FABRICATION OF UNIAXIALLY ALIGNED ELECTROSPUN NANOFIBRE USING A ROTATING COLLECTOR

MUHAMAD NASRUDDIN BIN AMIR HAMZAH

A report submitted
in fulfillment of the requirements for the degree of
Bachelor of Mechanical Engineering

Faculty of Mechanical Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2018

DECLARATION

I declare that this project report entitled "Fabrication of Uniaxially Aligned Electrospun Nanofibre Using A Rotating Collector" is the result of my own work except as cited in the references.

Signature : ……………………….

Author's Name : Muhamad Nasruddin bin Amir Hamzah

Date : ………………………..

APPROVAL

I hereby declare that I have read this project report and in my opinion this report is sufficient in term of scope and quality for the award of the degree of Bachelor of Mechanical Engineering.

Signature : ……………………….

Name of Supervisor : Dr. Nurfaizey bin Abdul Hamid

Date : ………………………..

DEDICATION

To my beloved mother, father and the loves one.

ABSTRACT

Due to the development of nanofiber in medical application, military equipment and many more, the aligned nanofiber required intensive knowledge and information in electrospinning process and rotating drum as collector of nanofiber. Yet, the normal process of electrospinning using flat plate as collector produced random of nanofiber due to whipping instability that occur when the electrostatic force is induced to the polymeric solution. Thus, formation of aligned nanofiber can be generated by using rotary collector with suitable speed. Therefore, this research aims to obtain the uniaxially aligned electrospun nanofiber by using collector and identify the suitable speed of rotating collector. Polyvinyl Alcohol (PVA) electrospun nanofiber was produced by using electrospinning machine fabricated by UTeM students. The speed of rotating collector was set up at 100, 200, 300, 400, 500 600, 700, 800, 900, 1000, 1100, 1200, 1300, 1400, 1500 and 1600 RPM for 7 minutes with 10 cm distance needle tip with rotating collector and set up feed rate at 0.5 ml/h. The voltage was set at 15kv. Scanning Electron Microscope (SEM) was used to examine the morphology of the fiber. While ImageJ software was used to analyze the characteristic of nanofiber. Alignment of electrospun nanofibers were fabricated but there were still random nanofiber were obtain. This is because the speed of rotating collector need to get alignment approximately 24 m/s but the speed of rotating collector that can that use for this study only can rotate at 8.46 m/s. Generally, to get aligned eelctrospun nanofiber, the speed of rotating collector must be same or higher with speed of whipping instability.

ABSTRAK

Oleh kerana pembangunan seratnano dalam aplikasi peruatan, peralatan ketenteraan dan banyak lagi, seratnano yang sejajar memerlukan pengetahuan dan maklumat yang intensif dalam proses pemintalan elektrik dengan dram yang berputar dalam pengumpulan seratnano. Namun, proses pemintalan elektrik yang biasa digunakan adalah plat rata sebagai pengumpulan serat dan ia menghasilkan serat yang rawak. Ini disebabkan, ketidakstabilan sebat yang berlaku apabila daya electrostatik diinduksi pada larutan polimer. Oleh itu, pembetukan seratnano sejajar boleh dihasilkan dengan menggunakan dram berputar sebagai pemungut serat dengan kadar kelajuan yang sesuai. Objektif kajian ini adalah untuk mendapatkan sejajar seratnano yang telah dipintal oleh elektrik dengan menggunakan pemgungut dram dan mengenal pasti kelajuan pemungut berputar yang sesuai. Polyvinyl Alcohol (PVA) yang telah dipintal, seratnano dihasilkan dengan menggunakan mesin pemintalan elektrik yang direka oleh pelajar UTeM. Kelajuan dram ditetapkan pada 100, 200, 300, 400, 500 600, 700, 800, 900, 1000, 1100, 1200, 1300, 1400, 1500, dan 1600 RPM selama 7 minit dengan 10 cm jarak antara hujung jarum dengan pemungut dram dengan kadar sesuapan di tetapkan pada 05 ml/j. Voltan juga ditetapkan pada 15 kV. Scanning Electron Microscope (SEM) digunakan untuk mengkaji morfologi serat. Perisian ImageJ digunakan untuk menganilisa sifat seratnano. Penjajaran seratnano telah dibuat tetapi masih terdapat seratnano dalam kedudukan secara rawak. Ini kerana kelajuan dram perlu berputar dalam 24 m/s tetapi kelajuan dram berputar yang boleh digunakan untuk kajian ini hanya boleh berputar pada 8.46 m/s. Secara amnya, untuk mendapatkan sejajar seratnano, kelajuan dram berputar mestilah sama atau leih tinggi dengan kelajuan ketidakstabilan serat.

ACKNOWLEGDEMENT

In the name of God, the most kind and merciful, first, I am grateful to Allah S.W.T for blessing me with a good health and well-being, opportunities and courage to complete my research and especially throughout all eight semesters in Universiti Teknikal Malaysia Melaka.

Next, I would like to express my deepest gratitude to my supervisor, Dr. Nurfaizey bin Abdul Hamid for giving me chances and trust to finish this project. Thank you for his time, guidance and willingness to share his ideas and views to make this research a reality. The encouragement and under supervise of Dr. Nurfaizey bin Abdul Hamid, I manage to understand and complete my final year project successfully.

Lastly, my gratitude goes to my lovely families for their encouragement, constant support and emotional understanding throughout my studies here. Their sacrifices and caring towards me are priceless. They are my source of strength to face all the obstacles in finishing this dissertation.

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LIST OF ABBREVIATION

DMF	Dimethylformamide
kV	Kilo Volts
PVA	Polyvinyl Alcohol
Wt.%	Weight Percentage
PVP	Polyvinylpyrrolidine
THF	Tetrahydrofuran

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CHAPTER 1

INTRODUCTION

1.1 Background Study

Nanofibers have a small size of structure and light of weight. The characteristics of nanofiber are enormous specific area, high porosity and small pore size. Nanofiber can create a new product with new properties by using various chemical or physical processes as nanofibers are able to modify the nanofibrous layer. In 1930, electrospinning was first discovered as simple technique making nanofiber from polymer solution with diameter between 50 to 500 nanometers (Katta *et al.*, 2004).. Nowadays, nanofiber have been used as chemical sensors for detecting biological species, in high-performance filtration, as a drug delivery system, and as a scaffold for tissue engineering (Kim and Kim, 2006). There has been globally production and fabrication of nano-size structure for used optical and electrical feedback. Electrospun nanofiber also used in military and civilian in filtration application as the nanofiber has small pore of surface tension.

Electrospinning is a nanofiber production using High Voltage Direct Current (HVDC) from polymer solution. Nanofibers can be produced in many ways, such as electrospinning, melt spinning, solution spinning and gel state spinning (Katta *et al.*, 2004). The advantage of electrospinning process is it has a greater porous structure that could allow drug molecule to disperse out from the matrix efficiently (Taepaiboon, Rungsardthong and Supaphol, no date). Other than that, the high surface area of nanofiber with small pore size are good for certain application.

Alignment of electrospun nanofiber can be determine by using rotating collector that fixed with alternating current (AC) electric field and spinning nozzle that connected to with cylindrical electrode (Lee, Yoon and Kim, 2009). Various frequency can be tested to form an align of nanofiber electrospun such as 20 Hz, 100 Hz, and 500 Hz. Moreover, to produce the alignment nanofiber, the collector speed need to modify or adjust until the aligned structure of nanofiber is obtained. As example, the speed rotating collector can be adjusted from 3.1 m/s to 8.4 m/s (Lee, Yoon and Kim, 2009). This is due whipping instability of polymer that stretch out by rotating collector. There various type of rotating collector was use to generate aligned nanofiber.

The type of collector used will gives different results for nanofiber either is random nanofiber or an align nanofiber Based on a study from (Safaeijavan *et al.*, 2014) shows that the rate of human Adipose-derived Stem Cell (ASC) is higher on aligned nanofiber compare to random nanofiber. ASC is the best cell stem cell-based in biomedical. The comparison between random nanofiber and aligned nanofiber as shown Figure 1.1 and Figure 1.2

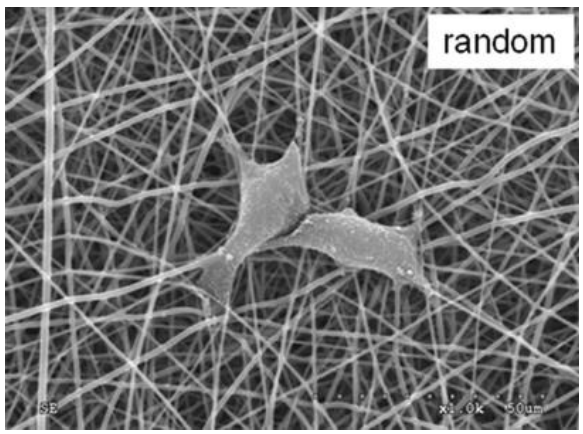

Figure 1.1 : Random nanofiber (Shin *et al.*, 2012)

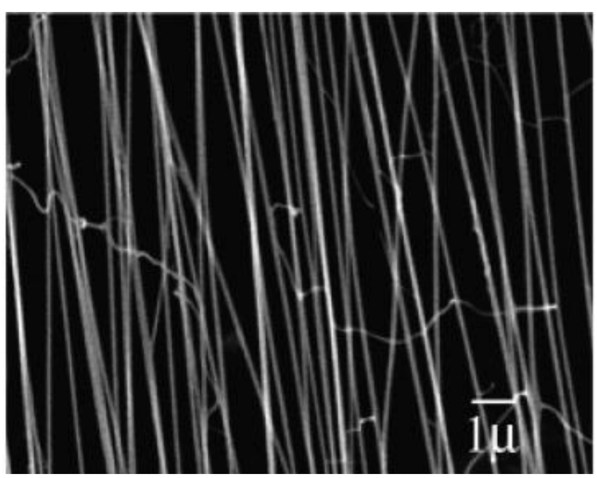

Figure 1.2 : Aligned nanofiber (Katta *et al.*, 2004)

1.2 Problem Statement

The problem that occur in common electrospinning with flat plate collector is formation of random nanofiber. This is because whipping instability cause from electrostatic interaction of the charge. The behavior of whipping instability is unpredictable because the behavior is affected by the viscosity of solution, induced of high voltage power supply and distance between collector. The uses of rotating collector to stretch the electrospun nanofiber once its reach the rotating collector. However, in flat plate collector only obtained random electrospun nanofiber as the collector static in one place. Thus, formation of aligned nanofiber can be generated by using rotating collector but the speed of rotating collector must be equal or more than of speed whipping instability in order to eliminates the accumulation of fiber at the collector. If the speed of the rotating collector is low, the accumulation of fiber at the collector occur and random nanofiber is obtained. Use of aligned nanofiber in engineering application is to hold promising potential as efficient cardio-inductive implants for cardiac tissue (Safaeijavan *et al.*, 2014). In some study, cell growth has shown improving in aligned formation of nanofiber and rapid oxygen exchange to heal wound. In this research will undergo uniaxially aligned nanofiber by using electrospinning process and observe the nanofiber under Scanning Electron Microscopy (SEM). Only the uniaxially aligned nanofiber will be fabricated in this study.

1.3 Objective

The objectives of this project are as follows:

1. To obtain the uniaxially aligned electrospun nanofiber by using a rotating collector.
2. To identify the suitable speed of rotating collector to get aligned nanofibers.

1.4 Scope of Project

The scopes of this project are:

1. To produce aligned polyvinyl alcohol (PVA) electrospun nanofiber by using electrospinning process with rotating collector.

2. To analyse morphology of nanofiber by using Scanning Electron Microscope (SEM)
3. To measure the fiber diameter and degree of oriented of electrospun nanofiber by using ImageJ software

1.5 Report Overview

The study begins with literature review on Chapter 2 that covers the historical background of electrospinning, an introduction to the electrospinning process, electrospinning parameter, application of nanofiber and current development. The basic experimental procedures are then presented in Chapter 3, to describe the process preparation of solution, preparation of electrospinning and analyse of electrospinning. Then, analysis of the result and discussion of the nanofiber was explained in Chapter 4. Finally, implication of the results and future research directions are also presented in Chapter 5.

CHAPTER 2

LITERATURE REVIEW

2.1 History of Electrospinning

The term of electrospinning was taken or derived from electrostatic spinning and the term has rapidly used in around 1994 (Bhardwaj and Kundu, 2010). Electrospinning is a process depend on electrostatic force to produce continues fibre from polymeric solution and polymer melt (Casasola and Raffaella, 2016). Electrohydrodynamic phenomenon (EDH) is one of example of electrospinning, EDH is that the charge an electric field that induces in fluid motion (Collins *et al.*, 2012).

The reactions between fluid motion and electric field were explored and documented the process of electrospray by William Gilbert in 1600 (Tucker *et al.*, 2012). The first patent the process of electrospinning were John Francis Cooley and William James Morton (Casasola and Raffaella, 2016). In 1934, Anthony Formhals was the first pioneer of western world who patent the procedure of electrospinning (Casasola and Raffaella, 2016). Between 1934 to 1944, Formhals issues several of patents that explained procedure of fabrication of polymers filaments using an electrostatic force (Huang *et al.*, 2003). Fabrication of yarn that made from cellulose acetate (CA) electro spun nanofibre using rotating collector that produced by Formhals (Casasola and Raffaella, 2016). Fabrication of yarns by Formhals is using CA with of monomethyl either ethylene glycol and a voltage of 57 KV (Bhardwaj and Kundu, 2010). Movable thread collection machine to collect threads in stretched condition was consist in Formhals'spinning method (Subbiah *et al.*, 2005).

Over 60 years, there were 50 patents for electrospinning polymer melts and solutions had been filed. In 1952, Vonnegut and Neubauer designed simple apparatus for electrical atomization and could produces droplet about 0.1 mm droplets with sources of variable Direct Current (DC) or Alternating Current (AC) voltage. In the early 1964, Taylor produced a study on behaviour of initial fluid droplet (cone) at needle tip when applied high electrical field. The studied shows that the droplet of cone with angle of 49.3º is produce when the surface tension is equilibrium with electrostatic force. Later in 1969, Taylor predict the critical electric potential required to change a cone of conducting fluid into an equilibrated cone by deriving a mathematical model.

In the 1971, Baumgarten produces an apparatus than can electrospun solution of polyacrylonitrile (PAN) into dimethyl formamide (DMF) with diameter range 0.05 – 1.1 μm and used various parameter such as solution viscosity , flow rates and distances to review the effect on nanofiber. The result shown that the viscosity of the solution is affected by the concentration of the solution itself thus it produced bigger nanofiber.

In the 1980, electrospinning method had regained more attention to among researchers as the fibre can be ultrafine fibre or fibrous structures from various polymer solution with diameter down to nanometres (Huang *et al.*, 2003). Over 200 colleges and research institutions all over the world are studying various parameter of electrospinning process and patent the electrospinning process has grown rapidly over years (Bhardwaj and Kundu, 2010). The companies such as eSpin Technologies, NanoTechnics and KATO Tech have close the gap of the advantages that offer from electrospinning while for air filtration product, company from Donaldson Company and Freudenberg has been using over two decades. Figure 2.1 show that increasing of publication of electrospinning over the years between 1967 to 2017.

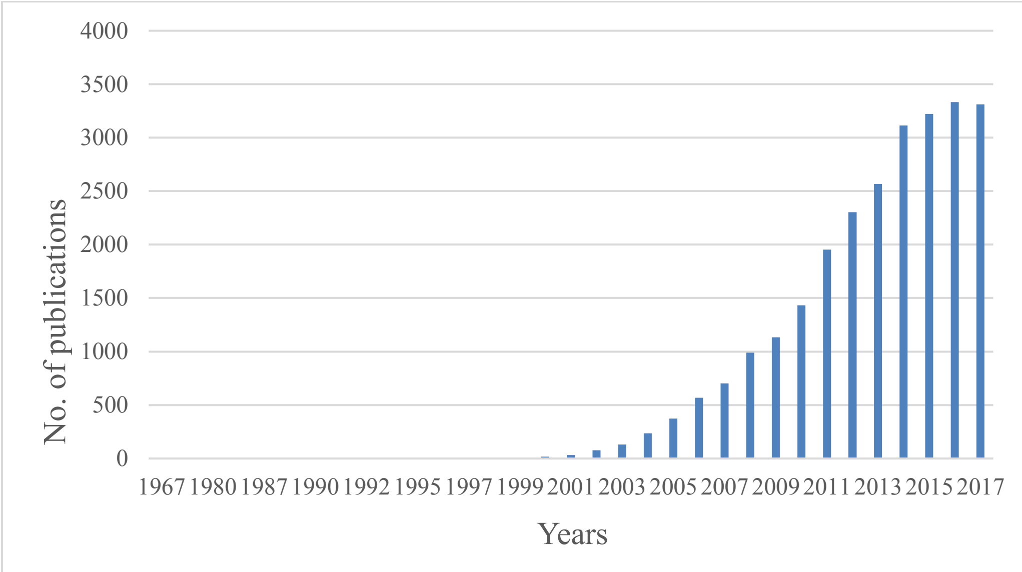

Figure 2.1: Publication of electrospinning over 50 years (Scopus, 19/11/2017. Search using keyword 'electrospinning')

2.2 Process of Electrospinning

Electrospinning is a spinning method that using electrostatic force to form small diameter of fibre (micrometre to nanometre) from a polymeric solution (Bhardwaj and Kundu, 2010). The fundamental setups of electrospinning involve with two electrodes which are source of electrode that connected with high voltage power supply and grounded conductive electrode (Nurfaizey, 2014). Basically, procedures of electrospinning involve with common apparatus consists of high voltage power supply, syringe, needle, syringe pump, conducting collector. The material of syringe can be plastic or metal to store the polymeric solution before inducing with high voltage power supply. The voltage can be in various voltage, between 5 to 39 kV (Casasola and Raffaella, 2016).

There are two types for set up the electrospinning which are horizontal and vertical as in Figure 2.2(a) and 2.2(b). Thus, researchers can fabricate nanofiber in more vigorous and complex way formation of nanofiber because development of electrospinning systems. Usually process of electrospinning take place in close chamber with good ventilation system because some solvent of polymer is hazardous and toxic to human being. Most fabrication of nanofiber are in room temperature. Furthermore, there also two types of set up of feed rate that sell in the market of electrospinning machine which are, constant pressure using gravity and constant flow rate using syringe pump as shown in Figure 2.3(a) and 2.3(b).

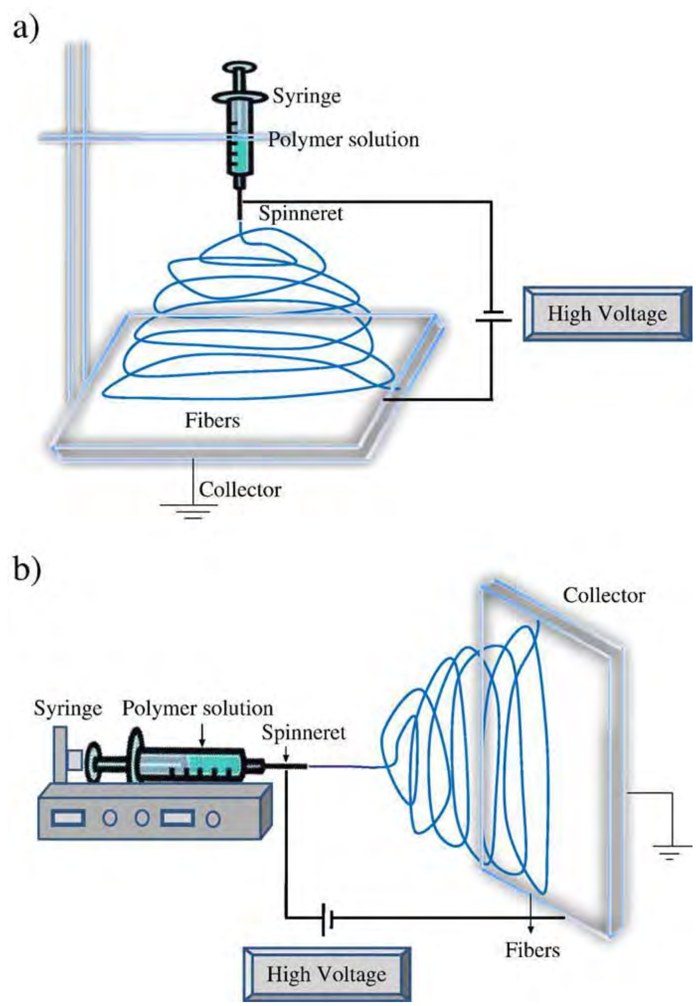

Figure 2.2: Set up of process electrospinning process (a) vertical set up of electrospinning (b) horizontal set up of electrospinning (Bhardwaj and Kundu, 2010)

At the end of spinneret, polymer droplet will form when applying suitable feed rate. Deformation of semi-spherical shape of droplet at the end of spinneret is deform when electrostatic force and molecules have same charges. However, the surface tension will not break up and deform a conical shape as known as Taylor Cone. Inducing a proper voltage will help braking down the surface tensions droplets due to high electrostatic forces. Then, at the summit of conical shape it will accelerating toward collector when the surface breaks up and emit by straight jet polymer as shown in Figure 2.3(a) and 2.3(b) (Nurfaizey, 2014).

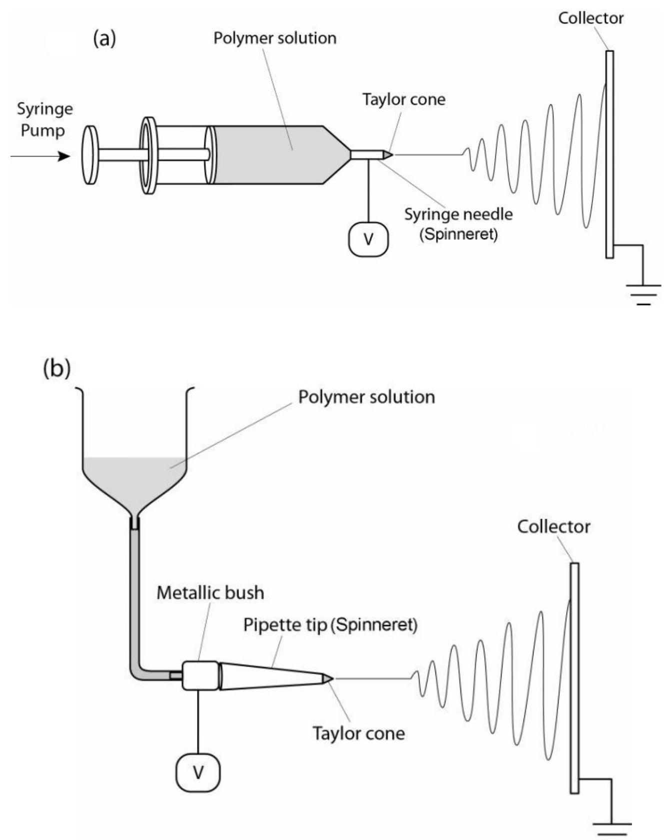

Figure 2.3 :Schematic drawing of feed rate system (a) constant flow rate using syringe pump (b) constant pressure by using gravity (Nurfaizey, 2014)

At the end of spinneret, polymer droplet will form when applying suitable feed rate. Deformation of semi-spherical shape of droplet at the end of spinneret is deform when