



Faculty of Mechanical Engineering

**THE DEVELOPMENT OF CARBON NANOTUBE/CARBON
NANOFIBRE FROM POLYACRYLONITRILE ELECTROSPUN
NANOFIBRE PRECURSOR FOR ELECTRONIC APPLICATIONS**

Nurain binti Abdul Munajat

Master of Science in Mechanical Engineering

2018

**THE DEVELOPMENT OF CARBON NANOTUBE/CARBON
NANOFIBRE FROM POLYACRYLONITRILE ELECTROSPUN
NANOFIBRE PRECURSOR FOR ELECTRONIC APPLICATIONS**

NURAIN BINTI ABDUL MUNAJAT

**A thesis submitted
in fulfilment of the requirements for the degree of Master of Science
in Mechanical Engineering**

Faculty of Mechanical Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2018

DECLARATION

I declare that this thesis entitled “The Development of Carbon Nanotube/Carbon Nanofibre from Polyacrylonitrile Electrospun Nanofibre Precursor for Electronic Applications” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :.....
Name :.....
Date :.....

APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Mechanical Engineering

Signature :

Supervisor Name :

Date :

DEDICATION

To my beloved mother and father

ABSTRACT

Carbon nanofibre (CNF) have attracted much attention among researchers due to their excellent properties such as high mechanical strength, thermal and electrical conductivity. CNF have been proposed for various applications such as filtration, smart material, tissue engineering, fuel cell, capacitors and sensors. Recently, electrospinning technique followed by pyrolysis process of the precursor material has been proposed as a simple and economic alternative for fabricating CNF. In this study, the best parameters on fabrication process of CNF with and without the inclusion of multi-walled carbon nanotubes (MWCNT) fillers were determined and their properties were characterised. MWCNT was selected due to its superior electrical properties. Previously, considerable amount of effort have been made on studying the physical, chemical, and mechanical properties of electrospun CNF. However, there are limited studies dedicated to investigating the electrical properties of the CNF especially in terms of conductivity, complex permittivity (dielectric constant and loss factor) and loss tangent. Therefore, the scope of this research is to investigate the relationship of electrical properties with physical and chemical properties of the fibres. Polyacrylonitrile (PAN) precursor nanofibre were prepared using electrospinning technique. The best parameters for electrospinning were investigated by preparing the samples at electrospinning distances of 5 cm to 30 cm and applied voltage of 5 kV to 20 kV. Furthermore, the best pyrolysis process was determined by varying the carbonisation temperature of 800 °C, 1000 °C and 1200 °C with heating rate of 3 °C/min and 5 °C/min in a nitrogen filled furnace. As the optimum parameters were achieved, nanofibre samples with and without MWCNT were prepared. The characterization of the electrospun CNF was carried out using scanning electron microscopy (SEM), transmission electron microscope (TEM), ImageJ software, Fourier transform infrared spectroscopy (FTIR), four-point probe methods and dielectric probe. Based on fibre diameter, morphology, and deposition amount; the optimum electrospinning distance was found to be between 10 cm to 20 cm with an applied voltage between 15 kV to 20 kV. The results also suggest that increase in carbonisation and heating rate during pyrolysis process would increase the rate of elimination of non-carbon elements. This is evidenced by flatter FTIR spectrum and higher electrical conductivity of the samples which were carbonised at 1200 °C and heating rate of 5 °C/min. The electrical conductivity of CNF was significantly increased with the inclusion of MWCNT. The highest electrical conductivity was showed by CNF with 0.1 wt% of CNT with value 155.90 S/cm. However, samples with higher amount of MWCNT (> 0.1 wt%) showed reduced electrical conductivity to 21.56 S/cm. This could be explained by the formation of broken fibre network and agglomeration of MWCNT as observed using SEM and TEM. Finally, complex permittivity values of pure CNF and MWCNT-filled CNF were highest with dielectric constant value of 338.38 and loss factor value 488.72 at 1 GHz frequency. The knowledge gained from this study would extend the use of electrospun nanofibre in electronic applications such as sensors and other nano-sensing applications.

ABSTRAK

Karbon nanoserat (CNF) telah menarik perhatian ramai penyelidik kerana sifat-sifatnya yang bagus seperti kekuatan mekanikal, kekonduksian termal dan elektrik yang tinggi. CNF telah dicadangkan untuk berbagai aplikasi seperti penapisan, bahan pintar, kejuruteraan tisu, sel bahan, kapasitor dan sensor. Pada masa ini, teknik pemintalan elektro diikuti dengan proses pirolisis untuk prekursor serat yang disintesis telah dicadangkan sebagai satu alternatif yang mudah dan ekonomi untuk menghasilkan CNF. Dalam kajian ini, parameter terbaik untuk proses fabrikasi CNF dengan dan tanpa penambahan nanotub karbon berbagai dinding (MWCNT) ditentukan dan sifatnya dicirikan. Sebelum ini, pelbagai usaha telah dilakukan untuk mengkaji sifat fizikal, kimia dan mekanikal bagi CNF terpintal. Walau bagaimanapun, terdapat kajian yang terhad khusus untuk menyiasat sifat elektrik CNF terutamanya dalam soal kekonduksian, ketelapan kompleks (pemalar dielektrik dan faktor kehilangan) dan tangen kehilangan. Oleh itu, skop penyelidikan ini adalah untuk mengkaji hubungan sifat elektrik dengan sifat fizikal dan kimia. Prekursor nanoserat Poliakronitil (PAN) telah dihasilkan menggunakan teknik pemintalan elektro. Parameter terbaik untuk proses pemintalan elektro disiasat dengan menyediakan sampel pada jarak elektrospinning 5 cm hingga 30 cm dan voltan terpakai 5 kV hingga 20 kV. Tambahan pula, proses pirolisis terbaik ditentukan dengan mengubah suhu karbonisasi 800 °C, 1000 °C dan 1200 °C dengan kadar pemanasan 3 °C / min dan 5 °C / min dalam relau yang diisi dengan gas nitrogen. Oleh kerana parameter optimum telah dicapai, sampel nanoserat dengan dan tanpa CNT telah disediakan. Pencirian CNF terpintal dilakukan menggunakan mikroskop pengimbasan elektron (SEM), mikroskop penghantaran elektron (TEM), perisian ImageJ, Fourier transform spektroskopi inframerah (FTIR), kaedah empat titik prob dan prob dielektrik. Berdasarkan diameter serat, morfologi dan jumlah pemendapan, jarak optimum pemintalan elektro adalah antara 10 cm hingga 20 cm dengan voltan yang dikenakan antara 15 kV hingga 20 kV. Hasilnya menunjukkan peningkatan kadar karbonisasi dan pemanasan semasa pirolisis akan meningkatkan kadar penghapusan unsur-unsur bukan karbon. Ini dibuktikan dengan spektrum FTIR yang lebih rata dan kekonduksian elektrik yang lebih tinggi daripada sampel yang berkarbonat pada 1200 °C dengan kadar pemanasan 5 °C/min. Kekonduksian elektrik CNF meningkat dengan ketara setelah CNT dimasukkan. Kekonduksian elektrik tertinggi telah dihasilkan oleh CNF dengan 0.1 wt% CNT dengan jumlah 155.90 S/cm. Bagaimanapun, sampel dengan jumlah yang lebih tinggi mengandungi CNT (> 0.1 wt%) menunjukkan kekonduksian elektrik yang rendah kepada 21.56 S/cm. Ini dapat dijelaskan oleh pembentukan rangkaian serat pecah dan pengumpulan CNT seperti yang diperhatikan menggunakan SEM dan TEM. Akhirnya, nilai kebolehtelapan kompleks CNF dan CNF yang dipenuhi CNT adalah tinggi dengan jumlah pemalar dielektrik 488.72 dan jumlah faktor kehilangan 488.72 pada frekuensi 1 GHz. Pengetahuan yang diperoleh dari kajian ini akan memperluaskan penggunaan nanoserat terpintal dalam aplikasi elektronik seperti alat pengesan dan aplikasi nanopengesan yang lain.

ACKNOWLEDGEMENTS

In the name of Allah, the Most Gracious and the Most Merciful

Alhamdulillah, all praises to Allah for the strengths and His blessing in completing this thesis. Special appreciation and sincerest gratitude to my supervisor, Dr Nurfaizey bin Abdul Hamid, for his invaluable supervision, guidance, kindness, encouragement, and financial support throughout my study. I am also very thankful to my co-supervisor, Dr Siti Hajar binti Sheikh Md. Fadzullah for her guidance, advice and motivation.

I would like to extend my thanks to my fellow postgraduate colleagues for their support, invaluable help and for providing an excellent research atmosphere during the course of my Master candidature. My sincere appreciation also extends to all freinds and others who have provided assistance at various occasions. Importantly, my study would not be possible without my sponsors. I would like to acknowledge the Ministry of Higher Education (MOHE), Universiti Teknikal Malaysia Melaka (UTeM), and Tabung Pendidikan Sarjana PDT, for the scholarships and the research grants.

I am deeply indebted to my parents Abdul Munajat bin Ab Rahman, Nor Harlin binti Samian, husband, and family members for their unconditional love, continuous support, encouragement and prayers during my life and throughout the course of this study. Last but not least, I would like to extend my gratitude to everyone who have been directly and indirectly involved in the successful completion of this thesis. Thank you so much!

TABLE OF CONTENTS

	PAGE
DECLARATION	
APPROVAL	
DEDICATION	
ABSTRACT	i
ABSTRAK	ii
ACKNOWLEDGEMENTS	iii
TABLE OF CONTENTS	iv
LIST OF TABLES	vi
LIST OF FIGURES	viii
LIST OF ABBREVIATIONS	xiv
LIST OF PUBLICATIONS	xv
CHAPTER	
1. INTRODUCTION	1
1.1 Research background	1
1.2 Problem statement	3
1.3 Research objectives	4
1.4 Scope of research	4
1.5 Hypothesis	5
1.6 Contributions of research work	5
1.7 Thesis organisation	7
2. LITERATURE REVIEW	9
2.1 Introduction	9
2.2 Electrospinning process	10
2.2.1 Applied voltages	12
2.2.2 Feed rate of the polymer solutions	13
2.2.3 Solution and surrounding temperature	14
2.2.4 Electrospinning distance	15
2.3 Carbon nanofibre	15
2.3.1 Fabrication method of carbon nanofibre	16
2.3.2 Carbon nanofibre precursor and filler addition	17
2.3.3 Pyrolysis of PAN precursor	19
2.3.4 Characterisation of the carbon nanofibre	21
2.3.4.1 Morphological analysis	21
2.3.4.2 Chemical analysis	21
2.3.4.3 Conductivity	22
2.3.4.4 Complex permittivity	22
2.3.4.5 Loss tangent	23
2.4 Desired properties studies on electrospun CNF from PAN precursor	24
2.4.1 Preparation of electrospinning polymer solutions	25
2.4.2 Recent applications of PAN electrospun nanofibre	25
2.4.3 Fabrication of electrospun carbon nanofibre	29

2.4.4	Recent studies on characterisation of electrospun carbon nanofibre	33
2.5	Summary	42
3.	RESEARCH METHODOLOGY	43
3.1	Introduction	43
3.2	Research flow chart	43
3.3	Materials	46
3.4	Experimental parameters	47
3.4.1	Preparation of electrospinning solutions	47
3.4.2	Fabrication of electrospun nanofibre	48
3.4.3	Pyrolysis process of nanofibre	50
3.5	Characterisation of electrospun nanofibre	52
3.5.1	Morphological study of electrospun nanofibre	52
3.5.2	Determination of functional group of electrospun nanofibre	54
3.5.3	Electrical conductivity analysis of electrospun carbon nanofibre	55
3.5.4	Dielectric analysis of electrospun carbon nanofibre	56
3.6	Summary	57
4.	RESULT AND DISCUSSION	58
4.1	Electrospinning parameters analysis on electrospun nanofibre	58
4.1.1	The effect of electrospinning distance on electrospun nanofibre	58
4.1.2	The effect of applied voltage and electrospinning distance of electrospun nanofibre	64
4.1.3	Optimum electrospinning parameters	68
4.2	The effects of carbonisation temperature and heating rate during pyrolysis process	71
4.2.1	Physical properties of the nanofibre	71
4.2.2	Morphological properties of electrospun nanofibre	72
4.2.3	Chemical properties analysis of electrospun nanofibre	75
4.2.4	Sheet resistance and electrical conductivity of the nanofibre	78
4.2.5	Dielectric properties of the nanofibre	81
4.2.6	Optimum pyrolysis parameters	87
4.3	The effect of MWCNT addition on the properties of carbon nanofibre	89
4.3.1	Physical properties of the nanofibre	89
4.3.2	Morphological properties of electrospun nanofibre	91
4.3.3	Further observations on morphological analysis	95
4.3.4	Chemical properties analysis of electrospun nanofibre	97
4.3.5	Sheet resistance and electrical conductivity of the nanofibre	101
4.3.6	Dielectric properties of the nanofibre	103
4.3.7	Optimum weight percentage of MWCNT addition	107
4.4	Summary	109
5.	CONCLUSION AND RECOMMENDATION	110
5.1	Conclusion	110
5.2	Recommendation	111
	REFERENCES	113

LIST OF TABLES

TABLE	TITLE	PAGE
2.1	Experimental property values of SWCNTs and MWCNTs.	19
2.2	Classification of materials based on loss tangent value.	24
2.3	Recent applications of PAN electrospun nanofibre.	28
2.4	Summary of recent studies related to pyrolysis of PAN electrospun nanofibre.	32
2.5	FTIR's peaks and corresponding groups/bonds of pure PAN fibres.	36
2.6	The summary of recent characterisation studies of electrospun carbon nanofibre.	41
3.1	Composition of the MWCNT used in the experiment.	46
3.2	Summary of addition of MWCNT in electrospun carbon nanofibre.	48
3.3	Electrospinning parameters used in Experiment 1.	49
3.4	Summary of carbonisation process.	51
4.1	The average fibre diameters and SEM micrograph when electrospinning at a fixed distance of 5 cm and different applied voltages of 5 kV, 10 kV, 15 kV, and 20 kV.	60
4.2	The average fibre diameters and SEM micrograph when electrospinning at a fixed distance of 30 cm and different applied voltages of 5 kV, 10 kV, 15 kV, and 20 kV.	62

4.3	The micrographs of SEM and average fibre diameters at applied voltage of 5 kV, 10 kV, 15 kV and 20 kV with different electrospinning distance of 10 cm, 15 cm, 20 cm, and 25 cm.	67
4.4	Table of SEM micrographs of PAN-MWCNT before and after pyrolysis process (a)PAN/MWCNT/0, (b) PAN/MWCNT/0.1, (c) PAN/MWCNT/0.3, (d) PAN/MWCNT/0.5, (e) PAN/MWCNT/0.7, and (f) PAN/MWCNT/1.0.	93

LIST OF FIGURES

FIGURE	TITLE	PAGE
1.1	A chart showing the gap in knowledge which this study aims to address.	6
2.1	SEM micrograph of polyacrylonitrile electrospun nanofibre at $\times 10000$ magnification by using electrospinning process.	10
2.2	Schematic diagram of electrospinning process.	11
2.3	Formation of Taylor cone.	12
2.4	Formation of fibres with beads caused by excessive polymer feed rate (a) 0.5 ml/hr (b) 2ml/hr.	13
2.5	Diagram showing the effect of ambient temperature on surface morphology and diameter of electrospun PAN nanofibre.	14
2.6	The electrospinning distance h between the spinneret and the collector.	15
2.7	TEM micrographs of CNF produced using (a) vapour growth carbon nanofibre (VGCNF) and (b) electrospinning of CNF precursor followed by thermochemical treatment.	17

2.8	Schematic illustrations of (a) single-walled carbon nanotube (SWCNT), (b) double-walled carbon nanotube (DWCNT) and (c) multiwalled carbon nanotube.	18
2.9	The conversion of $C\equiv N$ bonds in PAN polymer chains to $C=N$ bonds of the ladder structure.	20
2.10	Carbonisation process of nanofibre.	21
2.11	Typical power transmission response with and without a sample.	23
2.12	Schematic diagram of an antimicrobial filter using Ag-filled PAN electrospun nanofibre.	27
2.13	SEM (left) and TEM (right) micrograph of PAN/MWCNT composite polymer.	34
2.14	SEM micrograph of (a) pure PAN (590 nm), (b) PAN/CNT (650 nm), (c) agglomeration of nanofibre strips, (d) formation of beads on nanofibre strips.	35
2.15	The FTIR results of pure PAN and PAN with 3wt% of MWCNT filler.	36
2.16	The FTIR spectrums of pure PAN and PAN-g-GMA fibres.	37
2.17	The FTIR spectrums of PAN before and after pyrolysis process at different carbonisation temperatures.	38
3.1	Flow chart of fabrication and characterisation of electrospun carbon nanofibre.	45
3.2	A schematic layout of the tube furnace.	50

3.3	A Model JSM-6010PLUS/LV scanning electron microscope (Jeol Ltd., Japan).	53
3.4	SEM micrograph of polyacrylonitrile electrospun nanofibre at $\times 5000$ magnification.	53
3.5	A TEM Model FEI Helios Nanolab TM (Thermo Fisher Scientific, USA)	54
3.6	Grounding and mixing of the samples.	55
3.7	A hand press (PIKE Technologies).	55
3.8	A typical four-point probe machine.	56
3.9	A dielectric probe Model Keysight 85070E with a network analyser Model Keysight E5071C.	57
4.1	A schematic diagram of electrospinning process at a short distance.	59
4.2	Average fibre diameter as a function of voltage at electrospinning distance of 5 cm.	61
4.3	A schematic diagram of electrospinning process at a long distance.	62
4.4	Average fibre diameter as a function of voltage at electrospinning distance of 30 cm.	63
4.5	The graph of average fibre diameters at applied voltage of 5 kV, 10 kV, 15 kV and 20 kV with different electrospinning distance of 10 cm, 15 cm, 20 cm, and 25 cm.	68
4.6	The colour change of the nanofibre before and after heat treatments (a) electrospun PAN electrospun nanofibre (NF), (b) stabilised nanofibre (SNF), and (c) carbonised nanofibre (CNF).	72

4.7	SEM micrograph of (a) electrospun PAN nanofibre, (b) stabilised nanofibre, carbonised nanofibre at temperature and heating rate of (c) 800 °C and 5 °C/min, (d) 800 °C and 3 °C/min, (e) 1000 °C and 5 °C/min, (f) 1000 °C and 3 °C/min, (g) 1200 °C and 5 °C/min, and (h) 1200 °C and 3 °C/min.	73
4.8	The relationship between average fibre diameter with temperature and heating rate of carbonisation process.	75
4.9	FTIR spectrum of electrospun carbon nanofibre under different heat treatment.	76
4.10	The relationship between sheet resistance, temperature and heating rate of carbonisation process.	79
4.11	The relationship between electrical conductivity, temperature and heating rate of carbonisation process.	81
4.12	Dielectric constant ϵ' (real part permittivity) of the nanofibre as a function of frequency.	83
4.13	Imaginary part of the complex permittivity ϵ'' (loss factor) of the nanofibre as a function of frequency.	84
4.14	Loss tangent of the nanofibre as a function of frequency.	86
4.15	The colour change after addition of MWCNT (a) 0 wt% PAN electrospun nanofibre (PAN/MWCNT/0), (b) 0.1 wt% PAN electrospun nanofibre (PAN/MWCNT/0.1), (c) 0.3 wt% PAN electrospun nanofibre (PAN/MWCNT/0.3), (d) 0.5 wt% PAN electrospun nanofibre (PAN/MWCNT/0.5), (e) 0.7 wt% PAN	90

electrospun nanofibre (PAN/MWCNT/0.7), and (f) 1.0 wt% PAN electrospun nanofibre (PAN/MWCNT/1.0).

4.16	Summary of average fibre diameter PAN-MWCNT before and after pyrolysis process.	94
4.17	SEM micrograph of electrospun nanofibre with distorted distributions.	94
4.18	Transmission electron microscope (TEM) micrographs of electrospun carbon nanofibre with different MWCNT addition (a) PAN/MWCNT/0.1, (b) PAN/MWCNT/0.3 (c) PAN/MWCNT/0.5, and (d) PAN/MWCNT/1.0.	96
4.19	Schematic diagram of well aligned MWCNT along the nanofibre thread based on TEM micrograph on 0.1 wt% MWCNT.	97
4.20	FTIR spectrum of electrospun nanofibre with different MWCNT concentrations before pyrolysis process.	98
4.21	FTIR spectrum of electrospun nanofibre with different MWCNT concentrations after pyrolysis process.	100
4.22	Graph of sheet resistance versus MWCNT concentration.	101
4.23	Graph of conductivity versus MWCNT concentration.	102
4.24	Dielectric constant (real part permittivity) of electrospun carbon nanofibre with different MWCNT concentration graph.	104
4.25	Loss factor (imaginary part permittivity) of electrospun carbon nanofibre with different MWCNT concentration graph.	105
4.26	Loss tangent of electrospun carbon nanofibre with different CNT concentration graph	107

LIST OF ABBREVIATIONS

ACNF	-	Activated Carbon Nanofibre
CNF	-	Carbon Nanofibre
CNT	-	Carbon Nanotube
CNT	-	Chemical Vapour Deposition
DMF	-	Dimethylformamide
DWCNT	-	Double-wall Carbon Nanotube
FE-SEM	-	Field Emission Electron Microscope
FTIR	-	Fourier Transform Infrared Spectroscopy
MWCNT	-	Multi-wall Carbon Nanotube
PAN	-	Polyacrylonitrile
PANI	-	Polyaniline
PLA	-	Polyactic Acid
PU	-	Polyurethanes
PVA	-	Polyvinyl Alcohol
PVDF	-	Polyvinylidene Fluoride
SEM	-	Scanning Electron Microscope
SWCNT	-	Single-wall Carbon Nanotube
TEM	-	Transmission Electron Microscopy
VNA	-	Vector Network Analyzer

LIST OF PUBLICATIONS

The research papers produced and published during the course of this research are as follows:

Journals:

1. N. A. Munajat, A. H. Nurfaizey, M. H. M. Husin, S. H. S. M. Fadzullah, G, 2018. The effects of different carbonization temperatures on the properties of electrospun carbon nanofibre from polyacrylonitrile (PAN) precursor. *Journal of Advanced Research in Fluid Mechanics and Thermal Sciences (ARFMTS)*, 2(49), pp. 85-91. (*Scopus*). (published)
2. N. A. Munajat, A. H. Nurfaizey, A. A. M. Bahar, K. Y. You, S. H. S. M. Fadzullah, G. Omar, 2018. High frequency dielectric analysis of carbon nanofibre from PAN precursor at different pyrolysis temperatures. *Microwave and Optical Technology Letters (MOTL)*, 60(9), pp. 2198-2204. (*Scopus/ISI*). (published)

Conference papers

1. N.A. Munajat, A.H. Nurfaizey, S. H. S. M. Fadzullah, 2018. The preparation and morphological investigation of polyacrylonitrile electrospun nanofibre with different loading of carbon nanotube. *1st Colloquium Paper: Advanced Materials and Mechanical Engineering Research (CAMMER'18)*. (published)
2. N. A. Munajat, A. H. Nurfaizey, S. H. S. M. Fadzullah, G. Omar, J. Jaafar, N. S. A. Roslan, 2017. Fabrication and characterization of carbon nanofibre from

polyacrylonitrile precursor. *Proceedings of Mechanical Engineering Research Day 2017*, Melaka, Malaysia, pp. 362-363. (published)

CHAPTER 1

INTRODUCTION

1.1 Research background

Recently, there was a growing research interest in nanofibrous materials as evidenced by the increasing number of publications with regard to nanofibre. One of the main reasons for this growing trend is due to the unique properties and superior capabilities of nanofibre. Nanofibrous materials have been proposed for various applications such as filtration, sensor, drug delivery, super capacitors and energy storage (Ramesh Kumar et al., 2012, Lee et al., 2014, Su et al., 2014, Zhou et al., 2014, Myung et al., 2015). Nanofibre is defined as ultrafine fibre with average fibre diameters typically in the range from 100 nm to a few microns.(Nataraj et al., 2012). At this scale, nanofibre offer unique properties such as high surface area, high porosity, light weight, as well as outstanding structural and mechanical properties (Zhang et al., 2014, Haider et al., 2015, Poveda and Gupta, 2016, Yalcinkaya et al., 2016).

One of the nanofibre research areas that has been particularly interesting is in the synthesis of carbon nanofibre (CNF). CNF is similar with other one-dimensional nanostructures such as nanotubes and nanowires in terms of high length-to-diameter ratio (Pashaloo et al., 2009). Traditionally, carbon fibre materials can be prepared by using several processes including post heat treatment of precursor materials. Precursor materials can be natural or synthetic materials which fulfil certain condition such as the ability to withstand high temperature during pyrolysis process in an inert environment (Cho et al., 2007). Polyacrylonitrile (PAN) precursor is one of the most popular precursor materials for the

fabrication of CNF. In term of processing method, electrospinning has been opted as the preferred choice for fabricating nanofibre (Huang, 2009). Electrospinning is a simple and versatile technique for producing ultra-fine fibres from polymeric solution or melt using electric charge (Chronakis, 2005). It is a very cost-effective technique compared to other nanomaterial synthesis technique such as chemical vapour deposition. This topic will be discussed in more detail in Chapter 2.

In some studies, carbon nanotubes (CNT) were added into the CNF in attempts to enhance the mechanical and electrical properties of CNF. CNT is known as allotropes of carbon with cylindrical molecular structure of carbon atoms with a length to diameter ratio larger than 1,000,000 (Hirlekar et al., 2009). Due to the very high surface area, stiffness, strength, and resilience, CNT offers novel properties that are highly potential for numerous applications in nanotechnology (Hirlekar et al., 2009). A study by Pasanen et al. (2009) reported that inclusion of CNTs in polymers would improve the polymer's tensile strength, elastic modulus, chemical resistance, and thermal shrinkage during stabilisation and carbonisation process. However, to the best of the author's knowledge, there is limited information available regarding electrical properties of CNT-filled CNF especially in terms of electrical conductivity, relative permittivity, and loss tangent.

The focus of this study is to fabricate and characterise CNF and CNT-filled CNF electrospun fibre membranes. Pure PAN and CNT-filled PAN precursor solutions were prepared prior to electrospinning process. After the electrospinning process, the as spun PAN and CNT-filled PAN nanofibre underwent a thermochemical process at elevated temperature to transform it into CNF and CNT-filled CNF. Characterisation of the membrane will be carried out to determine the physical, chemical, and electrical properties of the membrane. The main hypothesis of this study is that the inclusion of CNT into CNF would improve the

electrical properties of the membrane. The knowledge that will be gained from this study is important to extend the application of electrospun nanofibre in electronic applications.

1.2 Problem statement

A proper selection of material for electronic applications such as sensors is critical to make sure that the product has the desired range of capabilities in terms of sensitivity, selectivity, and reliability (Luoh and Hahn, 2006). In general, sensor materials should possess characteristics such as good electrical conductivity and high effective surface area. In this regard, the very high surface area and high porosity of electrospun nanofibre membranes are the main attractive attributes that make electrospun nanofibre membranes as highly potential candidates for ultrasensitive sensors and other nano-sensing applications (Ding et al., 2010). A significant amount of effort has been dedicated by researchers in studying the effects of high surface area of the material on the performance of the sensors (Llobet, 2013). One of the popular choice of materials from these studies were CNF and CNT-filled CNF. To an extent, the physical, chemical and mechanical properties of the CNF and CNT-filled CNF are well characterised (Ra et al., 2005, Guo et al., 2010, Qiao et al., 2011, Chien et al., 2014, Rubia et al., 2014, Kaur et al., 2016). However, there were few studies dedicated on the electrical properties especially in terms of conductivity, permittivity and loss tangent of the electrospun CNF, and their relationships with physical and chemical properties of the fibres. Therefore, a comprehensive study is required to give an insight knowledge about the topic.

1.3 Research objectives

The objectives of the research are as follows:

1. To determine the optimum electrospinning parameters of polyacrylonitrile precursor material.
2. To evaluate the morphological, chemical, and electrical properties of carbon nanofibre (CNF) with different pyrolysis parameters.
3. To characterise the morphological, chemical, and electrical properties CNF with different weight percentage of multi walled carbon nanotube (MWCNT).

1.4 Scope of research

The scopes of the research are as follows:

1. Polyacrylonitrile (PAN) polymer was chosen as the precursor material and dimethylformamide (DMF) as a solvent.
2. PAN electrospun nanofibre were produced using electrospinning process.
3. The range selection of electrospinning parameters for electrospinning distance was 5 cm to 30 cm and applied voltage was 5 kV to 20 kV.
4. The range selection of pyrolysis parameters for carbonisation temperature was 800 °C, 1000 °C and 1200 °C with heating rate 3 °C/min and 5 °C/min in a nitrogen filled furnace.
5. The physical and morphological characteristics of the nanofibre were characterised using scanning electron microscopy (SEM) and transmission electron microscope (TEM).
6. The fibre diameter of electrospun nanofibre before and after pyrolysis process were measured by using Image J software.