The Effects of Intrinsic Parameters on the Formation of Electrospun Polycaprolactone Fibre

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Abstract*: *Electrospinning process involved strong deformations of polymer fluid at rapid fibrous structure formation within mili seconds by several influence from the distinctive spinning parameters. This study has been conducted to achieve a desirable flow rate and optimum tip-to-collector needle distance for the electrospinning of polycaprolactone (PCL). For this aim, the morphological structure of fibre spun from 10 wt% PCL solution dissolved in dichloromethane (DCM) at 0.05 – 0.20 ml/min flow rate and 10 – 15 cm tip-to-collector needle range were characterized by FESEM. The results obtained show that average diameter of electrospun PCL fibres decreases with increasing flow rate at any needle to collector distance. In addition, the tip-to-collector needle range at 15 cm produced more beads-on-string fibres compared to 10 cm distance. The fibre thickness is reduced as both of the flow and the distance of needle tip-to-collector increased. All of the obtained electrospun PCL fibres are in non-uniform, long, porous beaded fibrous structure for all flow rate and needle distance tip-to-collector values.

Keywords* : *Electrospinning, Fibre morphology, Flow rate, Polycaprolactone, Needle distance.

I. INTRODUCTION

Electrospinning is a method for creating synthetic polymer filaments by using electrostatic forces that can generate the fibre submicron variety [1]. A long fibre with a high speed is one of the greater advantages for electrospinning aside from the best way of producing nano and micro size scale of fibres. The electrospinning instrument comprises of a power supply with high voltage, a syringe pump and a conductive grounded collector. In electrospinning, a method that utilized elevated electrical field voltage to generate the electrical charging jets from a liquid polymer or melts [2]. The liquid polymer which is usually ions, when free charges applied they migrate to the electrical field in reaction and rapidly pass a force to the polymer liquid [3]. The stretching of the liquid polymer together with evaporation forms few of nano and micro size scale of fibres to be collected onto the collector. There are many parameters that can influence the formation of electrospun fibres.

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Each of the electrospinning parameters are producing variety morphology of fibres which beads, thin and thick generally. There are several factors that affect the formation of electrospun fibres which are solution, electrospinning parameters and environmental parameters. The main parameter that will produced a high different in morphology basically is an intrinsic parameters in an electrospinning process which are applied voltage, tip-to-collector needle distance, solution flow rate [4]. For instance, the parameters are required to be controlled to get optimum parameters for a desirable application. The growing in literature review for electrospinning process creates advancement in wide end-applications from various field such as bioengineering, biomaterials, medical, sensors and electronics [4].

Electrospun fibres which a small-scale fibre ranging in nano and micro size fibres exhibited several outstanding properties such as high surface area to volume ratio, good flexibility in surface functionalities, higher stiffness and tensile strength, are suitable as substrate for bone regeneration application [5]. The porous fibre that can be formed by using electrospinning to produce small-scale fibre can allow the membrane to improve cell adhesion, proteins and drugs [6]. Due to the easy process configuration of electrospinning, the exceptional properties that small-scale fibres can acquire make it popular and have a greater demand. The electrospun nanofibre basically can be extruded from an organic and synthetic polymer which depends on the desire applications. In a medical application, gelatin and collagen are always being used in a research as well as poly(glycolic acid) (PGA), poly(lactic acid) (PLA) and polycaprolactone (PCL) from the synthetic polymer which has a good properties and it is easily to get and restore compared to the organic polymer [7].

Polycaprolactone (PCL) is one of the polymer materials appropriate for use in surgery and medicine applications due to its biodegradability. Because of its low melting point of around 60℃, PCL is biodegradable and the polymer has been commonly used in drug delivery and other medical applications [2]. PCL has superior mechanical properties such as good biocompatibility, complete degradation to nanotoxic by products, rubbery and slower erosion rate [8]. PCL is also a synthetic polymer fiber that function as a hemostatic device mean that the material is capable of stopping blood flow so that it can treat wound or burn human skin [9-10].

Synthetic polymer fibres, organic fibres, composite fibres are some of the more effective fields in application. The preparation of polymer dispersion of separate materials in composite electrospun fibres is very simple. Therefore, the different fibres with their own and new features can be produced. This research work objective is aiming to characterize the formation of electrospun polycaprolactone fibres with different intrinsic parameters in electrospinning process which are vary in needle tip-to-collector range (ml/min) and solution flow rate (cm). From previous study by Kattimani et al. [11], porosity and pore size is the impact factor that makes fibres become highly demand in the field of medical for fibres in nano and micro scale which the fibres can be forms by electrospinning. Nevertheless, the fibre diameter and thickness are measured and analyzed due to the diameter and thickness measurement can influence pore size and porosity of the electrospun membrane fibres so the membrane fibres can be used in tissue engineering for future.

II. ELECTROSPINNING PARAMETERS

A. Intrinsic parameters

The effect on the formation of electrospun fibre is influenced by the electrospinning parameters both extrinsic and intrinsic. The most crucial parameter gives high effect to the fibre is intrinsic parameters [12]. As in the theory of electrospinning, it is clear that the electrospinning process has controlled variables which are solution concentrations, electric field strength, flow rate, needle tip-to-collector distance and collector composition [1]. The intrinsic parameters focused in this research work are flow rate and distance of needle tip-to-collector. By varying the parameters in the electrospinning, the morphology of fibre will be changed. The investigation is focused on the effect of each parameter as mention earlier on electrospun polycaprolactone fibre in the fibre morphology and sizes in diameter and thickness.

B. Flow rate

The flow rate or feed rate is indicating the amount of solution dispensed per unit time. Flow rate can give effect to the formation of electrospun nanofibre in morphological structural, fibre diameter and fibre thickness. The relationship between solution and flow rate can be investigated in the fibre morphological structure and size. High resulting of beaded fibre can be formed with higher flow rate due to the fibre does not have a chance to get dry to reach the collector [13].

C. Needle tip-to-collector distance

Formation of electrospun fibre can be influenced by distance of needle tip-to-collector whereas the higher the distance of needle the fibre diameter will be decreased. The fibre produced can be affected when the distance of needle tip-to-collector shorter which the fibre will be wet due to the polymers melts which it cannot has a complete evaporate and will produced bead structures along the fibre [14]. The distance of needle tip-to-collector is too long; the fibre breakage can be increase. The distance of needle tip-to-collector can control the fibre morphological structure and diameter in the formation.

III. MATERIALS AND METHOD

A. Preparation of polymer solvent

Polycaprolactone (PCL) has been produced by dissolving with dichloromethane (anhydrous, ≥ 99.8 %). The solution concentration was chosen based on the previous study by Ibrahim et al. [15] where they reported 10 wt% of solution concentration can produced a cylindrical fibre. Briefly, PCL was mixture with dichloromethane in a Schott bottle 50 ml and allowed to be dissolved in shaker. The PCL solution was shaken at room temperature for 24 hours at the speed of 150 rpm. The solution then was left for about another 24 hours in a room temperature before handling the electrospinning process to fabricate the fibres.

B. Fabrication of membrane fibre

The polymer solvent was electrospun by using electrospinning process with controllable parameters on the flow rate (0.05, 0.10, 0.15 and 0.20 min/ml) and distance of needle tip-to-collector (10 and 15 cm). The constant applied voltage was used which 15 kV. The condition of electrospinning was carried out at room temperature. The PCL fibres were deposited on a cylindrical drum covered with aluminum foil for three hours . Fig.1 illustrated an electrospinning system setup in this research. The membrane fibre was left dried for 24 hours in desiccator at 10℃.

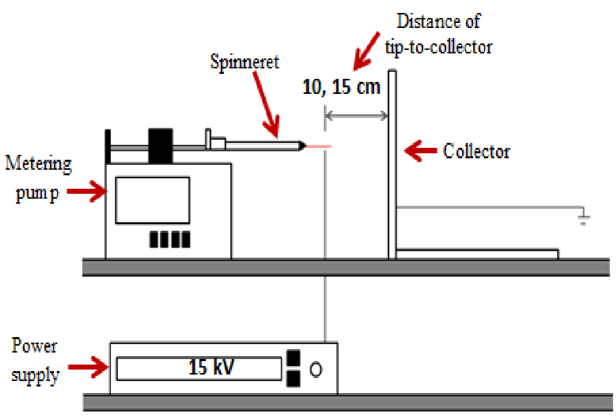


Fig. 1. An electrospinning system

C. Characterization of electrospun fibres

To characterize the electrospun PCL fibre, testing was handled for fibre morphology, fibre diameter and fibre thickness. The morphology of the electrospun PCL fibres was observed by Field Emission Scanning Electron Microscope (FESEM). The magnification used is 500x and 10,000x with a constant acceleration voltage of 5 kV. The samples of electrospun PCL fibres were coated with gold for 50 seconds before observation to increase conductivity of the samples.

The diameters of fibres were observed by using ImageJ software. 50 readings of electrospun PCL fibres were analyzed for each sample (8 samples).

Membrane fibre thickness was measured by using digimatic micrometer gauge. At least 10 readings were analyzed for each sample in different areas.

IV. RESULTS AND DISCUSSION

A. Effect on morphological structure

A variety of cross-sectional shapes of electrospun fibres such as round fibre, hollow fibre, flat fibre, split fibre and collapsed fibre may be produced by electrospinning [14]. As from the research conducted with different intrinsic parameters in electrospinning process on the distance of needle tip-to-collector and solution flow rate, the morphological structure of electrospun polycaprolactone fibre (Fig.2) showed non-uniform thick fibres with a slight amount of beads along the fibre. The fibres are all deposited and collected to the collector for about three hours each. As for the slower flow rate which is 0.05 ml/min (Fig.2a) showed the fibres produced a large number of thick fibres compared to the higher flow rate (0.20 ml/min) as shown in Fig.2d where a higher number of thin fibres is obviously showed in the figure taken from Field Emission Scanning Electron Microscope (FESEM). In addition, the fibre was deposited non-uniformly and producing thick fibre. The changes in the morphology of fibre are due to the solvent evaporation and the longitudinal of flow of jet and it is supported with a previous study by Chen [14]. The distribution of electrical force and surface tension is gradually changing, making a shape of jet unstable. By ejecting a lower jet from the main jet surface, this unstable jet can decrease its local charge per surface area unit. This breakdown leads to branched fibre formation.

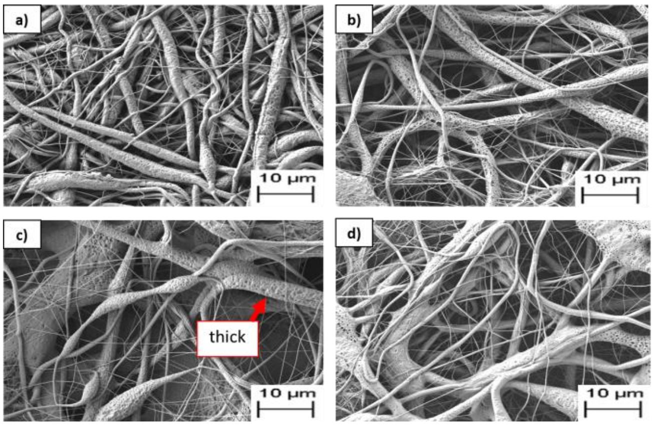


Fig. 2. Typical FESEM images at mag. 500x of the formation of electrospun PCL fibres at 10 cm distance of needle tip-to-collector a) 0.05 ml/min, b) 0.10 ml/min, c) 0.15 ml/min, d) 0.20 ml/min

At 15 cm distance of needle tip-to-collector as shown in Fig.3, the electrospun PCL fibres become smaller with several beads along the fibre. A number of beaded fibre is high when the distance of needle tip-to-collector (15cm) is high. The results showed are supported by Sill and Recum [8]. A possible reason of this is due to the evaporation of solvent at different flow rate which are from 0.05 to 0.20 ml/min where the lower flow rate attributed to the higher drying time of the fibre which is supported by Teo and Ramakrishna [12]. When increasing the flow rate from 0.10 ml/min to 0.20 ml/min, the fibre starts to form a higher amount of thin fibres with lower number of beads along the fibre. These results meet an agreement with the previous study by Hsu [14] where they reported that when the distance of needle tip-to-collector increases, more thin fibres produced and high number of beads was observed.

Fig.2 and Fig.3 showed the morphologies of polycaprolactone fibres at different distances of the needle tip-to-collector (cm) and flow rate (ml/min) formed non-uniform fibres. The distance in deposition of fibre also has a significant effect on the morphology and fineness of the electrospun fibres. Furthermore, the resultant fibres was observed that electrospun PCL fibres at 10 cm distance of needle tip-to-collector (Fig.2) formed a large fibre diameter with slight amount of beads compared to fibres with 15 cm (Fig.3) distance. At lower distance (10 cm), solvent from the polymer solution is expected to have slower evaporation compared to the solvent at longer distance (15 cm). As a result, a mixture of large fibre and small amount of beads is deposited onto the collector. The fibre morphology resultant formation is supported by Reneker and Chun [16] where the formation of beads along fibre will be produced at the higher needle tip-to-collector distance. This morphological formation of electrospun PCL fibres resultant is supported by Wannatong et al. [12] which beads formation is caused by the poor interaction between the flow rate parameter and the solvent molecules.

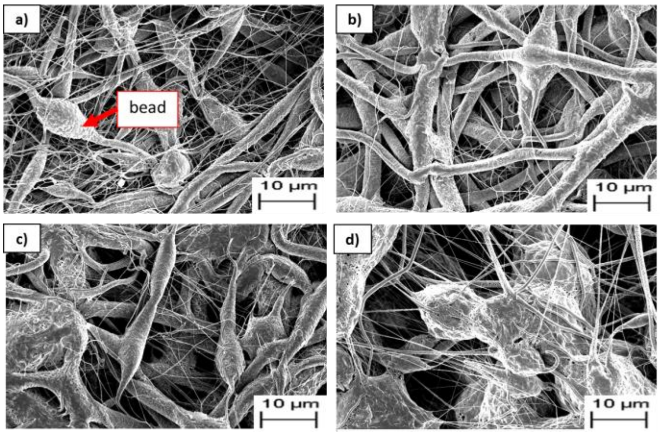
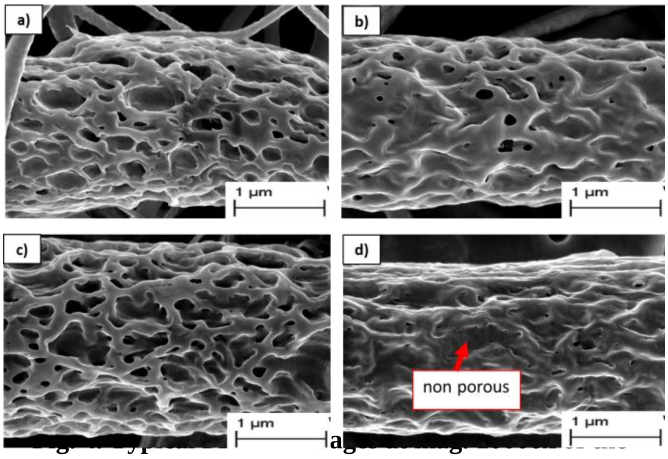


Fig. 3. Typical FESEM images at mag. 500x of the formation of electrospun PCL fibres at 15 cm distance of needle tip-to-collector a) 0.05 ml/min, b) 0.10 ml/min, c) 0.15 ml/min, d) 0.20 ml/min

As the fibre morphology on the single fibre formation of electrospun PCL fibre as shown in Fig.4, the porous fibre is produced at lower flow rate (0.05 ml/min). From the effect on fibre morphology results in Fig.4, the porosity as shown by a single fibre did not found to have a significant effect when flow rate increased from 0.10 to 0.20 ml/min (Fig.4b, Fig.4c and Fig.4d). The amount of excess polymer solution form the tip of the needle is higher when the flow rate increased formed a non-porous fibre started at flow rate 0.10 ml/min (Fig.4b, 4c and 4d). The non-porous formation of fibre may due to the slow evaporation by the solvent. The resultant of porous fibre is supported by Lipol and Rahman [17] which electrospun fibres can be produced by electrospinning when the solvent evaporated to the environment, the porous structure of fibre will be formed. Porous fibre is highly demand in medical application because the porosity can deliver a drug and promote cell adhesion

[12,17].



formation of electrospun PCL fibres at 10 cm distance of needle tip-to-collector a) 0.05 ml/min, b) 0.10 ml/min, c) 0.15 ml/min, d) 0.20 ml/min

In addition, electrospinning had been used by a variety of synthetic and natural polymers especially for biodegradable polymers and collagen to produce a porous fibre which can help in tissue engineering. For the resultant in different needle distance, porous fibre was shown by higher tip-to-collector distance (15 cm) where at high flow rate (0.15 and 0.20 ml/min) the porosity is obtained. Pore size was obviously formed by 0.20 ml/min flow rate at 15 cm distance of needle tip-to-collector.

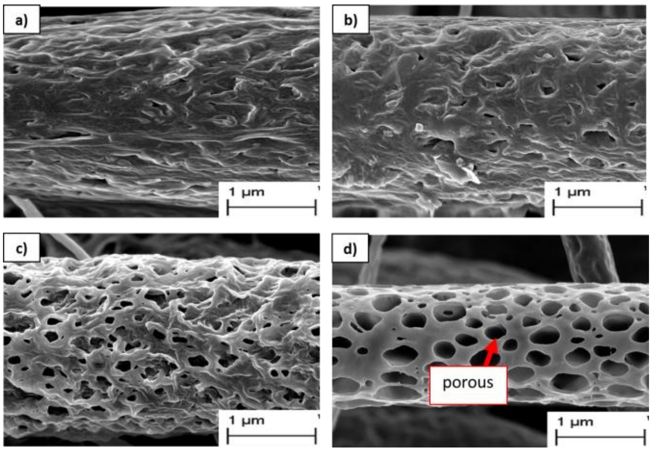


Fig. 5. Typical FESEM images at mag. 10000x of the formation of electrospun PCL fibres at 10 cm distance of needle tip-to-collector a) 0.05 ml/min, b) 0.10 ml/min, c) 0.15 ml/min, d) 0.20 ml/min

The higher porous fibres formed is due to the higher evaporation rate happened when the flow rate increased. From the research data, small-scale fibre with nano and micro size added with porous fibre which exhibit a high surface area to volume ratio can be made for medical applications which the attention recently is focused on the use of porous fibre scaffold for repairing and regenerating of tissues and cells [18].

B. Effect on fibre diameter

Fig.6 depicts the bar graph of fibre diameter against the distance of needle tip-to-collector of electrospun PCL fibre at different flow rates. It shows that the fibres diameter decreased with an increase of flow rate (ml/min). At 10 cm distance, the fibre diameter decrease from 3.807 μm ± 2.97 to 3.151 μm ± 3.32 when the flow rate increases from 0.05 ml/min to 0.15 ml/min. This could be due to the fibre has enough time to dry before reaching to the collector which influenced by the solidification of solvent Kanani and Bahrami [19]. Nevertheless, from the bar graph as shown in Fig.6 obvious trend shows the fibre diameter is decreasing as the flow rate increasing. This is happened due to the shorter distance of deposition fibres with a stable solvent concentration and makes it sufficient time to dry. The formation of fibres is consistent compared to the higher distance of needle tip-to-collector (15 cm). For 15 cm distance of needle tip-to-collector where the diameter is increasing from 3.01 μm ± 2.70 to 5.68 μm ± 4.05 then the diameter dropped to 3.64 μm ± 4.33 to 2.95 μm ± 5.28. Apparently, from Fig.6 the diameter of fibre at high distance (15 cm) is larger than the shorter distance (10 cm) even the higher distance produce more thin fibre which due to the beaded fibres produced by the higher deposition distance of needle tip-to-collector is high compared to the short distance. The fibre diameter starts to decrease at 0.15 ml/min with an increase of flow rate to 0.20 ml/min. As mentioned earlier, it is due to the polymer melts before reaching to the collector get dry completely. Thus, the smaller diameter of fibre is produced more than large fibre diameter but beaded fibre is produced due to the high distance of deposition for the fibre to reach the collector. The results in the formation of electrospun polycaprolactone fibre in fibre diameter showed the higher distance of needle tip-to-collector produced higher fibre diameter and increase in a number of beads fibre is supported by Reneker and Chun [16].

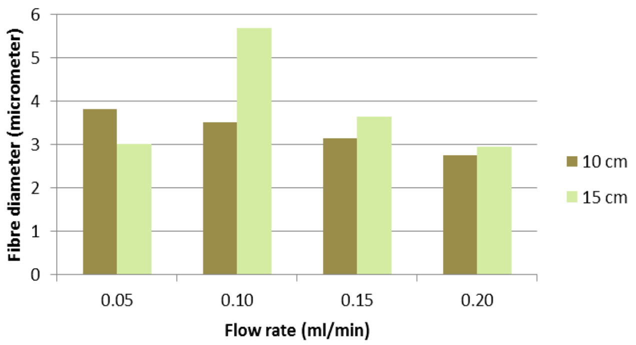


Fig. 6. Bar graph of fibre diameter (μm) against flow rate (ml/min) for each 10 and 15 cm distance of needle tip-to-collector

Another possible reason for the fibre diameter of electrospun PCL nanofibre been increasing as well as the distance of needle tip-to-collector and flow rate is due to the applied voltage that has been applied to the electrospinning which is 15 kV. The applied voltage is high so the study from Katsogiannis et al. [20] agreed with the results when the fibre diameter increasing, the applied voltage is high.

C. Effect on fibre thickness

The deposition of electrospun PCL fibre membranes onto the collector is observed its fibre thickness as shown in the Fig.7. The fibre produce by electrospinning give a high thickness until 0.371 μm ± 0.120 for 15 cm distance of needle tip-to-collector compared to 10 cm distance which the thickness of fibre is around 0.202 μm ± 0.086.

The thickness at low distance of needle tip-to-collector (0.10 cm) is increasing from 0.118 μm ± 0.039 to 0.202 μm ± 0.086 as the flow rate increased which is 0.05 to 0.10 ml/min respectively. Nevertheless, the thickness of fibre decreased starts from 0.15 ml/min at 10 cm distance of needle to-collector 0.146 μm ± 0.024 to 0.104 μm ± 0.009 due to the higher flow rate that deposited the fibre not evenly which the fibre deposited focused at the middle of the collector. The situation also happened at high distance of needle tip-to-collector (15 cm) where the thickness of fibre deposited to the collector is decreasing from 0.371 μm ± 0.120 to 0.213 μm ± 0.034 at high flow rate (0.20 ml/min). The thickness of fibre can be influenced by the fibre diameter which the higher fibre diameter can lead to high fibre thickness and vice versa. This resultant in fibre thickness is supported with a study by Chen [14]. Typically, low deposition distance (10 cm) corresponds to the wet fibre. This effect can cause the fibre to collapse and flatten which promote to intra- and inter-layer bonding because the evaporation may not entirely support the fibre.

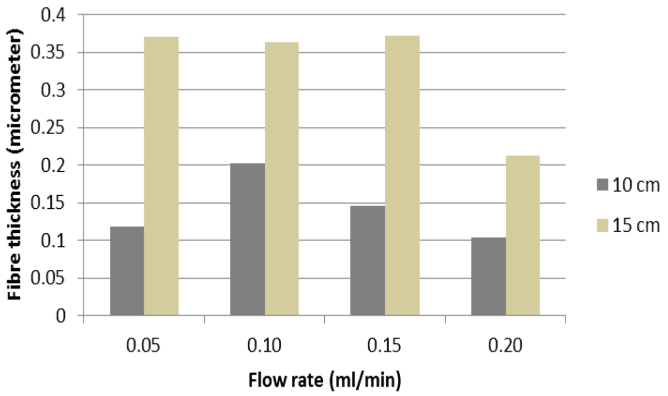


Fig. 7.Bar graph of fibre thickness (μm) against flow rate (ml/min) for each 10 and 15 cm distance of needle tip-to-collector

V. CONCLUSION

The research electrospinning experimental parameters of flow rate and tip-to-collector needle range have shown a significant effect on the morphology of the resultant fibre formation. Non-uniform, long, porous, beaded fibrous structure has been observed in the formation of electrospun PCL fibre in this study. The formation of beads was found to increase as the range of needle tip-to-collector increased. Whereas, the porous surface of electrospun PCL fibre were increased with the increment of flow rate. This research may provide improved understanding of the formation of electrospun PCL fibre to provide functional architecture fibre morphology for specific geometric architecture of natural bone in synthetic scaffold.

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