

## Design and Construction the Melt Electrospinning System for Fabricating Polymer Electrospun Fibers

### การออกแบบและสร้างระบบอิเล็กโตรสปินนิงแบบหลอมสำหรับผลิตเส้นใยพอลิเมอร์

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#### ABSTRACT

Melt electrospinning is a suitable process for fabricating fibers by molting polymers. The melt electrospinning system includes a high voltage supply, a syringe pump, a fiber collector and a heating system. Therefore, the purpose of this study was to design and construct the syringe pump and the heating system of the melt electrospinning system. The syringe pump was driven by a stepper motor that controlled by a microcontroller "Arduino". A temperature controller of the heating system controlled the maximum temperature up to 400 °C through a band heater. To demonstrate the functionality of the fabricated melt electrospinning system, polycaprolactone (PCL) fibers were fabricated. The results showed that the melt electrospinning system could successfully fabricate PCL fibers which had slightly rough surface with diameter of micrometer range.

#### บทคัดย่อ

อิเล็กโตรสปินนิงแบบหลอมคือกระบวนการที่ใช้ในการผลิตเส้นใยจากการหลอมพอลิเมอร์ ในระบบอิเล็กโตรสปินนิงแบบหลอมประกอบไปด้วย แหล่งกำเนิดศักย์ไฟฟ้ากำลังสูง อุปกรณ์ฉีดพอลิเมอร์ หลอมเหลว วัสดุรองรับเส้นใยและระบบทำความร้อน ดังนั้นในการศึกษานี้มีวัตถุประสงค์เพื่อที่จะออกแบบและสร้างอุปกรณ์ฉีดพอลิเมอร์หลอมเหลวและระบบทำความร้อนสำหรับระบบอิเล็กโตรสปินนิงแบบหลอมที่สามารถผลิตเส้นใยได้จากการหลอมพอลิเมอร์ โดยอุปกรณ์ฉีดพอลิเมอร์หลอมเหลวถูกควบคุมการขับเคลื่อนด้วยการหมุนของสเต็ปเปอร์มอเตอร์ ซึ่งสเต็ปเปอร์มอเตอร์จะถูกควบคุมการหมุนด้วยโปรแกรมที่ทำงานบนไมโครคอนโทรลเลอร์ตระกูล Arduino และระบบทำความร้อนประกอบไปด้วย ส่วนควบคุมอุณหภูมิที่สามารถควบคุมอุณหภูมิของฮีตเตอร์ได้สูงสุด 400 องศาเซลเซียส ส่วนควบคุมอุณหภูมิจะคอยควบคุมการป้อนกระแสผ่านฮีตเตอร์แบบรัดท่อ ระบบอิเล็กโตรสปินนิงแบบหลอมที่ได้สร้างขึ้นมานี้ได้ทำการทดสอบในการผลิตเส้นใยพอลิคาโพรแลคโตน พบว่าสามารถผลิตเส้นใยพอลิคาโพรแลคโตนได้ โดยเส้นใยมีผิวขรุขระเล็กน้อยและมีขนาดเส้นผ่านศูนย์กลางของเส้นเฉลี่ยในระดับไมโครเมตร

**Keywords:** Melt electrospinning, Polymer fiber, Polycaprolactone

**คำสำคัญ:** อิเล็กโตรสปินนิงแบบหลอม เส้นใยพอลิเมอร์ โพลีคาโพรแลคโตน

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## Introduction

Micro-/nano fibers possess several amazing properties such as a high surface to volume ratio, high porosity, flexibility in surface, etc (Ramakrishna et al., 2005). Electrospinning is one of the processes for fabricating micro-/nano fibers by electrostatic force. It can be divided into two sub-technologies: solution electrospinning and melt electrospinning. The solution electrospinning, consisting of a high voltage supply, a syringe pump and a fiber collector, can fabricate fibers with diameter ranging from hundreds of micrometer to nanometer from various polymer solution. However, the major challenges of solution electrospinning are potential environmental pollution of solvent accumulation and residue of solvent in fibers. These drawbacks largely influence mass production and biomedical applications. Meanwhile, the melt electrospinning, which requires a heating system, is an ecofriendly process. It fabricates nontoxic fibers from molten polymer. Therefore, electrospun fibers from molten polymer without a residue of solvent provide opportunities in biomedical applications such as tissue engineering, wound dressings, drug deliveries, filtration and textiles (Muerza-Cascante et al., 2014; Lee et al., 2006). However, the melt electrospinning fabricates fibers with diameters in a micrometer range because resistivity and viscosity of molten polymer are higher than these of polymer solutions.

Currently in our laboratory at the Department of Physics, we have built many electrospinning systems. However, those electrospinning systems can only fabricate fibers from polymer solution since the heating components material which is used were not included. This prevents the opportunities for fabrication of polymers from molten state and limits the potentials for some applications.

In this study, we fabricated the melt electrospinning system. A syringe pump and a heating system of the melt electrospinning system were designed and constructed. For demonstration of the constructed machine, polycaprolactone (PCL) fibers were fabricated in laboratory scale. The morphologies of the PCL fibers versus the processing parameters were observed.

## Objective of the study

The aim of this study was to construct the melt electrospinning system and to demonstrate the fabrication of electrospun fibers from such system.

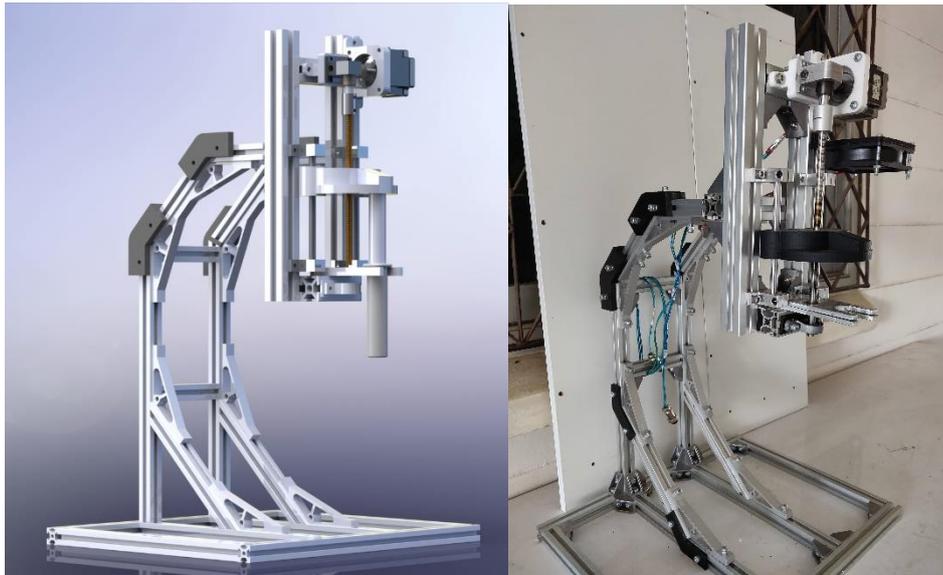
## Material and methods

### Melt electrospinning system

The melt electrospinning system consists of a high voltage supply, a syringe pump, a fiber collector and a heating system. The high voltage supply was already available at the Department of Physics. The high voltage supply provides a maximum voltage output of 25 kV. However, other components of the melt electrospinning system

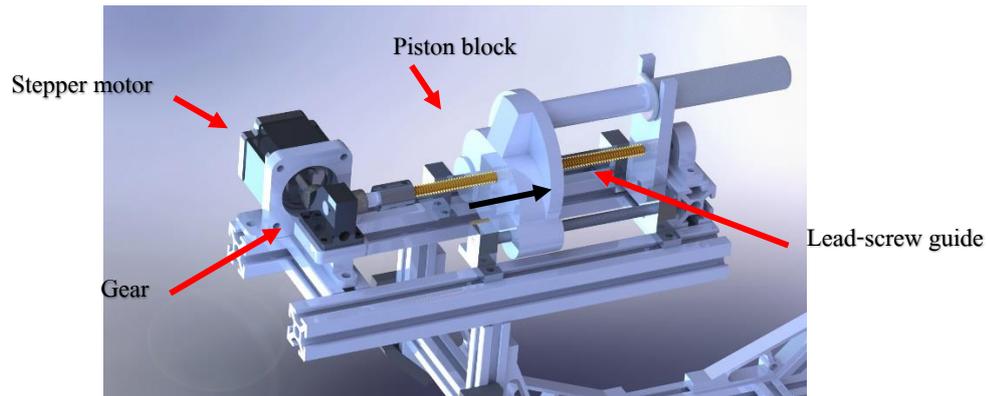
were not available. Therefore, a syringe pump, a fiber collector and a heating system were constructed in this study and assembled to make the melt electrospinning system.

The syringe pump of the melt electrospinning system was designed to perform in a vertical direction. The model of syringe pump is shown in Figure 1.



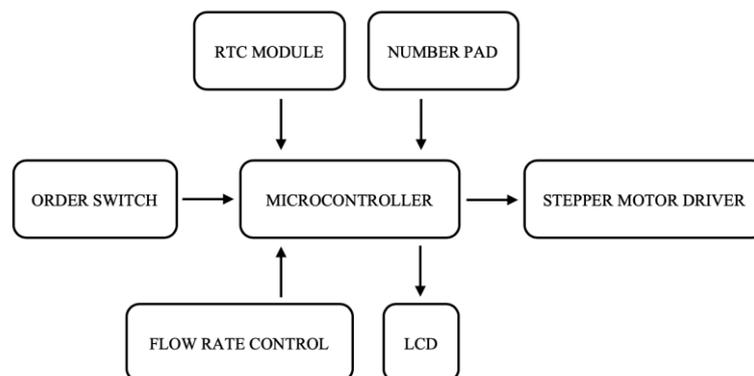
**Figure 1** The model (left) and the construction (right) of the syringe pump in the melt electrospinning system

The mechanism of the syringe pump is demonstrated in Figure 2. The piston block is driven by a stepper motor (1.8 degree/step). The stepper motor is connected to piston block through a gear. The rotation of the axial stepper motor is converted to a linear motion by a lead-screw guide (pitch = 1 mm). The lead-screw guide moves up or down depending on the direction of rotation of stepper motor. When the lead-screw guide moves down, the piston block moves downward, that means the piston block pushes the molten polymer in the syringe to the nozzle. Therefore, the flow rate of the molten polymer is determined by the rotation speed of the stepper motor.

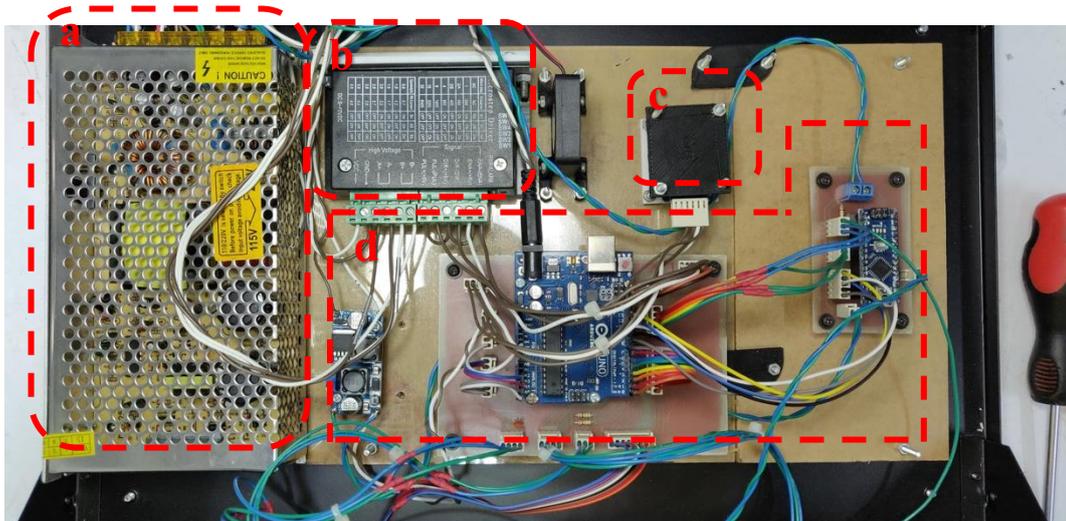


**Figure 2** The mechanism of the syringe pump

Figure 3 and 4 show a circuit diagram and an electronic circuit unit for the syringe pump control. A microcontroller, Arduino (Figure 4d), is employed to control the stepper motor by sending the voltage output through the stepper motor driver (Figure 4b) and receives the voltage input from an order switch (Figure 5), a potentiometer (Figure 5), a real time clock (RTC) module (Figure 4c) and a number pad (Figure 5). Three order switches control the operation of the stepper motor such as start operation, stop operation and move backward operation. To control rotation speed of the stepper motor, the potentiometer which is an adjustable resistor is used to alter the delay time between steps of the stepper motor. To set the time to stop operation of the syringe pump, the RTC module is added on the circuit board. Users can set time to stop operation by a number pad in the front panel control (Figure 5). When the time to stop operation corresponds to the real time, the piston block stops for preventing damage to the device. Lastly, the status of the syringe pump such as a status of operation, a flow rate, a real time and a time to stop operation is shown on a liquid crystal display (LCD) in the front panel control (Figure 5).

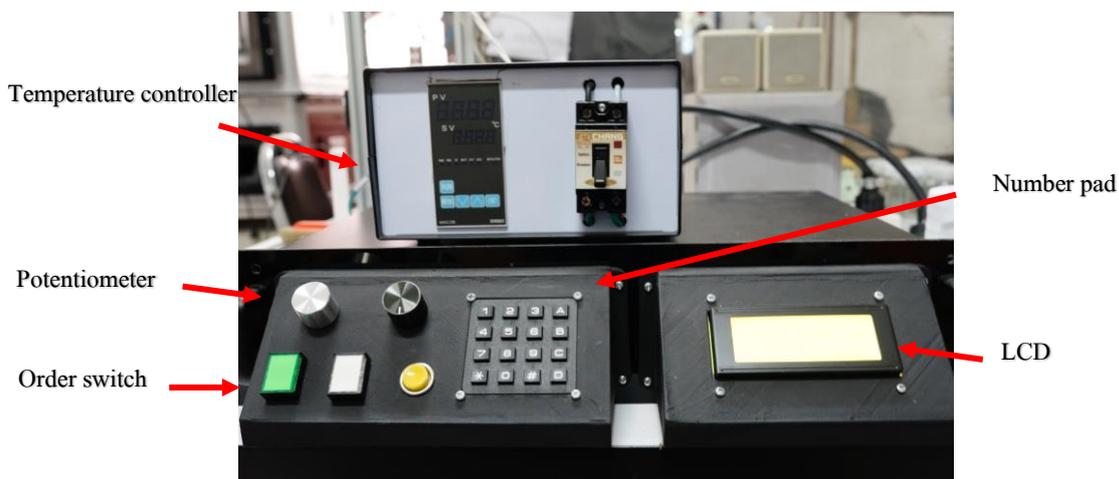


**Figure 3** A circuit diagram of the syringe pump control



**Figure 4** An electronic circuit unit of the syringe pump control (a) power supply unit, (b) stepper motor driver, (c) real time clock module and (d) microcontroller unit

The heating system that constructed in this study is composed a temperature controller, a band heater and a thermocouple. The temperature controller (Figure 5) can control the temperature up to 400 °C through a band heater (190 w). The temperature is measured using a K-type thermocouple.



**Figure 5** The front panel control and the temperature controller of the syringe pump

## Material

To demonstrate the fabrication of electrospun fibers, the polycaprolactone (PCL) with average  $M_w$  of 48000, average  $M_n$  of 45000 and melting point of around 60 °C was used in this study.

## Electrospinning conditions

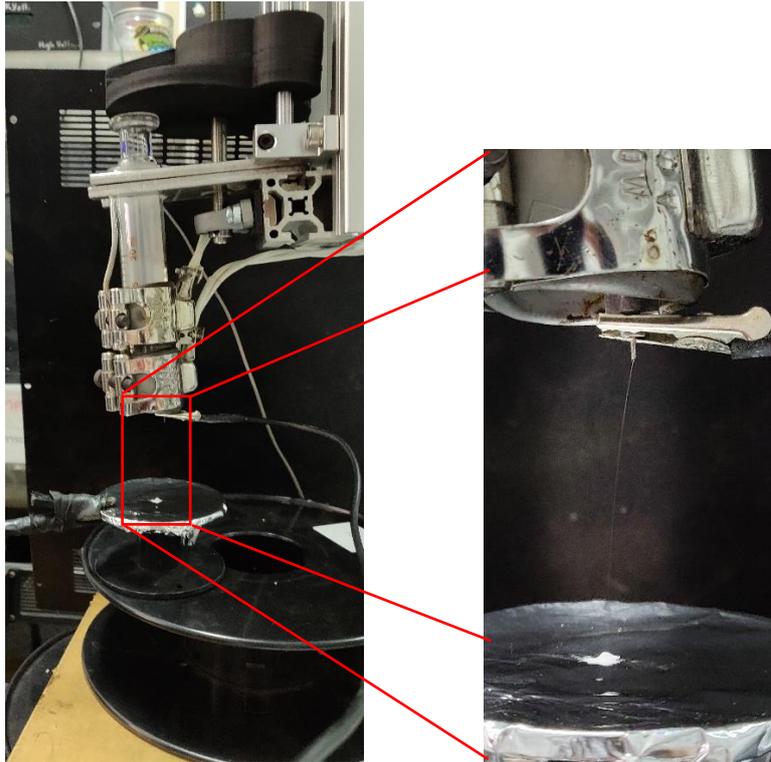
The PCL pellets-like were loaded into the glass syringe and heated to 120 °C for about 25 minutes. To reduce the interference between the high voltage supply and the heating system, a negative voltage was applied to the nozzle of the glass syringe and a positive voltage of 20 kV applied to the fiber collector. A fiber collector was placed at 45 mm from the nozzle and the nozzle size was 0.7 mm. The molten polymer was pumped with a flow rate of 0.04 mL/h. For studying the effect of high voltage on fiber diameter, a high voltage was varied from 16 kV to 20 kV, while other processing parameters were kept constant, i.e. spinning temperature of 120 °C, distance of fiber collector of 45 mm, nozzle size of 0.7 mm and flow rate of 0.04 mL/h.

## Characterization

The morphologies of electrospun PCL fibers were observed by scanning electron microscopy (SEM: SNE-4500M, SEC). The average fiber diameter and standard deviation (SD) were measured from the SEM image.

## Results and Discussions

The fiber collector which made from plastic stub with size of 8 cm x 8 cm was covered by aluminum foil. The fiber collector was placed directly below the glass syringe and once report, the molten jet of polymers can be visualized. This demonstrated that our home-made melt electrospinning system worked and could produce polymer fibers from molten polymer (Figure 6). From Figure 7, the electrospun PCL fibers was deposited on the fiber collector in a small area because the molten polymer was ejected as a straight jet towards the fiber collector (Dalton et al., 2007). This is unlike in the solution electrospinning process where the electrospun fibers were formed spreading to other parts of the system due to the electrostatic force (Larrondo et al., 1981).



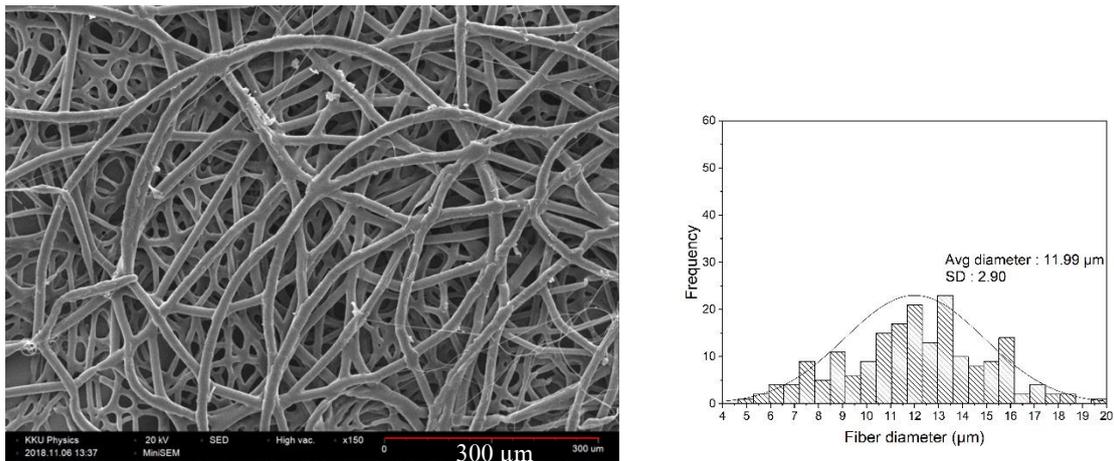
**Figure 6** The melt electrospinning process of the electrospun PCL fibers



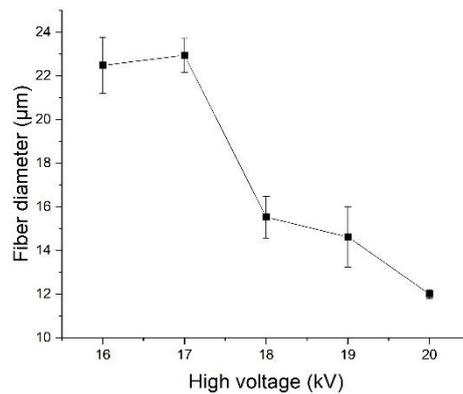
**Figure 7** The electrospun PCL fibers on fiber collector

The surface morphology and the fiber diameter distribution of the electrospun PCL fibers are demonstrated in Figure 8. It can be observed that the fibers have slightly rough surfaces but relatively uniform dispersed. The average fiber diameter of the electrospun PCL fibers and the SD were 11.99  $\mu\text{m}$  and 2.90, respectively.

Figure 9 shows relationship between high voltage and average fiber diameter of the electrospun PCL fibers. As the high voltage increases, the average fiber diameter decreases, corresponding to the previous studies (Tian et al., 2009; Nazari et al., 2018). At the high voltage of 16 kV, the electrostatic force was enough to overcome the surface tension and viscosity force of molten polymer, but a whipping motion of melt jet was suppressed, leading to a large average fiber diameter around 22  $\mu\text{m}$ . At higher high voltage, the whipping motion of melt jet appeared in the region near the fiber collector (Zhou et al., 2006) leading to a lower average fiber diameter.



**Figure 8** The SEM image of electrospun fibers in 150 order of magnitude and fiber diameter distribution



**Figure 9** The effect of high voltage on average fiber diameter

## Conclusion

In this study, the melt electrospinning system has been successfully constructed. The syringe pump was designed and fabricated. The syringe pump performs in the vertical direction and work by the operation of stepper motor which controlled via a microcontroller. Users can control operation of syringe pump by adjusting input voltage from a switch, a potentiometer and a number pad in the front panel control. Apart from that, the heating system was integrated. To demonstrate the machine, the electrospun PCL fibers were produced with a diameter of around 12  $\mu\text{m}$ . By studying the effect of high voltage on average fiber diameter, it was shown that the higher the voltage, the smaller fiber diameter.

## Acknowledgement

This study was supported by the Research Fund for Supporting Lecturer to Admit High Potential Student to Study and Research on His Expert Program Year 2017, the Thailand Research Fund (TRF) in cooperation with Khon Kaen University (RSA5980014) and the Institute of Nanomaterials Research and Innovation for Energy (IN-RIE), Khon Kaen University and the National Nanotechnology Center (NANOTEC), NSTDA, Ministry of Science and Technology, Thailand, through its program of Research Network NANOTEC (RNN).

## References

- Dalton PD, Grafahrend D, Klinkhammer K, Klee D, Moller M. Electrospinning of polymer melts: Phenomenological observations. *Polymer* 2007; 48(23): 6823-6833.
- Larrondo L, John Manley RS. Electrostatic fiber spinning from polymer melts. I. Experimental observations on fiber formation and properties. *Journal of polymer science : polymer physics edition* 1981; 19(6): 909- 920.
- Lee S, Obendorf SK. Developing protective textile materials as barriers to liquid penetration using melt-electrospinning. *Journal of applied polymer science* 2006; 102: 3430-3437.
- Muerza-Cascante ML, Haylock D, Hutmacher DW, Dalton DP. Melt electrospinning and its technologization in tissue engineering. *Mary Ann Liebert, Inc.* 2014; 00(00): 1-16.
- Nazari T, Garmabi H. The effects of processing parameters on the morphology of PLA/PEG melt electrospun fibers. *Society of chemical industry* 2018; 67: 178-188.
- Ramakrishna S, Fujihara K, Teo WE, Lim TC, Ma Z. *An introduction to electrospinning and nanofibers.* Singapore: World Scientific Publishing Co. Pte. Ltd; 2005.
- Tian S, Ogata N, Shimada N, Nakane K, Ogihara T, Yu M. Melt electrospinning from poly(L-lactide) rods coated with poly(ethylene-co-vinyl alcohol). *Journal of applied polymers science* 2009; 113: 1282-1288.
- Zhou H, Green TB, Joo YL. The thermal effects on electrospinning of polylactic acid melts. *Polymer* 2006; 47(21): 7497-7505.