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ABSTRACT

CURRENT MEASUREMENTS IN ELECTROSPINNING

By

Clinton K. Lien

Electrospinning provides an economical method of producing nanofibers. The current carried by the main spinning jet is found to be one of the factors in determining the diameter of the fiber product. However, current sources such as the corona discharge and secondary jets will lead to a systematic overestimation of the actual current value.

In this research, experiments with different configurations are set up to investigate the influence of various parameters on the measured current. It is noticed that the measured current is nearly independent on the flow rate of the solution and the external cover. A large amount of current is detected even the syringe is empty when the experiments are carried out using conductive needle. By substituting the standard electrode with penetrating electrode, the current dropped to zero when the syringe was empty. An average of 0.26E-6 Amps reduction on the amount of measured current is observed when the syringe is filled with methylene chloride. In all cases that non-conductive Teflon needle is applied, a significant lower current is observed. However, experiments conducted using 12 wt% PCL polymer solution show nearly undetectable current value in all configurations. For the current behavior on outer collector plate, the measured current on the outer plate are extremely low regardless of needle type, electrode type, and syringe content conditions.

CURRENT MEASUREMENTS IN ELECTROSPINNING

by

Clinton K Lien

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Submitted to Faculty of
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TABLE OF CONTENTS

TABLE OF CONTENTS
(Continued)

Chapter **Page**

LIST OF TABLES

LIST OF FIGURES

LIST OF FIGURES

(Continued)

Figure **Page**

CHAPTER 1

INTRODUCTION

1.1 Objective

There is a wide variety of potential uses of nanofibers, which have diameters in the range from several micrometers down to tens of nanometers. Electrospinning, considered as an economical technique to produce nanoscale fibers, is a process by which a charged liquid polymer solution is introduced into an electric field.

The interaction between the charged liquid and electric field causes a jet of liquid to be ejected, elongated, and deposited on the collector plate. The current on the jet was found to be one of the factors in determining the diameter of the fibers obtained in electrospinning process [1]. Therefore, the measurement of the current on the polymer jet is an interesting topic in the electrospinning process. Unfortunately, the current measured from the collector usually does not represent the actual charge carried by the spinning jet due to various factors.

Previous studies showed that the current measured at the collector can be composed of several components such as (1) the current carried by the solution in the main jet; (2) the current that carried by secondary jet, which is sprayed from the main jet at the undulation points [1]; and (3) the current produced by the corona discharge from the conductive needle [2, 3]. Therefore, it is logical to assume that the current coming from the secondary jet, corona discharge or other sources will lead to a systematic overestimation on the current carried by the main jet. Considering the current measurements, aspects of the charge flow other than the main jet, has not being explored in detail. Further, the generation and transport of the charges are rarely discussed. To enhance understanding on

the nature of the current in electrospinning process and to manipulate the final quality of fiber product, it is important to investigate the composition and sources of the currents measured at the collector. The objectives of this research include, identifying and describing multiple sources of charges that may affect the accuracy of main spinning current measurement, evaluating the current measured at the collector by designing various experimental configurations. Observations from previous research have shown that current is present not only during electrospinning but also present when there is no liquid contents inside the syringe [2], therefore the current generated from the needle was emphasized in this research.

1.2 Nanofibers

In recent years there has been a push towards nanotechnology in many engineering disciplines. Nanotechnology comprises technological developments on the nanometer scale, usually 0.1 to 100 nm [4]. Fiber creation using polymers has evolved through this growing technology.

Fibers in nano scale offer several properties, including high surface area to volume ratio, high interstitial spacing, flexibility in surface functionalities, and superior mechanical performance [5]. These outstanding properties of electrospun nanofibers make them to be the good candidates for a wide range of important applications [6].

There is a wide variety of potential uses of nanofibers. One of the many important applications of nanofibers is in nanosensors [7, 8] that utilize the electronic properties associated with these fibers. Nanofibrous materials with specific interstitial spacing can be used as chemical and mechanical filters. These are ideally suited for filtering submicron particles from air or water. Properly designed fibrous mat can trap and dissolve certain

chemical and biological components through chemical reactions. Therefore, nonofiber becomes a good material to make nonsensors.

Nanofibers are also used in medical applications, which include, drug and gene delivery, artificial blood vessels, artificial organs, tissue engineering and medical facemasks [9]. For example, carbon fiber hollow nanotubes, smaller than blood cells, have potential to carry drugs in to blood cells.

Other applications of nanofibers include supercapacitors, separators in lithium ion battery, fuel cells, and solar cells [10]. These are just a few of the numerous ways that nanofibers can be used.

Conventional fiber spinning techniques, which rely on mechanical forces to produce fibers by extruding polymer melt or solution through a spinnerette and subsequently drawing the resulting filaments as they solidify or coagulate [11]. However, conventional mechanical fiber spinning techniques have difficulties producing fibers with diameters less than 2 μm robustly [12].

In contemporary technology, many applications require fibers in nano size, which has diameter in nm range. Electrospinning offers a different approach to produce fiber as small as few nanometer (see Figure 1.1).

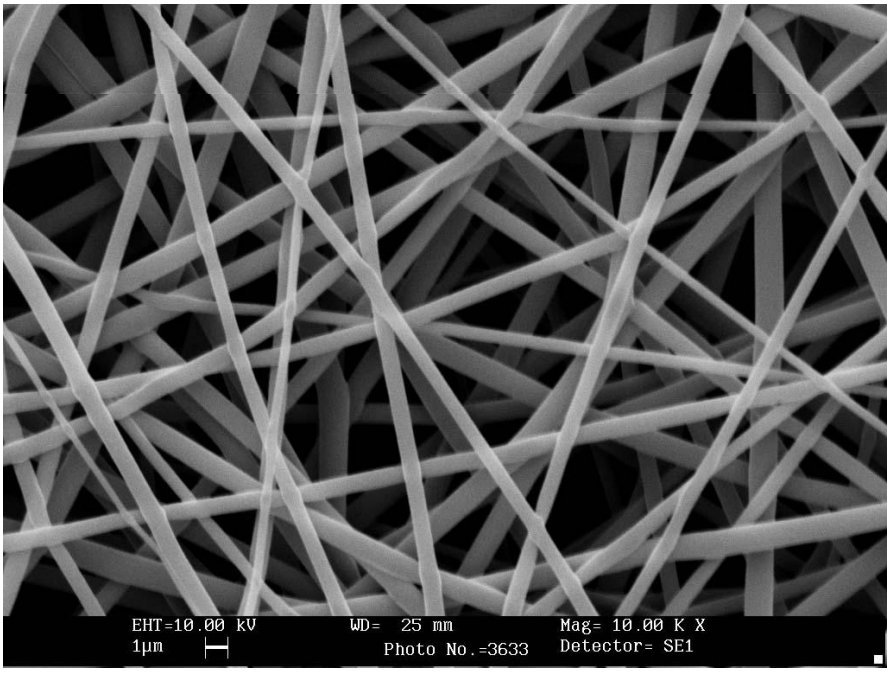

Figure 1.1 Micrograph of electrospun fibers [13].

1.3 Electrospinning

Electrospinning is an economical and simple technique that can be used to produce polymeric fibers of various morphologies and sizes, inexpensively. The fibers prepared by this method have diameters typically ranging over several orders of magnitude, from the micrometer range down to the nanometer range. These diameters are comparable to the fiber diameters of native components of the extracellular matrix.

Electrospinning involves the use of a high voltage to initiate and accelerate a liquid jet from the tip of a syringe, and the fabrication process is based on the principle that stronger electrical force overcomes weaker surface tension of a solution at certain threshold to eject a liquid jet. Therefore, a typical electrospinning apparatus consists of three major components: a syringe with a conductive needle connected to a pump device, a collector, and a high voltage power source. The schematic illustration of the basic setup for

electrospinning is shown in Figure 1.2. A syringe containing liquid polymer solution is mounted a fixed distance away from a collector. The polymer solution loaded in the syringe usually consists of natural or synthetic polymer combined with volatile organic solvent [14]. The needle of the syringe is connected to a high voltage power device. The voltage supplied to the solution provides a mechanism for generating a volume electric charge.

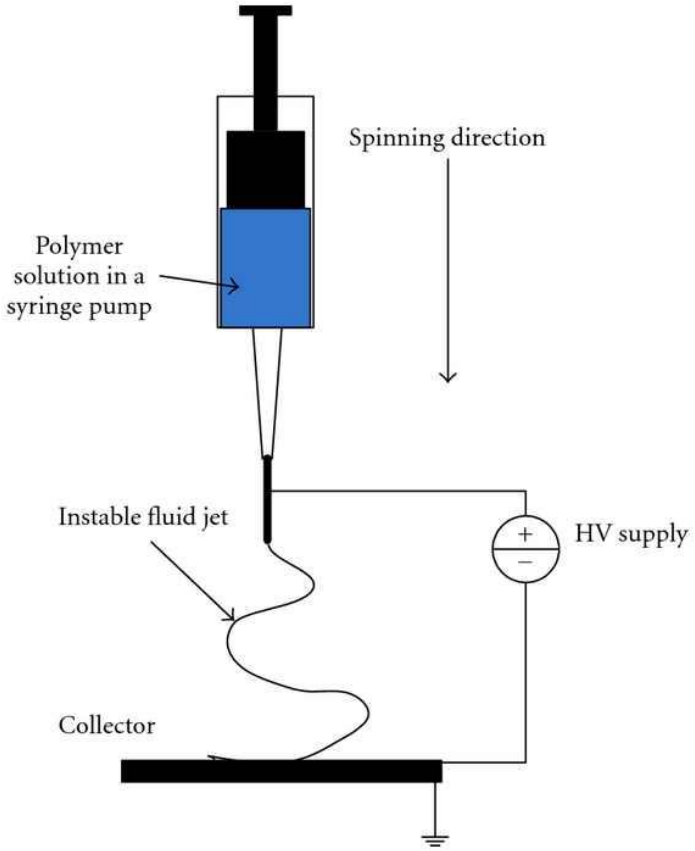

Figure 1.2 Standard experimental setup of electrospinning process in vertical direction [15].

In the process of electrospinning, the polymer solution is pumped at a constant rate from the syringe by the pump device. A droplet accumulates at the tip of the needle and the surface is stabilized by surface tension. By applying a high positive voltage on the needle,

the collector generates a background electric field with negative charges distributed on the plate. Meanwhile, a separation of charges is created inside the needle; negative charges were induced and attract near the needle wall, whereas the remaining positive charges converge in the center area.

Due to the separation of charges and existence of electric field, an electrostatic attraction force will be produced on the droplet which offsets the surface tension once the voltage is applied. As the electrostatic force is dominating, the droplet of solution at the tip of the needle is deformed into a conical shape, typically referred as a Taylor cone. The Taylor cone becomes increasingly unstable as voltage is increased as shown in Figure 1.3. The competition between the electrostatic force and the surface tension continues until the strength of electric field has surpassed a certain critical value which the surface tension can no longer maintain its static equilibrium. At this point, a liquid jet is ejected entangled polymer molecules that drawn into a tube. The electrified liquid jet is highly attracted, accelerated, and finally deposit on the grounded collector. During the traveling path, the jet undergoes a series of electrically induced bending instabilities and displays a whipping motion that result in the stretching of the jet in to a fiber [16]. The jet is thus drawn and its diameter is gradually reduced due to the evaporation of the solvent. The solidified fiber is often deposited as a randomly oriented, nonwoven mat of nanofibers [17].

Figure 1.3 Formation of the Taylor cone. Voltage increases with each stage until equilibrium between surface tension and the electrostatic force is approaching to the limit [18].

Although the setup for electrospinning is extremely simple, the detailed experimental and theoretical analysis reveals that the whole process is highly complex since it is controlled by various types of instabilities such as the Rayleigh instability, an axisymmetric instability, and whipping or bending instability [12]. The whipping or bending instability, which is mainly caused by the electrostatic interactions between the external electric field and the surface charge on the jet, was identified as the one that controlling the acceleration, stretching, elongation and thinning of the electrospun nanofibers [19]. The other types of instability give rise to fluctuations of the radius of the jet and may eventually result in droplet formation.

1.4 Sources of Charge Generation and Transport

Previous research shows that, in an electrospinning process, charges are transported by the main jet, secondary jet, and corona discharge. All of them play an important role in an electrospinning process and deserve a deeper investigation.

1.4.1 Main Jet

The main jet is formed by the flow of the polymer solution ejected from the needle and is deposited on the collector. When the polymer solution is ionized to carry positive charges by the high voltage source, it is attracted by the electromagnetic force induced by the grounded collector to eject out of the needle and deposit on the collector.

1.4.2 Secondary Jet

Previous papers document secondary jetting from the surface of the electrospinning jet [20]. A smooth jet with a circular cross section is stable only at low electric fields. As the electric potential difference between the electrodes is increased, undulations can occur on the surface of the jet. Yarin et al. [20] argued that these undulations grow in amplitude, giving rise to secondary jets that emanate from the surface of the main jet. If the charge density in such regions exceeds a certain threshold, secondary jetting may occur. Bhattacharjee et al. [1] did a measurement based experiment to study the details of secondary jets. They found that second jets can be viewed as the results of an electrospray effect, and is composed primarily of the solvent. Their experiments verified the existence of secondary, polymer-free electrospray that occurs simultaneously during electrospinning. The process of secondary jetting can therefore causes an overestimation of the charge on the main jet during electrospinning.

1.4.3 Corona Charging

Another common method used to charge polymer films was corona charging. This was used to charge the films made from the same solution used to produce the electrospun mats. In corona charging, the polymer sample is placed on an electrode between a high voltage

supply with a conducting needle electrode. The electric field strength around the needle surpasses the ionization threshold of the surrounding atmosphere. This creates a local ionized field, in which the positive and negative charges of the surrounding atmosphere become separated. The ions of the same charge as the voltage supply will be repelled, and travel in discrete paths towards the polymer sample, where they will become trapped within defects [21, 22].

Previous research shows that there is evidence of a corona during electrospinning [2, 18]. This implies that there are at least two sources of space charges: immediate injection of charges into the solution, and corona charge deposition.

The discharge phenomenon can come from various factors; such as the gas ions in the space between the electrodes create a current which could affect the background electric field. The sharp edges at the opening end of the conductive needle also present the possibility for field emission of charge [2].

A substantial current could be detected by measuring the current in the absence of liquid contents [2, 3]. It was assumed that the corona current is the first event to occur once the high voltage is applied.

CHAPTER 2

EXPERIMENTAL

The main focus of the research is to analyze the source of the charges that are transported to the collector plate. In order to develop a quantitative description of the current measured in the electrospinning process, an experimental setup was designed at NJIT to measure the current transport via the spinning jet.

2.1 Materials

To investigate the charge transport more specifically, the experiments were conducted using the basic syringe contents conditions i) without any contents, ii) with solvent only, and iii) with polymer solution in the syringe, respectively. The solvent used was methylene chloride (CH_2Cl_2) from J.T. Baker and the solute applied was Polycaprolactone (PCL) from Aldrich. The polymer solution was created by mixing a 12.0 wt% (13.5 g of PCL and 136.5 g of CH_2Cl_2) solution on a magnetic stirrer for 2 hours at speed 3.

2.2 Apparatus

A schematic diagram and photograph of the experimental setup are shown in Figure 2.1 and Figure 2.2. The design is similar to a standard electrospinning apparatus except an electrometer was included in the circuit to quantify the current transport to the plate. An isolation on the exposed conductive material around the connecting area between the electrometer and the collector plate was attempted. To avoid the distortion on the background electric field, the voltage line was designed to place nearly close and parallel to the syringe stage instead of hanging it down. The experiment was carried out within a large

enclosure which has limited exposure to exterior environment to minimize the possibilities of unpredictable elements that can interfere the final results.

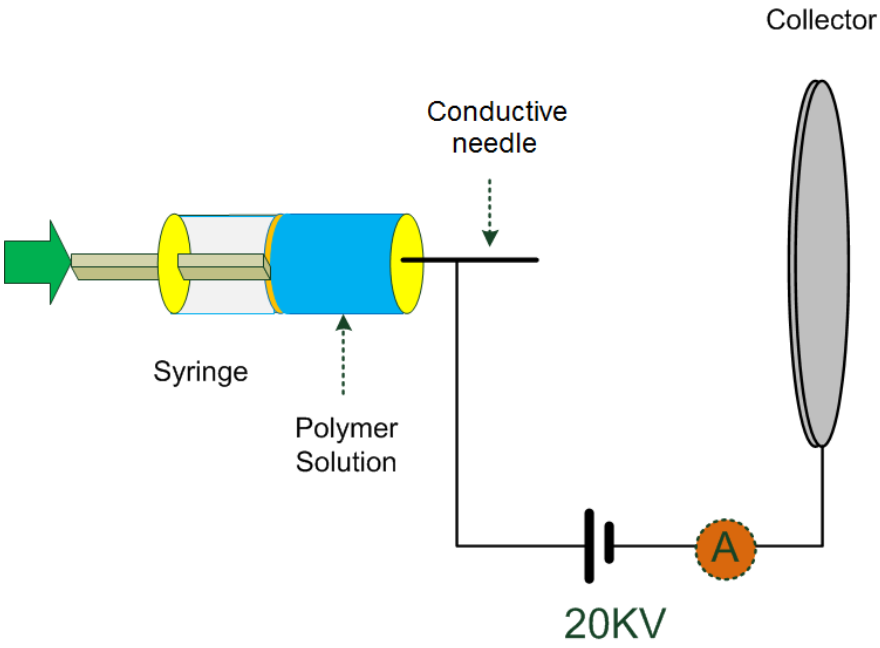

Figure 2.1 Schematic diagram of the basic setup for current measurement in electrospinning.

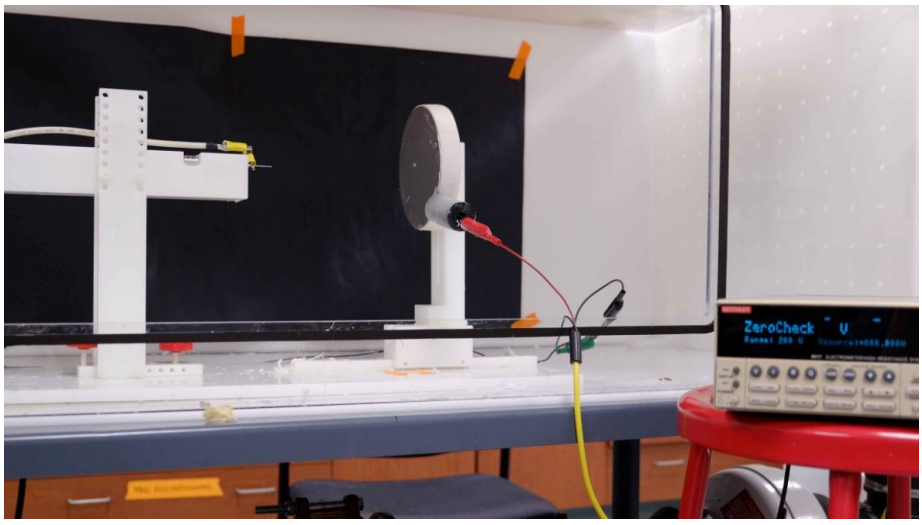

Figure 2.2 Photograph of the basic setup for current measurement in electrospinning.

A 10-mL syringe was used in the experiments. It was driven by a digital syringe pump (Harvard Pump 11 Plus Syringe Pumps), which was set up to control the infusion of the solution at a constant rate. Initially, the experiment was conduct without polymer solution.

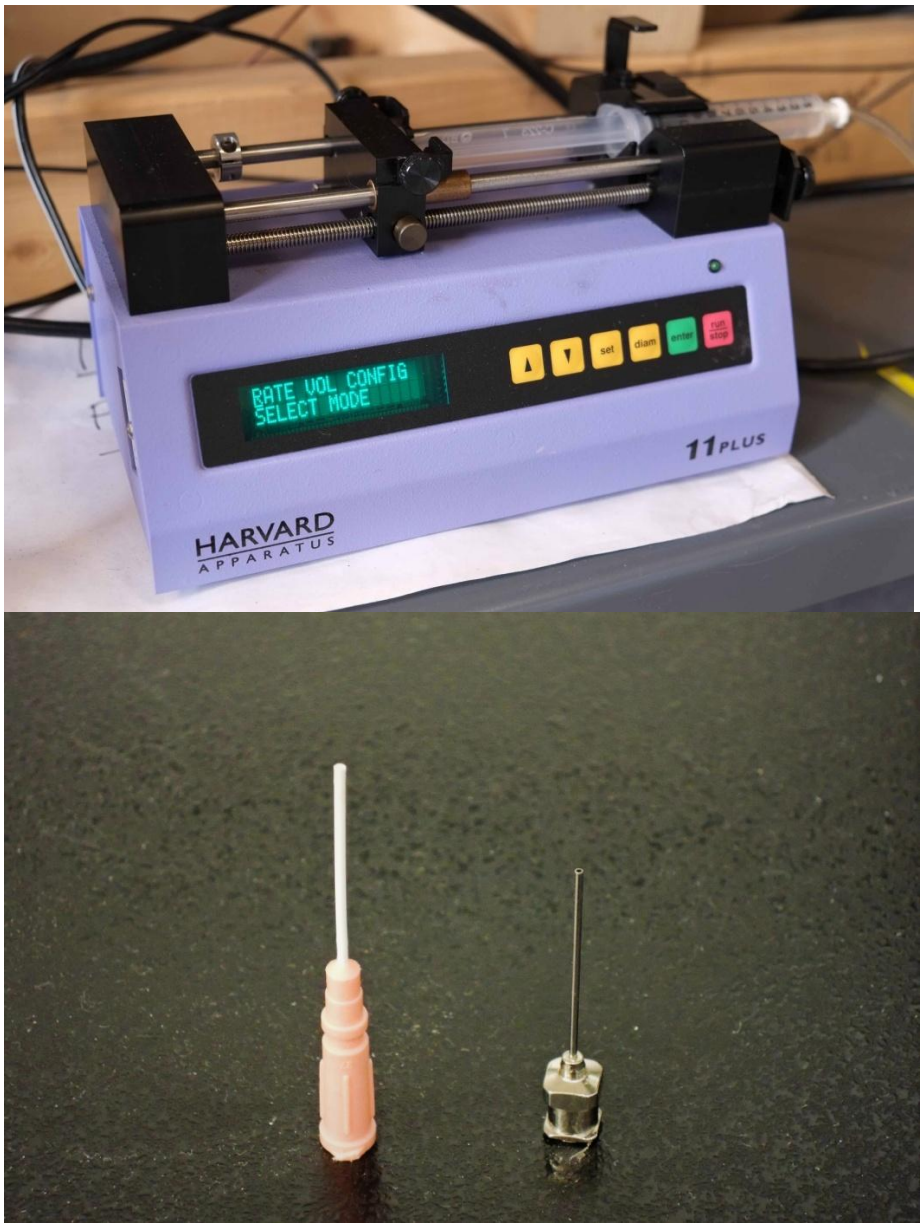

Figure 2.3 Electronic syringe pump used to eject and control the flow rate of the liquid solution (top). Metal needle and Teflon needle used in the experiments (bottom).

A conductive needle with inner diameter of 0.51 mm and a non-conductive Teflon needle with inner diameter of 0.60 mm as shown in Figure 2.3 were used in the experiments to investigate the correlation between the charge transport and needle type. The conductive needle was also used as the electrode of positive voltage (cathode). Figure 2.4 shows the 20 kV voltage supply used in the experiment, which was applied to the solution by connecting the positive voltage to the cathode.

Figure 2.4 High Voltage supply.

A round stainless steel disk of diameter 15.2 cm, was fixed on a Polyethylene stand for collecting purpose. Note that the collector plate is also considered as an anode in a typical electrospinning process after voltage is applied. The setup is shown in Figure 2.5.

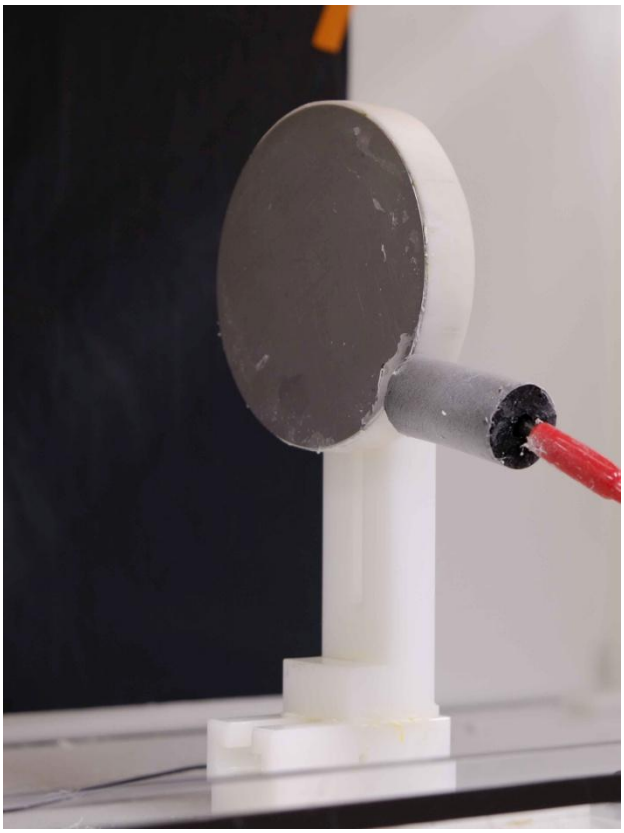

Figure 2.5 Collector plate.

To observe the current profile under different configurations, a device is required to record the current passing through the collector plate. This can be achieved by utilizing a programmable electrometer.

A programmable electrometer (6517A) manufactured by Keithley as shown in Figure 2.6, was used in the experiments to measure the current passing through the collector plate. The measured results were then exported to PC for further analysis.

Figure 2.6 Programmable electrometer used for current measurement in the electrospinning process.

The distance between the tip of the needle and the collector plate was fixed at 17.8 cm. Polymer solution was loaded into a syringe, and was pumped out from the needle at a constant rate. The experiments were set up with five adjustable parameters: a) needle type; b) contents in the syringe (without any solution, with solvent only, or with both solvent and solute); c) charge generation electrode configurations; d) add-on of external Styrofoam cover on the syringe; and e) flow rate of the polymer solution. Thus, several modifications were applied on standard electrospinning apparatus to investigate the charge transport dependencies on various factors.

For the purpose of reducing the possibilities of discharge phenomena generated from the needle, three modifications were applied, 1) a conductive pin was punched into the syringe body, which provides the syringe an interface connecting to the voltage source. The pin is then being used as a new cathode, called penetrating electrode, as shown in Figure 2.7, after the high voltage line is attached. The configuration was modified as shown in Figures 2.8 and 2.9. 2) replacing conductive needle with a non-conductive Teflon needle;

3) a design-to-fit Styrofoam cover as shown in Figure 2.10, was used to cover the syringe body. This modified configuration was intended to minimize any possibility that may cause the collector attract charges from place other than the needle.

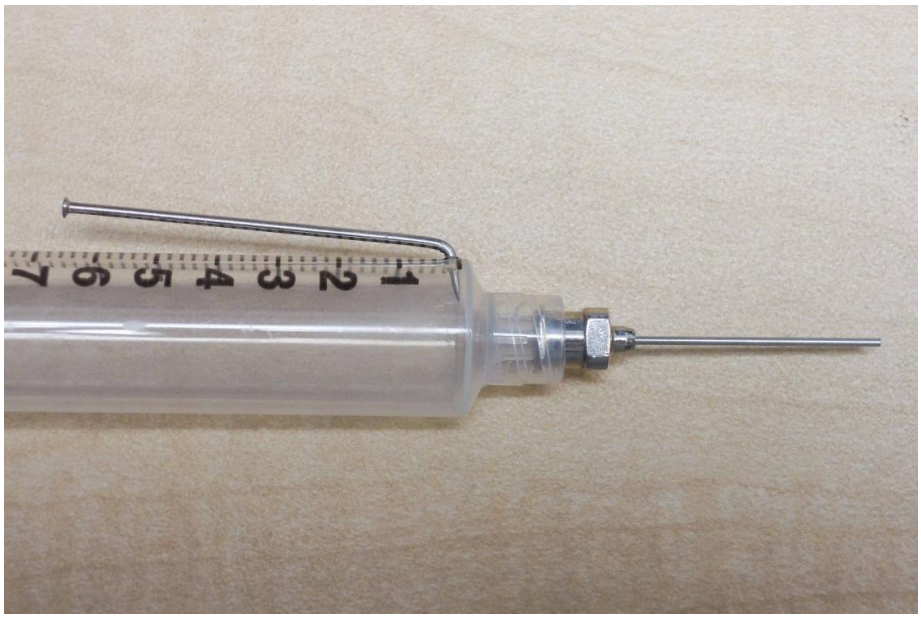

Figure 2.7 Modified syringe assembly used for electrospinning process.

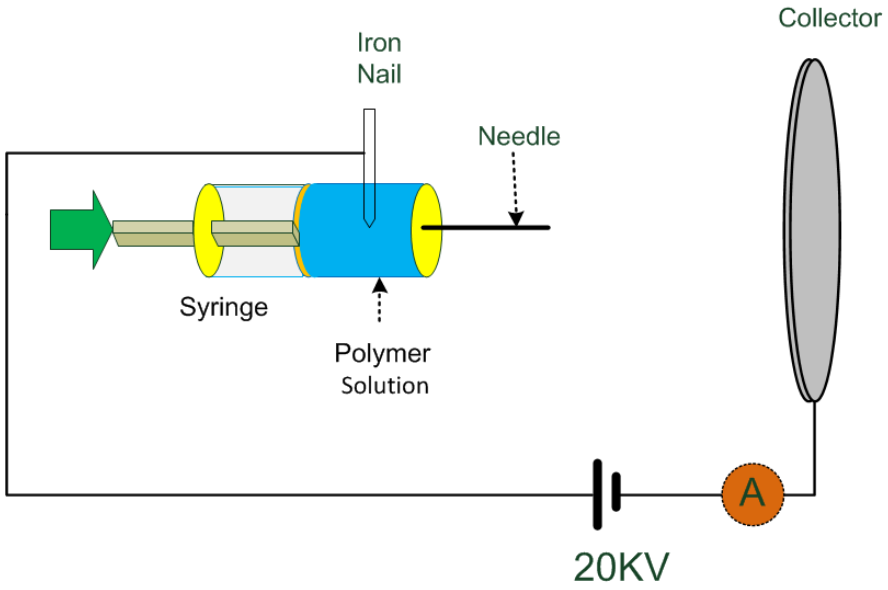

Figure 2.8 Schematic diagram of experimental setup for penetrating electrode.

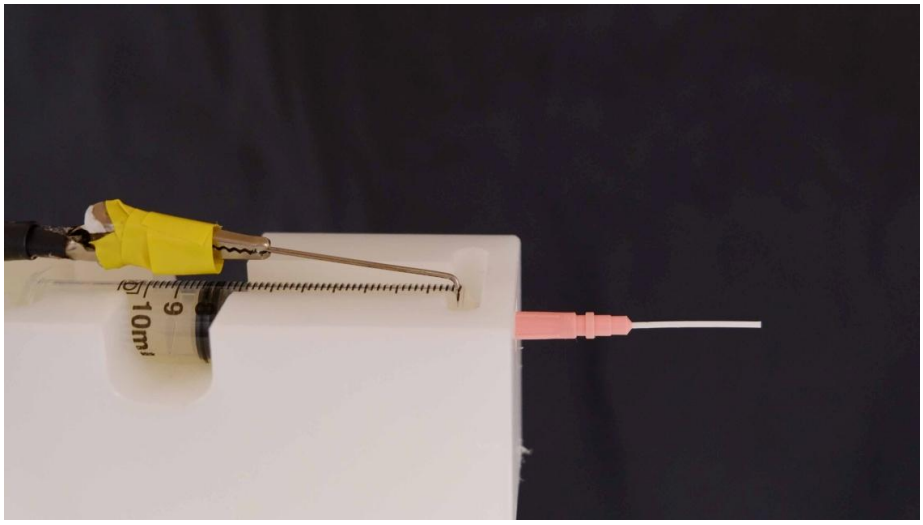

Figure 2.9 Close-up image of experimental setup for penetrating electrode and Styrofoam cover.

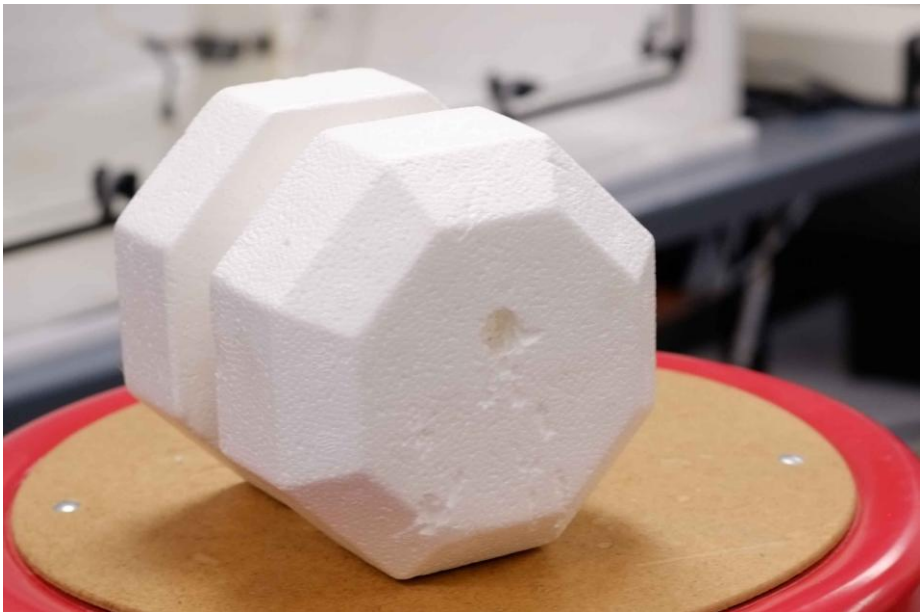

Figure 2.10 Styrofoam external cover.

To verify the dependencies of the results on flow rates, each configuration was repeated twice under two different flow rates: 5.0 and 7.5 mL/hr.

To understand the current distribution better, the collector configuration in the experiments was modified; an additional 21.6-cm diameter stainless steel plate was attached behind the original 15.2-cm diameter plate, the two concentric round electrode plates were separated by a 2.54-cm polyethylene plate as illustrated in Figure 2.11. The exposed surface areas for the two electrodes were equal. The size of the inner plate was chosen to be large enough to ensure that electrospun fibers were deposited exclusively on it during each experiment. Inner plate is used to collect main jet and outer plate is used to collect secondary jet. Since only one electrometer is used, instead of collecting current profile from two plates simultaneously, the experiment was conduct by measuring current flowing through each plate separately. Figures 2.11 and 2.12 show the apparatus of the dual plate configuration.

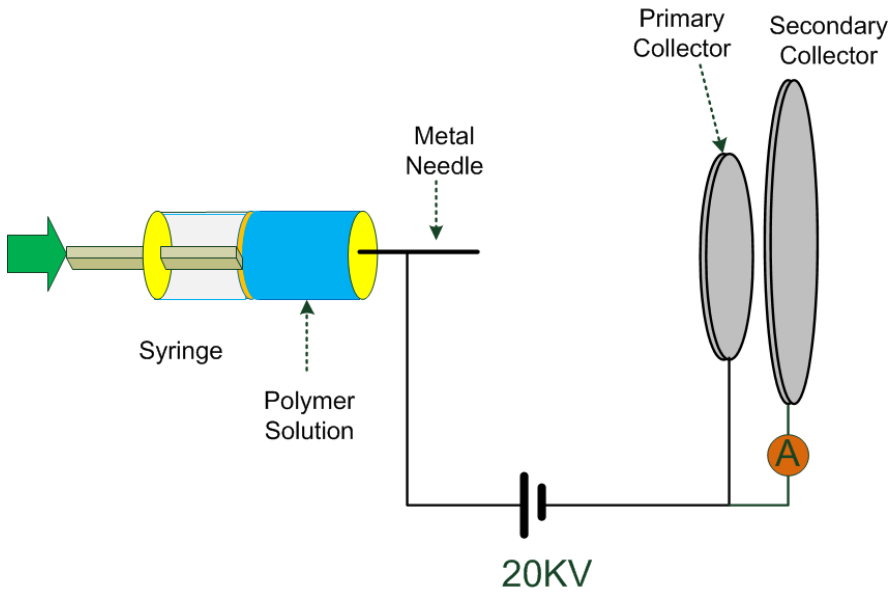

Figure 2.11 Schematic diagram of the dual plate setup for current measurement in electrospinning.

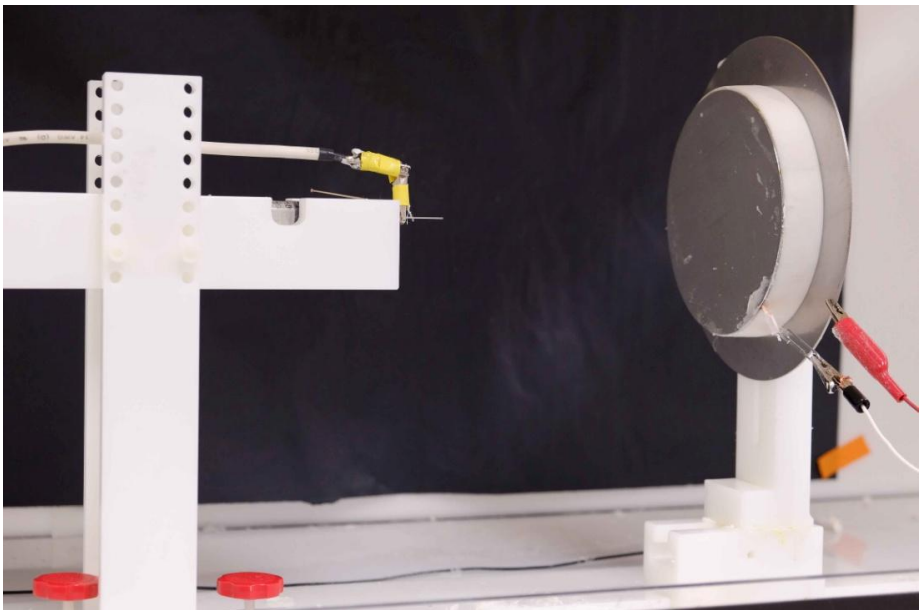

Figure 2.12 Image of the dual plate setup for current measurement in electrospinning.

2.3 Experiments

Table 2.1 shows that 18 sets of configuration were designed and carried out in this research, which are the combination of four parameters: 1) needle type; 2) contents in the syringe; 3) electrode configuration; and 4) add-on of external Styrofoam cover on the syringe. Each of the experiments was repeated under two different flow rates: 5.0 and 7.5 mL/hr, as well as the attachment of additional 21.6-cm outer plate. Each of these conditions was carried out for both single collector electrode and double collector electrode, and performed in triplicate, resulting in a total of 216 attempted experiments.

Table 2.1 Experimental Setup

Experiment	Setup			
	Needle type	**Syringe Contents**	**Penetrating Electrode**	**Styrofoam Cover**
1	Metal	×	×	×
2	Metal	×	×	O
3	Metal	×	O	×
4	Metal	×	O	O
5	Metal	CH_2Cl_2	×	×
6	Metal	CH_2Cl_2	×	O
7	Metal	CH_2Cl_2	O	×
8	Metal	CH_2Cl_2	O	O
9	Teflon	×	O	×
10	Teflon	×	O	O
11	Teflon	CH_2Cl_2	O	×
12	Teflon	CH_2Cl_2	O	O
13	Metal	PCL + CH_2Cl_2	×	×
14	Metal	PCL + CH_2Cl_2	×	O
15	Metal	PCL + CH_2Cl_2	O	×
16	Metal	PCL + CH_2Cl_2	O	O
17	Teflon	PCL + CH_2Cl_2	O	×
18	Teflon	PCL + CH_2Cl_2	O	O

Experiment 1 was conducted by using metal needle and without filling any polymer solution. Experiment 2 has the same configuration with Experiment 1 except the syringe was covered by Styrofoam. Both setups are illustrated in Figure 2.13.

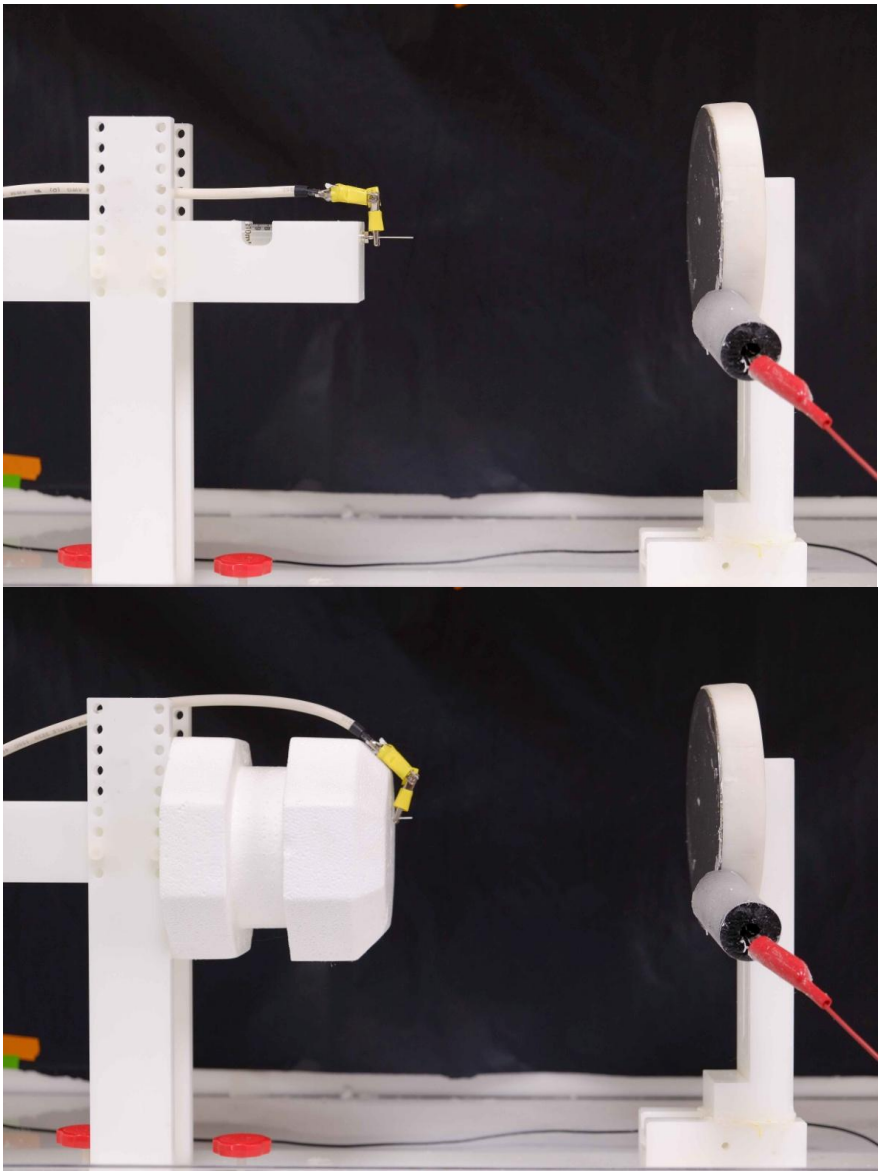

Figure 2.13 Configuration of Experiments 1 (top) & 2 (bottom).

Experiment 3 has same configuration as Experiment 1 except a penetrating electrode was applied on the syringe body to connect to high voltage. The setup of Experiment 4 was kept the same except adding Styrofoam cover. Both setups are illustrated in Figure 2.14.

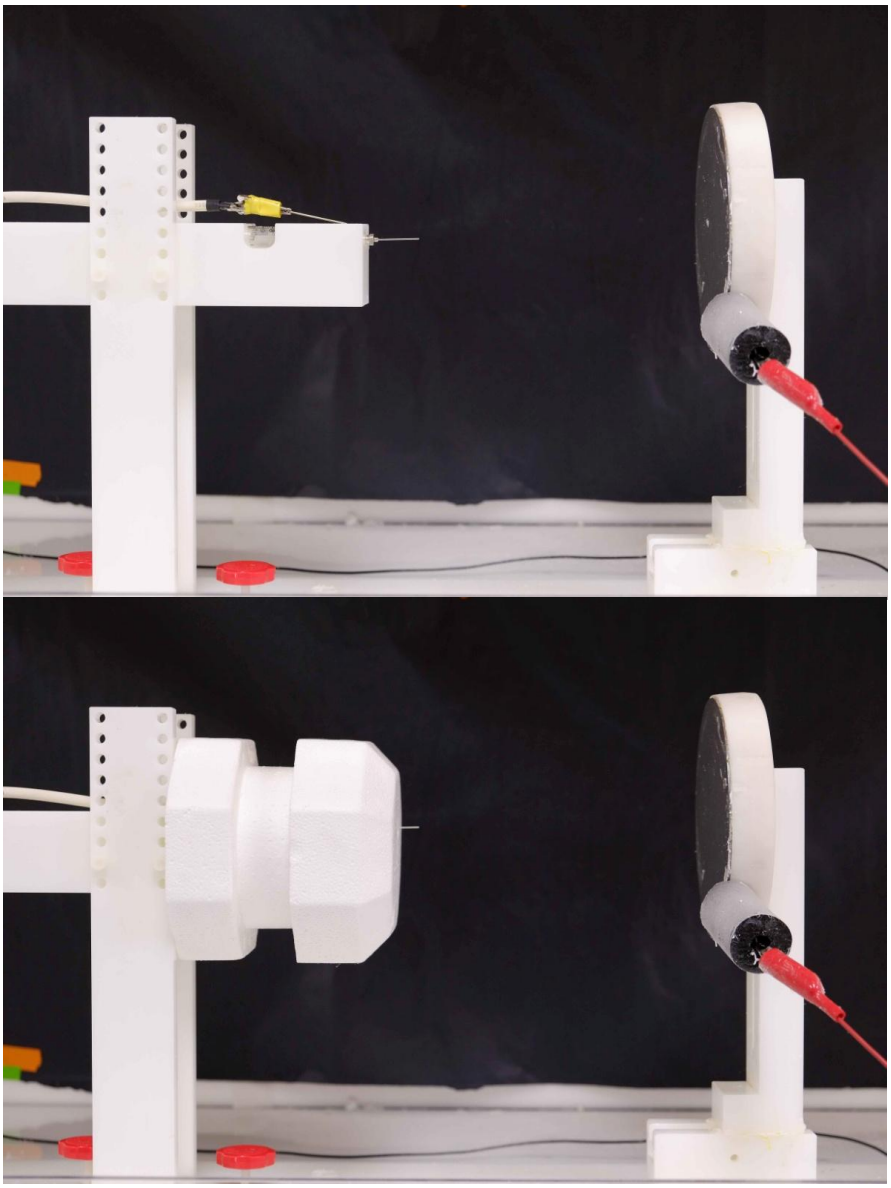

Figure 2.14 Configuration of Experiments 3 (top) & 4 (bottom).

The setups of Experiment 5-8, Experiment 13-16 were identical to Experiment 1-4 except the syringe was filled in with CH_2Cl_2 as solvent, and PCL polymer solution, respectively.

Experiments 9-12, 17, 18 were designed to be repeating process of previous experiment except these were conduct with Teflon needle.

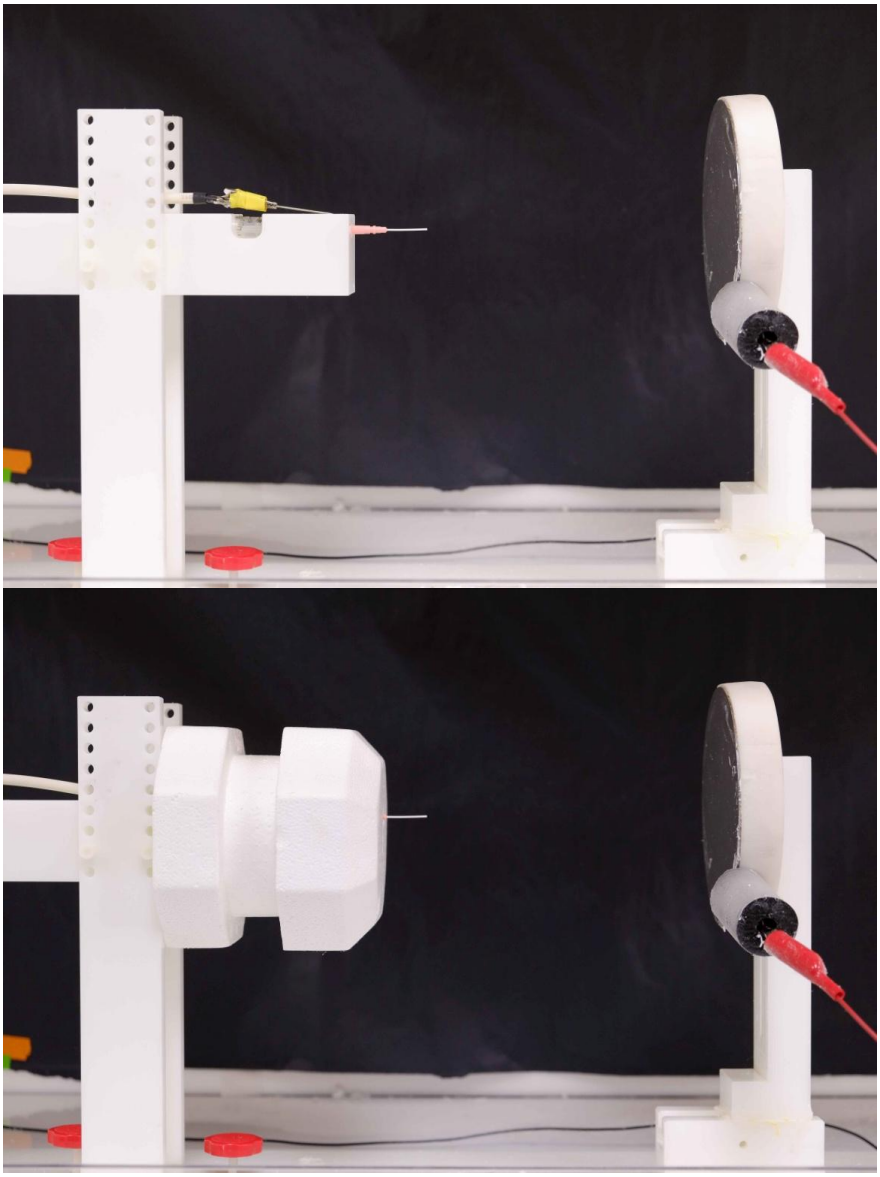

Figure 2.15 Configuration of Experiments 9 (top) & 10 (bottom).

CHAPTER 3

RESULTS AND DISCUSSION

In this chapter, the dependency of the current profiles on several configuration parameters are studied and analyzed. The configuration parameters under consideration are the cathode attachment type, needle type, masking, and flow rate. They were studied under both single plate in Sections 3.1 to 3.5 and dual-plate setups in the rest of the chapter.

3.1 Dependency of Flow Rate

In the experiments, each configuration setup is repeated for two different flow rates: 5 mL/hr and 7.5 mL/hr. Results from experiments carried out using a conductive needle in the standard electrode configuration are being discussed in this section. Figure 3.1 (a), (b), (c) represent the experiments conducted without contents in the syringe, CH_2Cl_2 in the syringe, and PCL solution in the syringe, respectively. The average current measured in experiments conducted without liquid contents in the syringe, are 1.48 μA, 1.51 μA, under flow rates of 5 mL/hr and 7.5 mL/hr, respectively. After filling methylene chloride into the syringe, the average currents under both flow rates are identical, 1.47 μA, as can be seen from Figure 3.1 (b). Similarly, as shown in Figure 3.1 (b), with PCL polymer solution, the observed amounts are the same for both 5 and 7.5 mL/hr. As can be seen from the Figure 3.1, there is no significant difference in the current observed from the two different flow rates in any configuration. These results suggest that the current profile is nearly independent from the flow rate of pumped polymer solution. It is speculated that the motion of the charges does not totally depend on the movement of the medium; charge motion is mainly induced by the applied electric field. This kind of response to the electric

field can be described as electrohydrodynamics. For the rest of the sections, only the results at 5 mL/hr flow rates will be analyzed and discussed.

Table 3.1 Average Current Measured in Experiments 1, 5, and 13, Single Plate

Experiment	**Syringe Content**	**Avg. Current (A) @ 5mL/hr**	**Avg. Current (A) @ 7.5mL/hr**
1	empty	1.48 E-06	1.51 E-06
5	CH_2Cl_2	1.47 E-06	1.48 E-06
13	PCL + CH_2Cl_2	6.80 E-08	6.20 E-08

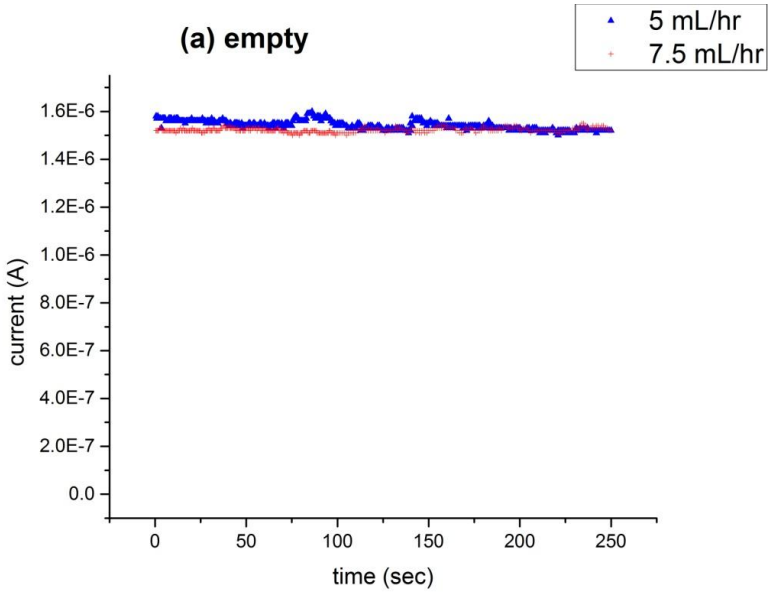

Figure 3.1 Current profile for flow-rate dependency test, conductive needle, standard electrode, single plate, (a) empty syringe, (b) CH_2Cl_2 in syringe, (c) CH_2Cl_2 + PCL in Syringe.

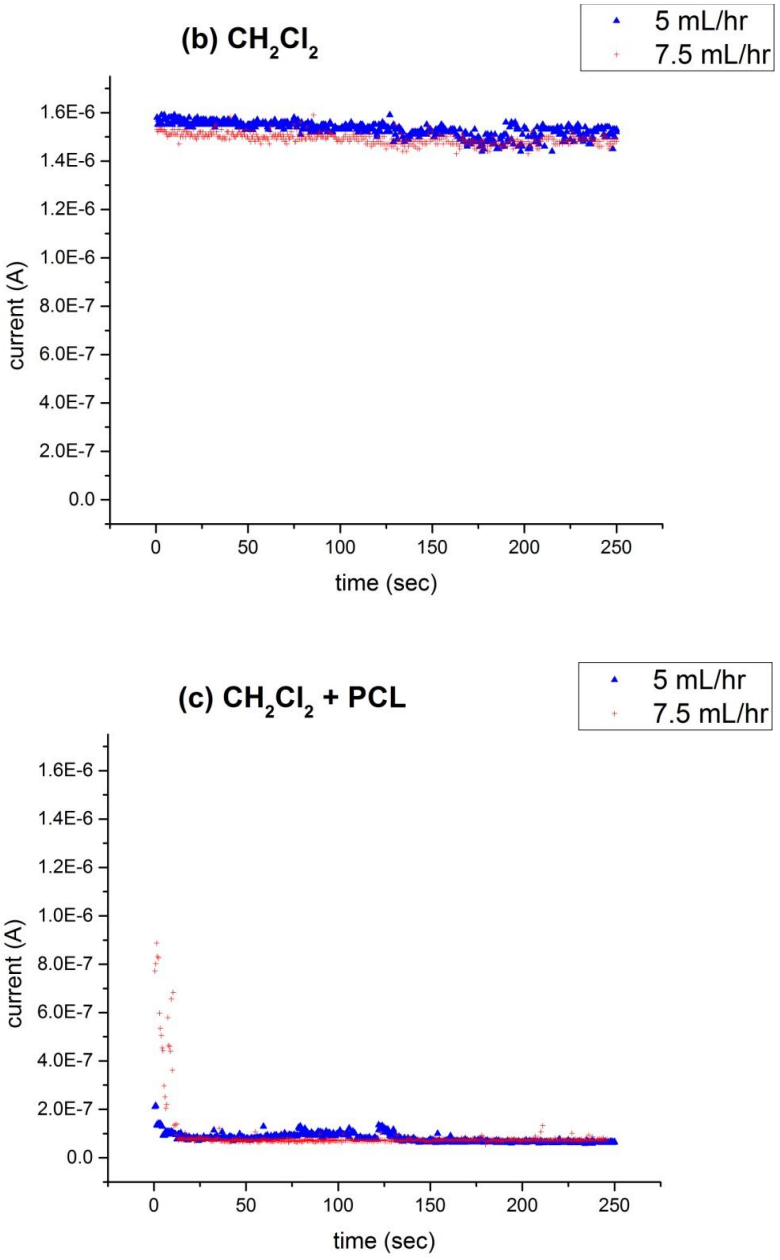

Figure 3.1 (Continued) Current profile for flow-rate dependency test, conductive needle, standard electrode, single plate, (a) empty syringe, (b) CH_2Cl_2 in syringe, (c) CH_2Cl_2 + PCL in Syringe.

3.2 Dependency of Cathode Attachment

In this section, dependency of electrode configuration will be discussed. Note that these experiments were conducted using the conductive needle, because the high voltage lead cannot be attached to a non-conductive Teflon needle. The selected configuration setups are summarized in Table 3.2.

Table 3.2 Summary of Selected Configuration in Section 3.2

Experiment	Needle Type	Electrode Configuration
1, 5, 13	metal	standard electrode
3, 7, 15		penetrating electrode

Figure 3.2 shows the current profile on the single collector plate with different electrode attachment type at a fixed flow rate of 5 mL/hr. The three current profiles are the results of the experiments without any content, with CH_2Cl_2 as solvent, and with PCL + CH_2Cl_2 as polymer solution in the syringe, respectively.

It is evident from Figure 3.2 (a) that a detectable current of an average value of 1.63E-6 Amp was observed even the syringe is empty. There is no conducting path between the two electrodes, so that the most likely description of the phenomenon is the "point discharge" effect. It is initiated when the electric charges accumulate at the needle, and the potential difference between the needle and the surrounding air is high enough to ionize the atmosphere and therefore provides a conducting path allowing charges transport from the tip of the needle to the collector. This is illustrated in Figure 3.3.

To minimize the unexpected sources of charges, the first modification was applied by substituting the standard electrode with a penetrating electrode. The result from Table 3.3 shows that the current was too small to be detectable by the electrometer when the

syringe is empty. It is believed that the point discharge is no longer taking place around the needle since the high voltage lead is connected to the pin located in the plastic syringe body. Figure 3.2 (b) shows detectable current values for both cases after filling methylene chloride (CH_2Cl_2) into the syringe. An average current of 1.32E-6 Amp was observed from the penetrating electrode attachment method, which is still smaller than what was measured if a standard electrode was used. In the case of using standard electrode, it is believed that the sharp edges of the needle present the possibility for field emission of the separated charges under high voltage effect. The motions of these charges are illustrated in Figure 3.4 (A). In the penetrating electrode configuration, by inserting a pin in the syringe body, charges are being directly injected into the solvent after the application of the high voltage as shown in Figure 3.4 (B) [2]. The electrified solvent enables the mobility for discrete charges transport to the collector. Further, according to the results, partial field emissions from the edge of the needle were avoided. After filling 12 wt% of PCL solution into the syringe, both cathode attachment types present largely reduced currents with insignificant differences as shown in Figure 3.2 (c). Fibers are collected at the plate. The possible explanation is that the charges are being trapped in the polymer molecule and thus doesn't have a chance to be grounded. The high current level at the beginning of the process may caused by the certain time period required for Taylor cone to be stabilized under interaction of surface tension and electrostatic attraction force.

Table 3.3 Average Current Measured in Experiments 1, 3, 5, 7, 13, and 15

Experiment	**Syringe Content**	**Avg. Current (A) Standard Electrode**	**Avg. Current (A) Penetrating Electrode**
1, 3	empty	1.63 E-06	0.00
5, 7	CH_2Cl_2	1.58 E-06	1.32 E-06
13, 15	PCL + CH_2Cl_2	2.09 E-08	2.86 E-08

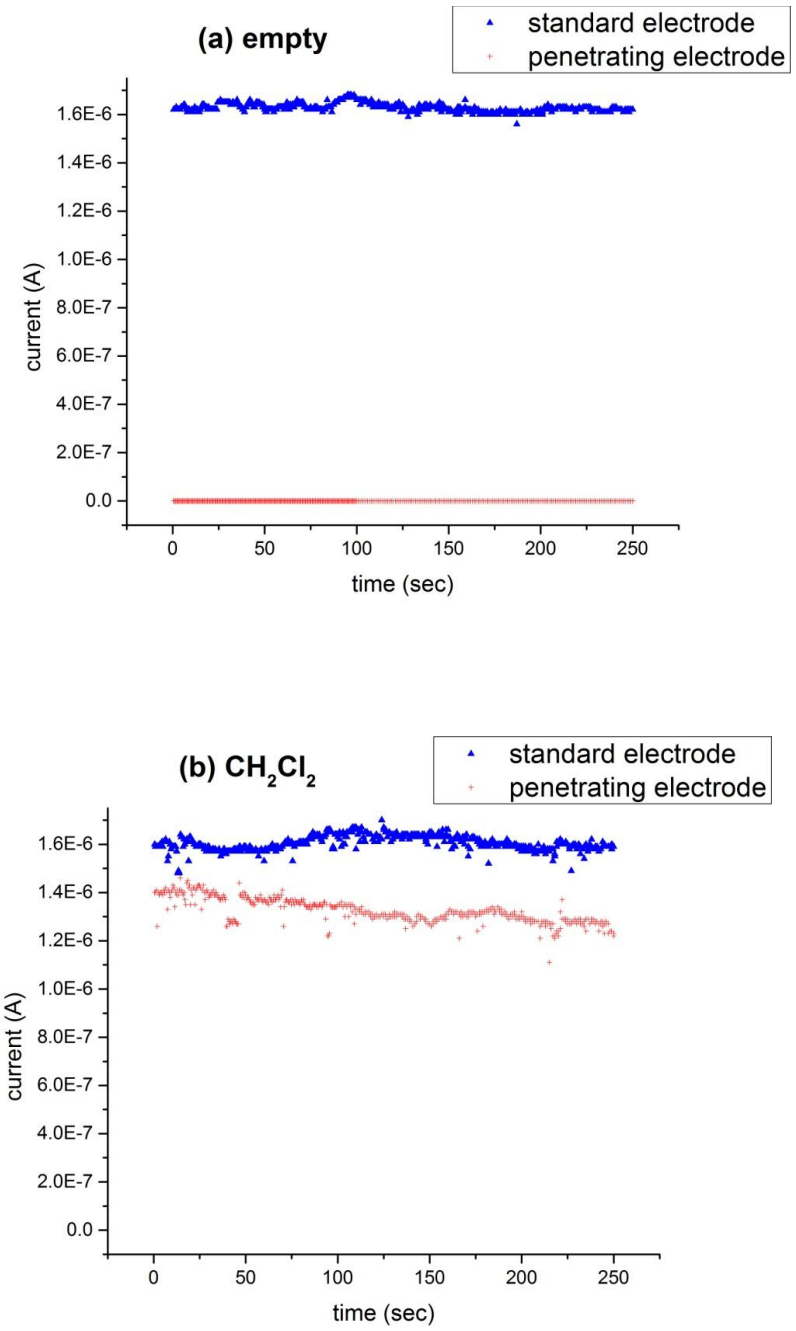

Figure 3.2 Current profile for cathode attachment type dependency test with a conductive needle, single plate, 5 mL/hr, (a) empty syringe, (b) CH_2Cl_2 in syringe, (c) CH_2Cl_2 + PCL in Syringe.

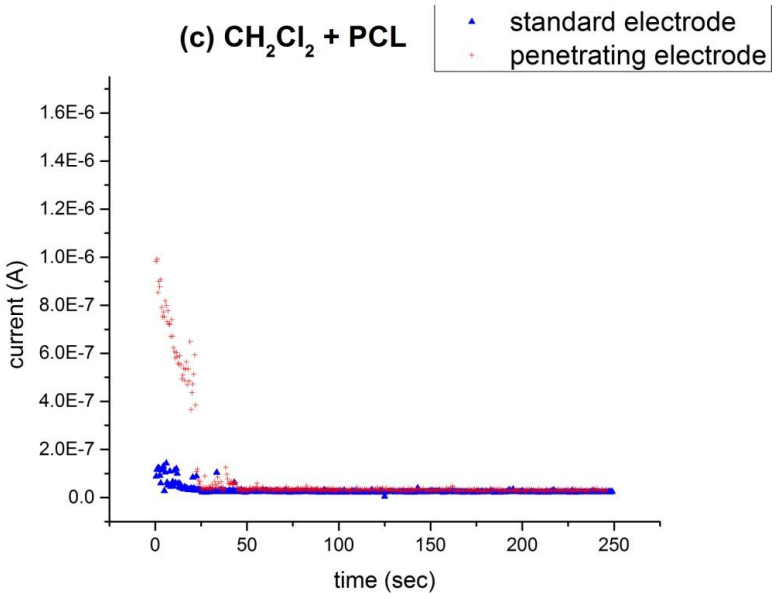

Figure 3.2 (Continued) Current profile for cathode attachment type dependency test with a conductive needle, single plate, 5 mL/hr, (a) empty syringe, (b) CH_2Cl_2 in syringe, (c) CH_2Cl_2 + PCL in Syringe.

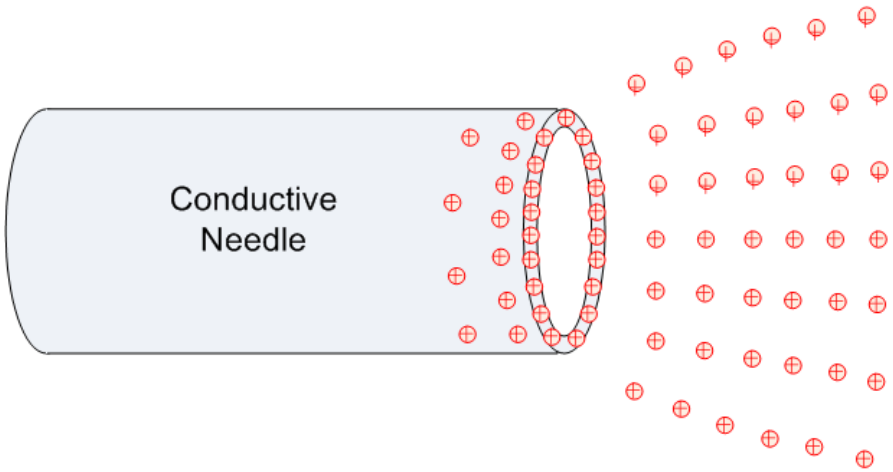

Figure 3.3 Point discharge effect.

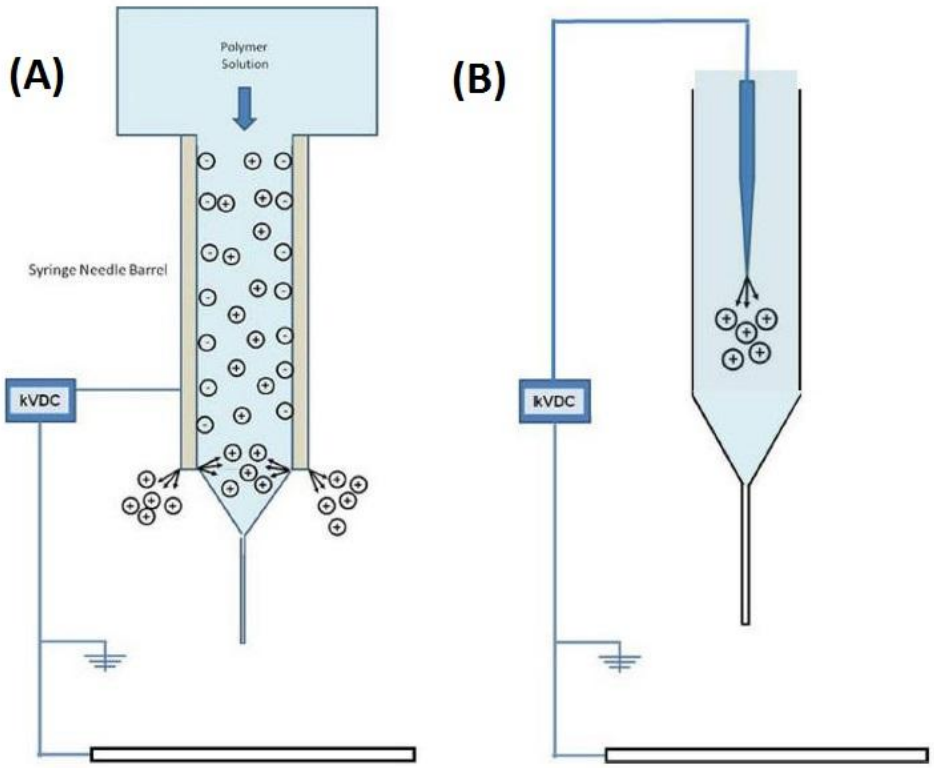

Figure 3.4 Schematic diagram illustrates the charge motion in (A) standard electrode (B) penetrating electrode configuration [2].

3.3 Dependency of Needle Type

In addition to the alternative cathode attachment method, a second modification for the purpose of minimizing the point discharge effect was applied in this section, which is the replacement of the conductive needle with a non-conductive Teflon needle. The experiments repeated in the rest were conducted using a penetrating electrode. The selected configuration setups are summarized in Table 3.4.

Table 3.4 Summary of Selected Configuration in Section 3.3

Experiment	Needle Type	Electrode Attachment Type
3, 7, 15	conductive	penetrating electrode
9, 11, 17	Teflon	

Table 3.5 summarizes the average values from the selected experiments. Figure 3.5 (a) illustrates the current profiles when there is no liquid in the syringe. Current was not detectable in the case of either conductive needle or Teflon needle. Figure 3.5 (b) shows that detectable current was observed for both needle types after filling methylene chloride into the syringe. The experiment conducted with a Teflon needle has an average current value of 4.01E-8 Amp, which is significantly lower than what the experiments operated with a metal needle can have, which is 1.12E-6 Amp. The result suggests that a non-conductive needle can further reduce the emitting of charges from the edge of the needle. Similar to what was found in previous sections, the average current reduces to a low value after PCL solution is filled as shown in Figure 3.5 (c).

Table 3.5 Average Current Measured in Experiments 3, 7, 9, 11, 15, and 17

Experiment	**Syringe Content**	**Avg. Current (**A**) Metal Needle**	**Avg. Current (**A**) Teflon Needle**
3, 9	empty	0.00	0.00
7, 11	CH_2Cl_2	1.12 E-06	4.01 E-08
15, 17	PCL + CH_2Cl_2	1.05 E-07	6.28 E-08

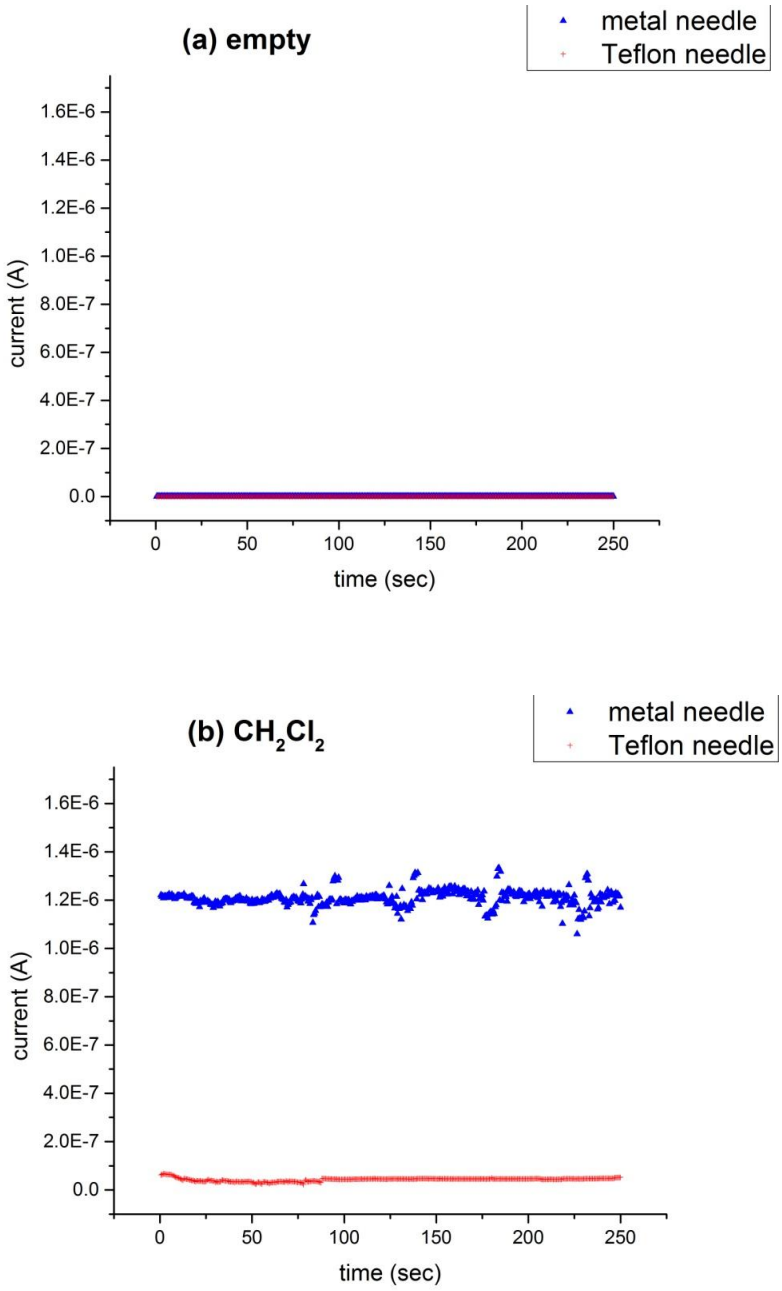

Figure 3.5 Current profile for needle type dependency test, single plate, 5 mL/hr, (a) empty syringe, (b) CH_2Cl_2 in syringe, (c) CH_2Cl_2 + PCL in Syringe.

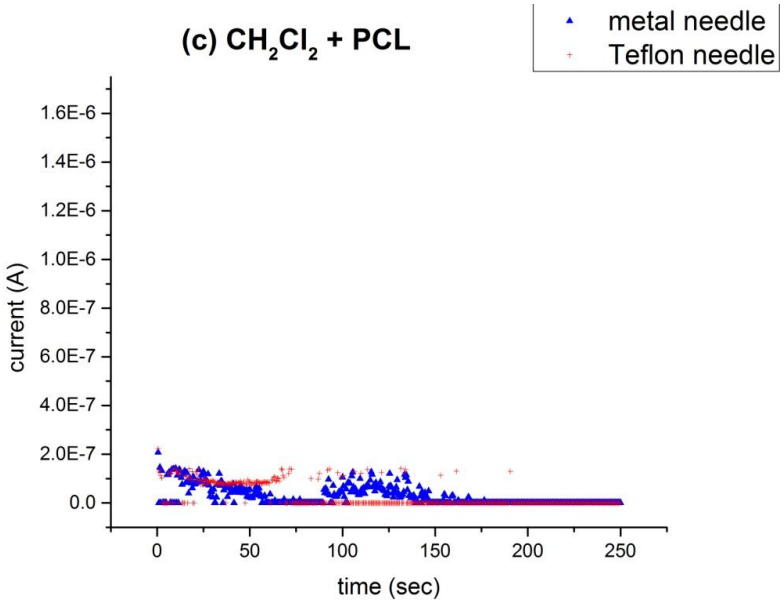

Figure 3.5 (Continued) Current profile for needle type dependency test, single plate, 5 mL/hr, (a) empty syringe, (b) CH_2Cl_2 in syringe, (c) CH_2Cl_2 + PCL in Syringe.

3.4 Dependency of Masking

In this section, a designed external cover was applied on the syringe body to determine the dependency of the current profile on masking. Experiments were carried out using conductive needle in the standard electrode configuration. A minor reduction on the current profile can be observed from Figure 3.6 (a) after covering the syringe stage with a Styrofoam cover. The same results are observed when the solvent is added into the syringe (not shown). Figure 3.6 (b) shows results from experiments carried out using a conductive needle in the penetrating electrode configuration. Similar current values were observed for both cases and thus suggest that the external cover is very likely an unessential factor to the current profile after applying penetrating electrode; this is probably attributable to the transport medium created by ionized solvent which allows the discrete charges to mobilize, and therefore decrease chances of current leaking from the electrode.

Table 3.6 Average Current Measured in Experiments 1,2,7, and 8

Experiment	**Syringe Content, Electrode Config.**	**Avg. Current (A) without Cover**	**Avg. Current (A) with Cover**
1, 2	empty, standard electrode	1.35 E-06	1.16 E-06
7, 8	CH_2Cl_2, penetrating electrode	1.12 E-06	1.15 E-06

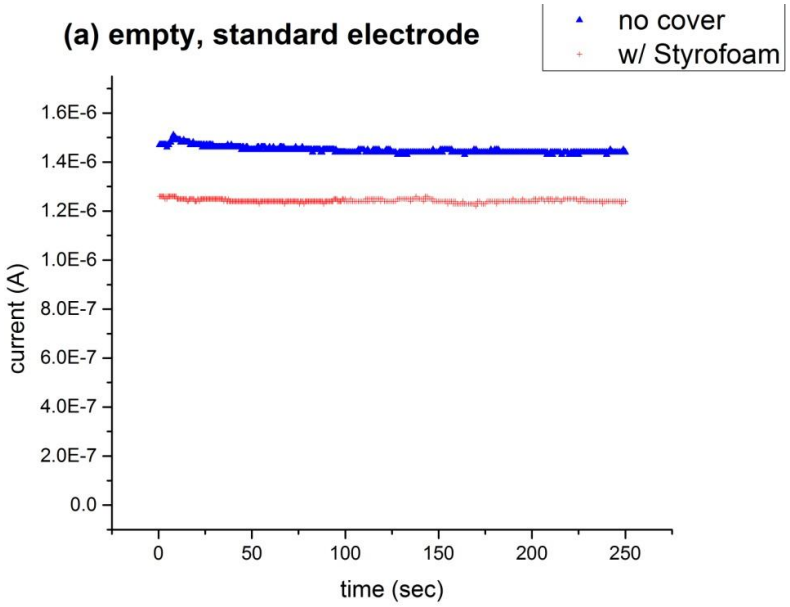

Figure 3.6 Current profile for masking material dependency test, conductive needle, single plate, 5 mL/hr, (top) empty syringe, (mid) CH_2Cl_2 in syringe, (bottom) CH_2Cl_2 + PCL in Syringe.

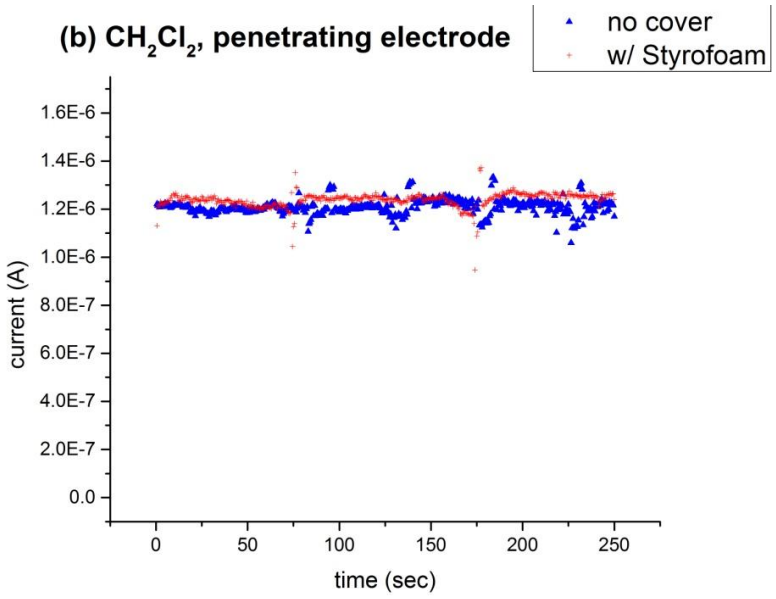

Figure 3.6 (Continued) Current profile for masking material dependency test, conductive needle, single plate, 5 mL/hr, (top) empty syringe, (mid) CH_2Cl_2 in syringe, (bottom) CH_2Cl_2 + PCL in Syringe.

3.5 Current Behavior on the Outer Collector

In this section, Figure 3.7 represents the experiments conducted with empty contents in the syringe, CH_2Cl_2 in syringe, CH_2Cl_2 + PCL in the syringe with (a) conductive or (b) Teflon needle attached. It appears from the figure that the differences between all three syringe content types are negligible. For the case using conductive needle, small amount of current was detected in the beginning, and then decays to an undetectable low current value. By replacing the conductive needle with a Teflon needle, current drops to value closing to zero regardless of their syringe types. Further, no fiber deposit on outer plate was found in these experiments. This data suggests that nearly all current coming from the needle was fully deposited on the inner plate.

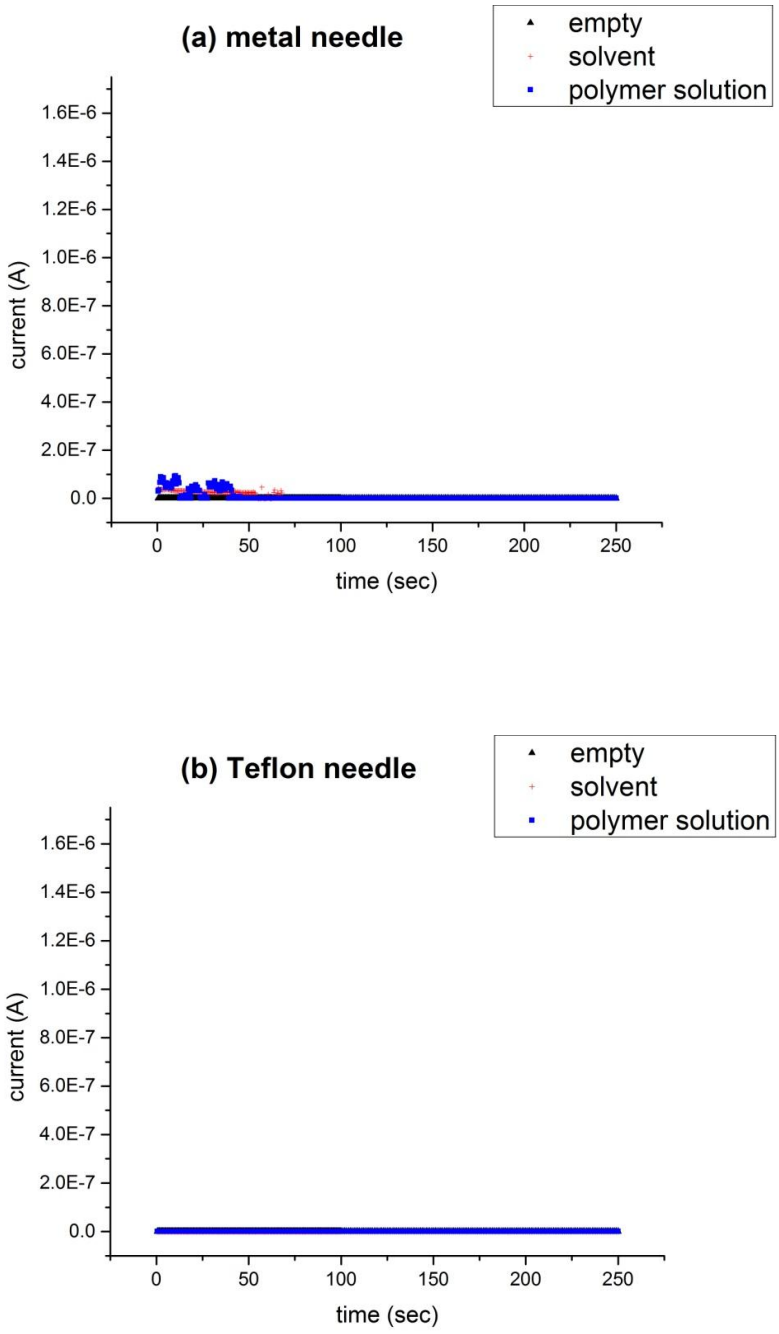

Figure 3.7 Current profile on outer collector based on experiment conduct with empty contents in the syringe, CH_2Cl_2 in syringe, CH_2Cl_2 + PCL in Syringe using (top) conductive needle, (bottom) Teflon needle.

CHAPTER 4

SUMMARY AND CONCLUSIONS

4.1 Summary of Observations and Conclusions

Previous research have shown that current is present not only during electrospinning but also present when there is no solution inside the syringe. Thus, to enhance the understanding of the nature of the current in electrospinning process, the main objective of this research is to identify the current sources from the needle jet.

In this research, the dependency of the current behavior on various factors such as needle type was evaluated; electrode configuration; flow rate; and add-on of external Styrofoam cover on the syringe by designing customizable setups for electrospinning.

Results from experiments indicate that the measured current is nearly independent on the flow rate of the solution set by pump device.

A large amount of current was detected even the syringe was empty when the experiments were carried out using standard electrode and conductive needle. The results are consistent with Grewal's et al. observations [2, 3]. A reasonable explaining is that the point discharge effect taking place around the needle after the application of high voltages. By substituting the standard electrode with penetrating electrode, the current dropped to zero when the syringe was empty; an average of 0.26E-6 Amps reduction on the amount of measured current can be observed even the syringe was filled with methylene chloride. This is due to the charges are being directly injected into the solvent from the syringe body, and possibilities for field emission of the charges occurred at the needle edge were reduced. However, experiments conduct using 12 wt% PCL polymer solution shows nearly

undetectable current value, this is perhaps due to reason of that, the charges are being trapped in the polymer molecule and thus don't have a chance to be grounded. This can result in positive charge accumulation. As the amount layers of deposited fibers increase, a discharge phenomenon is expected to take place within the molecules and has been previously reported [23]. This was not observed in the experiments reported here. That discharge will depend on the dielectric properties of the material and how it was collected on the collector. It may be that for the system used for these experiments, more fiber material has to be collected before the local field in strong enough to cause this discharge.

In all cases that non-conductive Teflon needle was applied, a significant lower current can be observed. The result suggests that regardless what configurations were used, conductive needle always allows chances for charge emitting from the opening edge of the needle. And the Teflon needle can efficiently eliminate this issue.

The add-on of external cover becomes unessential factor to the current profile after applying penetrating electrode, which decrease chances of current leaking from the electrode.

For the current behavior on outer collector plate, no fibers were deposited on outer plate in the experiments, which is consistent with Bhattacharjee et al. [1] findings. However, results from this research show some different outcomes compared with previous studies. It appears that the measured current values on the outer plate under all three syringe content conditions are detected to be extremely low, and present the possibility that nearly all current coming from the needle was fully deposited on the inner plate. The experimental setup in this research was designed to measure current from the needle itself and eliminate as much current from other sources as possible, which is one of

the factors may not be accounted for most of the previous studies. While the experiments described in this research specifically attempted to isolate the sources of current, previously reported experiments did not attempt this isolation of current source. In addition, the configuration of previous apparatus is different from the configuration used in this work. One of the consequences of this difference may be that there is not an agreement in measured current, because of the difference in the configuration of the experimental apparatus.

4.2 Impact

The use of conductive needle always allowing charges emitted from the edge of the needle regardless what configurations were used. A reasonable suggestion to the current measurement in electrospinning process is to avoid using a conductive needle.

The low result in polymer solution indicates charges are trapped in polymer mat and are unable drain to ground. The accumulated charges may represent important factors that can affect the electrical properties and degrade the overall performance on the polymer product. For this aspect, the evaluation of the discharge from accumulated charges is deserved to be investigated and may become an indication on the final product quality.

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