FABRICATION AND CHARACTERIZATION OF POLYACRYLONITRILE ELECTROSPUN NANOFIBERS WITH CONDUCTIVE PARTICLES

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DECLARATION

I declare that this project report entitled ―Fabrication and Characterization of Polyacrylonitrile Electrospun Nanofibers with Conductive Particles” is the result of my own work except as cited in the references

Signature : ....
Name : Mhd Alif Bin Mhd Sharif
Date : ....

APPROVAL

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

Signature :…………………….

Supervisor Name : Dr. Nurfaizey Bin Abdul Hamid

Date :……………………..

ABSTRAK

Kajian ini adalah mengenai penghasilan dan pencirian polyacrylonitrile (PAN) dengan zarah konduktif. Kebanyakan kajian tentang pemintalan elektrik nanoserat ini menggunakan asas polimer terkenal seperti pendahulunya PAN untuk menghasilkan nanoserat. Walaubagaimanapun, penambahan zarah konduktif sebagai pengisi dalam polimer boleh meningkatkan sifat-sifat nanoserat untuk membentuk bahan komposit. Dalam kes ini, terdapat dua objektif yang perlu dicapai dalam kajian ini iaitu menghasilkan nanoserat PAN dengan zarah konduktif yang merupakan nanotube karbon berbilang dinding (MWCNT) dan untuk mengkaji ciri-ciri nanoserat yang telah melalui pemintalan elektrik. Tiga ciri utama nanoserat PAN/CNT yang telah disiasat dalam kajian ini adalah sifat-sifat kimia, kekonduksian dan morfologi nanoserat. Pembuatan nanoserat dilakukan dengan menggunakan mesin pemintalan elektrik dengan nilai voltan yang tetap, jarak antara jet jarum dan pengumpul yang tetap, dan kadar aliran yang tetap. Sementara itu, kepekatan MWCNT dalam larutan PAN/CNT mempunyai pelbagai nilai seperti 0.00wt%, 0.03wt% , 0.05wt%, 0.07wt% dan 0.10wt%. Ciri-ciri nanoserat disiasat dengan menggunakan FTIR, empat titik probe dan mesin pemeriksaan mikroskop elektron (SEM). Keputusan menunjukkan bahawa kehadiran partikel konduktif (MWCNT) telah meningkatkan struktur rantaian molekul nanoserat PAN/CNT dalam sifat-sifat kimia. Oleh itu, ia juga telah meningkatkan kekonduksian nanoserat dengan peningkatan kepekatan MWCNT dalam larutan PAN/CNT. Di samping itu, diameter serat PAN/CNT juga meningkat apabila kepekatan MWCNT bertambah manakala diameter serat PAN/CNT menjadi lebih kecil selepas menjalani rawatan haba dalam proses penstabilan dan proses karbonisasi.

ABSTRACT

This study is about the fabrication and characterization of polyacrylonitrile (PAN) with conductive particles. Most of the research about the electrospinning of nanofibers were using basic well-known polymer such as PAN precursor to fabricate nanofibers. In fact, the addition of conductive particles as filler in the polymer may enhance the properties of the electrospun nanofibers to form composite fibers. In this case, there were two objectives needed to be achieved in this study which are to fabricate the electrospun nanofibers of PAN with conductive particle which was multi-walled carbon nanotube (MWCNT) and to investigate the characteristics of the electrospun nanofibers. The three main characteristics of the PAN/CNT electrospun nanofibers that have been investigated in this study were the chemical characterization, the conductivity and the morphology of the nanofibers. The fabrication of the nanofibers were done by utilizing electrospinning machine with the constant value of applied voltage, distance between needle jet and collector, and flow rate while the concentration of MWCNT in PAN/CNT solution was varied which were 0.00wt%, 0.03wt%, 0.05wt%,0.07wt% and 0.10wt%. The characteristics of the nanofibers were investigated by using FTIR, four point probe and scanning electron microscope (SEM) machine. The results showed that the presence of conductive particles (MWCNT) had improved the molecule chain structure of PAN/CNT nanofibers in chemical characterization. Hence, it also had enhanced the conductivity of nanofibers with the increased in the concentration of MWCNT in PAN/CNT. Furthermore, the fiber diameter of PAN/CNT nanofibers was increased as the concentration of MWCNT increased while the fiber diameter of PAN/CNT became smaller as undergo heat treatment in stabilization and carbonization process.

DEDICATION

To my beloved father,

Mhd Sharif Bin Omar,

My beloved mother,

Hayati Bt Mat.

ACKNOWLEDGEMENTS

In the name of ALLAH, the most gracious, the most merciful, with the highest praise have given me the opportunity to complete this final year project successfully without any difficulties.

I am deeply indebted to my supervisor, Dr. Nurfaizey Bin Abdul Hamid for his kind supervision, great advice, meaningful guidance and helpful support as well as exposing me with valuable experiences throughout this entire final year project.

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LIST OF ABBREVIATIONS

AMCHALS	Advance Material Characterization Laboratory Center
CNF	Carbon nanofiber
CNT	Carbon nanotube
DMF	N,N-Dimethylformamide
DNA	Deoxyribonucleic acid
ECM	Extracellular matrix
HEPA	High efficiency particular air
MW	Molecular weight
MWCNT	Multi-walled carbon nanotube
PAN	Polyacrylonitrile
FTIR	Fourier Transform Infrared Spectroscopy
SEM	Scanning Electron Microscope
SNF	Stabilized nanofiber
UTeM	Universiti Teknikal Malaysia Melaka
3D	Three-dimensional

LIST OF SYMBOLS

Ω/sq	=	Resistance per square
wt%	=	Weight percentage
σ	=	Conductivity
°C	=	Celsius
A	=	Ampere
cm	=	centimeter
nm	=	nanometer
µm	=	micrometer
g	=	gram

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

Nanotechnology is the formation of useful materials, devices and systems through control of matter on the nanometer length scale and exploitation of novel phenomena and properties (physical, chemical, biological) at the length scale. Last few decades, the evolution of nanofibers has attracted significant interest due to its extraordinary properties especially on their highly surface area-to-volume ratio (Peresin *et al.*, 2010). Consequently, this characteristic of nanofibers has widely use applications in pharmaceutical, medical, catalysis field, biosensors lithium ion batteries and many more. A nanofiber is a fiber with a diameter of 100 nanometers or less as shown in Figure 1.1.

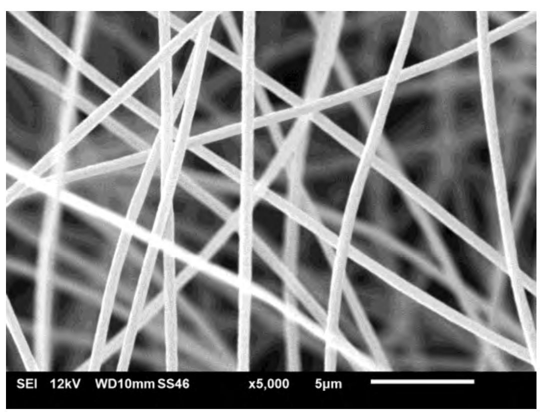

Figure 1.1: SEM Image of electrospun nanofibers

. Electrospun nanofibers of polymer polyacrylonitrile (PAN) are a new material of polymer that can be used for applications consists of filtration, medical, personal care, barrier, composites and energy storage. Electrospun nanofibers of PAN have characteristic of surface free energy which based on its special properties that has bigger surface area per unit mass (Wu *et al.*, 2002). The special characteristic of PAN nanofibers make it suitable for various applications such as in aerospace, drug delivery system, information technology and many more (Lau and Hui, 2002; Hsiao, Alms and Advani, 2003).

Composites nanofibers can be formed by using many kinds of additives and filler in electrospinning of nanofibers (Heikkilä and Harlin, 2009). Heikkila & Harlin stated that by using nano-size fillers in electrospinning nanofibers, the properties of electrospinning fibers can be modified. Well-known filler that are widely used in electrospinning of nanofibers is carbon nanotubes (CNTs) (Qin, 2008). CNT act as a reinforcement component in electrospinning of nanofibers and it also used in modification of electrical properties of electrospun nanofibers (Jeong *et al.*, 2006; Heikkilä and Harlin, 2009).

1.2 PROBLEM STATEMENT

Most of the research about the electrospinning of nanofibers were using basic well-known polymer such as PAN, PVA and many more polymer solution to fabricate nanofibers. PAN precursor has been widely used for the fabrication of nanofiber as it exhibit high conductivity for carbon nanofiber (CNF) especially in electronic applications. However, the addition of conductive filler can enhance the characteristics of electrospun nanofibers. The use of conductive particles as filler in the polymer solution may result in different properties of the solution such as it can be a charge carrier. Charge carrier or conductive filler particles in the solution can improve the conductivity of the solution and the charge density of the jet. The increase of the solution conductivity and the net charge density can enhance the instability of the jet that can lead to the formation of bead-free electrospun nanofibers and nanofibers with smaller diameter. Heikkilä & Harlin, 2009; Jeong et al., 2006 stated that basically, conductive particles such as carbon nanotubes (CNTs) as filler in polymer solution can serve as a reinforcement component and also used as a modification in the electrical properties of the electrospun nanofibers.

1.3 OBJECTIVE

The objectives of this study are:

1.3.1 To fabricate a new electrospun nanofibers by combining polyacrylonitrile (PAN) and conductive particles with various concentrations.

1.3.2 To investigate the characteristics of the electrospun nanofibers.

1.4 SCOPE OF PROJECT

The scopes of this project are concerns on fabricating electrospun nanofibers by using a mixture of polymer between polyacrylonitrile (PAN) with conductive particles. Multi-Wall Carbon nanotube (MWCNT) is the conductive particle that has been chosen as filler. The concentration of MWCNT will be varied while the process parameter such as applied voltage, distance between needle tip to the collector and flow rate will be fixed. This is due to examine the characteristics of PAN/CNT with different concentrations of MWCNT.

1.5 REPORT OVERVIEW

The structure of this report is outlined as follow:

Chapter 2 presents about the literature review that covers the historical of electrospinning process along with the system of electrospinning machine. Moreover, the effect of parameter toward the electrospun nanofibers also will be discussed, the application of electrospun nanofibers and lastly about the current development of electrospun nanofibers.

Chapter 3 gives a review of the experiment methodology consisting of preparation of the polymer solution, fabrication of electrospun nanofibers, process of thermal treatment and characterization of the electrospun nanofibers.

Chapter 4 presents the description on the result that have been obtained and discussions about the results.

Chapter 5 presents the conclusions along with the recommendations for future works.

CHAPTER 2

LITERATURE REVIEW

2.1 History of Electrospinning Process

Electrospinning is a unique technique that uses electrostatic force to produce fibers from a polymer solution. The history of electrospinning process started from William Gilbert who was the first observed the electrostatic attraction of liquid in 1600.

In 1934, Anton Formhals had managed to use the electric charge to spin small diameter of synthetic fiber. However, some improvements was needed on Formhals works due to the distance between the collector and the polymer solution was close that may causes the polymer solution not fully evaporate when the fiber stick to the collector. Then, Formhals had works on the second patent which the distance between the polymer solution and the collector was more greater (Sill & von Recum, 2008). Figure 2.1 shows some of Formhal's drawings that he inserted in his patent.

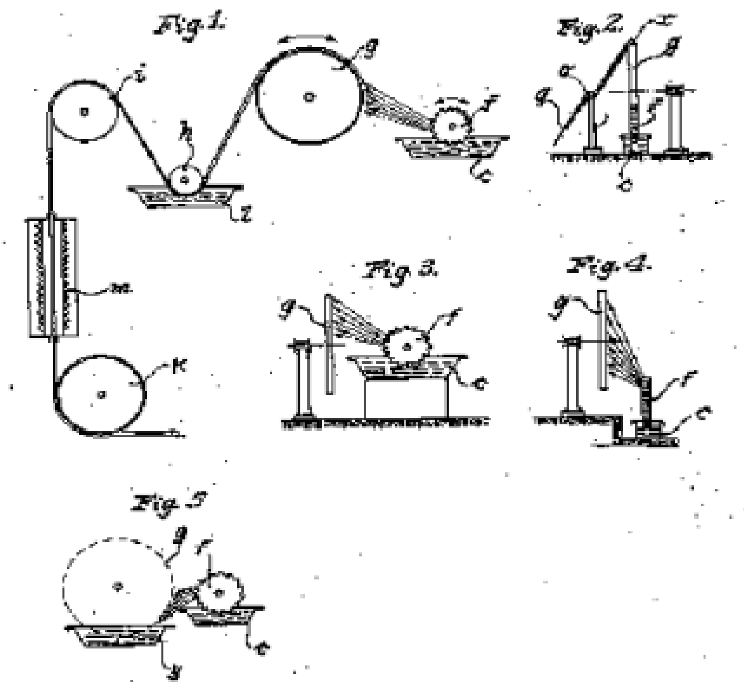

Figure 2.1: Some of schematic drawings by Formhals in his patent (Source: Vrieze & Clerck, 2009)

The electrospinning of nanofibers technology appears to be inactive after the revolution of Formhals until Simons design an apparatus that prove the feasibility of fabrication nanofiber. Simon shows that by using electrospinning process, ultrafine fiber will be produced. He also shows that lower viscous solution will produce shorter fiber but has a finer fiber while higher viscous solution will produce longer fiber but the fiber is thicker (Hwan, 2014).

There was another person has developed Formhals work on electrospinning at the same time as Simon. It was Taylor who had done the research about the jet forming process. In 1964, Taylor recommended that under an electric field at semi-vertical angle of 49.3° the equilibrium charged conducting fluid can exist in the form of cone (Nurfaizey *et al.*, 2014). This phenomenon of cone later became known as the Taylor Cone. Taylor also found out that when the fiber jet is ejected from the tip of the cone, it can produce fiber with smaller diameter (Sill & von Recum, 2008) as shown in Figure 2.2.

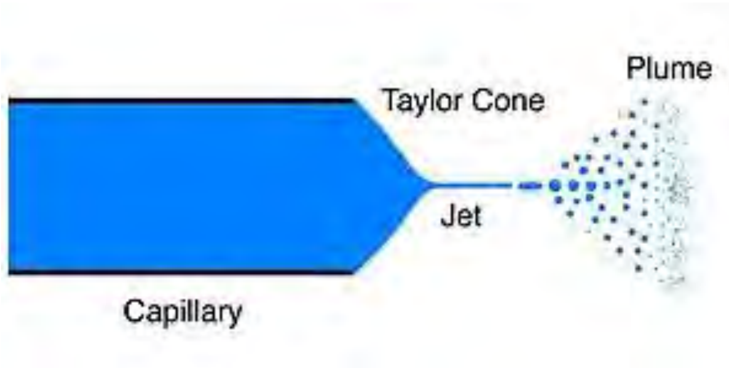

Figure 2.2: Taylor Cone formation at the tip of the needle (Source: en.wikipedia.org)

The research of electrospinning was continuing after Taylor and Simon by Baumgarten in 1971. Baumgarten had made substantial evidence about the effect of voltage applied to the solution as the fluid stream will be form instead of droplets (Hwan, 2014) as shown in Figure 2.3.

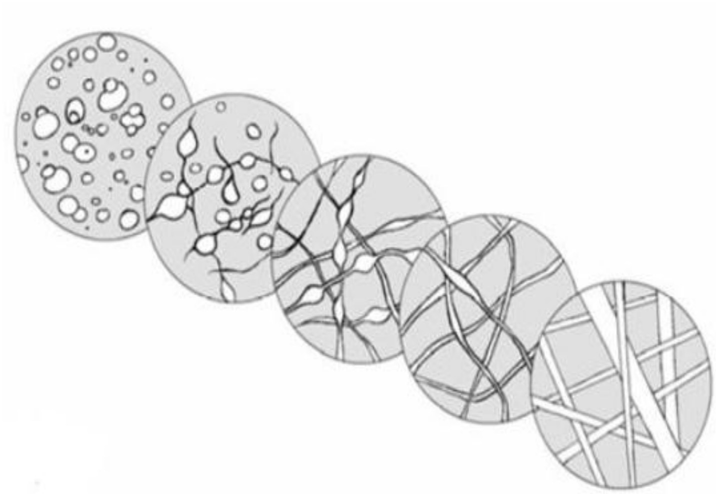

Figure 2.3: Formation from droplet to fluid stream (Source: Hwan, 2014)

Since then, many researches paper and patent have been done about the fabrication of electrospun nanofibers with various kind of polymer solution. The nanotechnology of fabricating nanofibers has grown widely from year to years.

2.2 Electrospinning Machine

Electrospinning machine is an effectual technology machine to produce fibrous membrane with nanometers unit of fiber diameter. Electrospinning machine is a fairly direct process to be setup. Han, Boyce and Steckl (2011) stated that there are three advantages of electrospinning machine system. An electrospinning machine has the ability to control:

1) Micrometer of fiber diameter to a nanometer dimensions.

2) Various fiber compositions.

3) Multiple fibers with the spatial alignment.

2.2.1 Basic component of electrospinning machine

The electrospinning machine consists of four crucial components which is source of electric field, syringe with a nozzle, collector and a pump. The polymer solution that will be electrospun is applied to the system via the nozzle of the syringe. Then, it is pushed by the pump. Next, a high electrical voltage present between the nozzle and the counter electrode will be subjected to the polymer. The source generates the high electrical voltage that causes a cone-shaped deformation of the drop of polymer solution. On its way to the collector, the solvent in the solution will evaporates.

2.2.2 Type of electrospinning machine

There are three types of electrospinning machines which are shaft type, converse type and horizontal type. Shaft type and converse type are included in the category of vertical type of electrospinning machine. The differences between these three types are based on its geometrical arrangement of ejecting capillary polymer solution and the collector. The ejecting capillary for the shaft type will be on the top while the collector will be on the bottom as shown in Figure 2.4. For the converse type, the ejecting capillary position will be on the bottom and facing the top while its collector position will be on the top as shown in Figure 2.5. Next, the horizontal type is more different from the other two types which are converse and shaft type. For the horizontal type the ejecting capillary will be horizontally parallel same position as the collector as shown in Figure 2.6.

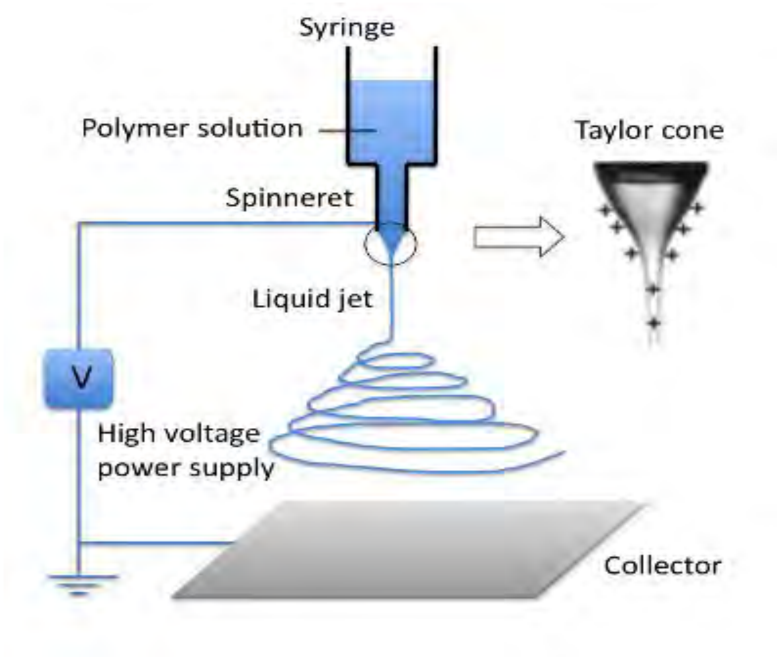

Figure 2.4: Illustration of electrospinning system of shaft type (Source: Athira, Sanpui, & Chatterjee, 2014)

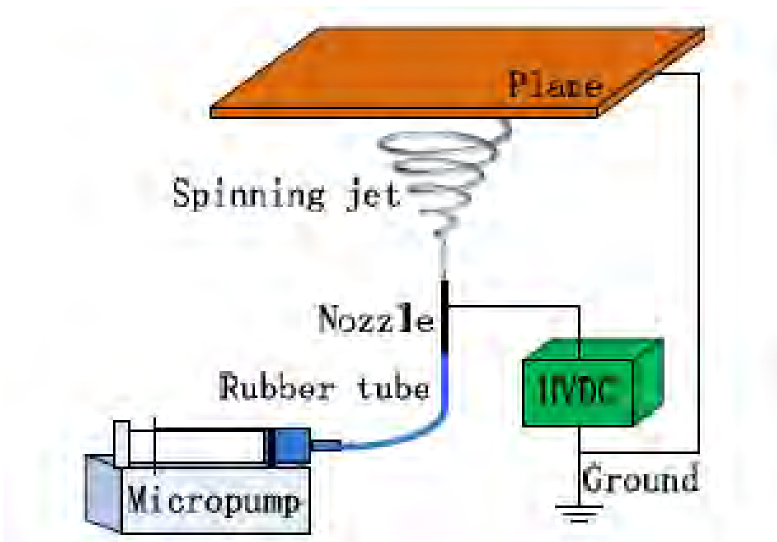

Figure 2.5: Illustration of electrospinning system of converse type (Source: C. Yang et al., 2009)