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**PROPERTIES OF CHEMICALLY MODIFIED GRAPHENE
THROUGH THERMAL, COMPOSITIONAL AND
MORPHOLOGICAL ANALYSIS**

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

by

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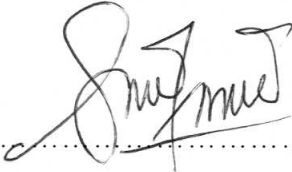
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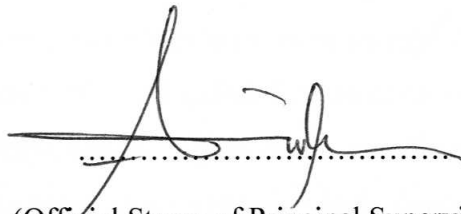
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ABSTRAK

Objektif utama kajian ini adalah melaksanakan modifikasi dan fungsionalisasi permukaan (penyalutan polimer) pada *Graphene Nanoplatelet* (GNP) dengan menggunakan bahan termoplastik melalui kaedah pencampuran larutan. *Graphene* telah digunakan secara meluas kerana prestasinya yang baik dan merupakan pengisi baru yang berpotensi untuk komposit polimer fungsian. Pengaruh modifikasi kimia pada taburan GNP dikaji melalui pemerhatian visual dan analisis kemikroskopan electron penghantaran (TEM). Kesan fungsionalisasi permukaan (formulasi & proses) terhadap sifat termal dan komposisi GNP termodifikasi dengan sifat-sifat dan kebolehcampuran bererti disokong oleh analisis melalui spektroskopi penjelmaan Fourier infra-merah (FTIR) dan permeteran kalori pengimbasan kebezaan (DSC). Modifikasi permukaan GNP memerlukan sonikasi di dalam larutan kloroform selama 1 jam untuk mendapatkan pencampuran seragam yang optimum. Penyalutan bahan polimer pada permukaan *graphene* yang telah dimodifikasi menunjukkan nisbah 1:4 memberikan tindak balas bukan kovalen yang terbaik. Setelah itu, komposit NR/EPDM di tetulang GNP-terfungsi dihasilkan untuk menunjukkan keserasian GNP-terfungsi dengan monomer getah NR / EPDM yang tinggi. Keputusan ini disokong oleh perincian dan analisis morfologi menggunakan kemikroskopan elektronimbasan (SEM) dan dapat disimpulkan bahawa propilena menunjukkan keserasian kimia terbaik dengan serakan *graphene* yang paling baik di dalam matriks NR / EPDM.

ABSTRACT

The main objective of this research is to perform surface modification or functionalization (polymer encapsulation) on graphene nanoplatelet (GNP) using thermoplastic material via solution mixing method. Graphene have been extensively used because of their excellent performance and is a new class of promising filler for functional polymer composites. The influence of the chemical modification on the graphene nanoplatelet dispersion was investigated by visual observation and Transmission Electron Microcopy (TEM) analysis. The effect of surface functionalization variables (formulation & process) to the thermal and compositional properties of modified graphene nanoplatelet with significant miscibility and properties were supported by Fourier Transform Infrared Spectroscopy (FTIR) and Differential Scanning Calorimetric (DSC). Surface modification of GNP requires sonication in chloroform for 1 hour to achieve the optimum uniform mixture. The encapsulation of polymeric materials with on modified graphene at ratio of 1:4 showed the best non-covalent reaction..Then, the NR/EPDM reinforced functionalized-GNP was prepared to show the high compatibility of functionalized-GNP with NR/EPDM rubber monomer. The results were supported by characterization and morphological analysis using Scanning Electron Microscopy (SEM) and it can be concluded that propylene monomer showed the best compatibility with high degree dispersion of graphene in NR/EPDM matrix.

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TABLE OF CONTENT

Abstrak	i
Abstract	ii
Acknowledgement	iii
Table of Content	iv
List of Tables	vii
List of Figures	x
List of Abbreviations	xii
List of Symbols	xiv
1. INTRODUCTION	1
1.1 Background	1
1.2 Problem Statement	3
1.3 Objective	5
1.4 Scope	5
1.5 Chapter Overview	6
2. LITERATURE REVIEW	7
2.1 Background of Nanomaterial	7
2.1.1 Zero-Dimensional Nanostructures	8
2.1.2 One-Dimensional Nanostructures	9
2.1.3 Two-Dimensional Nanostructures	9
2.1.4 Three-Dimensional Nanostructures	10
2.2 Polymer Nanocomposites	11
2.3 Carbon Material	11
2.3.1 The Graphene	12

2.3.2	Structural Properties of Graphene	12
2.3.3	Properties of Graphene	14
2.3.3.1	Chemical Properties	14
2.3.3.2	Mechanical Properties	14
2.3.3.3	Optical Properties	15
2.3.3.4	Electrical Properties	16
2.3.3.5	Electronic Properties	17
2.3.3.6	Thermal and thermoelectric properties	18
2.3.4	Applications of Graphene	20
2.4	Surface Modification of Graphene	21
2.4.1	Electrochemical Modification of Graphene	21
2.4.2	π - π Interaction of Graphene	23
2.4.3	Chemical Modification of Graphene	23
2.4.3.1	Covalent functionalization	23
2.4.3.2	Non-covalent functionalization	26
2.4.3.3	Stabilization in an ionic medium	27
2.4.3.4	Directly from graphite	29
2.5	Dispersion Method of Graphene	30
2.5.1	Solution Mixing Method	30
2.5.2	In situ polymerization	34
2.5.3	Melt blending	35
2.6	Analysis and Characterization	37
2.6.1	Thermal Analysis	37
2.6.1.1	Differential Scanning Calorimetric (DSC)	37
2.6.2	Compositional Analysis	37
2.6.2.1	Fourier Transform Infrared Spectroscopy (FTIR)	37
2.6.2.2	X-ray Diffraction (XRD)	36
2.6.3	Morphological Analysis	40
2.6.3.1	Transmission Electron Microcopy (TEM)	40
2.6.3.2	Scanning Electron Microscopy (SEM)	42

3.0	METHODOLOGY	45
3.1	Introduction	45
3.2	Raw Material	47
3.2.1	Graphene Nanoplatelet	48
3.2.2	Polypropylene (PP)	49
3.2.3	Polyethylene (PE)	50
3.2.4	Toluene	51
3.2.5	Tetrahydrofuran (THF)	52
3.2.6	Chloroform	53
3.2.7	Acetone	54
3.2.8	Ethanol	54
3.2.9	Natural rubber (NR)	55
3.2.10	Ethylene Propylene Diene Monomer (EPDM)	56
3.3	Sample Preparations and Processing	57
3.3.1	Dispersion study of GNP by dispersing the GNP in various solvent	58
3.3.2	Surface treatment of GNP by encapsulating GNP with various types of polymer	59
3.3.3	The incorporation of polymer-GNP with NR/EPDM	61
3.4	Analysis on Properties	66
3.4.1	Density Test	66
3.4.2	Differential Scanning Calorimetric (DSC)	67
3.4.3	Fourier Transform Infrared Spectroscopy (FTIR)	68
3.4.4	Transmission Electron Microcopy (TEM)	69
3.4.5	Scanning Electron Microscopy (SEM)	69
4.0	RESULT AND DISCUSSION	71
4.1	Introduction	71
4.2	Characterization of Raw Material	72
4.2.1	Density Test of Raw material	72
4.2.2	Transmission Electron Microcopy (TEM)	73
4.2.3	Fourier Transform Infrared Spectroscopy (FTIR)	75
4.2.4	Differential Scanning Calorimetric (DSC)	77

4.3	The dispersion study of Graphene Nanoplatelet (GNP) by dispersing the GNP in various type solvent	78
4.3.1	Visual observation of the dispersion Graphene Nanoplatelet (GNP)	78
4.3.2	Transmission Electron Microcopy (TEM) characterization analysis	83
4.4	Surface treatment of GNP to polymer matrix	86
4.4.1	Fourier Transform Infrared Spectroscopy Analysis	86
4.4.2	Differential Scanning Calorimetry (DSC) Analysis	92
4.5	The Incorporation NR-EPDM with GNP	95
4.5.1	Scanning Electron Microscopy (SEM)	95
5.0	CONCLUSION	
5.1	Introduction	101
5.2	Conclusion	101
5.3	Recommendation	103
	REFERENCES	105
	APPENDICES	

LIST OF TABLES

2.1	Functionalized graphene directly from graphite	29
3.1	Classification of Raw Material.	47
3.2	Characteristic and properties of GNP	48
3.3	General properties of polypropylene	49
3.4	General properties of polyethylene	50
3.5	The properties Toluene	51
3.6	The properties THF	52
3.7	The properties Chloroform	53
3.8	The properties Acetone	54
3.9	The properties Ethanol	55
3.10	The properties NR	56
3.11	The properties EPDM	57
3.12	Design matrix parameters of the Polymer/GNP encapsulation	61
3.13	Composition of GNP-NR/EPDM vulcanization	62
4.1	Density average of PE and PP.	72
4.3	The melting temperature and enthalpy of PE and PP.	77
4.4	The observation of the 30 minute of sonication time.	79
4.5	The observation of the 1 hour of sonication time.	80
4.6	The observation of the 2 hour of sonication time.	81
4.7	The observation of the 5 hour of sonication time.	82
4.8	The reduction agglomeration size of pure GNP and sonicated GNP	85

4.9	The functional group of PE, PP, GNP	87
4.10	The comparison of different melting temperature and enthalpy	92
4.11	The table of distribution of particle size under SEM in NR/EPDM blend with sample.	97

LIST OF FIGURES

2.1	A 2D honeycomb crystal is formed by placing a two atom basis to each point on a hexagonal lattice	13
2.2	Electrical conductivity of in situ CRGO/PS composites as a function of filler volume fraction.	17
2.3	Experimental electrochemical modification of grapheme	22
2.4	Photographs of (A) u-CCG sheets and (B) u-CCG sheets Dispersion in (a) water and (B) ethanol (c) DMF (d) DMSO (e) APTS respectively	25
2.5	Preparation of a homogeneous aqueous suspension of graphene sheets from a graphite stack	27
2.6	The schematic of ultrasonic treatment of the composite melt	31
2.7	The mechanism of FGS incorporation into polymer for melt blend technique	36
2.8	FTIR spectra of acidified graphite, GP and m-GP	38
2.9	FTIR spectra of Polyethylene	39
2.10	Schematic diagram of the layout of an analytical transmission electron microscope	40
2.11	(a–f) TEM and HR-TEM image of graphene	41
2.12	TEM images of (a) graphite and (b) graphene (c) High magnification TEM image of a graphene sheet,	42
2.13	Schematic diagram of the layout of a scanning electron microscope	43
2.14	SEM images of cry fracture surfaces of (a) PE–CNTs and (b) PE–GNs nanocomposites at 3 wt. % filler loading.	44

3.1	Flow chart of operational research framework	46
3.2	Graphene Nanoplatelet KNG-150	48
3.3	Polypropylene	49
3.4	Polyethylene (PE)	50
3.5	TolueneSYSTEM®ChemAR	51
3.6	THF	52
3.7	Chloroform SYSTEM®ChemAR	53
3.8	Natural Rubber (NR)	55
3.9	Ethylene Propylene Diene Monomer (EPDM)	56
3.10	Sample preparation by Ultrasonicator	58
3.11	The heated of polymer matrix	59
3.12	Polymer functionalized GNP	60
3.13	Drying oven	60
3.14	HAAKE RHEOMIX OS internal mixer machine	63
3.15	The product from the internal mixer process	63
3.16	The blend compound is placed in the mold	64
3.17	Gotech (GT 7014 – A) hot press machine	65
3.18	a) Specimen cutter machine and b) Specimen for tensile test.	65
3.19	Electronic Densimeter	67
3.20	Differential Scanning Calorimetric (DSC)	68
3.21	Fourier Transform Infrared (FTIR) and the specimen ATR	68
3.22	Scanning Electron Microscopy (SEM)	70
4.1	a) TEM image of pure GNP at magnification 20000X	74
	b) TEM image of pure GNP at magnification 10000X.	74
4.2	IR spectra of pure Graphene (GNP)	75
4.3	IR spectra of a) pure Polyethylene (PE) and b) pure Polypropylene (PP)	76
4.4	DSC graph of Pure Polyethylene (PE)	77
4.5	DSC graph of Pure Polypropylene (PP)	78

4.6	a)TEM image of sonicated GNP (mag. 30000X)	84
	b) TEM image of sonicated GNP (mag. 10000X)	
4.7	The encapsulation of PE with functionalized GNP	88
4.8	The encapsulation of PP with functionalized GNP	89
4.9	DSC graph of the encapsulation of PE with GNP	94
4.10	DSC graph of a the encapsulation of PP with GNP	94
4.11	The SEM image of NR/EPDM blend with a) pure GNP b) PE-f-GNP c) PP-f-GNP	96
4.12	SEM micrographs of fractured surfaces of NR/EPDM rubber blend with pure GNP	98
4.13	SEM micrographs of fractured surfaces of NR/EPDM rubber blend filled with PP-f-GNP under magnification blend filled with PE-f-GNP	99
4.14	SEM micrographs of fractured surfaces of NR/EPDM rubber blend filled with PP-f-GNP under magnification blend filled with PE-f-GNP	100

LIST OF ABBREVIATIONS

CCG	-	Chemically converted graphene
CO-890	-	Polyoxyethylene (40) nonylphenyl ether
CTE	-	Coefficient of thermal expansion
DSC	-	Differential Scanning Calorimetric
EPDM	-	Ethylene propylene diene monomer
FTIR	-	Fourier Transform Infrared Spectroscopy
GNP	-	Graphene Nanoplatelet
KOH	-	Potassium hydroxide
NR	-	Natural rubber
PE	-	Polyethylene
PP	-	Polypropylene
SEM	-	Scanning Electron Microscopy
TEM	-	Transmission Electron Microscopy
THF	-	Tetrahydrofuran
TS	-	Tensile strength
XRD	-	X-ray Diffraction

LIST OF SYMBOLS

$^{\circ}\text{C}$	-	Celsius
ms^{-1}	-	meter per second
%	-	percentage
kW	-	kilo watt
min	-	minute
kg	-	kilogram
mm	-	millimeter
μm	-	micrometer
s	-	second
nm	-	nanometer
g	-	gram
Hz	-	hertz
MPa	-	mega pascal

CHAPTER 1

INTRODUCTION

1.0 Background

Nanotechnology is utilized in various fields where the applications range anywhere from engineering to cosmetics. The leading attribute of this technology is the size that makes it so feasible to use in many different fields. The nanosize of the materials provides certain advantages like high surface area and low surface defects in the material, thus improving the material properties. For example in composites nanotechnology can be used in the filler material to increase the composite strength and to decrease the composite weight. Graphene is potential nanofiller that can dramatically improve the properties of polymer based composites at a very low loading and it has been attracting great interest due to its unique structure and properties. Moreover, polymer composites can be easily processed and fabricated into intricately shaped components with excellent preservation of the structure and properties of graphene using various processing methods. This is very important to make full use of the outstanding properties of graphene.

Graphene is a one-atom-thick planar sheet of sp^2 -bonded carbon atoms arranged in a hexagonal lattice with the basic structural unit of some carbon allotropes, including graphite, carbon nanotubes and fullerenes. It is believed to be the “thinnest and strongest material in the universe” and predicted to have remarkable physical and chemical properties (Jinhong *et al.*, 2012). Graphene, as nanofiller, may be preferred over other conventional nanofiller owing to high surface area, aspect ratio, tensile

strength (TS), thermal conductivity and electrical conductivity, EMI shielding ability, flexibility, transparency, and low thermal expansion (CTE). Furthermore, graphene has high conductivity and provides higher reinforcement than carbon nanotubes, low viscosity and non-toxicity (Stankovich., *et al.*, 2006).

The incorporation of graphene into the compound materials can provide them with the unique functions of graphene. Hereby a lot of graphene modified materials with enhanced performance are investigated and applied in different fields. In the automotive industry application, the incorporation of graphene as a nanofiller into NR/EPDM blend will produce thermally and electrically conductive on NR/EPDM composites that can help dissipate heat from the engine and prevent the accumulation of static charge. Natural rubber (NR) is widely used in shock and vibration isolators applications such as automotive engine mounts because of its high resilience (elasticity), high tensile, fatigue resistance, tear properties and low cost. In contrast, ethylene propylene diene monomer (EPDM) has excellent resistance to heat, oxidation and ozone, as well as to oils and chemicals, including ethylene glycol. NR/EPDM blend may provide a kind of rubber-rubber mixture, which may become technologically important as it combines the excellent outdoor properties of EPDM with the good elastic and dynamic properties of NR. In addition, the mixture of NR/EPDM with graphene will give good thermal conductivity also plays a vital role in improving the service life of engine mounts and could reduce costs and environmental impact.

Compared with CNT, graphene has a higher surface-to-volume ratio because of the inaccessibility of the inner nanotube surface to polymer molecules. This makes graphene potentially more favorable for improving the properties of polymer matrices, such as the mechanical electrical, thermal, and microwave absorption properties. More importantly, graphene is much cheaper than CNT, because it can be easily derived from a graphite precursor in large quantity. Therefore, graphene as a low cost alternative to CNT in nanocomposites is highly expected.

1.2 Problem Statement

Graphene has been touted as the "miracle material" of the 21st Century. The "thinnest" known material, graphene exhibits excellent electrical conductivity, mechanical flexibility, optical transparency, thermal conductivity, and low coefficient of thermal expansion (CTE) behavior (Kuila *et al.*, 2012). It has been widely used as nanofiller in the preparation of polymer nanocomposites materials. However, the preparation of homogeneous graphene-polymer composites entails some difficulties. To achieve high-performance of polymer nanocomposites, homogeneous dispersion of graphene nanoplatelet in the polymer hosts and proper interfacial interactions between the nanoplatelet and the surrounding matrix must be considered.

The main problem is, the nature of graphene itself is not compatible with the organic polymer matrix and the individual sheets tend to restack to form graphite through π - π stacking or tend to form irreversible agglomerates to their large specific surface area and the Van der Waals interactions between the interlayer of graphene, which lowers its effectiveness as a nanofiller for property improvements. Aggregation can be reduced by the attachment of other small molecules or polymers to the graphene sheets. Therefore, the prevention of aggregation was of particular importance for graphene sheets because most of their unique properties were only associated with individual graphene and keeping them well separated was required in the preparation and processing of graphene. To make the more easily dispersible in liquids, it is necessary to physically or chemically attach certain molecules or functional groups to their smooth sidewalls without significantly changing the nanomaterial desirable properties. In recent years, some modification has been used for obtaining graphene dispersion in aqueous media by functionalizing graphene surfaces.

The significant of the graphene has attracted so many attentions to be used in automotive industry. One of the possible utilization is graphene as filler for material of engine mounting. The graphene will tend to develop high shock absorption, thermally

conductive, with optimum mechanical performance and service environment resistance of NR/EPDM filled graphene composites. Thus, the chosen of the polymeric material such as Polypropylene and Polyethylene in this research is to show the high compatibility of propylene and ethylene monomer of NR/EPDM with the dispersion of graphene.

The processing of graphene with polymeric material involves choosing a proper blending method to reach a satisfactory dispersion of the nanofiller throughout the polymeric matrix. The three main methods of processing polymer encapsulated of graphene are in situ polymerization, solution mixing and melt blending. In situ polymerization is efficient method to uniformly disperse graphene and provide strong interaction between the graphene and polymer matrix. Generally, graphene is mixed with monomers or pre-polymers and then graphene/polymer composites with uniform dispersion and improved properties can be obtained by polymerizing the monomers or pre-polymers but the polymerization process is usually accompanied by a viscosity increase that hinders manipulation and limits load fraction. While, method is melt blending which is a versatile and commonly used method to fabricate polymeric materials, especially for thermoplastic polymers. However, this technique is that it may cause graphene buckling and even rolling or shortening due to the strong shear forces, thus reducing their aspect ratios, which is not favorable for achieving a low percolation threshold and high conductivity of the composites. For solution mixing method, graphene is generally dispersed in a solvent and then mixed with a polymer solution by mechanical mixing, magnetic agitation, or high-energy sonication. Subsequently, a composite can be obtained by vaporizing the solvent. This method is considered an effective means to prepare composites with uniform graphene dispersion. Hence, my research will be proceed with the solution mixing method due the polymeric materials to encapsulated with graphene are ideal choice because of their ease of fabrication by a low-cost room-temperature fabrication process.

Therefore, my research will focus on complicated efforts to utilize the surface functionalization on graphene, as well as in practical applications which require preparation of uniform mixtures of graphene with different organic, inorganic solvent and dispersion of polymeric materials with modified graphene by using solution mixing method.

1.3 Objectives

- To perform surface functionalization (polymer encapsulation) on graphene nanoplatelet using thermoplastic material via solution mixing method.
- To study the effect of surface functionalization variables (formulation & process) to the thermal and compositional properties of modified graphene nanoplatelet
- To study the morphological properties of NR/EPDM based rubber composite incorporated with modified graphene nanoplatelet.

1.4 Scope

This research will focus on properties of chemically modified graphene through thermal, compositional and morphological analysis via solution mixing method. The properties of pure graphene were compared with the chemically modified graphene. The findings were supported by the characterization and analysis on the thermal, compositional and morphological analysis of the sample are observed using Differential Scanning Calorimetric (DSC), Fourier Transform Infrared Spectroscopy (FTIR), Transmission Electron Microcopy (TEM) and Scanning Electron Microscopy (SEM).

1.5 Chapter Overview

This research is divided into five chapters that describe the analytical and experimental research performed. The First Chapter is an introduction to the study that brief about objectives, problem statement, significant of study and the thesis overview. Chapter Two present the literature review that relates to the theories on graphene and previous investigations on the issues and current method to be the chemically modified graphene. The important element that included in this chapter is about the materials and processing involved and also related experimental testing. Chapter Three, provides details explanations on the methodology used for overall research work, raw materials, characterization of the materials, samples preparation and procedure property analysis and testing. In Chapter Four, the result of the characterization and analysis on the properties for the chemically modified graphene were explained in details. The Chapter 5 summarizes major findings and offers some concluding remarks. The recommendation for future project also has been included in this Chapter 5.