



**EFFECT OF SILANE BASED FUNCTIONALIZATION ON
THERMAL AND ELECTRICAL PROPERTIES OF
NANOGRAPHENE/NYLON 6,6 COMPOSITE**

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

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APPROVAL

This report is submitted to Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirements for the degree of Bachelor of Manufacturing Engineering (Material Department) (Hons.). The members of the supervisory committee are as follow:

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ABSTRACT

Nowadays, the increasing demand of electronic devices causes undesired electromagnetic interference (EMI) to occur and lead to many problems including interference or multifunctioning of device. To overcome the problem, injection moulded thermoplastic in form of housing component of EMI shielding effectiveness (SE) material which is in conductive or magnetic state was used. In this research, nylon 6,6 was used as the polymer matrix while nanographene platelets (NGP) was selected as conductive filler. However, nanographene tends to cause agglomeration to itself and polymer matrix. Thus, introduction of silane treatment as functionalization or coupling agent can reduce the agglomeration of nanographene and enhance the adhesion properties. Objective of this research is to study the effect of silane based functionalization on thermal and electrical properties of nylon 6,6/nanographene composites. This research involves several stages consist of material selection, formulation, dry mixed master batch, pre-dried process, compounding process, injection moulding, characterization and analysis of sample. In formulation stage, the optimum weight percentage of NGP was treated with various weight percentage of Vinyltrimethoxysilane (VTMS) then the mixture was mixed with optimum weight percentage of nylon 6,6 in homogeneous form at dry mixed master batch stage. The mixture of nylon 6,6 and NGP was pre-dried to avoid moisture. Compounding process was used to compound the mixture in pallet form followed with ASTM D638 type 4 standard injection moulded. Then characterization and analysis of samples were performed. The sample of NGP/Nylon 6,6 treated with 20% VTMS showed the most thermally stable and highest electrical conductivity. It was observed that, 20% VTMS have enough energy to help NGP disperse well and reduce agglomeration.

ABSTRAK

Pada masa kini, permintaan tinggi untuk peranti elektronik dimana mengundang kepada gangguan elektromagnet yang menyebabkan banyak masalah termasuk mengganggu peranti electronic lain. Untuk menyelesaikan masalah ini, suntikan acuan berbentuk komponen perumahan bahan keberkesanan melindungi gangguan elektromagnet dimana bersifat konduktif digunakan. Dalam penyelidikan ini, penggunaan nylon 6,6 sebagai matrik polimer dengan nanographene platelets (NGP) sebagai pengisi konduktif dipertimbangkan. Bagaimanapun, NGP cenderung kepada penggumpalan kepada dirinya dan matrik polimer. Jadi, pengenalan rawatan silane sebagai gandingan ejen akan mengurangkan penggumpalan NGP dan meningkatkan sifat lekatan. Objektif penyelidikan ini adalah untuk mengkaji kesan rawatan silane terhadap sifat haba dan elektrik nanographene/nylon 6,6 komposit. Penyelidikan ini melibatkan beberapa peringkat iaitu pemilihan bahan, penggubalan, “dry mixed master batch”, proses pra kering, proses mengkompaun, pengacuan suntikan, pencirian dan analisis sampel. Dalam peringkat penggubalan, peratusan berat optimum NGP dirawat dengan pelbagai peratusan berat Vinyltrimethoxysilane dan dicampurkan dengan peratusan berat optimum nylon 6,6 dalam bentuk homogen. Proses pra kering digunakan untuk mengelakkan kelembapan campuran. Proses mengkompaun ke bentuk pallet diikuti dengan “ASTM D638 type 4 standard” menggunakan pengacuan suntikan. Pencirian dan analisis sampel dijalankan. Sampel NGP/Nylon 6,6 yang dirawat dengan 20% VTMS menunjukkan paling stabil dari segi haba dan kekonduksian elektrik tertinggi. Ia telah diperhatikan bahawa dengan kehadiran 20% VTMS, ia mempunyai tenaga yang mencukupi untuk membantu NGP tersebar dengan baik dan mengurangkan penggumpalan.

DEDICATION

To my beloved family, especially my mother, Nazumi Binti Nawawi and my father, Samsudin Bin Sapei that always supporting spiritually throughout my life. Equally important, to my lecturer, and friends whose give motivation, guided and inspired me to complete this project successfully.

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TABLE OF CONTENT

Abstract	i
Abstrak	ii
Dedication	iii
Acknowledgement	iv
Table of Content	v
List of Tables	x
List of Figures	xi
List of Abbreviations, Symbols and Nomenclatures	xiv

CHAPTER 1: INTRODUCTION

1.1 Background of Study	1
1.2 Problem Statement	5
1.3 Objectives	6
1.4 Scope	6
1.5 Organization of Thesis	7

CHAPTER 2: LITERATURE REVIEW

2.1 Introduction	8
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2.2	Electromagnetic (EMI) Shielding	9
2.2.1	EMI Shielding Materials	11
2.2.2	Polymer Composite as EMI Shielding Material	12
2.2.3	Types of Conductive Filler	13
2.2.4	Functionalization of Conductive Filler	14
2.2.5	Advantages of Functionalization	14
2.3	Introduction to Graphene	15
2.3.1	Properties of Graphene	17
2.3.1.1	Density of Graphene	17
2.3.1.2	Optical Transparency of Graphene	17
2.3.1.3	Thermal Conductivity of Graphene	18
2.3.1.4	Electrical Conductivity of Graphene	18
2.3.2	Applications of Graphene	19
2.3.3	Nanographene	20
2.3.3.1	Morphological of Nanographene Platelets	21
2.3.3.2	Thermal Characteristic of Nanographene Platelets	23
2.4	Silane	23
2.4.1	Typical Silane Applications	25
2.4.1.1	Coupling Agent	25
2.4.1.2	Adhesion	25
2.4.1.3	Hydrophobing And Dispersing Agent	26
2.4.1.4	Crosslinking Agent	26

2.4.1.5	Moisture Scavenger	26
2.4.1.6	Polypropylene Catalyst	26
2.4.1.7	Silicate Stabilizer	27
2.4.2	Properties of Silane	27
2.4.2.1	Thermal Properties of Silane	27
2.4.2.2	Electrical Properties of Silane	27
CHAPTER 3: METHODOLOGY		
3.1	Introduction	29
3.2	Flow Chart of The Project	30
3.3	Materials	31
3.3.1	Nylon 6,6	31
3.3.2	Nanographene Platelets	33
3.3.3	Silane	33
3.4	Sample Preparation	34
3.4.1	Formulation	34
3.4.2	Sonication	35
3.4.3	Dry Mixing Process of Master Batch Samples	36
3.4.4	Pre-Dried Process	36
3.4.5	Compounding Process	37
3.4.6	Injection Moulding Process	38
3.5	Characterization and Testing Analysis	39
3.5.1	Morphological Analysis	40

3.5.1.1	Scanning Electron Microscopy (SEM)	40
3.5.2	Instrumental Analysis	41
3.5.2.1	Fourier Transform Infrared Spectroscopy (FTIR)	41
3.5.3	Thermal Testing	42
3.5.3.1	Thermogravimetric Analysis (TGA)	42
3.5.3.2	Differential Scanning Calorimetry (DSC)	43
3.5.4	Electrical Testing	44
3.5.4.1	Electrical Conductivity	44
CHAPTER 4: RESULT AND DISCUSSION		
4.1	Introduction	46
4.2	Characterization of Silane (VTMS)	46
4.2.1	Fourier Transform Infrared Spectroscopy (FTIR)	46
4.3	Morphological Analysis	51
4.3.1	Scanning Electron Microscopy (SEM)	51
4.4	Thermal Analysis	56
4.4.1	Thermogravimetric Analysis (TGA)	56
4.4.2	Differential Scanning Calorimetry (DSC)	58
4.5	Electrical Analysis	63
4.5.1	Electrical Conductivity	63
CHAPTER 5: CONCLUSION AND RECOMMENDATION		
5.1	Conclusion	65

5.2 Recommendation	66
REFERENCES	67
APPENDICES	

LIST OF TABLES

1.1	Common properties of nylon 6,6 (http://www.plastic-products.com/nylon66.htm)	4
2.1	Electrical Conductivity of Metals (Tech,1987)	11
2.2	Type of nanographene platelets (NGPs) (J. Koo et. al.,2013)	21
3.1	Data sheet of Nylon 6,6 Zytel® 101F NC010 (Dupont)	32
3.2	Properties of Nanographene (Terra Techno Engineering)	33
3.3	Various weight percentage of VTMS functionalized with 30 grams of Nanographene (NGP)	34
3.4	Optimum weight percentage ratio of nylon 6,6 blended nanographene with VTMS as functionalization agent	34
3.5	Setting Parameter of Injection Moulding Process	37
4.1	The functional group of NGP and VTMS	47
4.2	The comparison of Tg, Tm, Tc, and Xc of pure Nylon 6,6 and Nylon 6,6/Nanographene composites treated varying weight percentage of VTMS	59

LIST OF FIGURES

1.1	Shielded and unshielded cable from Electromagnetic Interference	1
1.2	Structural formula of nylon 6,6	2
1.3	Nylon 6,6	2
1.4	Structural formula of graphene	3
1.5	Nanographene platelets (NGP)	3
1.6	Structural formula of Vinyltrimethoxysilane (VTMS) (www.struktol.com)	5
2.1	Electromagnetic radiation vector (Guo et. al., 2013)	10
2.2	Scanning Probe Microscopy of graphene honeycomb lattice pattern	16
2.3	Type of graphene-based polymer composites (T. Das and S. Prusty,2013)	17
2.4	Photograph of graphene in transmitted light	18
2.5	Application of graphene-based polymer composites (Yoo et. al, 2008)	19
2.6	SEM micrograph and magnifications of (a) NPG-1;2.65kX, (b) NPG-2;2.09kX, (c) NPG-3;2.67kX, (d) NPG-4;2.24kX, (e) NPG-5;2.05kX, (f) NPG-6;2.03kX, (g) NPG-7;2.08kX, (h) NPG-8;2.09kX, (i) NPG-9;1.94kX, (j) NPG-10;2.2kX, and (k) NPG-11;2.08kX (J. Koo et. al.,2013)	22
2.7	Carbon VS Silane Chemistry (D. Corning,2009)	23

3.1	Research methodology flow chart	30
3.2	Ultra Sonication Probe (Polymer Lab, Block 53, Agency Nuclear Malaysia)	35
3.3	High Speed Dry Mixer (Polymer Lab, Block 53, Agency Nuclear Malaysia)	36
3.4	SINO Alloy Twin Screw Co-Rotating Compounding Extruder	37
3.5	Ray-Ran test sample injection moulding press (Polymer Lab, Block 53, Agency Nuclear Malaysia)	39
3.6	The specimen Test Piece (ASTM D638 type 4 standard)	39
3.7	ZEISS EVO 50 Scanning Electron Microscopy (SEM)	40
3.8	BRUKER FT-IR Spectrophotometer (Polymer Lab, Block 53, Agency Nuclear Malaysia)	41
3.9	Perkin Elmer Pyris 6 TGA Analyzer (Polymer Lab, Block 53, Agency Nuclear Malaysia)	42
3.10	Perkin Elmer DSC7 device (Polymer Lab, Block 53, Agency Nuclear Malaysia)	44
3.11	LCR meter (Agilent E4980A)	45
4.1	IR spectra of nanographene (NGP) treated with varying weight percentage of VTMS	48
4.2	IR spectrum of nanographene treated with 0% VTMS	49
4.3	IR spectrum of nanographene treated with 15% VTMS	49
4.4	IR spectrum of nanographene treated with 20% VTMS	50
4.5	IR spectrum of nanographene treated with 25% VTMS	50
4.6	The SEM images of Nylon 6,6/Nanographene treated with 0% VTMS	52
4.7	The SEM images of Nylon 6,6/Nanographene treated with 15% VTMS	53

4.8	The SEM images of Nylon 6,6/Nanographene treated with 20% VTMS	54
4.9	The SEM images of Nylon 6,6/Nanographene treated with 25% VTMS	55
4.10	TGA curve of rate of weight loss of Nylon 6,6/Nanographene treated with varying weight percentage of VTMS	57
4.11	DTG curve of rate of weight loss during degradation of Nylon 6,6/Nanographene treated with varying weight percentage of VTMS	58
4.12	DSC curve of pure Nylon 6,6	60
4.13	DSC curve of Nylon 6,6/Nanographene treated with 0% VTMS	61
4.14	DSC curve of Nylon 6,6/Nanographene treated with 15% VTMS	61
4.15	DSC curve of Nylon 6,6/Nanographene treated with 20% VTMS	62
4.16	DSC curve of Nylon 6,6/Nanographene treated with 25% VTMS	62
4.17	Compared Electrical Conductivity for Nylon 6,6 and varying VTMS treated Nylon 6,6/Nanographene composites	64

LIST OF ABBREVIATIONS

EMI	-	Electromagnetic Interference
RFI	-	Radio Frequency Interference
ESD	-	Electrostatic Discharge
SE	-	Electromagnetic Interference Shielding Effectiveness
ASTM	-	American Society for Testing and Materials
MPa	-	Mega Pascal
VTMS	-	Vinyltrimethoxysilane
NGP	-	Nanographene Platelets
GNP	-	Graphene Nanoplatelets
SEM	-	Scanning Electron Microscopy
TEM	-	Transmission Electron Microscopy
TGA	-	Thermogravimetric Analysis
DSC	-	Differential Scanning Calorimetry
DMA	-	Dynamic Mechanical Analysis
CNT	-	Carbon Nanotubes
QHE	-	Quantum Hall Effect
LED	-	Light Emitting Diodes
OLED	-	Organic Light Emitting Diodes

CVD	-	Chemical Vapor Deposition
pH	-	Potential of Hydrogen
CTE	-	Coefficient of thermal expansion
T_{ult}	-	Ultimate temperature
T_m	-	Melting temperature
T_g	-	Glass transition temperature
T_c	-	Crystallisation temperature
NG ₀	-	Nanographene with 0 weight percentage of conductive filler
NG _{0.3}	-	Nanographene with 0.3 weight percentage of conductive filler
VNA	-	Vector Network Analyzer

LIST OF SYMBOLS

σ	-	Graphene sheet conductivity
μ	-	Mobility
n	-	Acoustic phonons at a carrier density
$\text{cm}^2\text{V}^{-1}\text{s}^{-1}$	-	Square centimetre per volts second
$\Omega^{-1}\text{cm}^{-1}$	-	Per ohm centimetre
S/m	-	Siemens per meter
$^{\circ}\text{C}$	-	Degree Celsius
$^{\circ}\text{F}$	-	Degree Fahrenheit
%	-	Percent
Kg	-	Kilogram
H	-	Magnetic Field
E	-	Electric Field
volts/m	-	Volts per meter
amps/m	-	Ampere per meter
λ	-	Wavelength
nm^2	-	Square nano meter
mg	-	Milligram
mg/m^2	-	Milligram per square meter

wt%	-	Weight percentage
mL	-	Millilitre
ΔH_f	-	Heat of fusion
ΔH_f°	-	Heat of fusion of 100% crystalline
Hz	-	Hertz
$^\circ\text{C}/\text{min}$	-	Degree Celsius per minute

CHAPTER 1

INTRODUCTION

1.1 Background of The Study

In this modern era, the demand of the electronic devices, in various sectors such as military, commercial, entertainment, and industrial have been increasing. Due to this situation, undesired electromagnetic interference (EMI), or electromagnetic radiation, or radio frequency interference (RFI) that can cause of many problems including interference or malfunctioning of device has also increased. Generally, most common cause of EMI that always happen in daily routine is electrostatic discharge (ESD) which are distortion of television broadcast reception, radio static and clicking sound when light in switched on mode. Hence, the EMI may cause the receiving devices and may reduce it performance. Therefore, the effective shielding material also known as EMI shielding effectiveness (SE) material which is injection moulded thermoplastic in form of housing component is high in demanding nowadays. The EMI shielding functioning by preventing the interference incoming and outgoing to/from devices. Absorption and reflection is one of the acts of electromagnetic wave interference which is can be prevent by EMI shielding effectiveness. However, in order to shield the ESD or EMI, the EMI shielding material should be an excellent conductivity in properties where the more excellent the conductivity material, the more the can absorb, reflect, and transmit the electromagnetic interference (EMI).

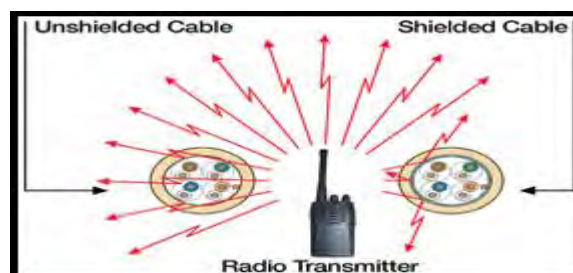


Figure 1.1: Shielded and unshielded cable from Electromagnetic Interference

Usually, housing components was made by plastics or rubber due to lightweight, low cost and ease to process. However, plastics and rubber are insulator materials to both heat and electrical which is transparent to electromagnetic interference (EMI) whereby do not absorb nor reflect EMI and most of energy wave not blocked. Thus, in order to shield the EMI by using this material, several methods can be considered to make the thermoplastic become the conductivity materials to both heat and electrical which is consist of conductive coating on plastic, compounding with conductive filler, and intrinsically conductive polymer. Among these methods, the most common method for EMI shielding is compounding polymer composites with the conductive fillers such as metal particles, metal flakes, stainless fibre, graphitized carbon particles, carbon fibre, graphene and many more. This method applied by incorporated the plastic materials and conducting material.

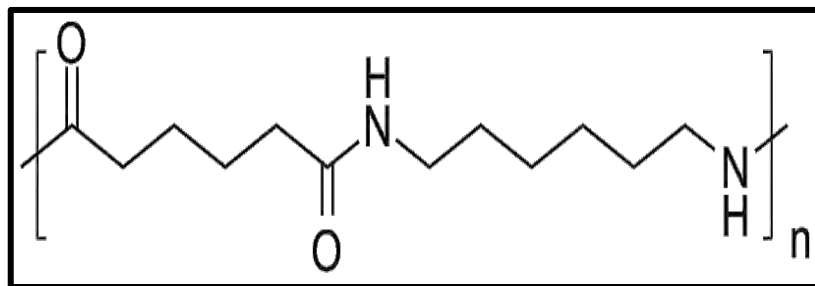


Figure 1.2: Structural formula of nylon 6,6



Figure 1.3: Nylon 6,6

Graphene with dramatic enhancement in properties at low filler content been very popular nowadays as a conductive filler. This two-dimensional honeycomb lattice graphene sheet conductivity is given by $\sigma = en\mu$ where mobility, μ have a theoretically limit to $200,000 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ and acoustic phonons at a carrier density, $n = 10^{12} \text{ cm}^{-2}$. Layer

thickness bulk conductivity of graphene is $0.96 \times 10^6 \Omega^{-1} \text{cm}^{-1}$ which is higher than the conductivity of metal copper that only $0.60 \times 10^6 \Omega^{-1} \text{cm}^{-1}$. Due to this reason, graphene is much more suitable material as a conductive filler thus make a graphene most suitable material as EMI shielding material than metal copper.

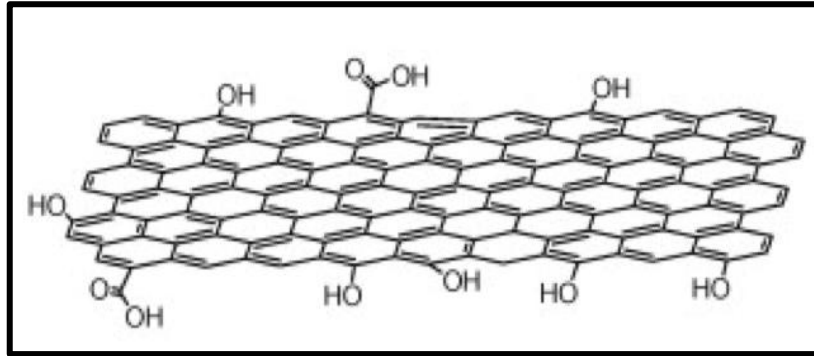


Figure 1.4: Structural formula of graphene



Figure 1.5: Nanographene platelets (NGP)

One of the most widely used polymer material as a component in many electronic devices is nylon 6,6 which has distinctive thermo-mechanical properties and has good resistance from severe atmospheric instability. (Pedro et. al.,2004). Generally, due to no free electron moving, nature of polymer material is good insulator but poor in electrical conductive. Beside very lightweight and easy to shape, it's also own better properties than steel coating EMI that easy to corrode and heavyweight. By adding the nanographene as conductive filler, nylon 6,6 enhanced in the electrical conductivity property, thus, make it an EMI shielding material that allow the current flow in the electrically shielding material.