

Parametric study of nanofiber fabrication by biocompatible polymers

S G Park¹ and H W Choi²

1 Graduate School of Mechanical Design, Keimyung University, Daegu, Dalgubeol-daero 1095, Republic of Korea

2 Department of Mechanical and Automotive Design, Keimyung University, Daegu, Dalgubeol-daero 1095, Republic of Korea

E-mail: psg5655@kmu.ac.kr

Abstract. This paper introduces a parametric study of nanofiber fabrication. Poly-Caprolactone(PCL) and Chloroform are basic material for nanofiber fabrication with electrospinning method. The device for electrospinning was composed of a syringe pump, syringe, high voltage generator and 32gauge plastic nozzle. PCL concentration, applied voltage, solution flow rate and the tip to collector distance (TCD) were parameters in electrospinning method. As a result of electrospinning experiment, it was found that as TCD and applied voltage increased, the diameter of fibres was decreased. In feed rate experiment, the size of fibres decreased when feed rate decreased. The optimized PCL concentration was found at 6w/v% for the minimum of fibres.

1. Introduction

Nanofibers can provide a highly porous mesh for the biomaterials. Their high surface area per unit volume is advantageous for the various applications. A porous structure made with nanofibers is useful platform where the pore size and form can be easily changed unlike regular medical solid structure. Also nanofibers can be referred to form a firm structure if it is required. For the porous mesh applications, the most adaptable and highly productive process can be electrospinning. Meshes of porous nanofibers produced by electrospinning have been identified for use in numerous applications [1].

In 1902, electrospinning was first patented in the U.S.[2]. However, this process was not generally used until the 1990s and it became popular with recent nanoscience and nanotechnology development. Researchers started the ES nanofiber again with new research concepts of nanofiber production using electrospinning after the 1990s [3].

Electrospinning method is one of the ways to manufacture nanofibers with polymer because it is low manufacturing cost and simple process. This method is in the attention as a useful method for fabricating functional thin porous product like fibers [4]. Electrospinning method can be different in experiment set up such as collector types or kinds of solutions. Several kinds of collectors exist like using mandrel, simple plate and parallel poles (Figure 1). Net arrangement of nanofibers is different according to the kinds of collectors [5-7]. Polycaprolactone(PCL) was used for the material of produced fibers by electrospinning. It is a bioresorbable polymer with possible applications for bone and cartilage repair. PCL has certain advantages relative to other polymers like PLA(poly lactic acid)



and it is more stable in its neighboring biomedical environments. It is also significantly low cost and is freely available in large quantities [8].

In this paper, the detail procedure of manufacturing process for the nanofibers is introduced. The nanofibers are produced with a solution of PCL and Chloroform. The PCL was used as bioresorbable polymers because it's hydrophobic, linear and synthetic that indicates high mechanical strength [9]. Nanofiber production using electrospinning method can be influenced by concentration of PCL, polymer solution feed rate, TCD (tip to collector distance) and applied voltage [10]. This paper provides the environment of experiment and introduces a parametric study of nanofiber fabrication applied with electrospinning for the biocompatible polymers.. If you don't wish to use the Word template provided, please use the following page setup measurements.

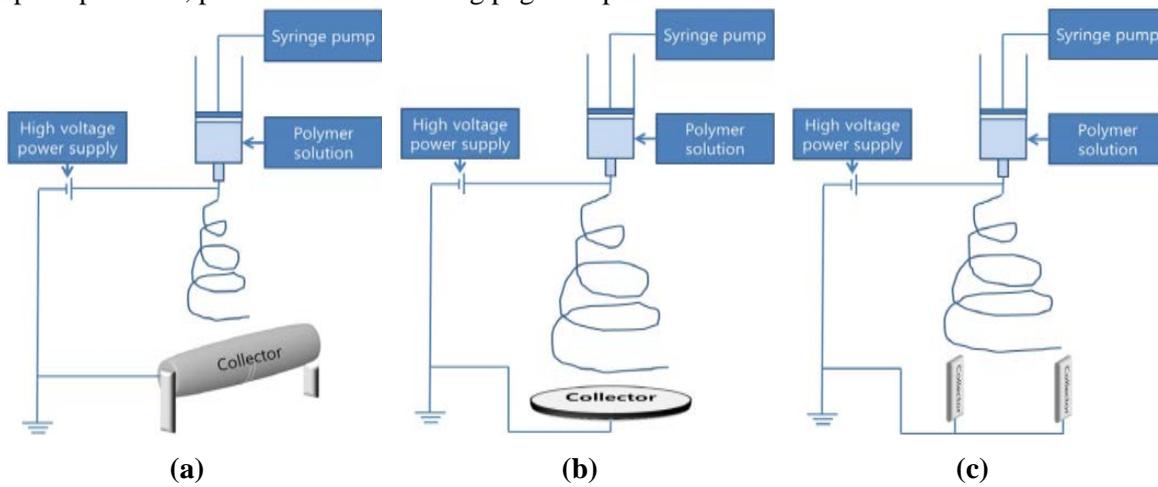


Figure 1. Schematics of collector types in electrospinning: (a) Mandrel type collector, (b) Simple plate type collector, (c) Parallel type collector.

2. Experiment

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2.1. Materials and methods

Commercial PCL pellets ($M_w=80,000$) and Chloroform (Sigma-Aldrich Inc.) were used in the experiments. Figure 2 is a molecular structure of PCL. In this paper, the device of experiment was chosen simple plate type collector in Figure 1 because it can have minimum parameter of electrospinning experiment like Figure 3. Figure 3 shows the concept design and parameters of electrospinning. The Chloroform was used as solution of PCL (Polycaprolactone). The collector was designed as flat aluminum plate with a diameter of 30mm. Fibers were electrospun from polymer solution containing 2, 4, 6 and 8% w/v PCL. The PCL solutions were transferred from 10ml syringes (HSW) and connected to a 32 gauge plastic nozzle. The needle tip was connected to a high voltage generator (NNC-HV30, NanoNC) operating at 5, 10 and 15kV. Polymer solution of PCL was fed into the needle at a rate of 1, 2, 4 and 6ml/hr by a syringe pump (KDS100, kdScientific). TCD (Tip and Collector Distance) was set at 90, 110 and 130mm.

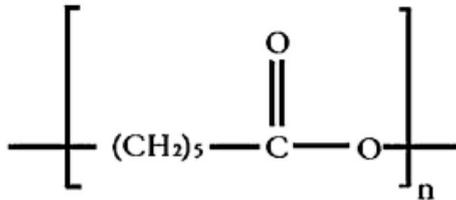


Figure 2. Molecular structure of PCL(polycaprolactone)[11].

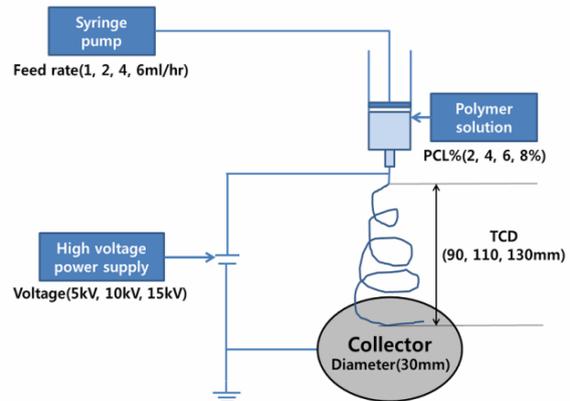


Figure 3. Experimental setup for electrospinning.



Figure 4. Plastic nozzle of 32 gauge.



Figure 5. Overall configuration of electrospinning apparatus: (a) Syringe pump, (b) Diode laser(532nm, Green), (c) Camera, (d) High voltage generator, (e) PC, (f) Plastic nozzle, (g) Collector.

2.2. Device of experiment

A syringe pump and a high voltage generator were used for electrospinning with polymer solution of PCL. Capacity of high voltage generator is maximum of 30kV. The installed syringe pump has minimum flow rate of 0.00635cm/hr and maximum of 76.18cm/hr. Syringe has a capacity of 10microliter to 60milliliter. The 532nm wavelength of diode laser was used for the visual monitoring of nanofibers electrospinning. A vision camera (zoom 7000, NAVITAR) was used for the real time monitoring via a computer. In electrospinning process, the material leaves from the nozzle to the collector. The size of nozzle was 32 gauge for the manufacturing of nanofibers as shown in Figure 4. The inner diameter of nozzle was 0.10mm and rack & pinion stage (Edmund Optics Inc.) was used for the movement of nozzle. It can move up to maximum traveling distance of 100mm. Figure 5 explains the setup of electrospinning apparatus.

3. Result

The platform of scaffold with PCL was prepared by electrospinning method. The parameters of ES were PCL concentration, applied voltage, feed rate and TCD, and the details of parametric studies are following.

3.1. PCL concentration

For the experiments, the concentration of PCL was changed from 2w/v% to 8w/v% to fine the optimum solution ratio of electrospun fibers. The samples of electrospun fibers were produced with variable concentration of PCL in solution. The flow rate of 2.0ml/h, TCD of 90mm and applied

voltage 15kV were fixed for the experiments. Electrospun fibers were observed like bead in concentration of 2w/v%. When concentration of PCL increased, the size of sample fibers was changed. It formed from bead shape to net shape fibers and the diameter of fibers decreased. In 8w/v% or higher concentration, the size of fibers increased like Figure 6 and Figure 7. Average values of fibers per PCL concentration is summarized with standard deviation values in Table 1. The minimum size of fibers was observed with 6w/v% concentration of PCL.

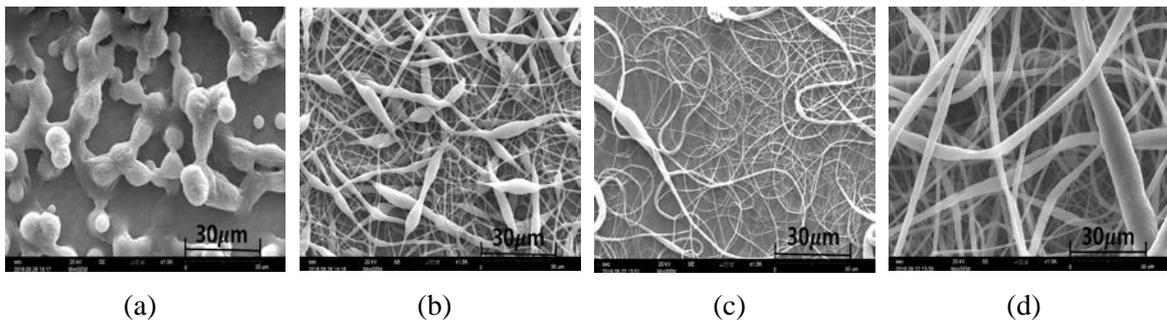


Figure 6. SEM of electrospun PCL composite scaffold with different PCL concentration: (a) 2w/v%, (b) 4w/v%, (c) 6w/v%, (d) 8w/v%.

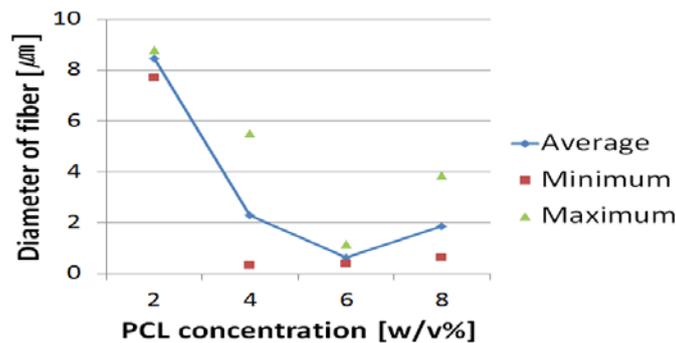


Figure 7. Fibre size distribution per PCL concentration.

Table 1. Average and standard deviation in experiment per PCL concentration.

Concentration of PCL [w/v%]	2	4	6	8
Average of fibres [µm]	8.461	2.293	0.634	1.846
Standard deviation [µm]	0.447	2.446	0.309	1.206

3.2. Applied voltage

In experiment of variable voltage, the fixed parameters were concentration of 6w/v% PCL, flow rate 8.0ml/h and TCD 90mm. When the applied voltage decreases, the fibres were getting thicker and the fibres were getting thinner in higher voltage. The maximum applied voltage was limited at 15kV for the safety of syringe pump which limits the applied voltage at 15kV. SEM images with variable applied voltage are summarized in Figure 8. Figure 9 is summary of fibre size variation per applied voltages by average, minimum and maximum fibres. The quantities of distribution with standard deviation is summarized in Table 2. The minimum size of fibers was observed with 15kV of applied voltage in this experiment with uniform size of fibers.

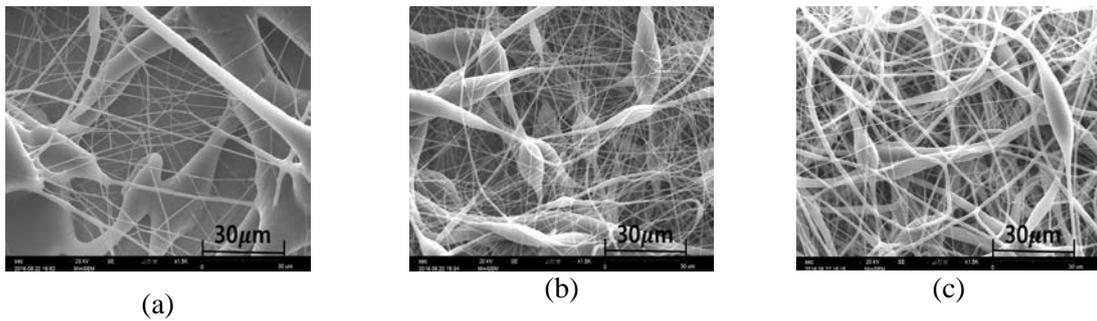


Figure 8. SEM of electrospun PCL composite scaffold with different applied voltage: (a) 5kV, (b) 10kV, (c) 15kV.

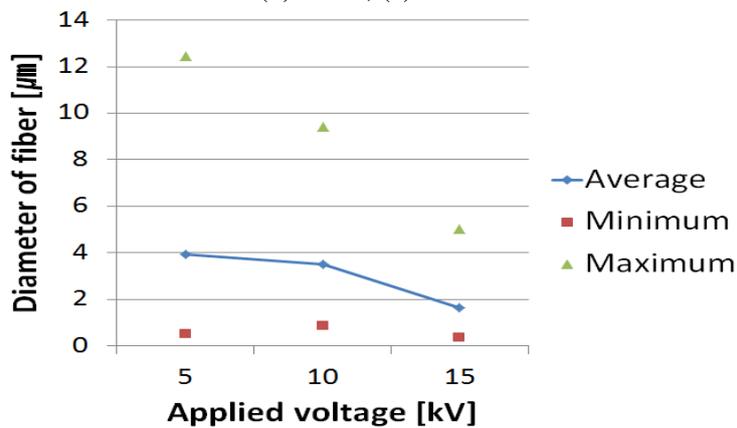


Figure 9. Fibre size distribution per applied voltage.

Table 2. Average and standard deviation in experiment per applied voltage.

Applied voltage [kV]	5	10	15
Average of fibres [µm]	3.932	3.527	1.653.
Standard deviation [µm]	4.999	3.843	1.923

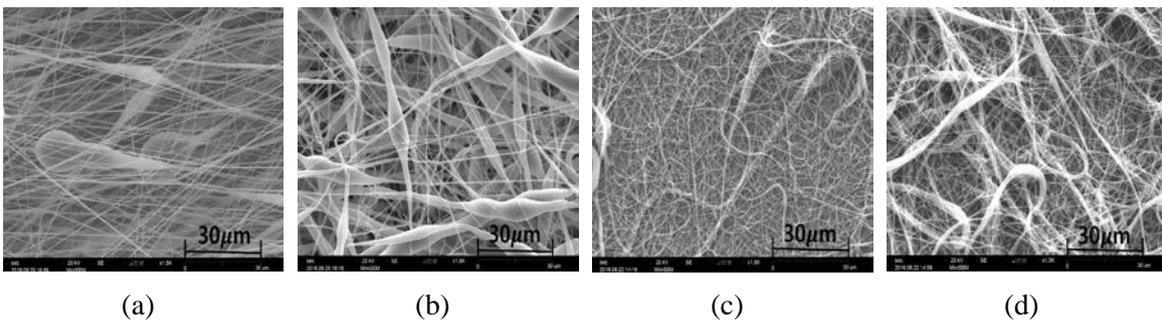


Figure 10. SEM of electrospun PCL composite scaffold with different flow rate: (a) 1ml/hr, (b) 2ml/hr, (c) 4ml/hr, (d) 6ml/hr.

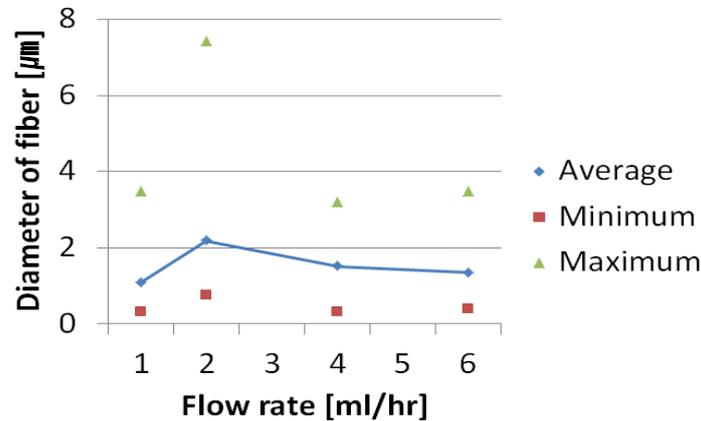


Figure 11. Fibre size distribution per flow rate.

Table 3. Average and standard deviation in experiment per flow rate.

Flow rate [ml/hr]	1	2	4	6
Average of fibres [μm]	1.107	2.196	1.544	1.343
Standard deviation [μm]	1.346	2.928	1.206	1.260

3.3. Flow rate

In this experiment, the flow rate was set as a variable. The concentration of PCL was 6w/v% with applied voltage of 15kV and TCD 90mm in experiment of flow rate. Based on the result of experiment data by the variation of flow rate, the smallest fibre size was observed when flow rate applied 1ml/hr in Figure 10 and Figure 11. The quantities of distribution with standard deviation is summarized in Table 3. The thickest fibre size was observed with 1ml/hr as shown in Figure 11 and Table 3.

3.4. TCD

In this experiment, TCD was set as a variable. The concentration of PCL was 6wt% with flow rate 1ml/h and applied voltage of 15kV. The range of TCD was from 90mm to 130mm. The effect of TCD for fibres size are summarized in Figure 12 and Figure 13. The quantities of distribution with standard deviation is summarized in Table 4. The thickest fibres were observed at 130mm of TCD in this experiment.

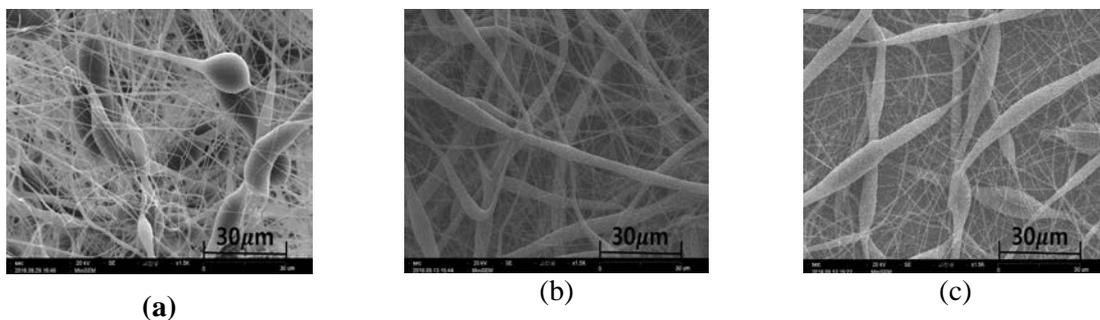


Figure 12. SEM of electrospun PCL composite scaffold with different TCD: (a) 90mm, (b) 110mm, (c) 130mm.

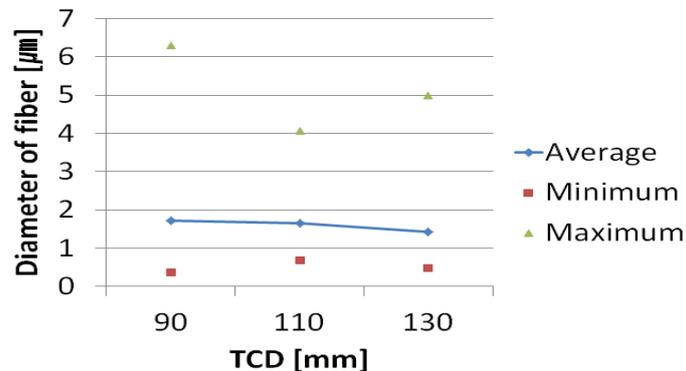


Figure 13. Fibre size distribution per TCD

Table 4. Average and standard deviation in experiment per TCD.

TCD [mm]	90	110	130
Average of fibres [μm]	1.716	1.660	1.439
Standard deviation [μm]	2.565	1.381	1.986

4. Conclusion

In this study, the general effects of nanofiber size by parameters of electrospinning was introduced. Based on the result analysis, it was found that as TCD and applied voltage increased, the diameter of fibres tended to decrease. In flow rate experiment, the size of fibres decreased when flow rate decreased. The optimized PCL concentration was found at 6w/v% for the minimum of fibres.

Acknowledgments

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