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

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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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TABLE OF CONTENT

Contents

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBERAVATIONS

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.

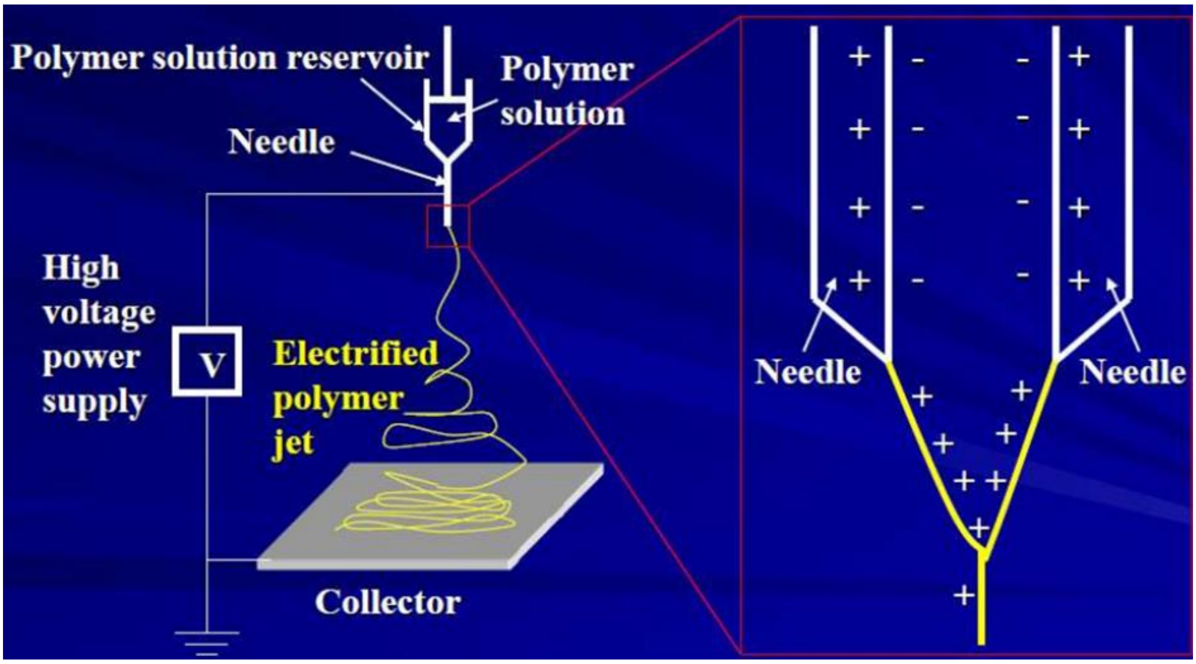

Figure2. 1: Schematic of the vertical setup of electrospinning process.

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.

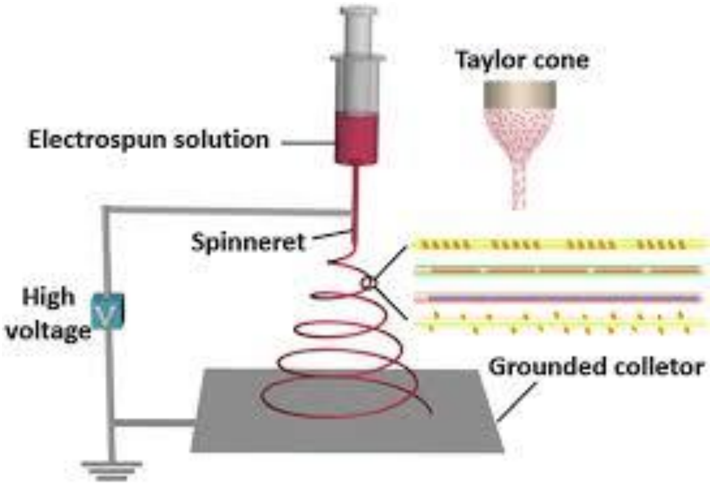

Figure 2. 2: The vertical electrospinning setup.

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