FABRICATION AND CHARACTERIZATION OF CARBONIZED ELECTROSPUN NANOFIBRES FROM POLYACRYLONITRILE AND POLYVINYL ALCOHOL PRECURSORS

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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

Faculty of Mechanical Engineering

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DECLARATION

I declare that this project report entitled “Fabrication and Characterization of Carbonized Electrospun Nanofibres from Polyacrylonitrile and Polyvinyl Alcohol Precursors” is the result of my own work except as cited in the references

Signature : ……..

Author‟s Name : AHMAD SYAHMI BIN AHMAD ZAINUDDIN

Date : ……..

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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 award of the degree of Bachelor of Mechanical Engineering (Structure & Materials).

Signature : ……..

Name of supervisor : ……

Date : ……..

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SUPERVISOR'S DECLARATION

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

Signature : ……..

Name of supervisor : ……

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ABSTRACT

Electrospinning is a simple and versatile technique for producing polymeric nanofibres. The technique has also been used to produce nanoscale carbon fibres through post heat treatment of precursor materials. However, a notable gap exists in carbon nanofibre production especially in understanding the effects of processing method on the properties of the carbon nanofibres. In this study, carbon nanofibres were produced carbon nanofibre from polyacrylonitrile (PAN) and polyvinyl alcohol (PVA) precursors. The main objectives of this study were to produce carbon nanofibres using electrospinning technique and to characterize the fibres based on physical, chemical and electrical properties of the nanofibres. . PAN solution was prepared by mixing PAN in N,N-dimethylformamide (DMF) and PVA solution was prepared by dissolving PVA in distilled water. The as-spun PVA nanofibres were first underwent iodination process to improve its thermal stability before heat treatment process. Both PAN and PVA nanofibre precursors were stabilized at 240°C with a ramping time of 1°C/min in an oxidative environment. Then, the nanofibres were carbonized at 1000°C in a nitrogen filled furnace. Different samples were produced at different heating rates of 5°C/min and 3°C/min for comparison. Finally, characterization of the carbon nanofibres was carried out using scanning electron microscope (SEM), four-point probe and Fourier transform infrared spectroscopy (FTIR). The results suggest that PAN precursor produce good carbon nanofibres characteristics. A flat line on FTIR spectra shows fully elimination of non-carbon element in PAN nanofibre. Carbon nanofibre produced has higher electrical conductivity with lower heating rate of carbonization. Diameter of PAN carbon nanofibre produce decreased after carbonization process. Carbonization of PVA produces unreliable carbon nanofibre characteristics due to higher temperature used. Characteristics of PVA carbon nanofibre undetected due to melting form of nanofibres. In short, both carbon nanofibre were successfully fabricated and characterized which achieve the main objective. Both precursor in producing carbon nanofibre need to used more effective method, parameters and proper machine to enhance accuracy of results.

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ABSTRAK

Electrospinning adalah teknik yang mudah dan serba boleh untuk menghasilkan polimer nanofibres. Teknik ini juga telah digunakan untuk menghasilkan serat karbon skala nano melalui rawatan haba bahan prekursor. Walau bagaimanapun, jurang yang ketara wujud dalam pengeluaran karbon nanofibre terutamanya dalam memahami kesan kaedah pemprosesan pada sifat-sifat karbon nanofibres. Dalam kajian ini, karbon nanofibres dihasilkan daripada pelopor polyacrylonitrile (PAN) dan polivinil alkohol (PVA). Objektif utama kajian ini adalah untuk menghasilkan karbon nanofibres menggunakan teknik elektrospinning dan mencirikan serat berdasarkan sifat fizikal, kimia dan elektrik nanofibres. . Larutan PAN disediakan dengan pencampuran PAN dalam N, N-dimetilformamide (DMF) dan larutan PVA disediakan dengan melarutkan PVA dalam air sulingan. Nanofibres PVA menjalani proses penyiaran pada awalnya untuk meningkatkan kestabilan haba sebelum proses rawatan haba. Kedua-dua pelopor PAN dan nanofibre PVA distabilkan pada 240°C dengan kadar pemanasan 1°C/min dalam persekitaran oksidatif. Kemudian, nanofibres dipanaskan pada suhu 1000°C dalam relau yang dipenuhi nitrogen. Sampel yang berbeza dihasilkan pada kadar pemanasan yang berbeza iaitu 5°C/min dan 3°C/min untuk perbandingan. Akhir sekali, pencirian karbon nanofibres dilakukan menggunakan mikroskop pengimbasan elektron (SEM), fourt point probe dan Fourier transform spectroscopy infrared (FTIR). Hasil pencirian mencadangkan bahawa pendahulu PAN menghasilkan ciri-ciri karbon nanofibres yang baik. Garis rata pada spektrum FTIR menunjukkan penghapusan elemen selain daripada karbon sepenuhnya dalam PAN nanofibre. Karbon nanofibre yang dihasilkan mempunyai kekonduksian elektrik yang lebih tinggi dengan kadar pemanasan karbonisasi yang lebih rendah. Diameter karbon nanofibre PAN menurun selepas proses karbonisasi. Karbonisasi PVA menghasilkan ciri-ciri karbon nanofibre yang tidak boleh dipercayai kerana suhu yang lebih tinggi digunakan. Ciri-ciri karbon nanofibre PVA tidak dapat dikesan oleh kerana bentuk nanofibres. Secara ringkasnya, kedua-dua karbon nanofibre berjaya dihasilkan dan dicirikan untuk mencapai matlamat utama. Kedua-dua pendahulu dalam menghasilkan nanofibre karbon perlu menggunakan kaedah yang lebih berkesan, parameter dan mesin yang betul untuk meningkatkan ketepatan keputusan.

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LIST OF ABBEREVATIONS

AC	Alternative Current
AMCHALS	Advance Material Characterization Laboratory
CNF	Carbon Nanofibre
DC	Direct Current 12
DMAc	Dimethylacetamide
DMF	Dimethylformamide
FESEM	Field Emission Scanning Electron Microscope
FTIR	Fourier Transform Infrared Spectroscopy
I-PVA	Iodinated Polyvinyl Alcohol
IR	Infrared
NF	Nanofibre
PA-6	Polyamide-6
PAN	Polyacrylonitrile
PEO	Polyethylene Oxide
PSF	Polysulfone
PVA	Polyvinyl Alcohol
SEM	Scanning Electron Microscope
SNF	Stabilized Nanofibre
SPE	Solid Phase Extraction

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Nanotechnology is a study with expansion of evolution for materials at nano-levels which about 100 nanometres (Subbiah, Bhat, Tock, Parameswaran, & Ramkumar, 2005). This technology is an appliance of study with an application on extremely undersized things and can be used for various fields of engineering and sciences. Nano-scale material involves on a capability to see small atoms or molecules especially in nanofibre. With capability to create nanofibre materials and sharpened quality, it can be importance for worldwide usage (Subbiah et al., 2005).

One of the favoured processes to create nanofibre is through electrospinning. Today research of the used of electrospinning in creating electrospun nanofibres have extensively known all over the world. Researcher agreed that electrospinning is a simple, versatile but useful in producing a nano-scale fibres meshes (Bin Mohamat, 2013). They also agreed that it is an ideal technique producing particles of material with biological nano-size scale. Electrospinning system subsist of three dominant basic parts which are a high voltage power supply, a spinneret and a spin collecting plate (Bin Mohamat, 2013).

Researchers used a device identical to Figure 1 with adjustment depending on their purpose (Subbiah et al., 2005). In general, the electrospinning process operated with a polymer solution being applied to high voltage sources through specific electrode, thereby charging the polymer solution. The charged ions applied to the electrode which immersed in the polymer solution. The polymer solution will be forced out from storage tube via a syringe pump to form a small drop of polymer at the tip of the capillary. Then, polymer being eject from the tip of cone with nano-scale polymer. The jet go through instable bending motion and spinning collector will collect the polymer (Raghavan, 2006).

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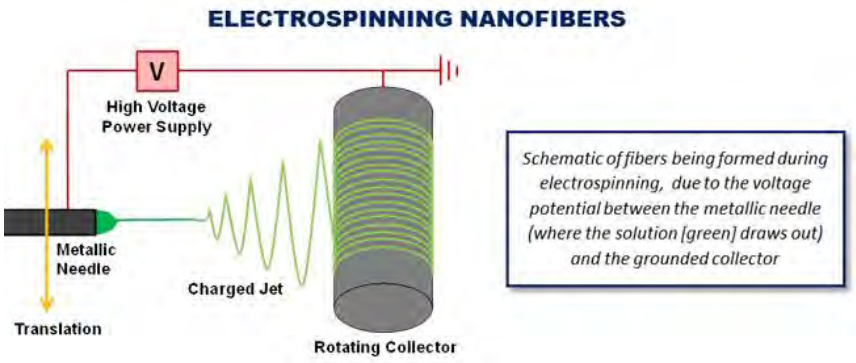

Figure 1: Schematic of electrospinning nanofibres being formed (Subbiah et al., 2005)

Electrospun nanofibres have self-assembly which is referred to manufacturing technique here small particle or molecules are added together to assemble a complete nanofibre (Rasel & Rizvi, 2015). It has features such as high porosity, large surface area and sectional interconnectivity of electrospun nanofibres (Rasel & Rizvi, 2015; Wang, Ding, & Li, 2013) .With that specific features, nanofibres are well appropriate for cell communication (Wang et al., 2013). Polymer fibres that produced by electrospinning has a diameter ranges from nanometre to micrometres where it is the main reason to be used for many applications (Rasel & Rizvi, 2015). It is also a material that being uses as a drug delivery, wound dressing, sensors, fuel cell, catalysis, filtration and tissue scaffolds (Bonino et al., 2011). However, synthetic polymer does not applicable for engineering of tissue.

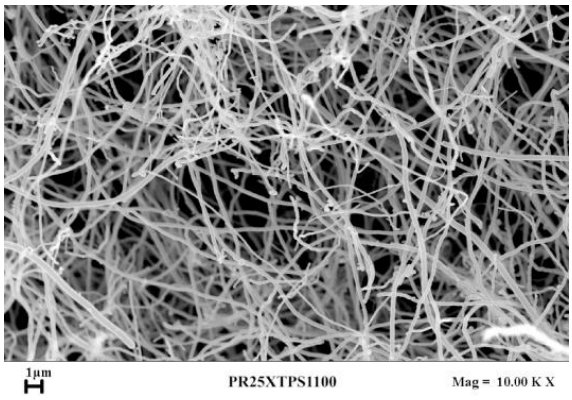

Figure 2: SEM image of nanofibres (Rasel & Rizvi, 2015)

Nanofibres size and structures can be controlled by many aspects. By customize the fibre collection, a 3D complex structure can be developed (Rasel & Rizvi, 2015). Nanofibres

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that produce by single jet needle have a different structure compared to multiple jets (Dosunmu, Chase, Kataphinan, & Reneker, 2006). Based on specific polymer, solvent and operation conditions, electrospun nanofibres have a differ diameters less than 100 mm to greater 1000 mm as shown in Figure 2. In a typical situation, electrospun nanofibres are delivered on the collector as a no specific orientation fibres (Xie, MacEwan, Schwartz, & Xia, 2010). In this cases, spinning collector are invented to simplify the distribution of nanofibres. With a precise rotation speed of collector, fibres align is more properly.

Almost all soluble polymers can be used for this method (Rasel & Rizvi, 2015). Polyvinyl alcohol (PVA) is used as a host material of electrospun nanofibres. It composed with graphitized conical platelets (Shehata, Madi, Al-maadeed, Hassounah, & Ashraf, 2015). To produce better electrospun nanofibres, polyvinyl alcohol (PVA) had to be dissolved in deionized water (Shehata et al., 2015). Others polymer like polyacrylonitrile (PAN) also uses electrospinning process for widely usage based on its capability such as hot gas filtration system or reinforced concrete. Both of these polymers will be carbonised to enhance their characteristics in producing carbon nanofibres.

Carbon nanofibres act as essential industrial materials for technology development which fascinated researchers. Carbon nanofibres are broadly used for various applications due to their unique properties including finest electrical and thermal conductivities (Zhou et al., 2009). Carbon nanofibres material is suggested as contender for nano composite manufactures for their ability to withstand squeezing without crack and twisting exaggerations. Either by pyrolyzing nanofibre from precursor such as polyacrylonitrile (PAN) or deposition of chemical vapour, carbon nanofibres can be produced (Zussman et al., 2005). Isotropic and phenolic resins also being recognized as carbon nanofibres precursors (Inagaki, Yang, & Kang, 2012). Chemical vapour deposition (CVD) method is rarely used due to high cost and limited capabilities of producing align nanofibres (Zhou et al., 2009). Carbon nanofibre advantages can be enhanced with proper technique and purposes.

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1.2 PROBLEM STATEMENT

In recent years, tremendous interest in nanofibre materials, development and improvement on electrospinning has increased dramatically. Especially on an innovative and contemporary carbonization process of carbon precursor nanofibre creates energy saving manner carbon which highly porous and have a high surface area. In general, carbon is a conductive material which is very helpful in electronic background while polymer is an insulator. Issues on converting insulator into electrical conductor get a lot of attention by previous researcher. Despite the previous research on nanofibres, producing carbon nanofibre from polymer of polyacrylonitrile (PAN) and polyvinyl alcohol (PVA) precursor is still a challenge. This is due to various suitable method and parameters that needed to produce carbon nanofibres.

The unique characteristics of carbon nanofibre precursor offer the promise of widespread adoption in numerous nanotechnologies area including sensors, fuel cell, catalysis and filtration. Previous researcher has issues different parameters result different analysis on carbon nanofibres. Depending on the application, issues arise from processing heating rate limitations for polymer which affect the characteristic of nanofibre, as well as handling technique and parameters. In fact, there still exists a notable gap which hinders the profile of carbon nanofibre from being investigated for enhancement of material. In order to realize its representative, extensive works are needed to be carried out.

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1.3 OBJECTIVE

The objectives of this project are as follows:

1. To produce electrospun nanofibres embedded with nanoparticles of carbonized polyacrylonitrile (PAN) and carbonized polyvinyl alcohol (PVA).
2. To observe characteristics of carbonized polyacrylonitrile (PAN) and carbonized polyvinyl alcohol electrospun nanofibres.
3. To determine an electrical conductivity and microstructure of carbonized polyacrylonitrile (PAN) and polyvinyl alcohol electrospun nanofibres (PVA).
4. To compare the characteristics between carbonized polyacrylonitrile (PAN) and carbonized polyvinyl alcohol electrospun nanofibres.

During the experiment, there is standard procedure to produce electrospun nanofibres and specific specimen is used to determine its characteristics. The experiment will be conducted 3 times to collect average result for each experiment. Since there are several characteristics needed to be recorded, the material that used is same.

1.4 SCOPE OF PROJECT

The scopes of this project are as follows:

1. To produce polyacrylonitrile (PAN) and polyvinyl alcohol (PVA) electrospun nanofibre using electrospinning process.
2. To produce carbon nanofibre (CNF) from polyacrylonitrile (PAN) and polyvinyl alcohol (PVA) electrospun nanofibre using stabilization and carbonization process.
3. To characterize an electrical conductivity of carbonize electrospun nanofibres using Four Point Probe.
4. To characterize chemical bonding of carbonize electrospun nanofibres using Fourier transform infrared spectroscopy (FTIR).
5. To characterize the morphology and microstructure of carbonize electrospun nanofibres using scanning electron microscopy (SEM).

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1.5 REPORT OVERVIEW

The PSM research begins with a literature review on Chapter 2 that covers the historical background of electrospinning, an introduction to the electrospinning process, carbonization of electrospun nanofibres and previous attempts to control deposition during electrospinning. The basic experimental procedures are then presented in Chapter 3 in order to describe the main experimental apparatus used, materials, sample substrate, and fibre characterization techniques.

Specific experimental results and discussion for each experimental work are presented in Chapter 4. Finally, the conclusions are presented in Chapter 5 along with recommendations for future works.

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CHAPTER 2

LITERATURE REVIEW

2.1 HISTORY OF ELECTROSPINNING PROCESS

„Electrospinning" term was derived from „electrostatics spinning" (Zheng Ming, 2003). Electrospinning identified as a dynamic method for polymer nanofibre fabrication. Before electrospinning being introduced to produce nano scale polymers, there is plenty of technique already used to performed production of nanofibre. A variety of the technique including self-assembly, phase separation, template synthesis and drawing used to produce nanofibre (Zheng Ming, 2003). However, the process takes a period of time to produce it. Moreover, previous technique has slightly low characteristics on nanofibre produced. Thus, a method which can be extra developed for mass manufacture of continuous nanofibre is electrospinning. It would not be conceivable that electrospinning affecting current electricity generation and insulation.

On late 1500"s, William Gilbert discover an electrical effects on liquid when he set up research and observation the phenomena of magnetism and electricity (Martin Masuelli, 2017). He demonstrated that electrostatic and magnetic attraction was a disparate phenomenon. Gilbert was a personal physician to the Queen Elizabeth 1 of England and also a Royal College of physician president (Tucker, Ph, Stanger, Staiger, & Ph, 1995). A droplet of water placed near a charged piece of Amber, it was drawn into a conical shape of water. This observation triggers small droplets of water which a phenomenon now called electrospaying.

Later on, in 1873 John William Strutt known as Lord Rayleigh conducted an experiment about electrical charges on water drops (Rayleigh & William, 1965). With theoretically calculation on charge, it causes certain size droplet to burst. Charles Vernon Boys was a talented instrument maker and physicist. He creates a torsion balance to measure

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gravitational constant based on stable fibre in 1888 (Boys, 1887). However, there are no mechanical properties in his electrospun fibres for the torsion balance.

Larmour define the stimulation of dielectric liquid effect on an electric charge using electrodynamics theory on 1898 (Knight, 1997). In 1902, John Francis Cooley created a device that by implement an electrical charge to spray liquids (Martin Masuelli, 2017). There are various indirectly charged spinning heads types that Cooley recommended including a coaxial head, an air assisted model, a conventional head and a spinneret. To collect fibres produced from jet in electrospinning method, a rotating collector was included in Cooley‟s patent. Viable fibre spinning technique is the origin of electrospinning can be drawn back to the early 1930s. Between 1907 and 1920, John Zeleny a physicist explored that a sharp point required a higher voltage to trigger discharge (Zeleny, 1914). He conducts an experiment on droplets of fluid behaviour at the tip of metal capillaries with mathematical model under electrostatic forces.

Graduate from University of Kyoto, Professor Kiyohiko Hagiwara adjust the molecular structure of colloidal liquid prior to spinning using electricity (Teo & Ramakrishna, 2006). By using a high frequency electrical discharge onto the solution, colloidal components are aligns and stick together.

Producing polymers filaments using electric charges is an invention that patented by Formhals in 1934 for the production of textile yarns (Shaikh, Mehmood, & Shaikh, 2010; Teo & Ramakrishna, 2006; Tucker et al., 1995). The solution between two electrodes bearing electrical charges of opposite polarity is where the polymer filaments were formed. By 1937, he focuses on his nozzle design which has conical taper and can be dismantle for cleaning. Although the technique using an electric field on producing polymer filament are explored for a long time, it wasn‟t favoured due to its spinning methods and short (Teo & Ramakrishna, 2006).

Formhals‟s process contained of adjustable collecting fibre like spinning hollow tube (Shaikh et al., 2010). This process was capable to achieve aligned thread parallel on the receiving device. Afterwards, Formhals licensed expansion system which different polymers generate composite fibre webs by electrostatically turning polymer fibres with respect to a

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moving base substrate (Raghavan, 2006; Subbiah et al., 2005; Zheng Ming, 2003). There are more latter electrospinning system that be drawn back to the design, researchers clearly gained knowledge deeply in electrospinning process. It is interesting to note an electrical engineer; Charles Ladd Norton illustrates melt-spinning using a joined electrostatic and air-jet assist method. Norton‟s patent the uses of auxiliary electrodes affect the path of fibres.

However, in 1964, an investigation about nature of fluid droplets on effect of an electric field was run by Ingram Taylor (Taylor, 1964). This investigation initiates studies on the operation of jet forming. Taylor illustrate that the jet originate by the creation of a conical shape (Niu, Wang, & Lin, 2011). Theoretically, Taylor conclude that a suitable cone formed need a semi vertical angle and demonstrate that the angle of cone almost reach his theoretical assumption. Simons patented the mechanisms using electrospinning for lightweight and ultra-thin fibres in 1966 (Shaikh et al., 2010).

In 1970, Peter Karl Baumgarten proclaimed that acrylic fibres produced by electrospinning has a diameter range of 0.05 until 1.1 μm (Baumgarten, 1971). He showed that by changing electric field, the diameters of fibres changed. In 1971, he studies the behaviour of surrounding gas, voltage and solution viscosity on diameter of fibre (Tucker et al., 1995). Larrondo create a melt electrospinner in 1981 (Larrondo & Manley, 1981). A fibre was drawn from the drop of polymer melt at spinning tip by electrostatic force.

Eletrospinning did not gain a lot of concern until 90‟s. When nanotechnology appearance in 1990, electrospinning method widely used for manufacture nano scale fibres. Electrospinning offers great benefit in formation of nanofibre. Since then, advertisement about electrospinning has been increasing epidemically through years.

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