractive to the fibres.

4. CONCLUSION

Optimal electrospinning distance and applied voltage for production of PAN electrospun fibres have been investigated. The results from this study suggest that the best combination of electrospinning parameters was between 10 to 20 cm of electrospinning distance with an applied voltage of between 15 to 20 kV. Electrospinning of PAN polymer at this range of parameters would produce defect-free fibres with an average fibre diameter of around 500 nm. It was also evidenced that a short electrospinning distance of 5 cm would only produce thicker fibres with uneven fibre diameter or beaded fibres. In contrary, electrospinning at a distance of over 20 cm produced less fibres compared to between 10 to 20 cm electrospinning distance.

The worst case was observed when conducting electrospinning at a distance of 30 cm with 5 kV of applied voltage. The findings from this study are crucial for understanding the effects of electrospinning distance and applied voltage on electrospun fibre quality.

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