

PREPARATION AND CHARACTERIZATION OF NANO-  
ALUMINA FILLED HDPE CONDUCTING POLYMER  
COMPOSITE

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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**PREPARATION AND CHARACTERIZATION OF NANO-ALUMINA  
FILLED HDPE CONDUCTING POLYMER COMPOSITE**

This report submitted in accordance with requirement of the Universiti Teknikal  
Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering  
(Engineering Materials) (Hons.)

by

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**2016**

## DECLARATION

I hereby, declared this report entitled “Preparation and Characterization of Nano-Alumina Filled HDPE Conducting Polymer Composite” is the results of my own research except as cited in references.

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## **APPROVAL**

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Department of Engineering Materials) (Hons.). The member of the supervisory committee is as follow:

.....  
**PROFESOR DR. QUMRUL AHSAN**  
(Official Stamp of Supervisor)

## ABSTRAK

Secara umumnya, polimer dikenali sebagai penebat elektrik, akan tetapi untuk menjadikan polimer yang mampu digunakan dalam aplikasi elektronik, penyelidikan dalam bidang pengaliran elektrik menjadi satu pendekatan yang menarik. Untuk tujuan ini, salah satu strategi spesifik adalah berdasarkan pengadunan penebat polimer konvensional, “HDPE” dengan pengisi organik yang konduktif, “Pani” dan “MWCNT” bagi membuat polimer komposit yang boleh mengalirkan elektirk. Cara pencampuran dari segi mekanikal diimplikasikan tanpa melakukan pengubahsuaian ke atas bahan yang digunakan. Kaedah pencampuran dengan kedua-dua pengisi organik telah menjanjikan prestasi elektrik pengaliran yang menunjukkan kos yang rendah sekali. Ini adalah disebabkan kelebihan polimer sendiri iaitu kemudahan dalam pemrosesan. Walau bagaimanapun, kelemahan pengisi konduktif termasuk penumpuan nanopartikel dan sensitif kepada kelembapan persekitaran akan menghalang kekuatan penagliran electkrik. Oleh itu, pengisi inorganik dalam nano saiz seperti alumina diperkenalkan untuk memasukkan ke dalam komposit polimer tidak organik yang konduktif untuk mengatasi isu-isu ini. Kekonduksian elektrik boleh diperolehi dengan menggunakan cara “volume resistivity testing”. Selain itu, penjelmaan Fourier inframerah (FTIR) dan teknik pengimbasan pembezaan kalorimeter (DSC) telah digunakan untuk menganalisis ciri-ciri elektrik dalam polimer komposit yang kondutif yang disediakan. Sampel Pani ( $6.69 \text{ Scm}^{-1}$ ) adalah lebih konduktif daripada sampel MWCNT, iaitu  $1.15 \text{ Scm}^{-1}$ . Walaubagaimanapun, kedua-dua sampel telah menunjukkan penurunan dalam konduktiviti. Tetapi, selepas pencampuran dengan nano saiz alumina, konduktiviti sampel telah distabilkan. Pada masa kini polimer konduktif digunakan untuk perlindungan untuk elektromagnet, perencat penghakisan, dalam electroluminescent dan paparan untuk telefon selular, untuk pelepasan statik, dan untuk pelbagai peranti pengesan. Akhir sekali, usaha

besar dalam bidang elektronik polimer telah membawa kepada perkembangan yang terbaharu dan ke arah peningkatan prestasi yang berterusan.

## ABSTRACT

Polymers are generally known as electrical insulators, but to enable their use in electronic applications, development of conducting polymer seems an interesting approach in recent years. For this purpose, one specific strategy is based on blending conventional polymeric insulator, HDPE with organic conductive fillers, polyaniline (PAni) and multiwalled carbon nanotube (MWCNT) to form conducting polymer composites, CPCs. The most basic mechanical mixing without modification on the original conductive fillers is used. This indigenous design guarantees an outstanding of electrical conductivity performance with low cost processing due to the advantage of polymer itself. However, the drawbacks of those conductive fillers including agglomeration of nanoparticles and sensitive to the humidity surrounding will inhibit the conductivity. Hence, nano-sized inorganic filler, alumina is called upon to incorporate into the CPCs to overcome these issues. Electrical conductivity is obtained by using the volume resistivity testing. Moreover, Fourier transform infrared (FTIR) and differential scanning calorimetry (DSC) techniques were used to characterize and analyse the electrical characteristics of the CPCs. In this case, PAni CPC shows higher conductivity than MWCNT CPC,  $6.69 \text{ Scm}^{-1}$  over  $1.15 \text{ Scm}^{-1}$ . However, conductivity of both CPCs is reduced after 6 weeks preparation. After incorporating with nano-alumina, the conductivity is then stabilised. CPCs are currently used for electromagnetic shielding, as corrosion inhibitors, in electroluminescent and cellular phone displays, for static dissipation, and for various sensing devices. Last but not least, immense efforts in the polymeric electronics field have led to unprecedented progress and to devices of ever increasing performance.

## **DEDICATION**

This thesis work is dedicated to my supervisor, Prof. Dr. Qumrul Ahsan, who has been a constant source of support and encouragement during the challenges of research study. This work is also dedicated to my senior Nurul Akmil binti Mustaffa for her consistent guidance. I am truly thankful for having you throughout this period. This work is also dedicated to my beloved parents and my partner, who have always loved me unconditionally and whose good examples have taught me to work hard for the things that I aspire to achieve.



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## LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURE

Al	-	Aluminum
ASTM	-	American Society for Testing and Materials
CB	-	Carbon Black
CNF	-	Carbon Nano Fiber
CNT	-	Carbon Nanotube
CPC	-	Conducting Polymer Composite
CSA	-	Camphorsulfonic Acid
DBSA	-	Dodecylbenzenesulfonic Acid
DSC	-	Differential Scanning Calorimetry
DWCNT	-	Double Walled Carbon Nanotube
EMI	-	Electromagnetic Interference
EMS	-	Electromagnetic Shielding
ESD	-	Electrostatic Dissipation
FTIR	-	Fourier Transformed Infrared Ray Spectroscopy
HCL	-	Hydrochloric Acid
HDPE	-	High Density Polyethylene
HMC	-	HDPE with MWCNT
HMCA	-	HDPE with MWCNT and Alumina
HP	-	HDPE with PANi
HPA	-	HDPE with PANi and Alumina
ICP	-	Intrinsically Conductive Polymer

KBr	-	Potassium Bromide
LED	-	Light Emitting Devices
MO	-	Metal Oxide
MPS	-	(3-Mercaptopropyl) Trimethoxysilane
MWCNT	-	Multiwalled Carbon Nanotube
PAni	-	Polyaniline
PE	-	Polyethylene
PEDOT	-	Poly(ethylenedioxythiophene)
PEO	-	Polyethylene Oxide
PMC	-	Polymer Matrix Composite
PMMA	-	Poly(methyl methacrylate)
PP	-	Polypropylene
PPP	-	Poly(Pphenylene)
Ppy	-	Polypyrrole
PS	-	Polystyrene
PTh	-	Polythiophene
PVA	-	Polyvinyl Alcohol
SI	-	Standard International
Sn	-	Stannum
SN	-	Sulfur Nitride
SWCNT	-	Single Walled Carbon Nanotube
THF	-	Tetrahydrofuran
$\mu\text{m}$	-	Micrometer
A	-	Ampere
A	-	Area
$\text{Al}_2\text{O}_3$	-	Alumina

C	-	Carbon
cm	-	Centimeter
eV	-	Electron Volt
G	-	Giga
g	-	Gram
H	-	Hydrogen
HRC	-	Hardness Value
I	-	Current
K	-	Kelvin
m	-	Meter
$\text{mAhg}^{-1}$	-	Discharge Capacity
Min	-	Minute
mL	-	millilitre
mm	-	Millimeter
N	-	Nitrogen
nm	-	Nanometer
O	-	Oxygen
$^{\circ}\text{C}$	-	Degree Celsius
Pa	-	Pascal
pH	-	Measurement of the concentration of hydrogen ions in a solution
R	-	Resistance
$R_m$	-	Measured Resistance
S	-	Siemen
T	-	Tera
t	-	Thickness

$T_c$	-	Crystallization Temperature
$T_m$	-	Melting Temperature
$U$	-	Potential difference
$V$	-	Volt
$W$	-	Watt
$Wt\%$	-	Weight Percent
$X_c$	-	Crystallinity Percent
$\Delta H$	-	Heat of Fusion
$\rho$	-	Resistivity
$\rho_v$	-	Volume Resistivity
$\sigma$	-	Conductivity
$\Phi_c$	-	Percolation Threshold
$\Omega$	-	Ohm

# CHAPTER 1

## INTRODUCTION

This chapter discusses the background study, problem statement, objectives and research scope of this project.

### 1.1 Background Study

In current trend of technology advancement in polymer industries, new components with low cost-high performance materials are highly demanding. Polymer matrix composite (PMCs) is widely developed due to its superior properties such as high strength-low weight ratio, good electrical insulation, capability to transfer load, and ease of processing. Additionally, another trend in advanced polymer industry which rapidly study by the researchers is the conducting polymers. In particular, it is a promising material in nano-scaled electronic devices which are advantageous over than metal.

Conducting polymer is the new class of material which can be categorized into two classes, intrinsically conducting polymers (ICPs) and conducting polymer composites (CPCs). ICPs are an organic polymer which can conduct electricity. It is discovered by Alan G. MacDiarmid, Hideki Shirakawa, and Alan J. Heeger over 150 years ago. It is known as semiconductor or “synthetic metal” as it can undergo insulator-conductor transition under different processing condition. Since then, intensive growth of research effort has been directed towards the development, synthesis, and characterization of new types of conducting polymer materials. (Song & Choi, 2013)

On the other ground, CPCs is the material in which conductive fillers are introduced into conventional insulating polymer matrix to enable electricity flow through the material. These organic-inorganic hybrids make up a new prospective material, owing to the advantageous properties of the polymer matrix and the embedded organic or inorganic particles. It is easily processed and thus considered more economically than ICPs in electronic application. As reported in Deng *et al.* (2014), a large variety of conductive fillers has been used to fabricate CPCs including most metals, carbonaceous fillers, metal fibres, metal-coated fibres and intrinsic conductive polymer.

Conjunctionally, High density polyethylene (HDPE) is one of the most commonly used thermoplastic in the world. (Khanam *et al.*, 2016) It consists of a linear structure with no or few side branches making it a denser polymer as well as more crystalline material. In that case, it has been a high strength yet lighter polymer than other PEs. As reported by Khanam *et al.* (2016) also, it exhibits excellent properties such as chemical inertness, near-zero moisture absorption, low coefficient of friction, low electrical properties and ease of processing and. Therefore, HDPE is widely used by researchers as polymer matrix in engineering composites (PMCs) because of its superior properties.

It is a well-known fact that, Polyaniline (PANI) is the most important industrial conducting polymer. (Molapo *et al.*, 2012) It is one of the ICPs which consist of diverse properties like relatively inexpensive monomer, easy preparation, and high yield of polymerization, environmental stability, and unique processability for device fabrication. (Nagaraju *et al.*, 2014) Moreover, it can be processed from solutions into thin film, with low synthetic cost which finds application in electronic and optoelectronic devices. Intensive studies have been done to the research of this class of material to improve its mechanical, electrical, thermal, gas barrier and other properties.

Moreover, with the growth of nanotechnology advancement, carbonaceous nano-fillers such as graphite, diamond and fullerene, and carbon nanotubes (CNTs) have established part of widespread research and challenging due to their superior