INVESTIGATION ON THE EFFECTS OF ELECTROSPINNING DISTANCE AND APPLIED VOLTAGE ON MORPHOLOGY OF POLY (LACTIC ACID) ELECTROSPUN NANOFIBRES.

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This report is submitted
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DECLARATION

I declare that this thesis entitled "Investigation On The Effects Of Electrospinning Distance And Applied Voltage On Morphology Of Poly (Lactic Acid) Electrospun Nanofibres" is the result of my own research paper except as cited in the references.

Signature :.....

Name :.....

Date :.....

SUPERVISOR'S DECLARATION

I have checked this report and the report now can be submitted to JK PSM to be delivered back to supervisor and to the second examiner.

Signature :…….....

Supervisor‟s Name :…….....

Date :…….....

DEDICATION

To my beloved mother and father.

ABSTRACT

Nanofibres are popular and have been subject of research study recent years. The special characteristic which are have small diameter with large surface area to volume ratio rather than human hair. Nanofibres have been developed in many applications such as filtration, biomedical, electronic and tissue engineering. Recent years, the electrospinning method which is simple and cost friendly was popular to produce nanofibres among researchers. In electrospinning process, the applied voltage and electrospinning distance are the two most important parameters that affect the quality of the fibres. In this study, these two parameters were studied. The effect of the solution concentrations on morphology electrospun nanofibres also have been studied in this research. Poly (lactic acid) electrospun fibres were produced using electrospinning technique. The morphology and fibre diameter of the fibres were examined using scanning electron microscopy and ImageJ. From the results, fibre diameter increased when electrospinning distance and applied voltage increased. The fibres diameter also increased as the concentration of the polymer solution increased. The best electrospinning parameters were 15 kV of applied voltage and 10 cm of electrospinning distance. The fine, smooth, defect free and homogenous size nanofibres also produced with best parameters.

ABSTRAK

Nanofibres popular dan tertakluk kepada kajian penyelidikan beberapa tahun kebelakangan ini. Ciri khas yang mempunyai garis pusat kecil dengan luas permukaan yang besar kepada nisbah volum daripada rambut manusia. Nanofibres telah dibangunkan dalam pelbagai aplikasi seperti penapisan, bioperubatan, elektronik dan tisu kejuruteraan. Tahun-tahun kebelakangan ini, kaedah elektrospinning yang mudah dan mesra kos adalah popular untuk menghasilkan nanofibres di kalangan penyelidik. Dalam proses elektrospinning, voltan yang digunakan dan jarak elektrospinning adalah dua parameter paling penting yang mempengaruhi kualiti gentian. Dalam kajian ini, kedua-dua parameter ini dikaji. kesan kepekatan larutan pada morfologi nanofibres electrospun juga telah dikaji dalam kajian ini. Poli (asid laktik) gentian elektrospun dihasilkan menggunakan teknik elektrospinning. Morfologi dan diameter serat gentian telah diperiksa dengan menggunakan mikroskop elektron pengimbasan dan ImageJ. Dari hasilnya, diameter serat meningkat apabila jarak elektrospinning dan voltan yang digunakan meningkat. Diameter serat juga meningkat apabila kepekatan larutan polimer meningkat. Parameter elektrospinning yang terbaik ialah 15 kV voltan terpakai dan 10 cm jarak elektrospinning. Nanofibres yang halus, licin, cacat bebas dan homogen saiz nanofibres dihasilkan dengan parameter terbaik.

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PLA	Poly lactic acid
PVA	Polyvinyl alcohol
PLLA	Poly (L-lactic acid)
SEM	Scanning electron machine

LIST OF SYMBOLS

kV	Kilovolt
cm	Centimeter
µm	micrometer

CHAPTER 1

INTRODUCTION

1.1 Background

Recently, biodegradable materials have been developed for many applications such as packaging, agriculture and medicine. Biodegradable materials are made from plant based raw material such as corn or other starch. One of the most common applications of biodegradable materials is packaging. Biodegradable packaging materials are able to disintegrate by anaerobic process that allows the materials to decompose without the presence of oxygen. Biodegradable plastic also estimated to be decomposed in a few months only while the traditional plastic took several hundred years to degrade. The end result of which the biodegradable packaging is less negative impact to the environment compared to the conventional plastic packaging (Gross and Kalra, 2002).

Polylactic acid (PLA) is one of the biodegradable polyester. In the production of Polylactic acid in an industrial scale, the choice of carbohydrate sources may be from maize starch, sugar cane or cassava root to make it sustainable and renewable. Thus, Polylactic acid material became the main choice in environmental friendly plastic production. Moreover, the process by which Polylactic acid is made is also famous in biomedical areas as it ability to be safely absorbed biologically (Gupta, Revagade and Hilborn, 2007).

In line with the capabilities of biodegradable materials, nanotechnology that used biodegradable material also gained popularity since it has high potential in many purpose. A biodegradable nanofibre is also important technology that can bring innovation in many

applications. Fibres with diameter 1000 nm or 1 µm are classified as nanofibres that smaller than microfibers. Several unique characteristics of nanofibres are high porosity, high surface area and superior mechanical performance (Zhu *et al.*, 2017). This special characteristic also motivates many researchers to develop nanotechnology deeply for different application. The nanofibres technology gives a huge impact and escalates momentum in material science area. Examples of major applications are bio technology and environmental engineering, bio engineering and medical science, electronics, and energy (Zhou, Green and Lak, 2006). Recent years, the more specific, nanofibres or nanotechnology has various applications to comply and meet the needs of the community in line with technological change such as in filtration system, tissue engineering, chemical and optical sensors or wound healing (Khude, 2017).

There are several techniques or method to produced nanofibres such as template synthesis, drawing, phase separation, self-assembly and electrospinning („Technology of Nano-Fibers : Production Techniques and Properties - Critical Review", 2018). However, the electrsopinning process is the most popular techniques among researchers as the techniques is very simple and cost friendly yet adaptable techniques (Pham, Sharma and Mikos, 2006). Electrospinning method was widely used in nanotechnology since 1990 (Reneker and Yarin, 2008). Electrospinning is a famous method to produce continuous nanofibres. The electrospinning process is actually inspired by process of electrospying that involving the electrical potential when the charged liquid imposed to the electrostatic than attracted to the collector which is has apposite charge with polymer solution (Pillay *et al.*, 2013).

In this study the effect applied electrospinning voltage and the distance between the tip of the capillary and the collector on morphological electrospun Polylactic acid nanofibres was investigated. The change of nanofibres diameter and characteristic from the

micrograph of electrospun Polylactic acid under scanning electron microscopy (SEM) were observed. Three concentration of Polylactic acid solution also have been used to investigate the relationship between effect concentration solutions on the nanofibres diameter.

1.2 Problem statements

Electrospun nanofibres have special characteristic such as high in mechanical properties, small pores and high in porosity (Oliveira *et al.*, 2013). These special characteristic make the electrospun nanofibres have high potential in applications such as a filtration system, tissue engineering, optical sensor, and wound healing. The morphology of the electropsun nanofibres depend on the electropspinning parameters such as solution properties, process parameters and the ambient parameters (Pillay *et al.*, 2013),(Doshi and Reneker, 1993). The morphological nanofibres could be beaded, connected between the fibre strains and these depend on the process variables (Oliveira *et al.*, 2013). Aside from morphology, the electrospun fibres diameters are also affected by the electropspinnning parameters. A few studies showed that parameters such as polymer concentration, electrospinning distance and applied voltage would give impact on the nanofibres diameter (Tan *et al.*, 2005) , (Pillay *et al.*, 2013).

As discussed in a study review by (Pillay *et al.*, 2013), the effect of applied voltage on nanofibres morphology varies from one solution to another. P.K. Baumgarten As quoted in (Pillay *et al.*, 2013) found that the diameter fibre initially decreased then increased as the applied voltage increased. The ultrathin nanofibres will be produced when the critical voltage reached. Thus, this experiment investigates the critical voltage of the nanofibres that can produce thin nanofibres. The effect of the electrospinning distance,

applied voltage and the polymer concentration on the morphology of PLA nanofibres also investigate in this experiment.

1.3 Objectives

The aim of this study is

1. To investigate the effect on Polylactic acid electrospun nanofibres morphology when apply different electrospinning voltage and distance between the tip of nozzle and the collector.
2. To determine best parameters to produce fine nanofibres through electrospinning method.
3. To investigate the effect of Polylactic acid concentration solution on nanofibres diameter

1.4 Scopes

The scopes of the final year project are:

1. Investigate the morphology electrospun nanofibres based on the objectives through micrograph under scanning electron microscopy (SEM).
2. The range of the applied voltage in electrospinning process was in between 10kV to 20kV to investigate the effect of the nanofibres diameter.
3. The range of distance or gap between the tip of nozzle and the collector was between 75mm to 175mm.
4. The flow rate of the solution flow fixed to 3.00 ml/h.

5. The concentrations of the polymer solutions used were 10% wt, 13% wt and 16% wt.

CHAPTER 2

LITERATURE REVIEW

2.1 Electrospinning background

Electrospinning method was widely used in nanotechnology since 1990 (Reneker and Yarin, 2008). Electrospinning is a famous method to produce continuous nanofibres. In 1897, the electrospinning technique was firstly introduced by Rayleigh then develops in detail by Zeleny. Zeleny state that the nanofibers are produced by the emission of the charged liquid when imposed to the electrical potential. Then, the electrospinning apparatus was invented by Formhals in 1934 that showed the electrical field can imposes a uniaxial stretching of a viscoelastic jet derived from the polymer solution to continuously reduce the diameter and leads to formatting nanofibres (El-newehy, no date).

The electrospinning process is actually inspired by process of electrospraying that involving the electrical potential when the charged liquid imposed to the electrostatic than attracted to the collector which is has apposite charge with polymer solution (Pillay *et al.*, 2013). Before applying the electrical charge in electrospinning process, the polymer droplet appeared at the tip of the capillary is due to the surface tension of the liquid. Once electrical field applied, the "Taylor Cone" shape of the liquid polymer formed when surface tension is equal to the electrostatic force. As the electrical field is strong enough to overcome the surface tension, the fine fibre will eject from the tip of capillary or Taylor Cone to the grounded collector which is appositely charge with the polymer liquid. During polymer liquid travels, it is imposed to the atmosphere and evaporated before reached the collector. The Figure 2.1 below shows illustrate of electrospinning process.

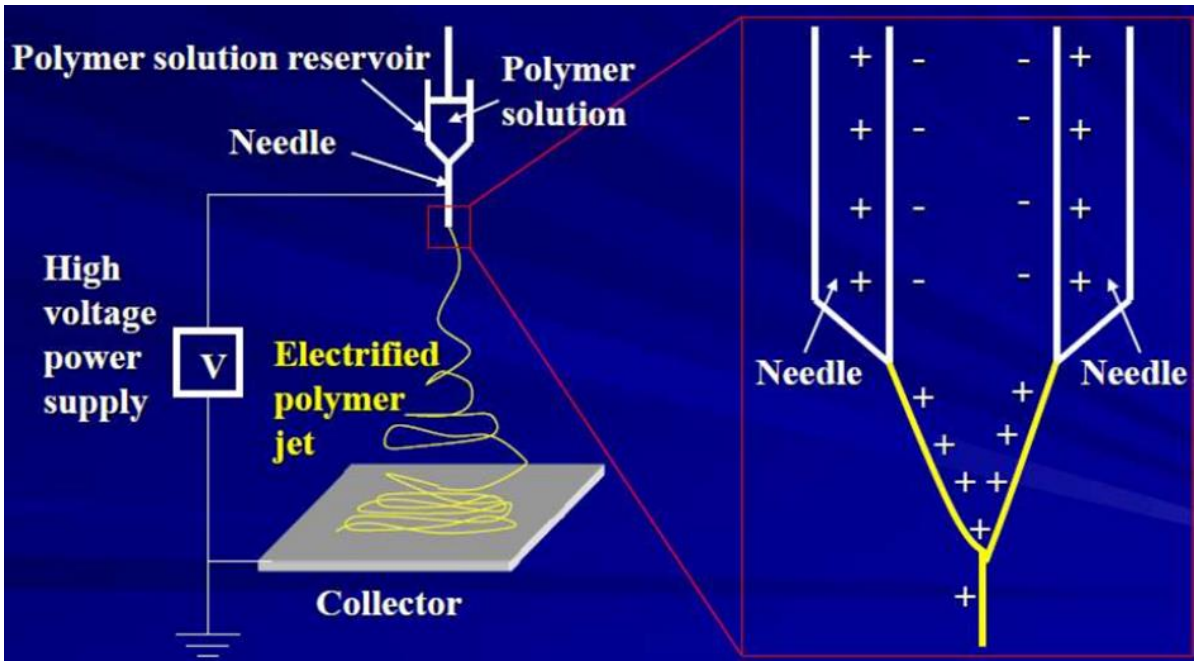


Figure2. 1: Schematic of the vertical setup of electrospinning process.

Electrospinning was the famous method to produce the nanofibres due to non-complicated process (Pillay *et al.*, 2013). The electrospinning is also versatile and inexpensive techniques that successfully to produce the thin nanofibres. This factor motivated many researchers to develop the nanofibres using electrospinning techniques.

2.2 Electrospinning process or apparatus.

The electrospinning process begins when a high voltage is applied and the polymer solution imposed to the electric charges throughout the conductive metallic needle. The cone shaped of polymer solution droplet held in the tip of capillary when the electric filed is equilibrium to the surface tension of the solution. The cone shaped of the solution also called as "Taylor Cone". This caused when the solution is charged to electrical field, the surface charge is act appositely to the surface tension and produced the Taylor Cone. Once the surface tension defeated by the electric field, the sharp pointed of the polymer solution

at the tip of capillary accelerates directly to the grounded metal collector. In (Pillay et al., 2013) explained this happened because the electrical field is strong enough to overcome the surface tension of the polymer solution. So, it contributes the ejected fine fibre travels to the collector. A dry fine nanofibres will be collected at the metal collector because the ejected nanofibres will evaporates during travels to collector.

According to the (Haider, Haider and Kang, 2015) in research on effect electrospinning parameters and potential applications of nanofibre in biomedical and biotechnology explained the electrospinning basically consist of four major parts: a syringe filled up with the polymer solution , metal needle , electric charge and the metal collector. This statements also explained by the (Pham, Sharma and Mikos, 2006).

In electrospinning, there are two different collectors such as rotating drum collector and the static plate collector. By using rotating drum collector, commonly to collect the aligned nanofibres. In this case, the diameters of the nanofibres are also affected by the speed of the rotating drum collector. The diameter of the nanofibres would be decrease as increase the speed of the rotating drum collector. Plus, the rotating drum collector can collect aligned nanofibres in large surface area. However, the limitation by using the rotating drum collector is the thickness of the deposited nanofibres at collector. The thickness of the nanofibres would be thin in high speed of rotating drum collector.

Despite, the static smooth plate collector was used to collect nonwoven nanofibres and the behaviour of the nanofibre membrane can be tested. The nanofibres deposited at the static collector thicker than nanofibres deposited at the rotating drum in same period of time. This is because the static metal plate collector has limited surface area compared to the rotating drum.

2.2.1 Shaft or Vertical electrospinning setup

For vertical elecrospinning method setup, the grounded plate is placed at floor. The plate is known as collector where are the ejected nanofibres from the tip of nozzle deposited when exposed to the electric field. The syringe is located above the plate so that the electrical field generated by applied voltage is upright to the floor. In vertical setup, the fibre collected in the grounded collector is not in the centre or randomly attached to the collector when apply a high voltage due to the bending instability. The gravitational force in vertical setup electrospinning is affecting the shape of the solution droplet. However, the nanofibre produced in vertical setup electrospinning is the finest compared to the horizontal setup (Rodoplu, Mutlu and Ph, 2012). The Figure 2.2 below shows the schematic diagram for vertical electrospinning setup.

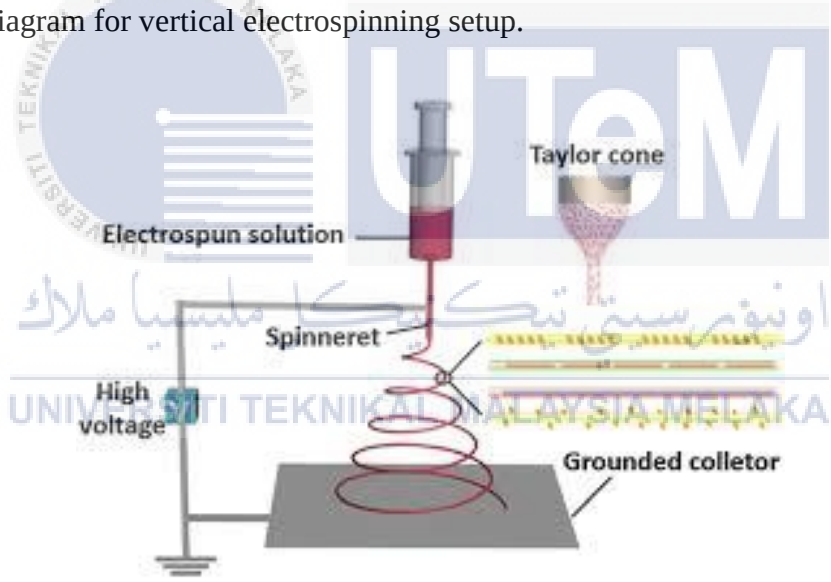


Figure 2. 2: The vertical electrospinning setup.

2.2.2 Horizontal electrospinning setup

For horizontal electrospinning setup, the grounded charged collector is placed perpendicular to the floor and in line with the syringe. The flow of the electric field produced by applied voltage is parallel to the floor. Different with the vertical setup, the

electrical field flow perpendicular to floor. The schematic diagram for electrospinning horizontal type is shown by Figure 2.3.

Based on the graph Figure 2.4 above, the diameter of the nanofibres produced by vertical (shaft type) electrospinning setup is smaller than nanofibres produced by electrospinning in horizontal setup for varies applied electrospinning voltage. According to the previous researchers, the gravity played rolls in the vertical electrospinning setup as it can strengthen the effect of electric field. Thus, the fibres jet can extend effectively then produced the thin nanofibres (Yang *et al.*, 2009).

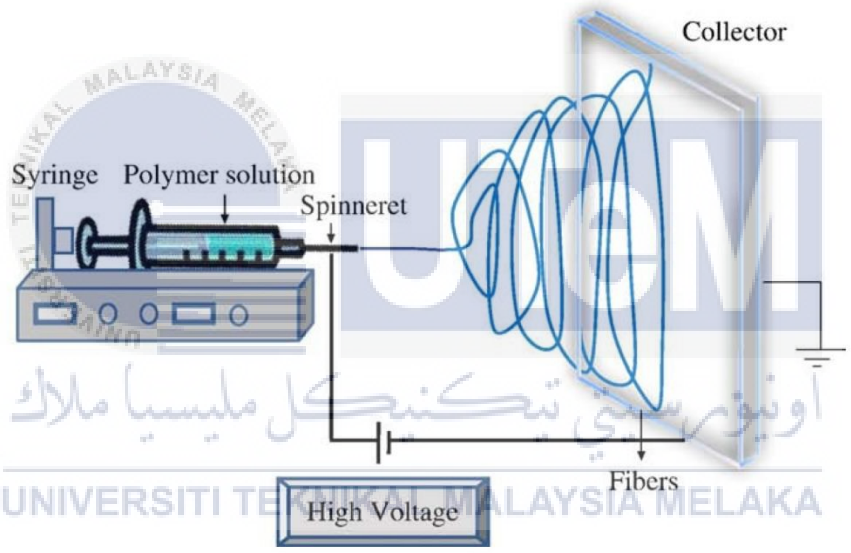


Figure 2. 3: The horizontal electropsinning setup.

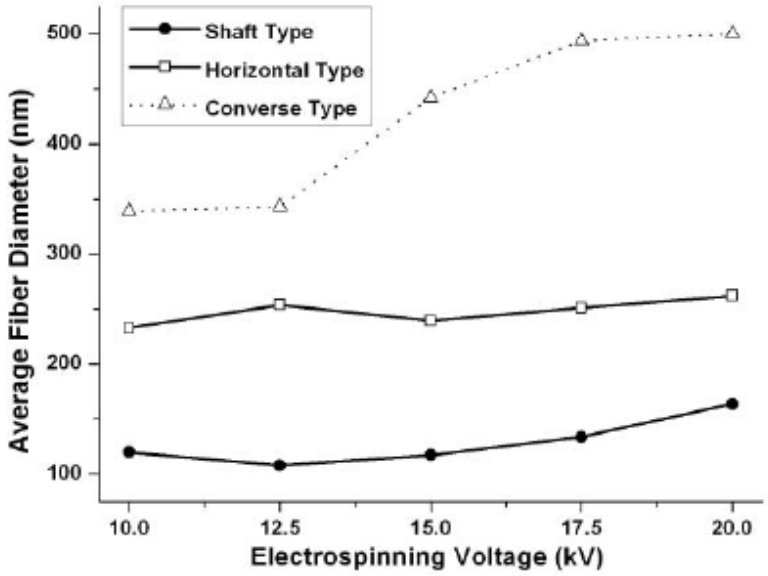


Figure 2. 4: The graph of the average fibres diameter (mm) versus electrospinning voltage in different type of setup.

2.3 Important parameters in electrospinning

In electrospinning process, there are several factors that influence the process. The factors can be divided into three categories i.e. electrospinning parameters, solution properties and ambient parameters. These parameters generally affect the formation of nanofibres. The Table 2.1 below shows electrospinning, solution properties and the ambient parameters.

Table 2. 1: The electrospinning, solution properties and ambient the parameters

Category	Parameter
Electrospinning	Electrospinning applied voltage Electropsinning distance Flow rate of the solution Diameter of metal needle

Solution properties	Concentration of the solution Conductivity Viscosity Solvent Molecular weight
Ambient	Humidity Temperature

2.3.1 Applied voltage in electrospinning

Basically, the electrical field is main role in electrospinning process. The electric field is generated from power supply and current flows to the solution through the metal needle. Generally, the applied voltage can affect the diameter and beads formation of nanofibres. The ultrafine nanofibres can be produced at the critical voltage initially but then the diameter of nanofibres increased after critical voltage. The critical voltage is diversified with the type of the polymer solution used and the ideal range of the voltage. In consonance with the result reported by Viness Pillay et al, the diameter of the fibre decreased initially but then increased as increasing in applied voltage at 50mm distance between tip of capillary and the collector. For distance 75 mm, the reduction of the nanofibres diameter to a minimum was not as apparent and the diameter increased slightly in response to an increase in the applied voltage. Initially, the diameter of the nanofibres decrease means at the critical voltage due to polymer solution stretching in interrelationship of increased the charge repulsion within the electrical field and the charge repulsion of polymer jet (Pillay *et al.*, 2013).

Adnan haider and co-workers (Haider, Haider and Kang, 2015) also reported the initially decreased of nanofibres diameter due to the polymer solution stretching and

relationship of the charge repulsion within the jet. Alsaid Ahmed Almetwally et al reported in their research as showed in the graph Figure 2.5, the diameter of nanofibres increase as increase of the applied voltage. Besides, Adnan Haider and co-workers reported the same statements and added in their report that the jet length increased due to increase of the applied voltage thus the diameter of nanofibres also increased (Haider, Haider and Kang, 2015).

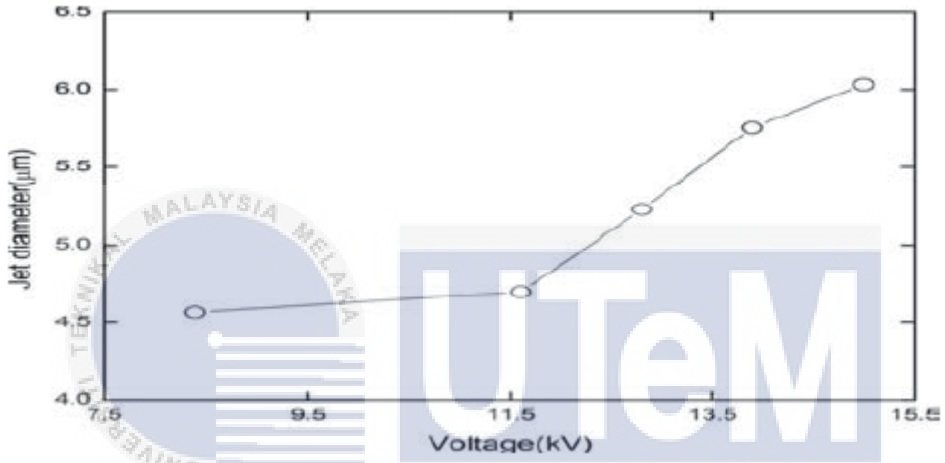


Figure 2. 5: The graph between jet diameter and applied voltage.

Despite, some researchers found that the applied electrospinning voltage was not dramatically changes the diameter of the nanofibres. In their study, the voltage used was in range 10 kV to 25kV and reveals that the high applied voltage would provide small diameter of nanofibres but non uniform in nanofibres diameter.

More than that, the low or high applied voltage than the critical voltage in electrospinning can contribute to the formation of the beaded nanofibres (Pillay *et al.*, 2013). Pursuant to previous researchers, the formation of beads in nanofibres and increased in diameter of nanofibres are due to the increased of applied voltage in electrospinning. These situations are attributed to the decrease of spherical drop or Taylor cone size and

high velocity of polymer jet (Haider, Haider and Kang, 2015). As reported by Viness Pillay et al, at low voltage range between 5.5 kV to 7.0 kV produced some beads defect in nanofibres. Meanwhile, the beads of nanofibres are getting more rampant at 7.0 kV of applied voltage and the density of beads increased at 9.0 kV. Similar results of beads increased as applied voltage increased as shown in Figure 2.6 below have been reported by Haghi et al (Haghi and Akbari, 2007).

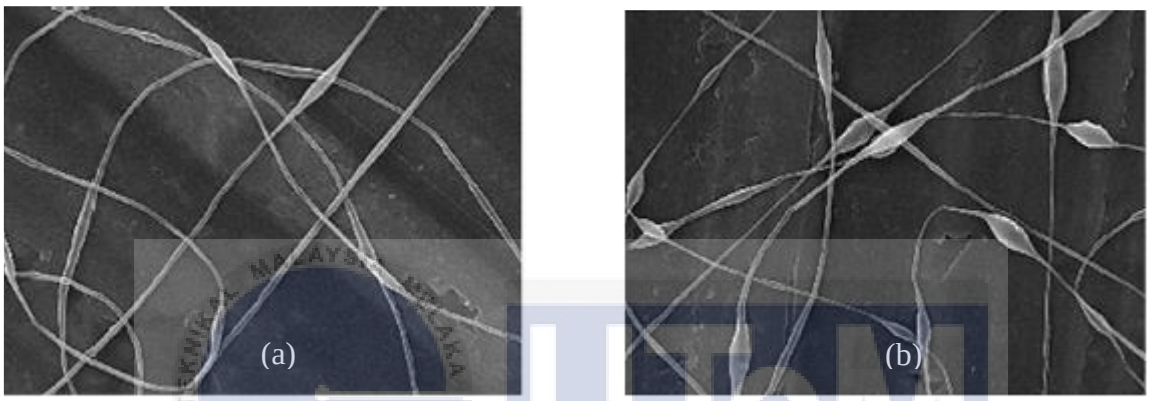


Figure 2. 6: The formation of beads (a) and the rampant beads as increased of applied voltage (b)

2.3.2 Distance between the tip of the needle and collector

The distance or gap between the tip of the needle and the collector also influence in nanofibres diameter and morphology. Commonly, the diameter of nanofibres is decreased as the electrospinning gap between tip of needle and collector increased. In other words, the diameter of nanofibres increased if the distance between the tip of the needle and the collector is small. This attributes to the polymer jet unable to evaporate completely during travel and caused the deposited nanofibres not completely dry (Pillay *et al.*, 2013). Despite, Amir Doustgani explained in research paper, the effect of the electrospinning distance on morphology nanofibres is not same.

$$E = V/d \qquad \text{Equation 2.1}$$

E= Electric field strength

V= Voltage

D= Distance

By referring to the Equation 1 above, extending the electrospinning distance would decrease the electric field as the electric field strength is inversely proportional to the distance. Decreasing the electric field strength will resulting the less jet acceleration that would produce the large fibres diameter (Doustgani, 2015b). So, this attracts attention to study the effect distance on nanofibres diameter in this paper. According to the Jayesh Doshi and Darrell H.Reneker, jet of nanofibres was unstable and small when the distance between the tip of capillary and collector is 30 mm (Doshi and Reneker, 1993). The researcher added the diameter of polymer jet decreased as the distance of gap from Taylor cone apex increased.

Besides, the distance of the gap between tip of needle and collector also affect the time of jet fly or travels. The large gap gives the much time for polymer jet to stretch in the electrical field before reach at the grounded collector (Doustgani, 2015a). Nonetheless, there were some cases reported by previous researchers that the change of distance between the tip of needle and the collector was not affect the change of the nanofibres diameter but there were beads formation as the distance decreased (Pillay *et al.*, 2013) (Haider, Haider and Kang, 2015). As stated by Quynh P.Pham et al, there were no significant impact of the gap from needle tip to the collector on the fibre diameter (Pham, Sharma and Mikos, 2006). So, the effect of the distance between the tip of capillary and the collector in electrospinning will be investigated in this paper.

2.3.3 Viscosity or concentration of the solution

The viscosity and concentration of the polymer solution also contributes to the change on the morphology of the nanofibres. Recent researchers have studied the correlation of the polymer concentrations and the morphological or diameter of nanofibres. As reported by previous researchers, the high concentration of the solution produced junction and bundles on nanofibres mat. This caused by the polymer solution still wet when deposited on the grounded collector. Besides, the droplet of the polymer solution dried out before ejected if the solution is too concentrated. More than that, by increasing the viscosity of the polymer solution also contributes to the bead formation and junctions of nanofibres (Pham, Sharma and Mikos, 2006). According to the H.Fong and co-workers, the beads formation decreased as the increased the viscosity but the beads diameter and average distance between the beads increased (Fong, Chun and Reneker, 1999). Some cases reported polymer with high concentration can affect the surface tension. But other observation, the bead formation decreased with increased of concentration. This observation tells that the varies of viscosity was affect the morphological nanofibres (Pham, Sharma and Mikos, 2006).

Furthermore, the viscosity of polymer solution is also influence the diameter of the nanofibres. According to the Quynh P.Pham et al, the diameter of the nanofibres deposited at the collector increased as well as increased of the concentration (Pham, Sharma and Mikos, 2006)

Table 2. 2: Table previous results for concentration effect on nanofibres diameter.

Solution	Concentration (wt %)	Diameter of nanofibres (nm)	Concentration (wt %)	Diameter of nanofibres (nm)
PLLA	1	100-300	5	800-2400
PVA	6	87	8	246

From the Table 2.2 result above, know that the diameter of the nanofibres increased with increased of concentration. Researcher added, the diameter of nanofibres increased attributed to the increased the surface tension (Pham, Sharma and Mikos, 2006). As reported by Viness Pillay and co-workers, the diameter of the nanofibres decreased as the concentration of the solution decreased. This is because the fibre jet is stretching due to the repulsive force from the electric charge. For the high viscosity of the solution, the diameter of nanofibres increased because of the viscoelastic force of the solution deviate the repulsive force.

From the previous researcher observation, the diameter of the nanofibres increased as the viscosity increased but fewer of the junction and bundles formation. Besides, low viscosity contributes to the decreased of the nanofibres diameter but increased in junction and bundle formation. This is due to the fibre jet not have sufficient time to evaporate before reaching the grounded collector.

2.3.4 Ambient parameters

The ambient parameters involve the humidity and the temperature in the electrospinning process. Several research papers have been studied to delve into the effect of the ambient parameters to nanofibres. Some researchers found the nanofibres diameter decreased when the temperature is increased. This relates to the decreasing of the polymer viscosity due to the increasing of the temperature.

For humidity effect, Adnan Haider and co-workers observed the effect humidity on nanofibres diameter by using PVA and PEO as their solutions. They reported the diameter of PVA nanofibers decreased from 667nm to 161 nm while PEO nanofibers decreased from 252nm to 75 nm when the humidity increased from 4% to 60%. The researchers also added there were beads formation in nanofibres if the humidity increased continually (Haider, Haider and Kang, 2015). Besides, according to the Alsaid Ahmed Almetwally and co-workers reported that average of the fibres increased as the humidity increased. This attributes to the higher humidity leads to increase the thickness of the fibre because the high electrostatic charge is adequate to split more fibres (Henton *et al.*, 2005).

Meanwhile, the humidity affects the porosity on the morphology nanofibres. The researcher added the increased of humidity leads to the small circular pores on the nanofibre surface. The further increasing of the humidity is yielding to the pores coalescing (Pham, Sharma and Mikos, 2006).

2.4 Poly lactic acid (PLA)

In this experiment, the poly lactic acid and chloroform are used as the type of polymer solution. As matter of fact, the use of poly lactic acid in polymer technology was famous and growth wildly in the development of biomedical and biotechnology. One of the interesting of characteristic poly lactic is biodegradable and environmental friendly. This biodegradable polymer is destroyed in physiological environment by organic macromolecular containing carbon into small end product. This degradation process also attributes by anaerobic or aerobic microorganism (Gupta, Revagade and Hilborn, 2007).

More than that, the poly lactic acid is made up from the chemical processing of lactic acid monomer. Commonly, lactic acid is produced through petrochemical feedstock which is non-renewable source. Around 1990, lactic acid produced from corn starch

through bacterial fermentation process which makes it more economic and used renewable source instead of petrochemicals (Gupta, Revagade and Hilborn, 2007).

The special biodegradable characteristic makes poly lactic acid the major choice in various applications such as biomedical, tissue engineering and manufacturing obviously. Additionally, the PLA has speciality in mechanical strength, thermoplastic processibility and biodegradability. Besides, the PLA promotes its biodegradable and nontoxic characteristic in biomedicine application which is not harm to human health.

In this experiment, the poly lactic acid will be dissolved in chloroform. As recorded by R.Casasola et al, PLA was dissolved in chloroform and obtained 10% w/v concentration of polymer solution. The experiment was set with 20kv of voltage, 15cm for the gap between the tip of capillary and the collector and 10 minutes for time taken. The feed constant was set in 1.00 ml/h. The Figure 2.7 below shows the result obtained by previous researchers.

Figure 2. 7: Polylactic acid solution in chloroform

2.5 Applications of Polylactic acid nanofibres and electrospinning

Electrospinning is simple, inexpensive yet versatile process to produce the fine nanofibres that has high demand on variety of applications. This process has been used widely for the fabrication which has develop the potential application in environmental protection, biomedical, sensor, electronic and clothing(Haider, Haider and Kang, 2015). Electrospinning process promotes great opportunity in designing the morphological and porosity of the nanofibres. In addition, electrospinning provides the nanofibres with special properties such as high surface area to volume ratio, its morphological and the porosity.

PLA is one of the biodegradable polymers that from natural resources such as corn starch. Plus, PLA is the great and excellent material for medical purpose. It is no wonder nowadays nanofibres is the popular approaches in all the tissue and organs i.e collagen, skin, dentine, cartilage and bone in biomedical fields. For biomedical, the tissue engineering, wound dressing and drug delivery were the famous applications. In tissue engineering, biodegradable scaffolds is more preferred than the conventional because of its natural environment.

In drug delivery application, it is important to provide the drug is smaller size so that the drug can be absorbed and digested easily by the target site. The electrospinning method helps a lot in drug delivery application. For example, anticancer agent, protein, antibiotics, RNA and DNA were in nanofibres form. Wound dressing is wound healing that plays main role to protect the wound site. There are varies of dressing according to the purpose. Commonly, the wounds exposed to the microbial growth. Here, the wound dressing must have special characteristic such as great moist environment to enhance the healing process and antibiotic resistant bacteria. Thus, electrospinning provides those characteristic in wound dressing rather than conventional method. This is because the electrospinning can prepared the nanofibres with great porosity and high surface area.

More than that, the beauty mask which are used for skin treatment or skin cleansing also can be prepared by electrospining (Haider, Haider and Kang, 2015).

Electrospinnig is also important in filtration application which can remove the heavy metal ion from the contaminant water. Plus, the ion released can cause harm to the human health and the environment. In reservoir, the heavy metal ion easily mixed with water. Therefore, filtration is very important to separate the ions from the water. Prakash Khude reported that the effectiveness filtration is relates to the fineness of nanofibres (Khude, 2017). Thus, electrospinning is a great and simple method that can produced the fine nanofibres.

In summary, nanofibres technology is widely used and has great potential in variety of application. Nanofibres technology also helps in biomedical development as can enhance the performance in tissue engineering and wound dressing obviously. Plus, electrospinning method is the simple and versatile method helps a lot in producing quality nanofibres.

CHAPTER 3

METHODOLOGY

3.0 Introduction

This chapter explained the methodology used in this experiment to obtain the data and result for the effect of electrospinning applied voltage and the distance on morphology PLA electrospun nanofibres. In this experiment, the procedure begun with solution preparation and continued with electrospinning process, platinum coated process, observation under scanning electron microscopy (SEM) and analysis the diameter of nanofibres using ImageJ software.

Starting with solution preparation, the Polylactic acid and chloroform were used as mixture in this experiment. The Polylactic acid used in this experiment was in pellet form while the chloroform was used as solvent to dissolve the Polylactic acid pellet. The procedure to prepare the solutions with concentration 10% wt, 13% wt and 16 % wt of Polylactic had been recorded in this chapter.

Continuing on, the horizontal electrospinning setup was used to run the experiment. The metal plate was used as collector which is the nanofibres deposited on. In this experiment, the flow rate of the solution was set to 3.00 ml/h by using automated flow rate machine. For different applied electrospinning voltage experiment, the range of voltage used was 10kV to 20kV while for different electrospinning distance was 75mm to 175mm. The total sample collected were 10 samples for every mentioned concentration.

The next procedure, the samples were coated with platinum by using auto fine coated machine. The samples were attached to metal stubs before coating process. The coating process was set to 240 seconds with current 20 miliampere (mA).

Next, the samples were investigated under scanning electron microscopy (SEM) to obtain the micrograph for every sample. In the scanning electron microscopy, the voltage used is 10kV with 100 and 1000 of magnification. The micrographs of the samples then were observed by using the ImageJ software to determine the nanofibres diameter.

3.1 Flow chart of process

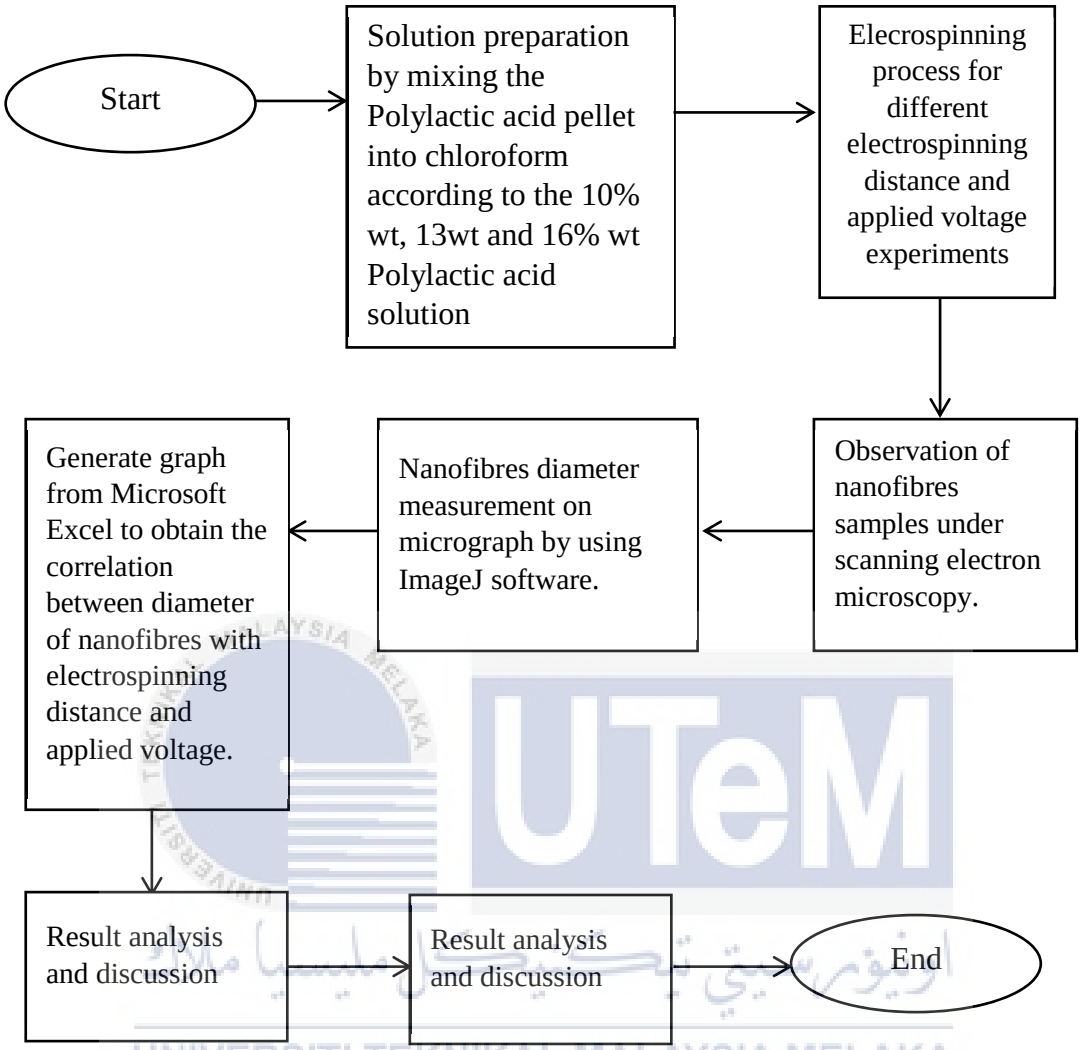


Figure 2. 8: The flowchart of process.

3.2 Solution preparation

In this experiment, the mixture of poly lactic acid and chloroform are used as mixture. The Polylactic acid pellet will be dissolved in the chloroform solvent. The solution with concentration of 10% wt, 13% wt and 16% wt of Polylactic acid were prepared. Hence, the following formula Equation 2 below was used to determine the amount of solvent needed in achieving 10%, 13% and 16% wt of Poly lactic acid.

Formula:

$$Wt\% = M1 / (M1 + M2) \qquad \text{Equation 3. 1}$$

M1 = Mass of Poly lactic acid pellet (gram)

M2 =chloroform as solvent (gram)

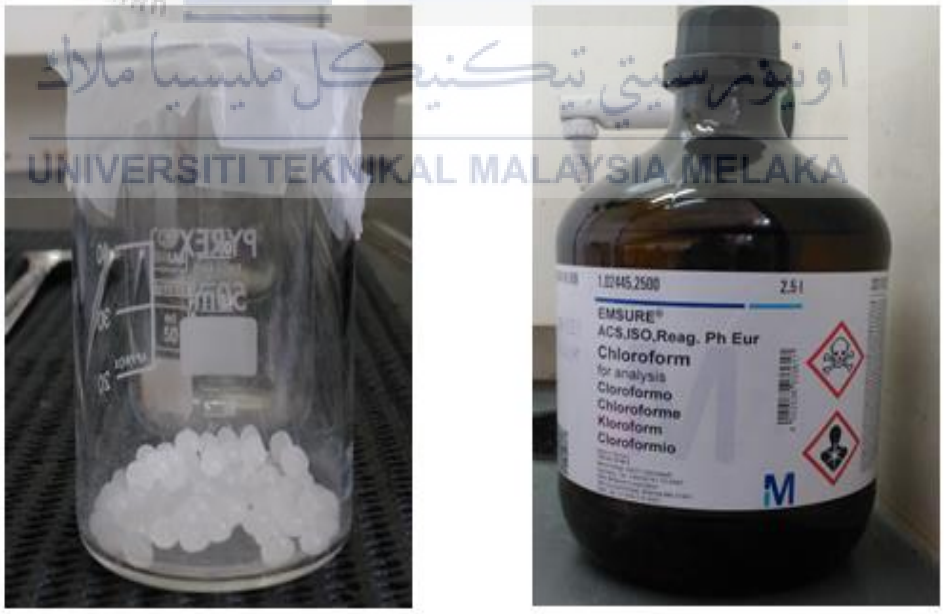


Figure 3. 1: (a) The Polylactic acid (PLA) pellets and (b) the chloroform (solvent)

In this experiment, 200g for every concentration were prepared. The materials were weighed by using weighing balance machine. The Table 3.1 below shows the weight of the Polylactic acid (PLA) and chloroform in grams to achieve 10%, 13% and 16% wt of Polylactic acid.

Table 3. 1: The details for the solutions

Concentration of Polylactic acid (%)	Weight of the Polylactic acid (PLA)	Weight of the chloroform (g)
10	20	180
13	26	174
16	32	138

After that, the solution was stirred gradually using magnetic stirrer to avoid the formation of residue that can affect the solution. To dilute the solution, the heat was not used in this experiment because the chloroform can easily degrade. The time taken to dissolve the Polylactic acid pellet in the chloroform is different according to the solution concentrations. The Table 3.2 shows the time taken to dissolve the Polylactic acid pellet in the chloroform for every concentration.

Table 3. 2: Time taken to dissolve Polylactic acid in chloroform.

Concentration solution (%)	Time taken to Polylactic acid (PLA) in chloroform (hours)
10	4
13	7
16	9

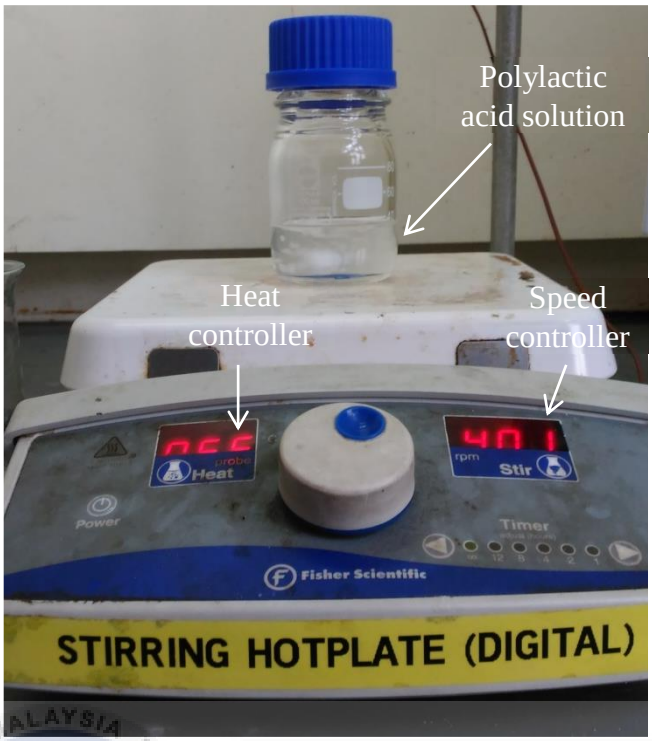


Figure 3. 2: The Polylactic acid solution gradually stirred by magnetic stirrer.

3.3 Electrospinning process

The electrospinning setup consist of four major components such as automated flow rate machine to control syringe pump, a metal plate collector, power supplier and metal needle. The Figure 3.3 below shows the electrospinning apparatus and process.

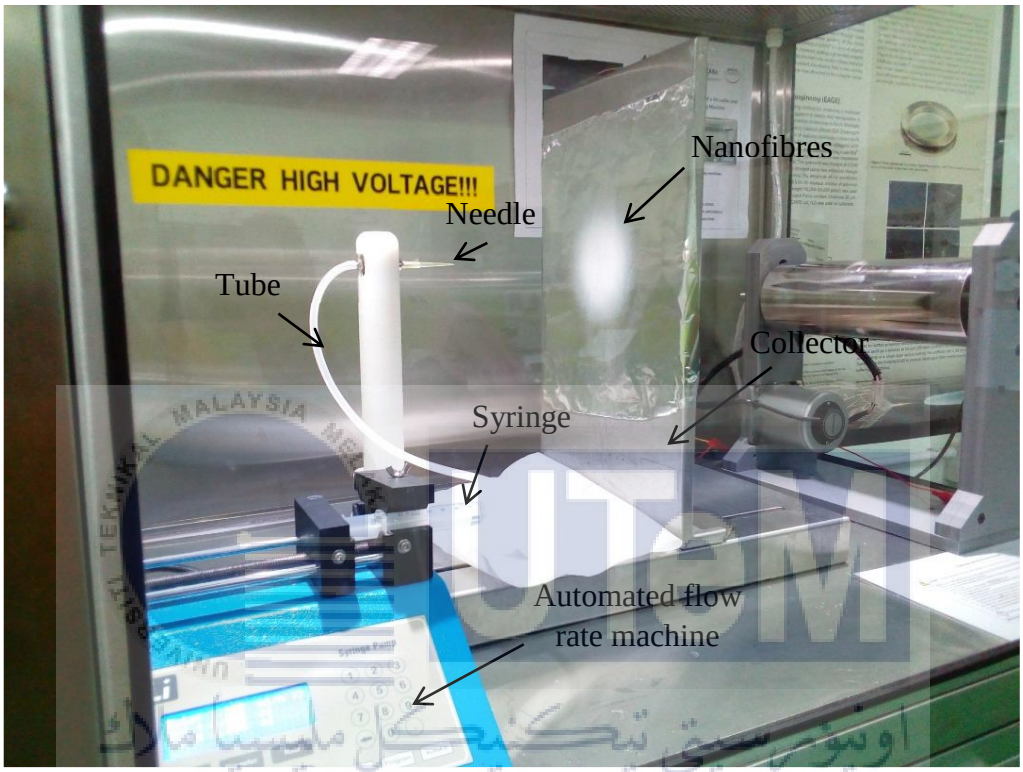


Figure 3. 3: The electrospinning process and apparatus.

Continuing on, the polymer solution was filled in the syringe and the flow rate then set to 3.00 ml/h. The flow rate is constant for both different electropsinning distance and applied voltage experiments. The metal plate collector was covered with the aluminium foil to collect the samples. For varies applied electrospinning voltage experiment, 10 kV, 12.5 kV, 17.5 kV and 20 kV of voltage were applied in electrospinning to collect five (5) samples of nanofibres. The electrospinning distance from the tip of the needle to collector was 100 mm.

Otherwise for different electrospinning distance from the tip of needle to collector experiment, the applied electrospinning voltage was set to 15 kV. The varies electrospinning distances for this experiment were 75 mm, 10 mm, 125 mm and 175 mm.

When the solution imposed to the charge, the polymer ejected from the tip and travel to the collected.The solid polymer fibres attached to the collector is called as scaffold or mesh.

3.4 Platinum coating process

Before the nanofibres samples were scanned under SEM, the sample will be coated with platinum by using JEOL JEC-300FC auto fine coated machine. The samples were cutted into small pieces as shown in Figure 3.5. Then nanofibres samples were attached to carbon tape and placed on the metal stubs before undergo the coating process. After that, the stub was placed in the chamber for coating process. The time for coating process is set to 240 seconds (s) with current 20 miliampere (mA).

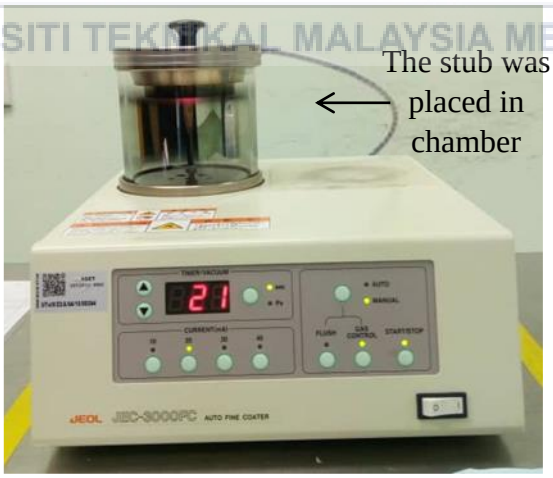
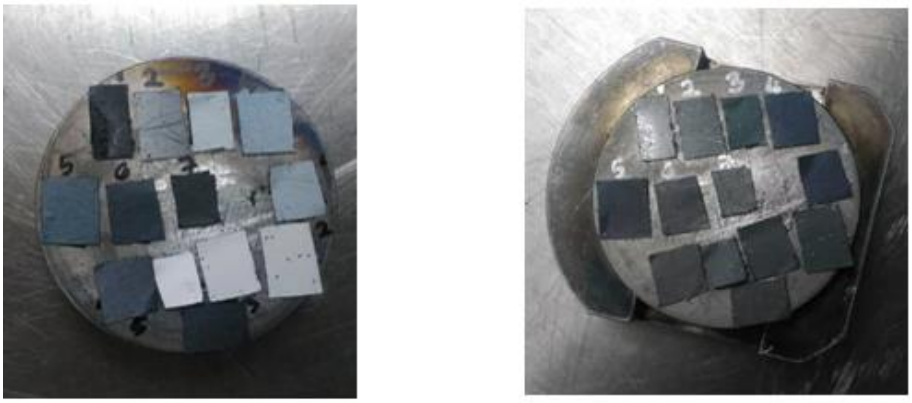


Figure 3. 4: Coating process using auto fine coated machine.



(a) (b)

Figure 3. 5: (a) before coating process and (b) after coating process.

3.5 Morphology observation under Scanning electron microscopy (SEM)

Scanning electron microscopy precedes the investigation of the nanofibres samples in vacuum mode. The samples were vacuumed in the vacuum chamber in the SEM machine. This step is to avoid the electron disperse during collision with other molecules. The micrograph of nanofibres will be observed under scanning electron microscope (SEM). The scanning electron machine (SEM) also operated at acceleration voltage 10 kV to 15 kV. The magnification was adjusted to obtain micrograph of the nanofibres samples.

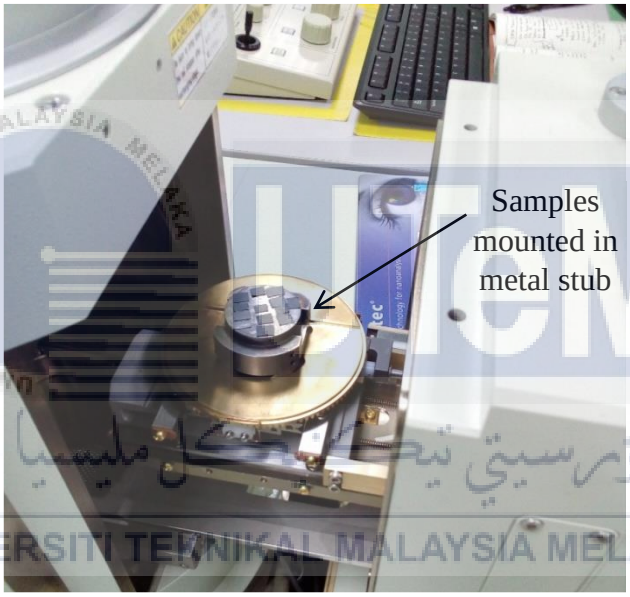


Figure 3. 6: The samples were placed in chamber of the Scanning electron microscopy.

Figure 3. 7: Scanning electron microscopy (SEM)

Lastly, the diameters of the nanofibres were determined through micrograph using ImageJ software. There were 100 measurements of nanofibres diameter at different location on micrograph to minimise the error. The following steps are the sequences to obtain the diameter of nanofibres.

Step 1: The micrograph was uploaded in Image J software and the line was drawn on the scale.

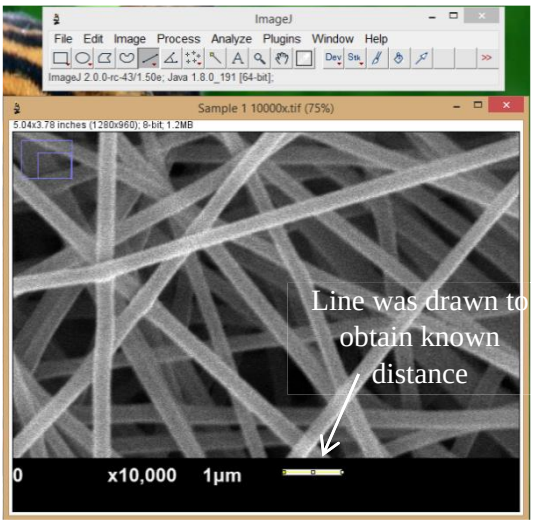


Figure 3. 8: Calibration of the nanofibres in micrograph.

Step 2: The scale known distance was set 1 µm (according to the micrograph) in set scale option.

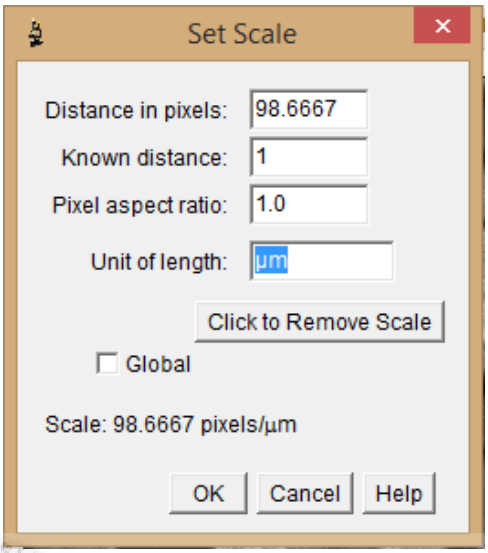


Figure 3. 9: The set scale option.

Step 3: 100 lines were drawn at the nanofibres to get average of the nanofibres diameters.

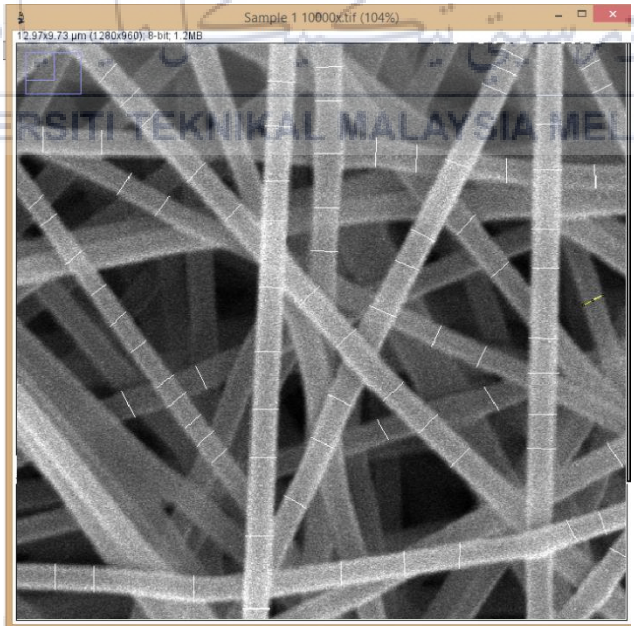


Figure 3. 10: The lines were drawn at the nanofibres micrograph.

Step 4: Then, the diameter of the nanofibres generated in the software by clicking the measure option. The 100 data were generated based on the drawn line.

File Edit Font Results

	Area	Mean	Min	Max	Angle	Length
1	0.003	171.182	99.667	224.801	-68.199	0.279

Figure 3. 11: The data diameter obtained in ImageJ software.

Step 5: The data generated after the entire 100 diameter were measured. The data included were mean, standard deviation, minimum and maximum diameter of nanofibres. The steps were repeated for each nanofibres sample.

Mean	0.003	150.707	107.086	191.452	-20.076	0.287
SD	2.601E-4	26.833	28.209	32.994	50.491	0.025
Min	0.002	75.944	41.650	97.840	-132.274	0.235
Max	0.004	203.492	166.482	248.935	62.447	0.357

Figure 3. 12: The data generated by the ImageJ sofware.

Step 6: Lastly, the data were analysed in Microsoft excel and two graphs were presented by diameter of the nanofibres. The data were analysed using Microsoft excel software then presented in two graphs that are for different applied electrospinning voltage and distance experiments.

CHAPTER 4

RESULT AND ANALYSIS

4.1 Result for electrospinning parameter analysis on electrospun nanofibres for 10 % wt of Polylactic acid.

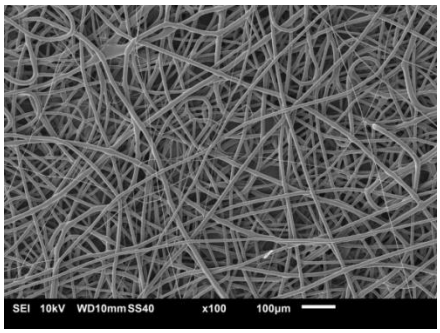
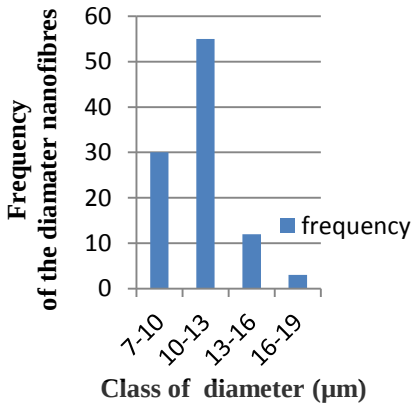
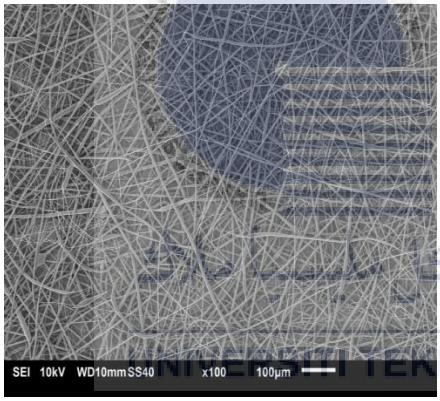
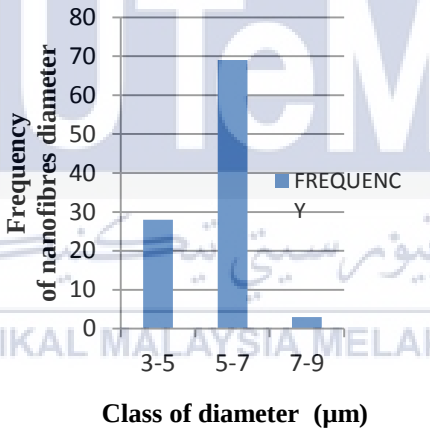
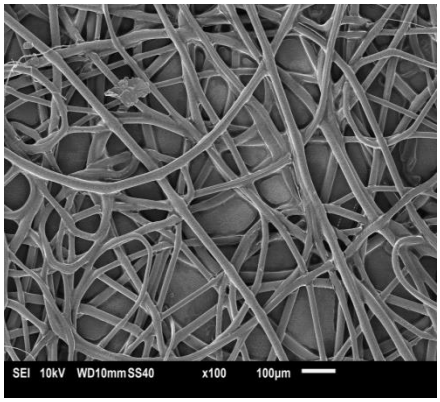
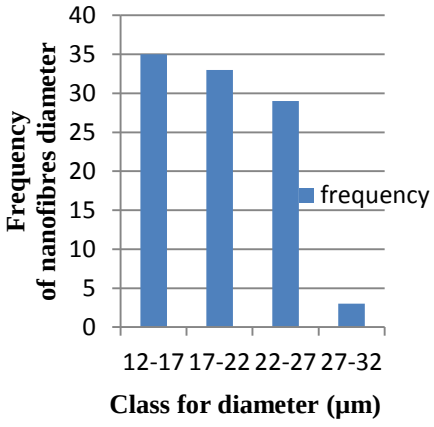
4.1.1 Result for different distance from the tip of nozzle to the collector.

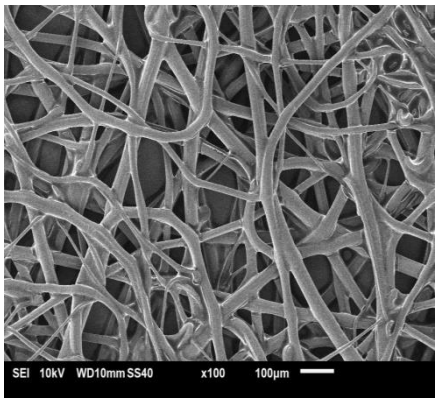
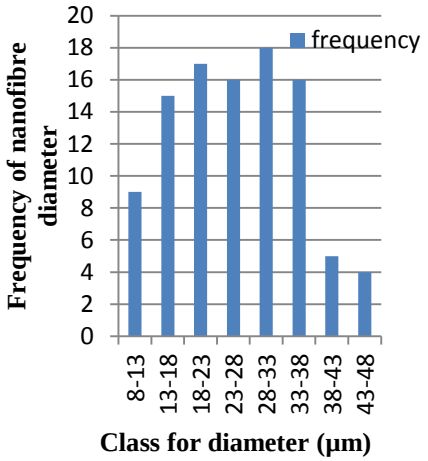
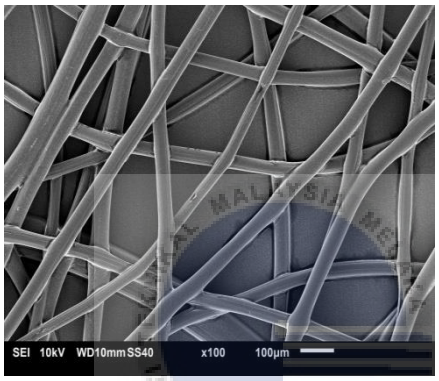
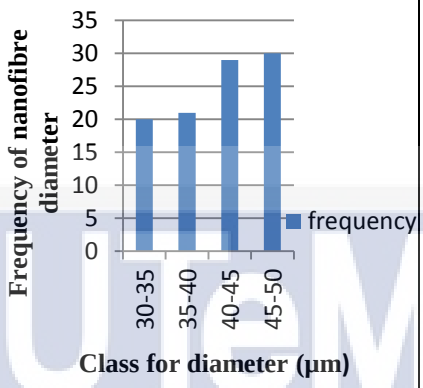
The concentration of the solution for this experiment is 10% wt of Polylactic acid (PLA). The parameters for every sample are as shown in Table 4.1 below.

Table 4. 1: Parameter for the Polylactic acid nanofibres sample

Sample	Distance between the tip of the nozzle to the collector (cm)	Applied electrospinning voltage (kV)	Flow rate of the solution (ml/h)
a	7.5	15	3.000
b	10.0		
c	12.5		
d	15.0		
e	17.5		

Table 4. 2: The micrograph, class of nanofibres diameter and mean diameter of the Polylactic acid nanofibres for different distance from the tip of nozzle to collector.

MICROGRAPH OF POLYLACTIC ACID NANOFIBRES	GRAPH OF CLASS DIAMETER	MEAN DIAMETER (µm)
 (a)		11.21±2
 (b)		5.53±0.8
 (c)		19.10±4.5

 (d)		25.80±9.2
 (e)		41.21±5.3

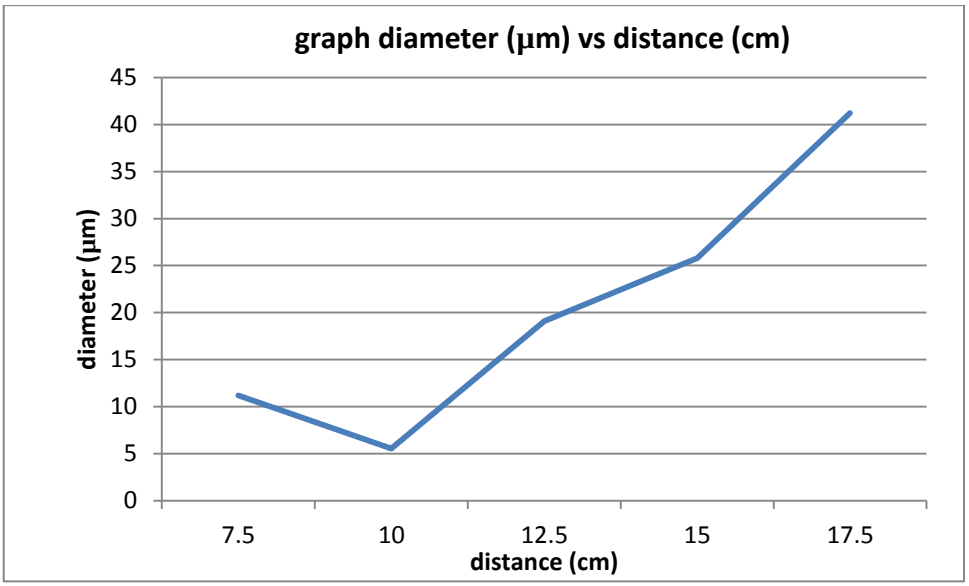


Figure 4. 1: The graph of diameter versus distance of Polylactic acid nanofibres.

Table 4.2 above shows the nanofibres micrograph with 100 times of magnification, the graph of class nanofibres diameter for different distance between the tip of the nozzle and the collector samples. For sample (a), most of the fibre strains have diameter 10 to 13 µm while only the small amount of fibres that have 16 to 19 µm. Next, the highest amount of nanofibres with diameter 5 to 7 µm was found in sample (b). For sample (c), (d) and (e), the diameter that have highest amount of fibre strains are 12 to 17 µm, 28 to 33 µm and 45 to 50 µm respectively.

From the micrograph above, the bead free, smooth and smallest fibres can be found in sample (b) with the average as smallest as 5.53 µm compared to other samples but the fibres are not homogenous. According to the micrographs with 100 times of magnification that have been analysed under scanning electron microscope (SEM), the fibres getting bigger and the production rate decreased when the distance from the tip of nozzle increased. Regarding to the graph Figure 4.1 above, the diameter of the nanofibres

increased start from the distance of 10 cm to 17.5 cm. This proof that the diameter of nanofibres increased as the distance from the tip of nozzle to collector increased.

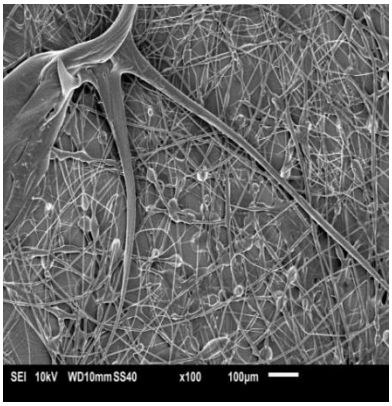
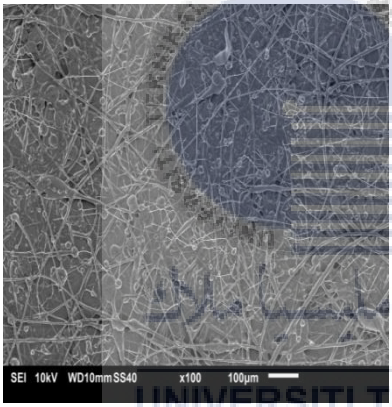
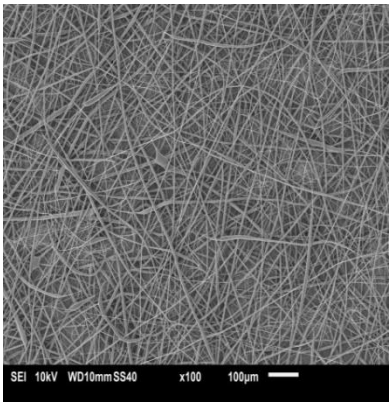
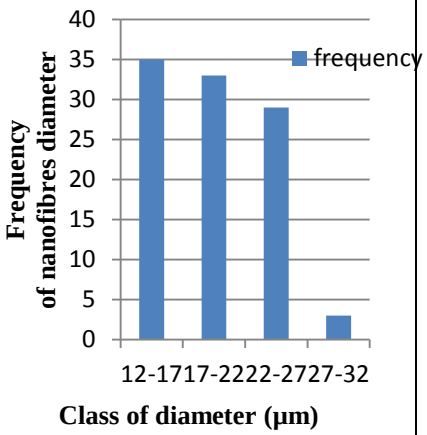
4.1.2 Result for different applied electrospinning voltage

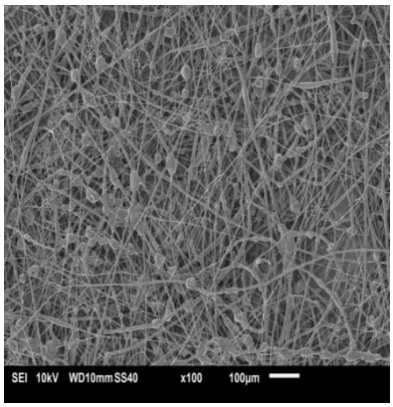
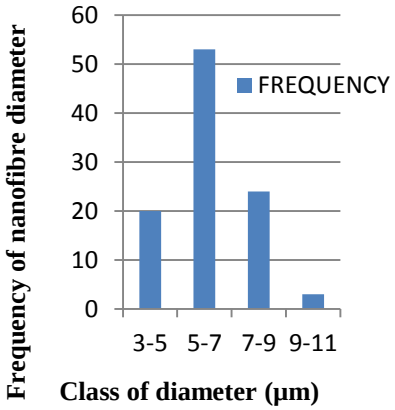
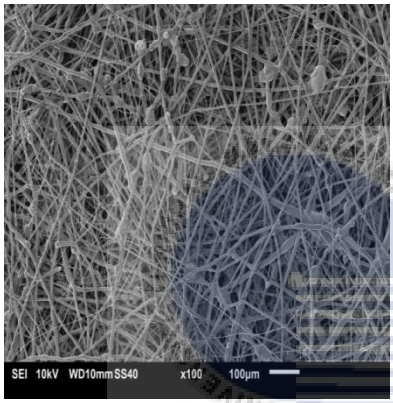
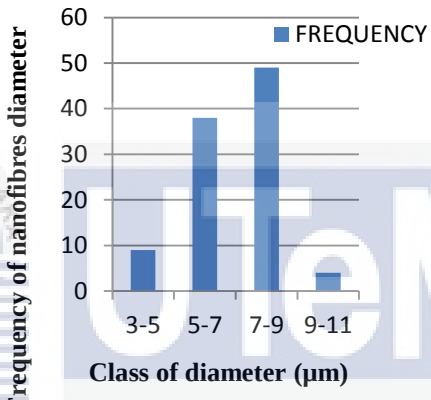
The parameters for every sample in different applied electrospinning voltage experiment are as shown in Table 4.3 below.

Table 4. 3: The parameters of Polylactic acid nanofibres samples.

Sample	Applied electrospinning voltage (kV)	Distance from the tip of the nozzle to the collector (cm)	Flow rate of the solution (ml/h)
a	10.0	10	3.000
b	12.5		
c	15.0		
d	17.5		
e	20.0		

Table 4. 4: The micrographs, graph of class of diameter and mean of diameter for the different applied electrospinning voltage.

MICROGRAPH OF POLYLACTIC ACID NANOFIBRES	GRAPH OF CLASS DIAMETER	MEAN DIAMETER (µm)
 (a)		Can't be measured
 (b)		Can't be measured
 (c)		5.53±0.8

 (d)		6.29±1.4
 (e)		6.9±1.3

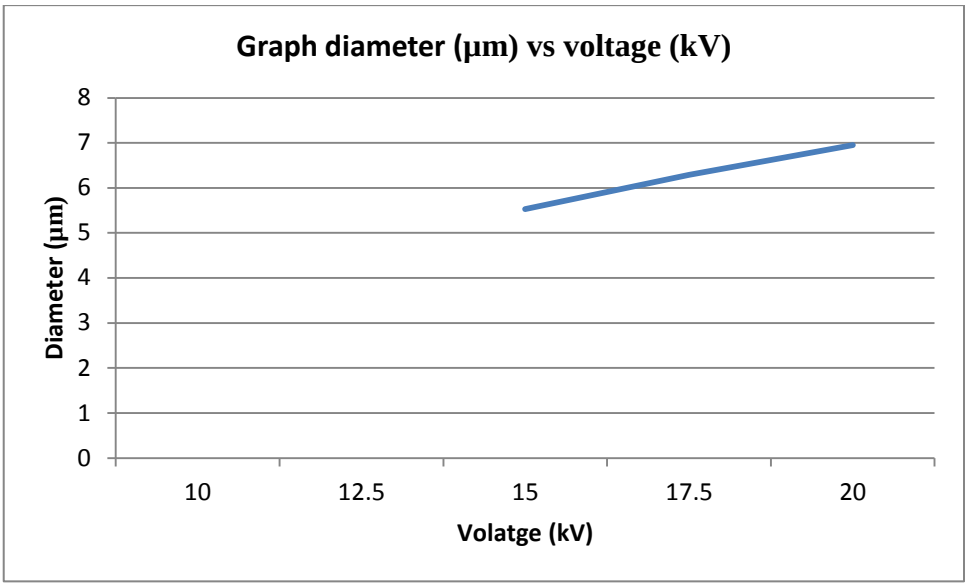


Figure 4. 2: The graph of Polylactic acid nanofibres diameter and applied electrospinning voltage

For sample (a) and (b), the diameter of the nanofibres cannot be measured because of the huge bead and totally wet nanofibres. For sample (c) and (d), the most fibres strains have diameter 5 to 7 µm. Besides, the highest amount of the fibres strain in sample (e) is 7 to 9 µm. By referring the micrographs, the sample (e) is the smallest and non-beaded nanofibres. However, the beads are getting more prevalent as increases of applied voltage. Regarding to the graph Figure 4.2 above, the diameter of the nanofibres gradually increased start from 10 kV of applied voltage in electrospinning. From the result in graph Figure 4.2, know that applied voltage on electrospinning can affect the diameter of the nanofibres which is the diameter of nanofibres increased as the applied voltage increased.

4.2 Result for electrospinning parameter analysis on electrospun nanofibres for 13 % wt of Polylactic acid.

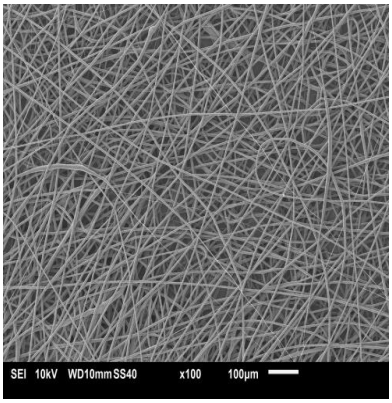
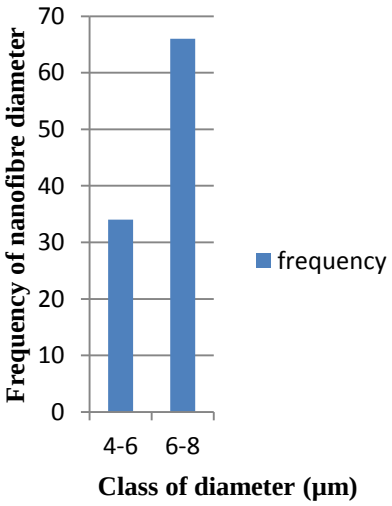
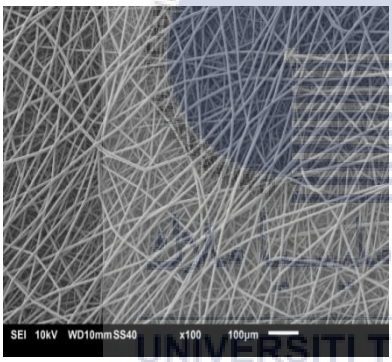
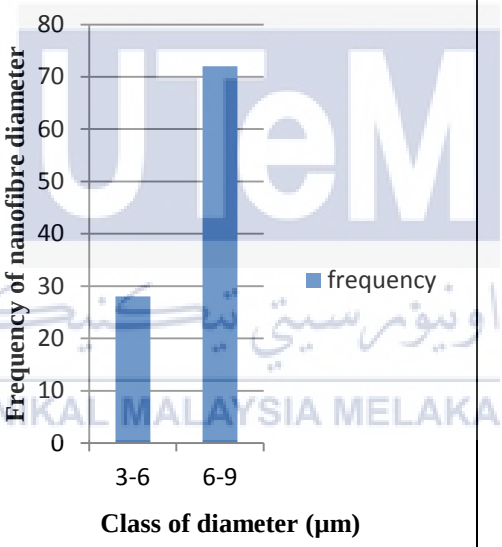
4.2.1 Result for different electrospinning distance.

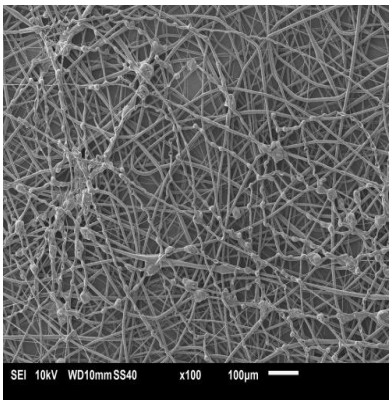
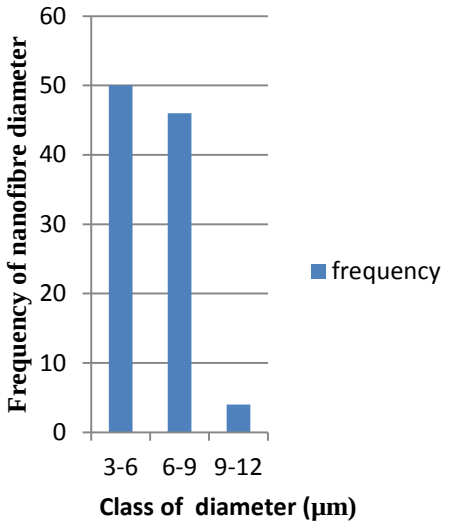
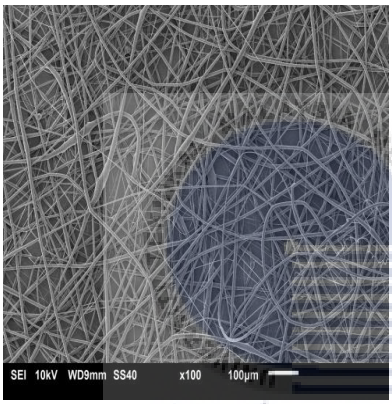
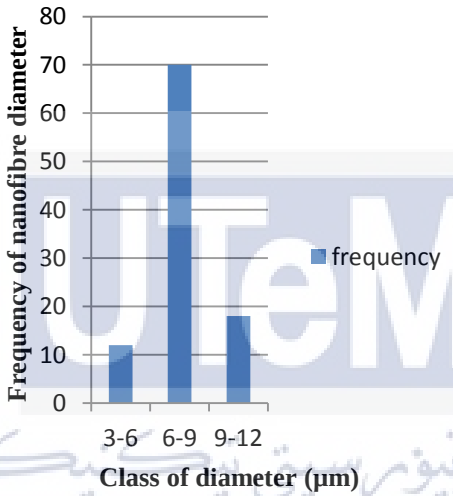
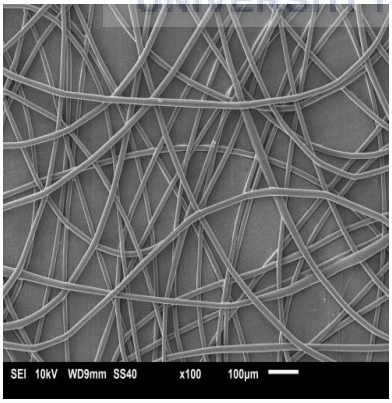
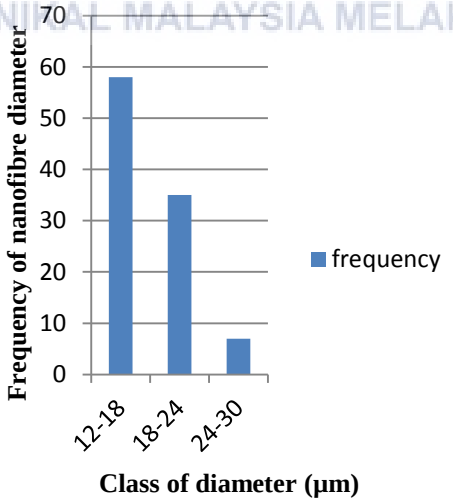
The parameters for every sample in different distance from the tip of nozzle to the collector experiment are as shown in the Table 4.5 below.

Table 4. 5 :The parameters for 13% wt of Polylactic acid samples

Sample	Distance between the tip of the nozzle to the collector (cm)	Applied electrospinning voltage (kV)	Flow rate of the solution (ml/h)
a	7.5	15	3.000
b	10.0		
c	12.5		
d	15.0		
e	17.5		

Table 4. 6: micrograph, class of diameter and mean diameter of 13% wt Polylactic acid

MICROGRAPH OF POLYLACTIC ACID NANOFIBRES	GRAPH OF CLASS DIAMETER	MEAN DIAMETER (µm)
 (a)		6.14±0.7
 (b)		6.76±1.3

 (c)		6.17±1.8
 (d)		7.56±1.4
 (e)		18.15±3.5

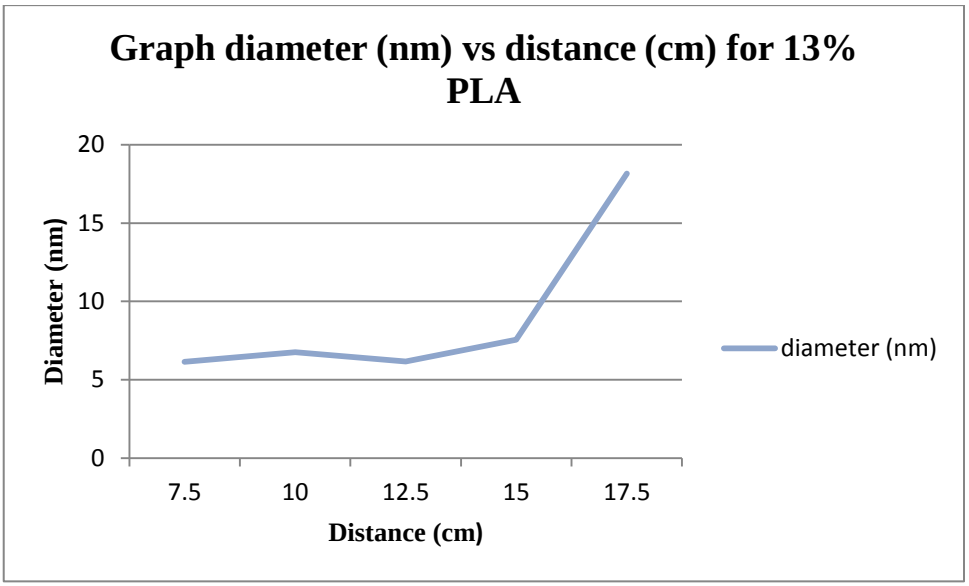


Figure 4. 3: The graph diameter versus the electrospinning distance for 13% wt PLA

From Table 4.5 above, the micrograph sample (a) and (b) show that there were no significant change in size of nanofibres. Both of the sample (a) and (b) micrographs showed that homogenous nanofibres in range 6.14 µm and 6.76 µm respectively. It showed that the thinnest nanofibres can be found in sample (a) with mean diameter 6.14 µm. For sample (c), the micrograph showed that there were beaded defect in nanofibres. The beaded nanofibres might be because of the wet nanofibres layered and stick each other. However, the thin nanofibres in range 3-6 µm can be found more in sample (c).For micrographs (d) and (e), the diameter of nanofibres increased as the distance increased. According to the micrographs (e), it showed that the production rate of nanofibres is less compared to other samples. It clearly showed that less fibre deposited in sample (e).

From the graph Figure 4.3 above, the diameter of the nanofibres for 13% wt of Polylactic acid increased as the distance from the tip of the tip of nozzle to the collector increased. As mention, increasing the electrospinning distance will decrease the electric

field strength as the formula in equation 1. Thus, the less acceleration will contribute to the large diameter of fibres.

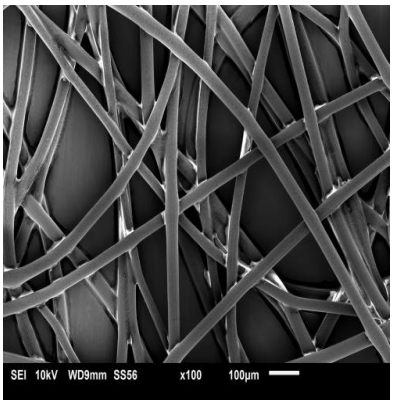
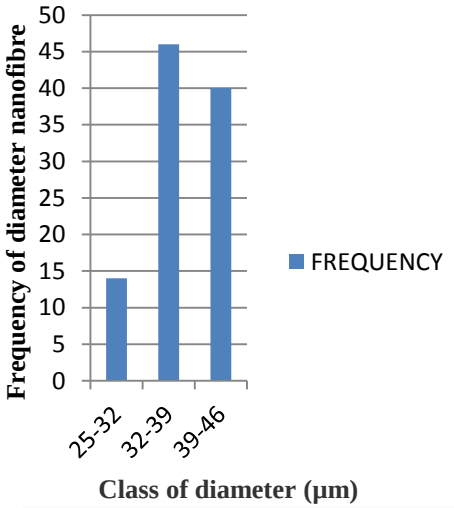
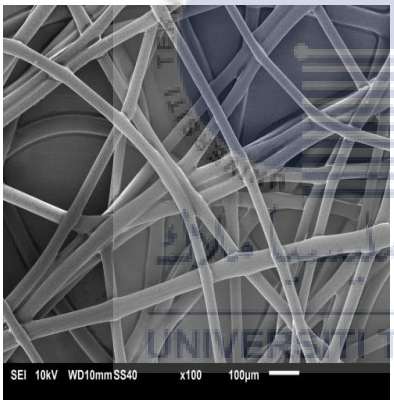
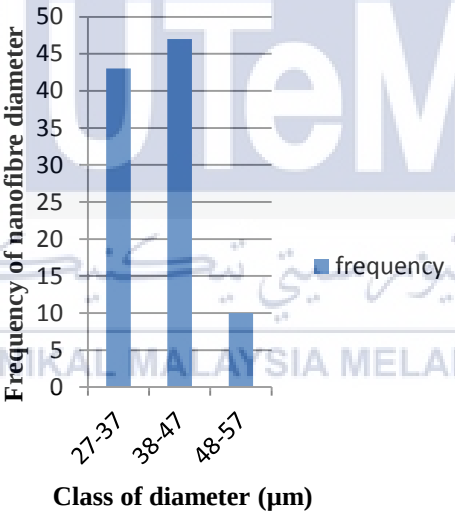
4.2.2 Result for different applied voltage.

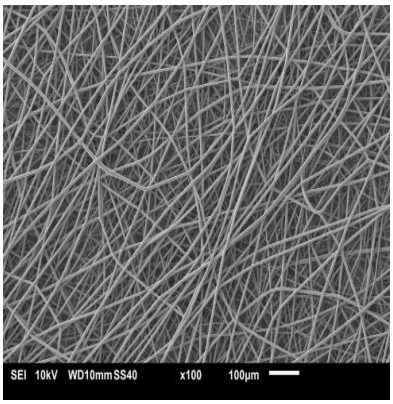
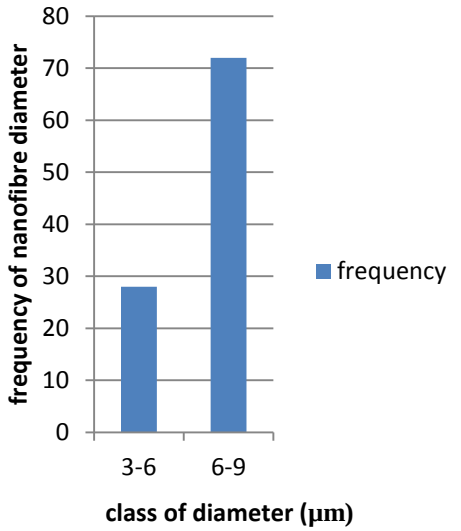
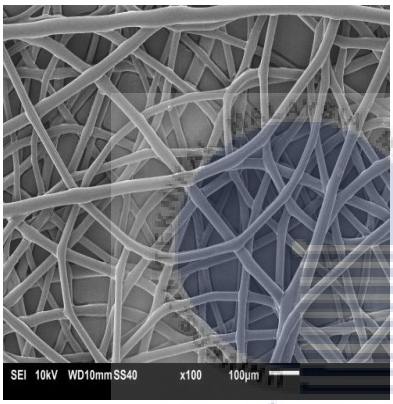
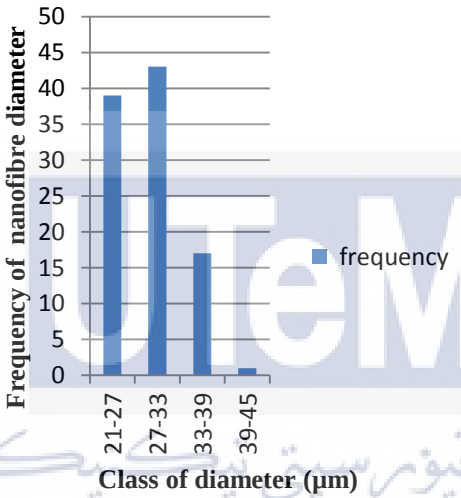
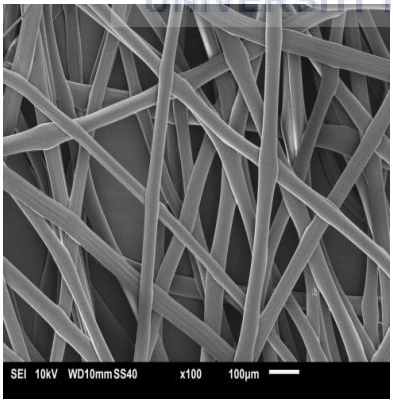
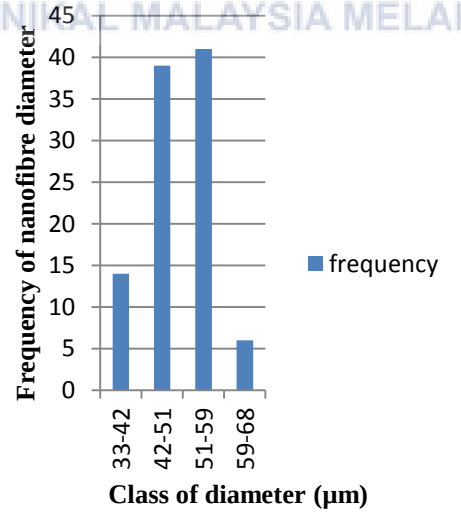
The parameters for every sample in different applied electrospinning voltage experiment are as shown in Table 4.7 below.

Table 4. 7: The parameters of the Polylactic acid nanofibres samples.

Sample	Applied electrospinning voltage (kV)	Distance from the tip of the nozzle to the collector (cm)	Flow rate of the solution (ml/h)
a	10.0	10	3.000
b	12.5		
c	15.0		
d	17.5		
e	20.0		

Table 4. 8: Micrographs, graph of class diameter and mean diameter for the different applied electrospinning voltage.

MICROGRAPH OF POLYLACTIC ACID NANOFIBRES	GRAPH OF CLASS DIAMETER	MEAN DIAMETER (µm)
 (a)		36.15±4.6
 (b)		38.79±5.3

 (c)		6.76±1.3
 (d)		29.09±4.0
 (e)		49.20±6.7

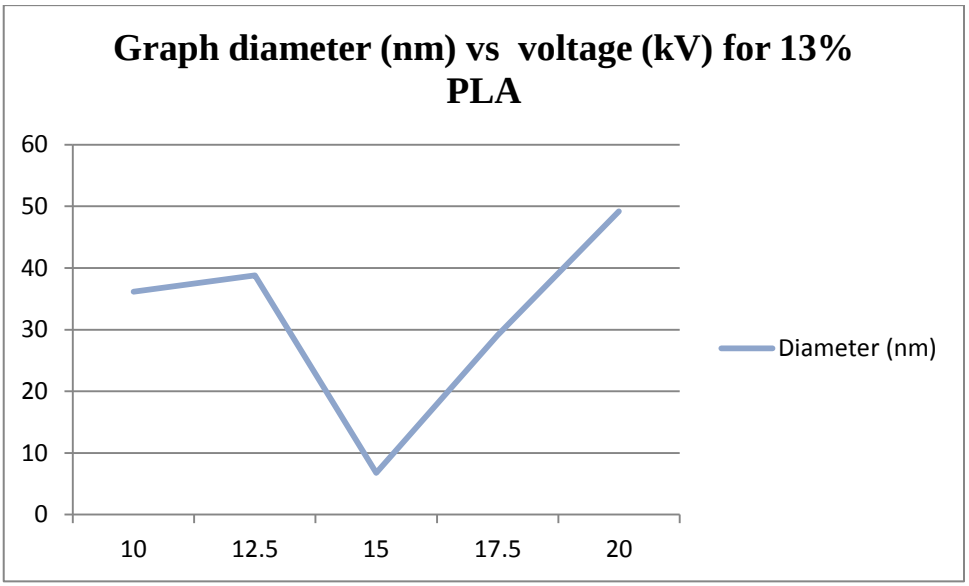


Figure 4. 4: The graph of nanofibres diameter vs electrospinning applied voltage for 13% wt PLA

By referring the Table 4.7 above, the wet fibres can be found in the sample (a). The wet fibre can be seeing clearly when the fibres layered on other fibres. For sample (c), the micrograph showed that more nanofibres produced and it was the thinnest nanofibres with diameter 6.76 µm. The diameters of the nanofibres increased start from sample (a) to sample (b) but became smaller in sample (c) and then continue bigger again from sample (d) to sample (e). The nanofibres in sample (c) were in the best or optimum parameter. This is because the characteristic of the nanofibres which are smooth, beads free, thin and homogenous in size.

From the graph Figure 4.4 above, the diameter of nanofibres increased as the voltage increased. However, the nanofibres diameter in sample (c) was the thinnest as it is achieved the optimum parameter. The parameters of the sample (c) were shown in Table 4.5 above.

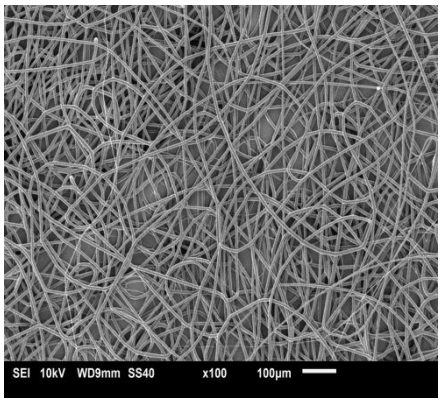
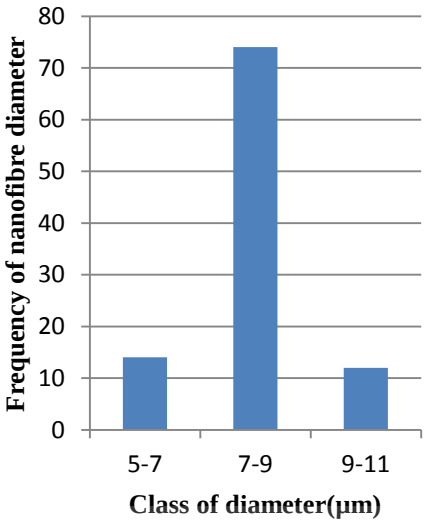
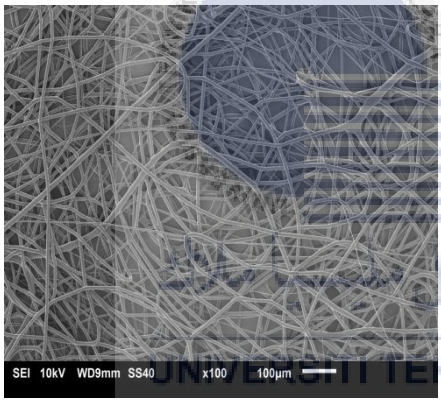
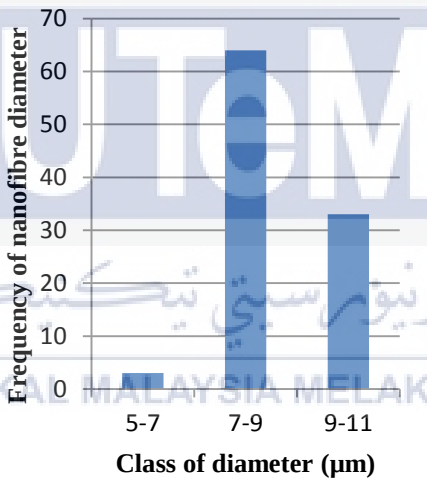
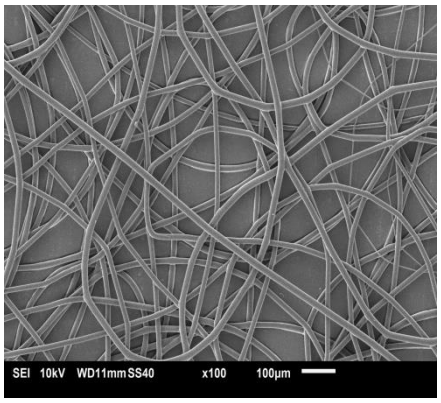
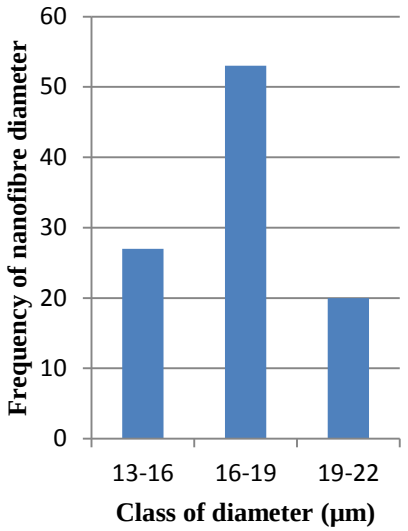
4.3 Result for electrospinning parameter analysis on electrospun nanofibres for 16 % wt of Polylactic acid.

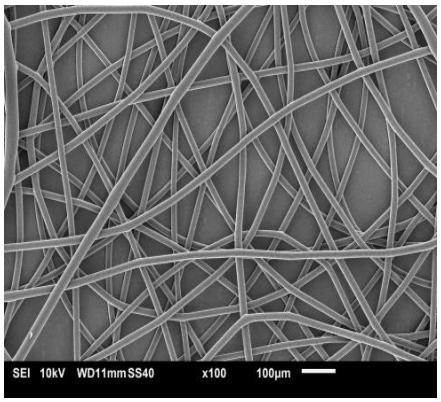
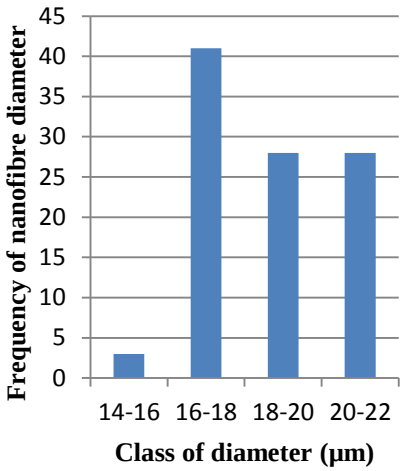
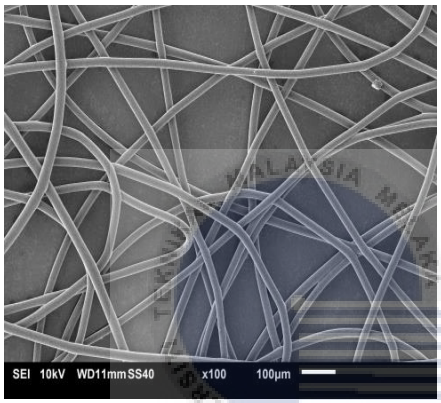
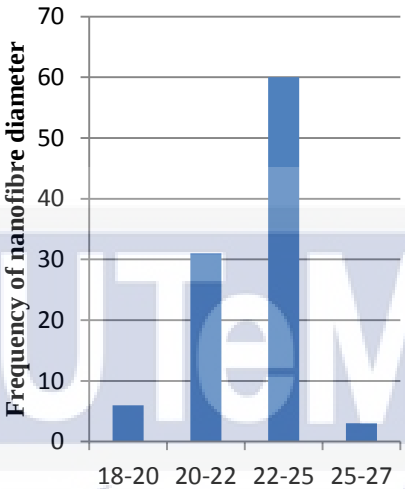
4.3.1 Results for different electrospinning distance experiment.

Table 4. 9: Parameters for different electrospinning distance experiment

Sample	Distance between the tip of the nozzle to the collector (cm)	Applied electrospinning voltage (kV)	Flow rate of the solution (ml/h)
a	7.5	15	3.000
b	10.0		
c	12.5		
d	15.0		
e	17.5		

Table 4. 10: Micrograph, class of diameter and mean diameter of PLA nanofibres.

MICROGRAPH OF POLYLACTIC ACID NANOFIBRES	GRAPH OF CLASS DIAMETER	MEAN DIAMETER (µm)
 (a)		7.90±0.9
 (b)		8.60±0.8
 (c)		17.30±1.9

 (d)		18.62±1.6
 (e)		22.38±1.5

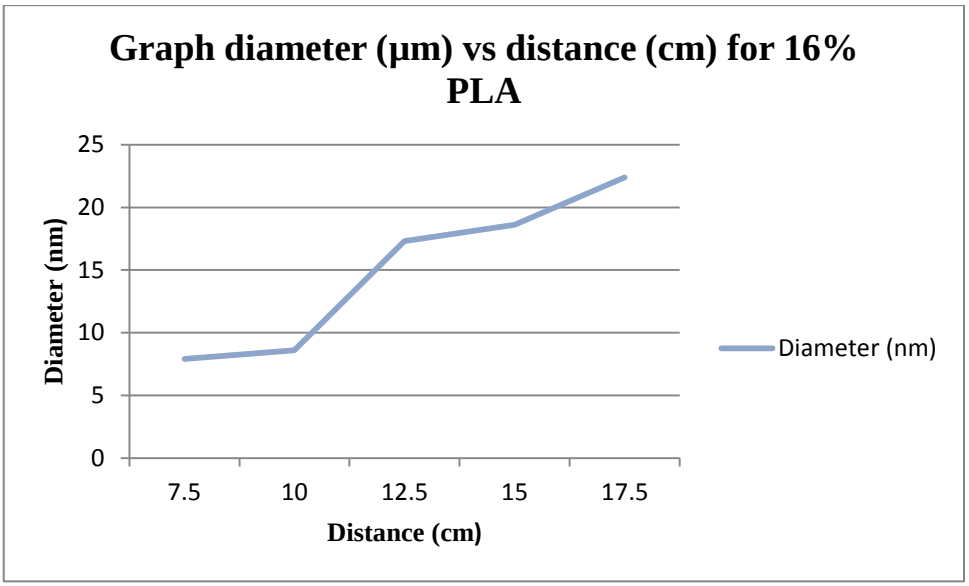


Figure 4. 5: The graph of diameter nanofibres vs electrospinning distance for 16% wt PLA.

Based on the Table 4.10 above, majority of the fibres in both sample (a) and (b) are of 7 to 9 µm diameter strands. For sample (c), the most fibres are in diameter 16 to 19 µm while for sample (d) are 16 to 18 µm. For the sample (e), the most fibres are in range 22 to 25 µm and it can be consider as the largest fibres compared to fibres in other samples. From the micrographs also show the production rate of the nanofibres decreased as the voltage increased. This can be seen through comparison micrograph sample (a) and (e). The amount of nanofibres produced in sample (e) is less than the nanofibres in sample (a).

From the graph in Figure 4.5, the diameter of the nanofibres increased as the electrospinning distance from the tip of nozzle to the collector increased. The diameter of the nanofibres start increased when the electrospinning distance 7.5 cm and continued increased until the distance 17.5 cm. The thinnest nanofibres can be found when the electrospinning distance is 7.5 cm which is the diameter 7.90 µm while the biggest diameter with the diameter 12.38 µm can be found when the electrospinning distance 17.5 cm. This proof that increasing the electrospinning distance will decrease the electric

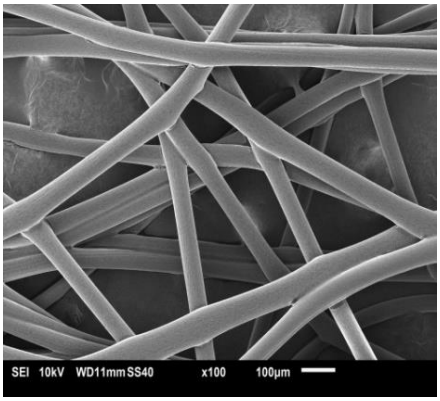
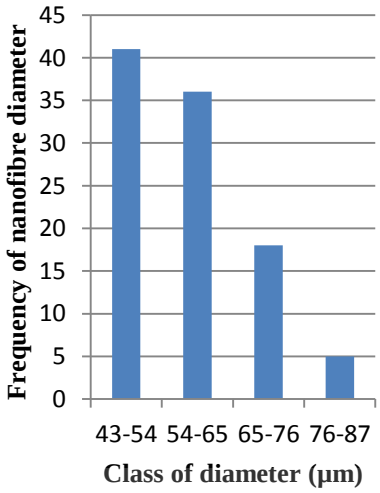
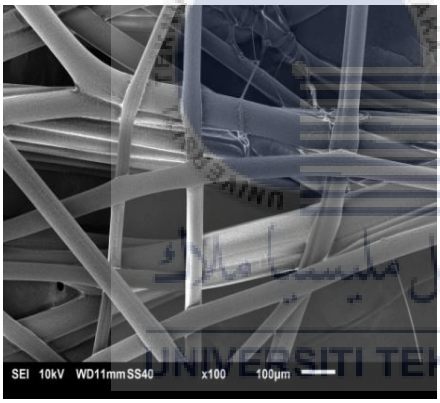
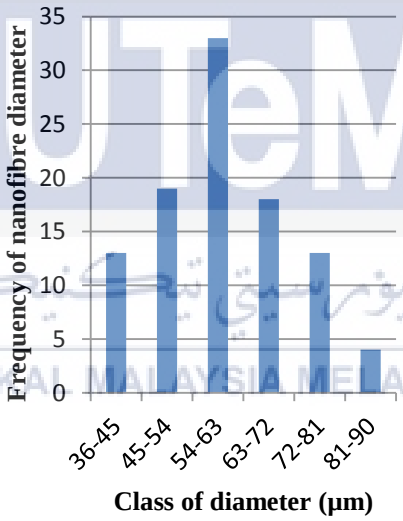
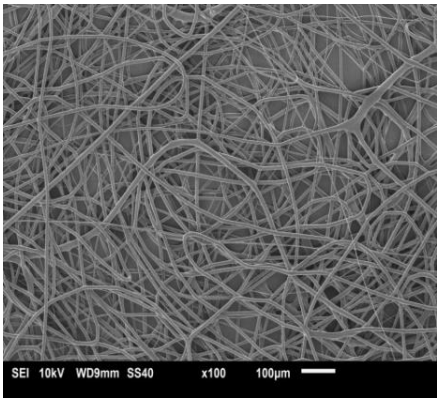
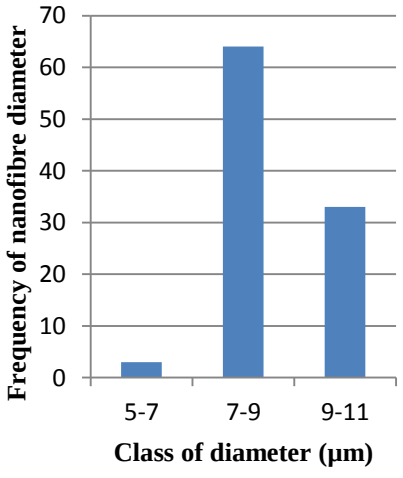
strength as the distance is inversely proportional to the electric strength. Thus, this contributes to the formation of bigger fibres.

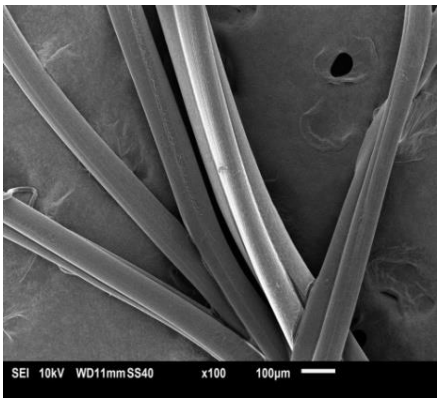
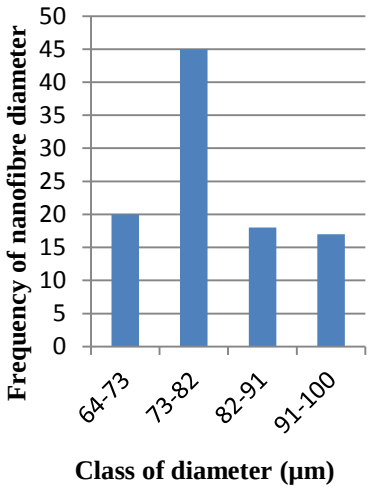
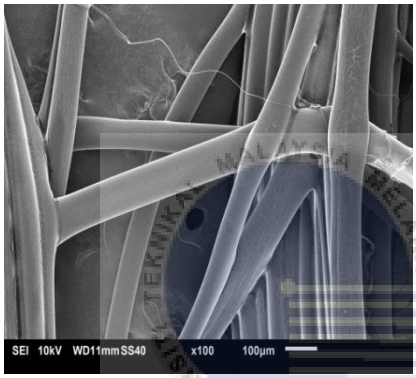
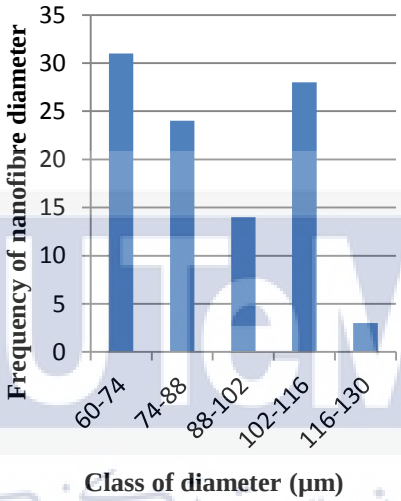
4.3.2 Results for different electrospinning applied voltage experiment.

Table 4. 11: The parameters for different applied electrospinning voltage

Sample	Applied electrospinning voltage (kV)	Distance between the tip of the nozzle to the collector (cm)	Flow rate of the solution (ml/h)
a	10	10	3.000
b	12.5		
c	15		
d	17.5		
e	20		

Table 4. 12: The micrograph, graph of class diameter and mean diameter of PLA nanofibres.

MICROGRAPH OF POLYLACTIC ACID NANOFIBRES	GRAPH OF CLASS DIAMETER	MEAN DIAMETER (µm)
 (a)		58.19±5.8
 (b)		58.92±1.1
 (c)		8.60±0.8

 (d)		80.84±0.8
 (e)		87.59±1.6

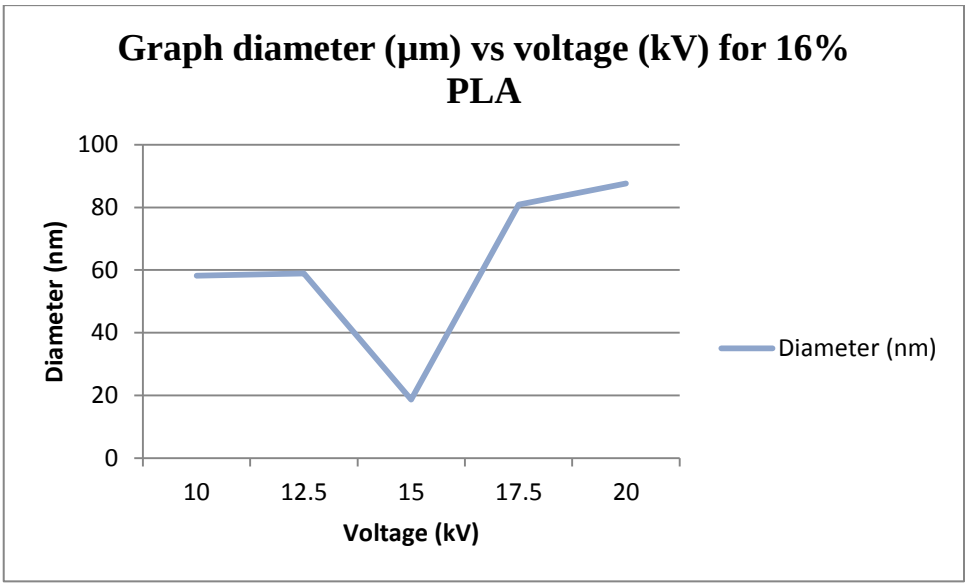


Figure 4. 6: The graph nanofibres diameter vs voltage for 16% wt PLA.

From the Table 4.13, the micrograph (a) showed the homogenous nanofibres with the mean diameter 58.19 µm but the production rate of the nanofibres is less since there were gap between fibre strains. For micrograph sample (b), the diameters of the nanofibres were not consistence and have interconnection between the fibres. By referring the graph in Figure 4.6 above, the sample (c) was the smallest nanofibres with diameter 8.60 µm. It is showed that in sample (c) the nanofibres produced with the optimum and best electrospinning parameters. In addition, there was significant different in the variation of nanofibres diameter for this experiments. While, the biggest nanofibres diameter can be found in 20 kV of applied electrospinning voltage with diameter 87.59 µm. For micrograph sample (d) and (e), the production rate of the nanofibres less as the applied electrospinning voltage increased. The diameter of nanofibres increased from 80.84 µm to 87.59 µm for sample (d) and (e).

From the graph in Figure 4.6, it shown that the diameter of nanofibres increased as the applied electrospinning voltage increased. By applying the high voltage, the electric strength will increase same goes with the acceleration of the nanofibres jet. However since

the distance from the tip of the nozzle to collector was limited in this experiment, the whipping time for the nanofibres travel also limited. Thus, it contributes to the increase of the nanofibres diameter.

Summary, the diameter of the nanofibres increased for both experiments different electrospinning distance from the tip of nozzle to collector and the different electrospinning applied voltage in these experiments. For different electrospinning distance experiment, the diameter increased when extending the electrospinning distance. This result was same as experiment conducted by previous researcher (Doustgani, 2015b). By increasing the electrospinning distance means increasing the whipping time of nanofibres. From the Equation 1, the electric field strength is inversely proportional to the distance. Thus, increasing the electrospinning distance will decrease the electric field strength that generated from power supply. Decreasing of the electric field strength will resulting the decrease of the nanofibres jet acceleration which can contribute to the formation of large nanofibres diameter. However, Viness Pillay and co-workers stated that fibres diameter decreased when extending the elecrospinning distance in their experiment (Pillay *et al.*, 2013). But Megelski and co-workers said that the electrospinning distance does not give significant effect on the nanofibres distance (Megelski *et al.*, 2002). Amir Dougstani explained that the final diameter of fibres will determined when the electrospinning distance balance with the nanofibres jet acceleration. Hence, the fibre diameter may or may not experiences any changes with increasing spinning distance (Doustgani, 2015b).

Very short electrospinning distance from the tip of the capillary to the collector formed the beaded or flattened nanofibres. This is because the nanofibres do not have enough time to evaporate during travel to collector. In other words, the whipping instability which is winding process travels in short time with limited electrospinning distance. This also contributes to the increased of the fibres diameter. In different applied

electrospinning voltage experiment, the fibres diameter increased as the applied electropsinning voltage increased. This proved the limited or short electrospinning distance will decrease whipping time of fibres. Thus, the premature of nanofibres produced. For example, nanofibres sample (a) in Table 4.4 is wet and beaded. By applying the higher voltage also produce greater amount of charge that will cause the nanofibres jet accelerate and move faster. Hence, more fibres produced and deposited at the collector. Applying the high voltage also increased the electrostatic force that will draw much more fibres with large diameter (Doustgani, 2015b).

Table 4. 13: The diameter of nanofibres for 10%, 13% and 16% wt of Polylactic acid.

Concentration of the Polylactic acid (PLA) (wt)	Different electrsopinning distance experiment.		Different applied electrospinning voltage experiment.	
	Lowest diameter (µm)	Highest diameter (µm)	Lowest diameter (µm)	Highest diameter (µm)
10 %	11.21	41.21	5.53	6.95
13%	6.14	18.15	36.15	49.20
16%	7.90	22.38	58.19	87.59

By comparing the concentration 10%, 13% and 16 % wt of Polylactic acid solution from the experiments, the diameter increased as the concentration of the solution increased. From the Table 4.13 above, it clearly showed that the highest concentration of the solution can contribute to the bigger nanofibres formation. For example, the diameter of the nanofibres in different applied electrospinning voltage increased as the concentration increased. The 10%, 13 % and 16% wt of Polylactic acid solution have diameter 5.53 µm, 36.15 µm and 58.19 µm respectively. This proved that the higher concentration of the solution lead to fibre formation with large diameter. This also support by the previous researcher that had gained the same result as this experiment (Pham, Sharma and Mikos,

2006). According to the researcher, the polymer with low concentration has low viscosity that will form beading and droplet formation in electrospinning process. In other words, increasing the solution concentration will produce the beaded free and few junction of fibres. In addition, the high concentration of the polymer solution increased the diameter of the nanofibres. However, the high concentration of solution with high viscosity dried up fast at the tip of the nozzle during the electrospinning process.

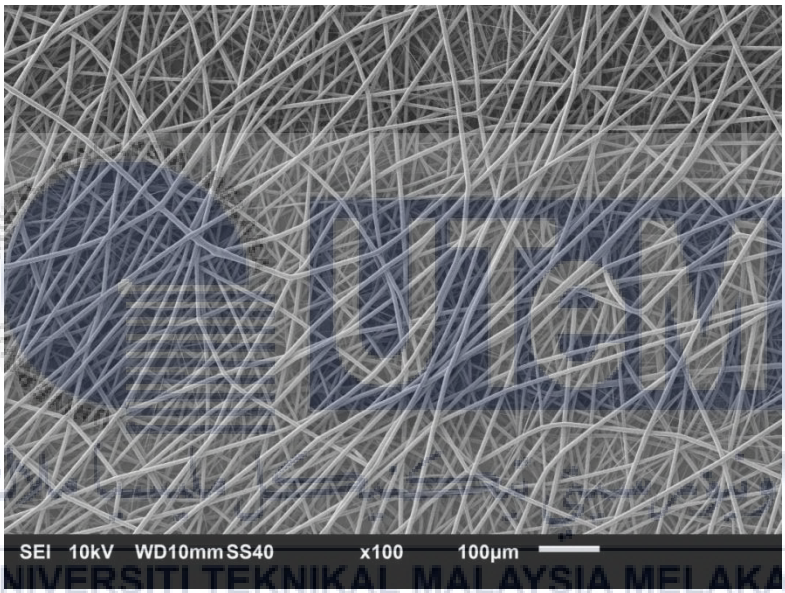


Figure 4. 7: The micrograph of nanofibres with optimum parameters

The Figure 4.7 above shows the micrograph of nanofibres with 100 times of magnification. From this micrograph, the nanofibres were smooth and beads free defect. Plus, the sizes of the nanofibres were homogenous with thin nanofibres diameter 6.76±1.3 µm. This micrograph of nanofibres shows the best parameter achieved. The parameters are as in Table 4.14 below.

Table 4. 14: The optimum parameters of nanofibres.

Sample	Distance (cm)	Voltage (kV)	Flow rate (ml/h)	Concentration (%)
b	10	15	3.000	13

CHAPTER 5

CONCLUSION AND RECOMENDDATION

5.1 Conclusion

The electrospinning process is influenced by several important parameters that can affect the morphology of electrospun nanofibres. The important parameters are applied electrospinning voltage, electrospinning distance, and flow rate of the solution and the concentration of solution. Based on the experiment, the diameter of the nanofibres increased as the electrospinning distance increased and the nanofibres diameter also increased when the applied electrospinning voltage increased.

The best and optimum parameters were obtained in these experiments. From the experiment, it can be concluded the optimum electrospinning applied voltage and distance for Polylactic acid were 15 kV and 10 cm respectively. The smooth, beaded free and homogenous diameters of nanofibres were obtained with optimum parameters. The diameter of the nanofibres was 6.76±1.3 µm.

The concentration of the solution also affects the nanofibres diameter. From the experiment, the high concentration of the solution will produced thick nanofibres through electrospinning process. Means, the diameter of the fibres increased as the concentration of the solution increased for material Polylactic acid. The diameter of the nanofibres for 10% wt, 13% wt and 16% wt of Polylactic acid were 5.53 µm, 36.15 µm, 58.19 µm respectively. The nanofibres diameters above are for different applied electrospinning

voltage experiment. For different electrospinning distance experiment also showed that increasing of nanofibres when polymer solution increased.

5.2 Recommendation

The conductivity is one of the parameter that affect the morphology and behaviour of the electrospun nanofibres. Many researchers claim that conductivity of the polymer solution will increased the charge of nanofibres jet. According to the previous research, by adding the NaCl into polymer solution will increased the conductivity of the polymer solution thus can affect the diameter of the nanofibres. The conductivity also depends on the material of the solution. Further research could be investigates the effect of the conductivity solution on the morphology electrospun nanofibres. The further research also could be undertaken on conductivity of different materials on nanofibres diameter.

Recent years, many researchers test multi-needles in electrospinning instead of single needle. The used of multi needle effect the deterioration of electric field at each tip of needle. The researchers claim that the more required electrospinning voltage to increase the production rate of the nanofibres. For further study, the research could be investigating the different single and multi-needles on production rate of the nanofibres and the optimum voltage to produce the fine and defect free nanofibres.

REFERENCES

Doshi, J. and Reneker, D. H. (1993) „Electrospinning process and applications of electrospun fibers", *Conference Record of the 1993 IEEE Industry Applications Conference Twenty-Eighth IAS Annual Meeting*, 35, pp. 151–160. doi: 10.1109/IAS.1993.299067.

Doustgani, A. (2015a) „Effect of electrospinning process parameters of polycaprolactone and nanohydroxyapatite nanocomposite nanofibers", *Textile Research Journal*, 85(14), pp. 1445–1454. doi: 10.1177/0040517514566109.

Doustgani, A. (2015b) „Optimization of mechanical and structural properties of PVA nanofibers", pp. 1–13. doi: 10.1177/1528083715601511.

Fong, H., Chun, I. and Reneker, D. H. (1999) „<1-s2.0-S0032386199000683-main.pdf>", *Polymer*, 40(16), pp. 4585–4592. doi: 10.1016/S0032-3861(99)00068-3.

Gross, R. A. and Kalra, B. (2002) „Biodegradable Polymers for the Environment", 297(4), pp. 803–808.

Gupta, B., Revagade, N. and Hilborn, J. (2007) „Poly(lactic acid) fiber: An overview", *Progress in Polymer Science (Oxford)*, 32(4), pp. 455–482. doi: 10.1016/j.progpolymsci.2007.01.005.

Haghi, A. K. and Akbari, M. (2007) „Trends in electrospinning of natural nanofibers", *Physica Status Solidi (A) Applications and Materials Science*, 204(6), pp. 1830–1834. doi: 10.1002/pssa.200675301.

Haider, A., Haider, S. and Kang, I. K. (2015) „A comprehensive review summarizing the effect of electrospinning parameters and potential applications of nanofibers in biomedical and biotechnology", *Arabian Journal of Chemistry*. King Saud University. doi: 10.1016/j.arabjc.2015.11.015.

Henton, D. E. *et al.* (2005) „Polylactic Acid Technology: A Review", *Natural Fibers, Biopolymers and Biocomposites*, 48674(23), pp. 527–578. doi: 10.1201/9780203508206.ch16.

Khude, P. (2017) „Nanofibers for High Efficiency Filtration", *Journal of Material Science & Engineering*, 06(06). doi: 10.4172/2169-0022.1000399.

Megelski, S. *et al.* (2002) „Micro- and Nanostructured Surface Morphology on Electrospun Polymer Fibers", pp. 8456–8466. doi: 10.1021/ma020444a.

Oliveira, J. E. *et al.* (2013) „Structural and Morphological Characterization of Micro and Nanofibers Produced by Electrospinning and Solution Blow Spinning : A Comparative Study", 2013.

Pham, Q. P., Sharma, U. and Mikos, A. G. (2006) „Electrospinning of Polymeric Nanofibers for Tissue Engineering Applications: A Review", *Tissue Engineering*, 0(0), p. 060509065116001. doi: 10.1089/ten.2006.12.ft-65.

Pillay, V. *et al.* (2013) „A review of the effect of processing variables on the fabrication of electrospun nanofibers for drug delivery applications", *Journal of Nanomaterials*, 2013. doi: 10.1155/2013/789289.

Reneker, D. H. and Yarin, A. L. (2008) „Electrospinning jets and polymer nanofibers", *Polymer*. Elsevier Ltd, 49(10), pp. 2387–2425. doi: 10.1016/j.polymer.2008.02.002.

Rodoplu, D., Mutlu, M. and Ph, D. (2012) „Effects of Electrospinning Setup and Process

Parameters on Nanofiber Morphology Intended for the Modification of Quartz Crystal Microbalance Surfaces", *Journal of Engineered Fibers and Fabrics*, 7(2), pp. 118–123. doi: 10.1177/155892501200700217.

Tan, S. *et al.* (2005) „Systematic parameter study for ultra-fine fiber fabrication via electrospinning process", 46, pp. 6128–6134. doi: 10.1016/j.polymer.2005.05.068.

„Technology of Nano-Fibers : Production Techniques and Properties - Critical Review" (2018), (May 2017).

Yang, C. *et al.* (2009) „Comparisons of fibers properties between vertical and horizontal type electrospinning systems", *Annual Report - Conference on Electrical Insulation and Dielectric Phenomena, CEIDP*, pp. 204–207. doi: 10.1109/CEIDP.2009.5377758.

Zhou, H., Green, T. B. and Lak, Y. (2006) „The thermal effects on electrospinning of polylactic acid melts", 47, pp. 7497–7505. doi: 10.1016/j.polymer.2006.08.042.

Zhu, J. *et al.* (2017) „Physical characterization of electrospun nanofibers", in *Electrospun Nanofibers*, pp. 207–238. doi: 10.1016/B978-0-08-100907-9.00009-X.