



**Faculty of Mechanical Engineering**

**EFFECT OF MULTIWALL CARBON NANOTUBE LOADING ON  
MULTI FILLER POLYMER COMPOSITE AS BIPOLAR PLATE**

**Aninorbaniyah binti Bairan**

**Master of Science in Mechanical Engineering**

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**ANINORBANIYAH BINTI BAIRAN**

**A thesis submitted  
in fulfillment 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 “Effect of Multiwall Carbon Nanotube Loading on Multi Filler Polymer Composite as Bipolar Plate” 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.

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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 family

## ABSTRACT

Apart from achieving homogeneous Multiwall Carbon Nanotube (MWCNTs) dispersion in nanocomposites and preventing agglomeration of MWCNTs due to Van der Waals attraction forces, the main challenge here is to enhance the interfacial compatibility between MWCNTs and nonpolar polypropylene (PP). Therefore, the aim of this study was to identify the most effective and suitable ratio of MWCNTs loading through two mixing methods. The ratio of fillers and binder was fixed at 80:20, while the conductive fillers of MWCNTs (0% up to 10%), G (45% up to 55%) and CB was set to 25% of the weight percentage of G/CB/MWCNTs/PP composite. The multi filler of Graphite (G), Carbon Black (CB) and MWCNTs composite using a medium density polypropylene (MDPP) as binder was mixed through melt compounding method. The second mixing method is through dry mixing method by using MDPP and low density polypropylene (LDPP) as binders. The composite were fabricated through compression molding. The results included the characterization of electrical and mechanical properties and analysis of the hydrogen gas permeability and surface morphology of the composites. The effective MWCNTs loading is in the range of 5 wt.% up to 7 wt.% of MWCNTs and based on the two methods, melt compounding method is better than dry mixing method in terms of its electrical conductivity and mechanical properties. For melt compounding method, it was found that using MWCNTs as a third filler at a loading of 5 wt.% in a G/CB/MDPP composite produced higher results of in-plane electrical conductivity; 518.90 S/cm, the flexural strength, density and shore hardness 61.43 MPa, 1.61 g/cm<sup>3</sup> and 65.1 (SH) respectively. Meanwhile, through dry mixing method for the MWCNTs/MDPP composite, the electrical conductivity is 158.32 S/cm with 6 wt.% MWCNTs content. The flexural strength of MWCNTs/MDPP increased from 22.95 MPa (3 wt.%) to 29.86 MPa (5 wt.%) with the increment of MWCNTs content. Results also indicated that there was no leaking gas occurred during the permeability test at 5 wt.% MWCNTs content for melt compounding and dry mixing (LDPP) and 6 wt. % MWCNTs for dry mixing (MDPP). These results confirm that melt compounding methods and the addition of MWCNTs lead to a significant improvement on the properties of the conducting polymer composite as bipolar plate.

## ABSTRAK

Selain daripada mencapai penyebaran homogen Multiwall Carbon Nanotube (MWCNTs) dalam nanokomposit dan mencegah penumpuan MWCNTs disebabkan oleh tarikan Van der Waals, cabaran utama di sini adalah untuk meningkatkan keserasian antaramuka di antara MWCNTs dan bukan polar polipropilena. Oleh itu, tujuan kajian ini adalah untuk mengenal pasti nisbah yang paling berkesan dan sesuai MWCNTs pengisi melalui dua kaedah percampuran. Nisbah pengisi dan pengikat ditetapkan pada 80:20, di mana pengisi konduktif MWCNTs (0% hingga 10%), G (45% hingga 55%) dan CB ditetapkan kepada 25% dari peratusan berat G/CB/MWCNTs/PP komposit. Pengisi iaitu pelbagai Grafit (G), Karbon Hitam (CB) dan MWCNTs menggunakan kristal polipropilena (MDPP) sebagai pengikat telah dicampur melalui kaedah percampuran lebur. Kaedah percampuran kedua adalah melalui kaedah percampuran kering dengan menggunakan (MDPP) dan amorfus Polipropilena (LDPP) sebagai pengikat. Komposit yang dihasilkan adalah melalui teknik pengacuan mampatan dengan dua kaedah pencampuran. Keputusan yang dibincangkan bukan sahaja untuk mencirikan sifat-sifat elektrik dan mekanikal, tetapi juga menganalisis kebolehtelapan gas hidrogen dan permukaan morfologi komposit. Pemuatan MWCNTs yang berkesan berada dalam julat 5 wt.% sehingga 7 wt.% berat MWCNTs dan berdasarkan kedua-dua kaedah, kaedah pengkompaunan leburan adalah lebih baik berbanding kaedah percampuran kering yang menghasilkan kekonduksian elektrik dan sifat-sifat mekanikal yang lebih baik. Untuk kaedah percampuran lebur, didapati bahawa, dengan menggunakan MWCNTs sebagai pengisi ketiga pada muatan 5 wt.% dalam G/CB/PP komposit, menunjukkan hasil yang lebih tinggi kekonduksian elektrik dalam-satah adalah 518.90 S/cm, kekuatan lenturan, ketumpatan dan kekerasan adalah 61.43 MPa, 1.61 g/cm<sup>3</sup> dan 65.1 (SH) masing-masing. Sementara itu, dengan menggunakan kaedah pencampuran kering, bagi komposit MWCNTs/MDPP, kekonduksian elektrik adalah 158.32 S/cm dengan kandungan MWCNTs 6 wt.%. Kekuatan lenturan MWCNTs/MDPP telah meningkat daripada 22.95 MPa (3 wt.%) kepada 29.86 MPa (5 wt.%) dengan peningkatan kandungan MWCNTs. Keputusan juga menunjukkan bahawa tiada kebocoran gas berlaku semasa ujian kebolehtelapan pada kandungan MWCNTs 5 wt.% untuk kaedah percampuran lebur dan percampuran kering (LDPP) serta kandungan MWCNTs 6 wt.% untuk kaedah percampuran kering (MDPP). Keputusan ini mengesahkan bahawa kaedah percampuran lebur yang digunakan dan penambahan MWCNTs membawa kepada peningkatan yang ketara pada prestasi sel plat dwikutub komposit polimer.

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## LIST OF ABBREVIATIONS AND SYMBOLS

PEMFC	-	Proton exchange membrane fuel cell
CPCs	-	Conductive polymer composite
ICPs	-	intrinsically conducting polymers
G	-	Graphite
CB	-	Carbon Black
MWCNTs	-	Multiwall Carbon Nanotubes
CF	-	Carbon Fiber
PP	-	Polypropylene
U.S. DOE	-	United State Department of Energy
SEM	-	Scanning Electron Microscope
DMFC	-	Direct methanol fuel cell
AFC	-	Alkaline fuel cell
PAFC	-	Phosphoric acid fuel cell
MCFC	-	Molten carbonate fuel cell
SOFC	-	Solid oxide fuel cell
MEA	-	Membrane electrode assemblies
GDL	-	Gas diffusion layer
PVDF	-	Polyvinylidene fluoride
PEEK	-	Polyether ether ketone
PET	-	Polyethylene terephthalate

DSC	-	Differential Scanning Calorimetry
TGA	-	Thermo-Gravimetry Analysis
MWNTs	-	Multi-walled Nanotubes
SWNTs	-	Single-walled Nanotubes
MA	-	Maleic anhydride
EG	-	Exfoliated graphite
xGnPs	-	Exfoliated graphite nanoplatelets
TEM	-	Transmission electron microscopic
ASTM	-	American Standard Test Method
LDPP	-	Low density Polypropylene
MDPP	-	Medium density Polypropylene
SSs	-	Stainless steels
Eq.	-	Equation
H <sub>2</sub>	-	Hydrogen
O <sub>2</sub>	-	Oxygen
e <sup>-</sup>	-	Electron
wt. %	-	Weight percentage
Scm <sup>-1</sup>	-	Siemen/centimeter
cm	-	centimeter
μA	-	micron Ampere
MPa	-	Mega Pascal
mK	-	mili Kelvin
°C	-	Degree Celcius
g/cm <sup>3</sup>	-	gram/centimeter <sup>3</sup>
~	-	is equavalent to
<	-	is less than
>	-	is greater than

$\Phi_c$	-	percolation threshold
$\sigma_m$	-	maximum electrical conductivity
F	-	filler
$\sigma_p$	-	conductivity of the polymer matrix
vol. %	-	Volume Percentage
psi	-	pound per square inch
e.g.	-	example
$\Omega m$	-	ohm meter
nm	-	nanometer
$\Omega \text{ cm}$	-	ohm centimeter
g	-	gram
rpm	-	revolutions per Minute
mm	-	milimeter
x	-	multiplied by

## LIST OF PUBLICATIONS

### JOURNAL

1. **A. Bairan**, M.Z. Selamat, S.N. Sahadan, S. D. Malingam and N. Mohamad, *Effect of MWCNTs on the Electrical and Mechanical Properties of Polymeric Composite as Pem Fuel Cell Bipolar Plate*. Jurnal Teknologi (Sciences and Engineering): Vol. 80: 6 November 2018. (Accepted)
2. **A. Bairan**, M.Z. Selamat, S.N. Sahadan, S. D. Malingam and N. Mohamad, *Electrical Conductivity and Mechanical Properties of Graphite/Carbon Black/Carbon Nanotube/Polypropylene nanocomposites*. Journal of Mechanical Engineering. (Submitted)

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2. **A. Bairan**, M.Z. Selamat, S.N. Sahadan, S. D. Malingam and N. Mohamad, *Effect of Polypropylene Type on G/CB/MWCNTs/PP Composites Properties as Bipolar Plate for PEM Fuel Cell*, Proceedings of Mechanical Engineering Research Day 2016, Melaka, 31 March 2016, pp. 157-158.
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2. **A. Bairan**, M.Z. Selamat, S.N. Sahadan, S. D. Malingam and N. Mohamad, *Effect of Polypropylene Type on G/CB/MWCNTs/PP Composites Properties as Bipolar Plate for PEM Fuel Cell*, Mechanical Engineering Research Day 2016, Melaka, 31 March 2016, pp. 157-158.
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## **CHAPTER 1**

### **INTRODUCTION**

#### **1.1 Background**

Proton exchange membrane fuel cells (PEMFCs) have been found to be a good choice and important source of energy for portable and automotive propulsion applications for their advantages of high power density, solid state construction, high efficiency of conversion of chemical to electrical energy, near zero environmental emissions and low temperature operation (de Oliveira et al., 2012; Gautam et al., 2015). However, the high cost of PEMFCs is still a major obstacle in its commercialization of transport applications. The bipolar plates is a major component of PEMFCs stack which takes a large portion of stack cost (Lee et al., 2007; Mathur et al., 2008). They can contribute 70-80% of the stack weight and up to 45% of the costs (Kakati et al. 2009). Hence, the investigation on cost/performance materials of bipolar plates has become an important area of research.

Basically, bipolar plates can be made from many different materials, such as pure graphite, metal or polymer composites with carbon or metal conductive as a main filler. Pure graphite is one of the more traditional materials used to produce bipolar plates due to their advantages of good thermal and electrical conductivity, excellent

chemical compatibility and good corrosion resistant. However, some problems with pure graphite include the high cost and time factor during fabrication process, especially the machining process of gas flow channels into the plate surface in which graphite has low mechanical strength properties (Cunningham and Baird, 2007; Du et al., 2010). Therefore, conductive polymer composite (CPCs) bipolar plate has gained a considerable interests among researchers to replace pure graphite bipolar plate for PEMFC.

CPCs are made of conductive fillers or multi fillers such as graphite (G), carbon black (CB) and Multiwall Carbon Nanotubes (MWCNTs) which are incorporated in Polypropylene (PP) matrix. Most researchers (Suherman and Bung, 2016; Selamat et al., 2011, 2013) reported only on high loading of fillers (more than 90 wt.%) and reaching electrical conductivity above 100 S/cm, which is targeted from Department of Energy (U.S. DOE). Higher loading of fillers causes change in rheological properties and increase the difficulties in polymer processing. This will decrease the electrical and mechanical properties of CPCs as bipolar plate.

Although there are many studies on CPCs as bipolar plates for PEMFCs, only few of them focus on MWCNTs as reinforced filler. Hence, it is necessary to do in-depth research on the combination of multi fillers bipolar plate materials to obtain a better electrical conductivity of the composite (Dweiri and Sahari, 2007; Bauhofer and Kovacs, 2009; Selamat et al., 2013b). Therefore, some conductive fillers like CB, G, MWCNTs and carbon fiber (CF) are commonly used as reinforced fillers to enhance overall performance of CPCs as bipolar plates (Ghosh et al., 2014; Dang et al., 2011). The interaction between fillers and polymer chains is the most important aspect that