EFFECT OF ELECTRON BEAM RADIATION ON THE DISPERSION OF GRAPHENE NANOPLATELETS IN NYLON66 BASED POLYMER COMPOSITES

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

By

MOHD FIRDAUS BIN AMRAN

B051410246

950622086419

FACULTY OF MANUFACTURING ENGINEERING

2018

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

Tajuk: **EFFECT OF ELECTRON BEAM REDIATION ON THE DISPERSION OF GRAPHENE NANOPLATELETS IN NYLON 66 BASED POLYMER COMPOSITES**

Sesi Pengajian: **2017/2018 Semester 2**

Saya **MOHD FIRDAUS BIN AMRAN (950622-08-6419)**

mengaku membenarkan Laporan Projek Sarjana Muda (PSM) ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

1. Laporan PSM adalah hak milik Universiti Teknikal Malaysia Melaka dan penulis.
2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.
3. Perpustakaan dibenarkan membuat salinan laporan PSM ini sebagai bahan pertukaran antara institusi pengajian tinggi.
4. *Sila tandakan (√)

☐ SULIT (Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysiasebagaimana yang termaktub dalam AKTA RAHSIA RASMI 1972)

☐ TERHAD (Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/ badan di mana penyelidikan dijalankan)

☑ TIDAK TERHAD

Disahkan oleh:

Alamat Tetap:
NO 6, JALAN PALMA RAJA 4,
TAMAN DATO' CHELLAM,
81800, ULU TIRAM, JOHOR.

Cop Rasmi:
Dr. Jeefferie Bin Abd Razak
Senior Lecturer
Faculty of Manufacturing Engineering,
Universiti Teknikal Malaysia Melaka,
Hang Tuah Jaya,
76100 Durian Tunggal, Melaka

Tarikh: 27 JUNE 2018

Tarikh: 2/7/2018

*Jika Laporan PSM ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali sebab dan tempoh laporan PSM ini perlu dikelaskan sebagai SULIT atau TERHAD.

DECLARATION

I hereby, declared that this dissertation entitled "Effect of Electron Beam Radiation on the dispersion of graphene nanoplatelets in nylon 66 based polymer composites" is the result of my own research except as cited in references.

Signature : ....

Author's Name : MOHD FIRDAUS BIN AMRAN

Date : 22 May 2018

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for the degree of Bachelor of Manufacturing Engineering (Hons).

The members of the supervisory committee are as follow:

…………………………………… 2/7/2018

(Supervisor) Signature & Stamp

Dr. Jeefferie Bin Abd Razak
Senior Lecturer
Faculty of Manufacturing Engineering,
Universiti Teknikal Malaysia Melaka,
Hang Tuah Jaya,
76100 Durian Tunggal, Melaka

ABSTRACT

Nowadays, the world is filled with electromagnetic radiation that can damage the internal component of electronic devices, but it can also affecting the human health such as insomnia and headache. The EMI shielding material was used by absorption or reflection of electromagnetic radiation. The nylon66 and graphene nanoplatelets have been used in this research as an EMI shielding material. Graphene nanoplatelets has been used as a conductive filler to improve the characteristic of nylon66 polymer which is an outstanding conductor of heat and electricity and has interesting light absorption behaviour. The purpose of this study is to prepare and characterize the effect of electron beam radiation on the dispersion of graphene nanoplatelets in nylon66 polymer matrix. The Nylon66/GNP nanocomposites were produced by dry mixing, followed by melt blending process, injection moulding and electron beam radiation applications. In this study, the percentage used of nylon66 is 100 wt% for control unfilled sample and 99.7 wt% for sample with addition of graphene nanoplatelets 0.3 wt% of filler addition. The test followed by formulating the sample with vinyltrimethoxysilane (VTMS) at 15% addition. Electron beam radiation was applied with various dosage which are 50, 100, 150, 200 kGy. For the testing are physical analysis, thermal analysis, morphological analysis, electrical properties and gel content analysis. It was found that the 100kGy dosage of EB radiation was significantly possessed outstanding physical properties in term of their intensity of crystallinity, transmittance and absorption peak during group arrangement and viscosity of the produced sample. Besides, it was found that the electrical conductivity of the sample that exposed to EB radiation has increased due to good dispersion of GNPs and molecular bonding between GNPs-nylon66. For the SEM analysis, 100kGy of EB radition shows good surface structure without any multilayer of the graphene nanoplatelets has been detected. The improvement in-terms morphological structure of graphene due to application of EB radiation has enhanced the interactivity of EMI shielding effectiveness for 50kGy and 100kGy dosage. At the end, this research has significantly important to be carried out as to provide the new ways to replace metal as an EMI shielding effectiveness.

ABSTRAK

Kini, dunia dipenuhi sinaran elektromagnetik yang boleh merosakkan komponen dalaman peranti elektronik, tetapi ia juga boleh menjejaskan kesihatan manusia seperti insomnia dan sakit kepala. Bahan perisai EMI yang digunakan sebagai penyerapan atau refleksi radiasi elektromagnetik dan telah digunakan di dalam penyelidikan ini sebagai bahan perisai EMI. Nilon 66 mempunyai ciri hebat yang merupakan penebat elektrik dan sifat mekanikal yang baik. Selain itu, nilon 66 ringan, tahan karat dan fleksibiliti yang lebih tinggi. Grafin telah digunakan sebagai bahan konduktif untuk meningkatkan ciri nilon 66 yang merupakan konduktor haba yang luar biasa dan mempunyai penyerapan cahaya yang menarik. Penyelidikan sebelum ini telah menggabungkan grafin dengan polipropilena untuk meningkatkan sifat-sifat mekanik bahan tersebut. Tujuan kajian ini adalah untuk menyediakan dan mengenal pasti kesan radiasi sinar elektron pada penyebaran grafin dalam Nilon66 / GNP dihasilkan oleh campuran kering, diikuti oleh proses campuran cair, pengacuan suntikan dan aplikasi radiasi rasuk elektron. Dalam kajian ini, peratusan yang digunakan dalam nilon66 adalah peratusan 100 berat dan peratusan 99.7 berat dan untuk grafin hanya digunakan peratusan 0.3. Ujian yang dilakukan dengan rumusan yang terlibat adalah viniltrimethoxysilane (VTMS) adalah sebanyak peratusan 15. Radiasi sinaran elektron digunakan dengan pelbagai dos iaitu 50, 100, 150, 200 kGy. Ia bermula dengan pemilihan bahan diikuti dengan rumusan bahan, persiapan sampel. Untuk ujian adalah analisis fizikal, analisis termal, analisis morfologi, sifat elektrik dan analisis kandungan gel. Didapati bahawa dos 100kGy sinaran EB mempunyai ciri-ciri fizikal yang sangat baik dari segi kekuatan keamatan cristalliniti, transmisi dan penyerapan semasa susunan kumpulan dan kelikatan sampel yang dihasilkan. Selain itu, didapati bahawa konduksi elektrik sampel yang dedah kepada radiasi EB telah meningkat kerana penyebaran ikatan molekul yang baik. Untuk analisis SEM, 100kGy menunjukkan struktur permukaan yang baik tanpa mana-mana tindihan bahan grafin. Peningkatan struktur morfologi grafin disebabkan penggunaan radiasi EB telah meningkatkan aktiviti keberkesanan perisai EMI untuk 50kGy dan 100kGy dos. Pada akhirnya, penyelidikan ini penting untuk dilaksanakan bagi menyediakan cara baru untuk menggantikan logam sebagai keberkesanan perisai EMI. Permohonan baru nilon 66 / GNP dengan penambahan radiasi EB telah meningkatkan sifat fizikal, kekonduksian elektrik dan keberkesanan perisai EMI.

DEDICATION

Dedicated to

My greatest mother and father, Norsafura Binti Ramli, Amran Bin Mustafa,

My supervisor, Dr. Jeefferie Bin Abd Razak,

My appreciated families and all my friends and colleagues for giving me moral support,

Cooperation, encouragement and also understanding.

Thank you So Much & Love You All Forever

ACKNOWLEDGEMENT

In the name of ALLAH, the most gracious, the most merciful, with the highest praise to Allah that I manage to complete this final year project successfully without difficulty.

My respected supervisor, Dr. Jeefferie Bin Abd Razak for the great mentoring that was given to me throughout the project. Besides that, I would like to express my gratitude to MINT lab manager, En. Iqbal Bin Syueb for their kind, advice and guidance as well as exposing me with meaningful experiences throughout the study.

Last but not least, I would like to give a special thanks to my friends who gave me much motivation and cooperation mentally in completing this report. They had given their suggestion and comments throughout my research. Thanks for the great friendship.

Finally, I would like to thank to everybody who was important to this FYP report, as well as expressing my apology that I could not mention personally each one of you.

TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURE

LIST OF ABBREVIATIONS

NyGNPs	-	Nylon 66/ GNPs nanocomposites
TRL	-	Thru Reflect Line
DAM	-	Digital Asset Management
ASTM	-	American Society for Testing and Materials
CBT	-	Cyclic Butylene Terephthalate
EMI	-	Electron Magnetic Interference
GNPs	-	Graphene Nanoplatelets
VNA	-	Vector Network Analysis
EBR	-	Electron Beam Radiation
FTIR	-	Fourier Transform Infrared Spectroscopy
MFI	-	Melt Flow Index
XRD	-	X-ray Diffraction
DMA	-	Dynamic Mechanical Analysis
SE	-	Shielding Effectiveness
Al	-	Aluminium
CNF	-	Carbon Nanofiber
PLA/PLLA	-	Polylactic Acid
POSS	-	Polyhedral Oligomeric Silsesquioxane
PP	-	Polypropylene
PC	-	Polycarbonate
PEI	-	Polyetherimide
TPU	-	Thermoplastic polyurethane

LIST OF SYMBOL

Cm	-	Centimetre
MPa	-	Mega pascal
N	-	Newton
%	-	Percentage
Wt%	-	Weight Percentage
kGy	-	Kilogray
mm	-	Millimetre
m	-	Metre
kHz	-	Kilohertz
°C	-	Celcius
Vol%	-	Volume percentage
S/m	-	Appearance power/metre
dB	-	Decibal
GHz	-	Gigahertz
μm	-	Micrometre
MeV	-	Mega electron volt
θ	-	Theta

CHAPTER 1
INTRODUCTION

1.1 Background of study

Electromagnetic interference (EMI) shielding material is a part of product that are commonly used in many electronic devices such as electronic systems and telecommunication devices. By increasing number of electronic devices nowadays, our environment was filled by electromagnetic radiation that can cause negative impact to human health such as insomnia and headache (Xia *et al.*, 2017). Besides, electromagnetic radiation also could destroy the internal component inside the electronic devices and could affecting the performance of the electronic devices by reducing their lifetime. Therefore, to overcome this problem, EMI shielding material is used to decrease the electromagnetic interference between electromagnetic field and sensitive component (Modak *et al.*, 2015). The function of EMI shielding is to absorb or reflect the electromagnetic radiation from attracting inside the electronic devices. The famous material types that was utilized as EMI shielding material is metal because it possessed higher shielding effectiveness and good mechanical properties (Kashi *et al.*, 2016). However, there are some weaknesses in metal such as heavy, expensive and prone to corrosion and difficult to manufacture. Besides, confidence level in metal decreasing due to their limitation in electromagnetic radiation absorber because it has a very shallow skin depth (Kashi *et al.*, 2016). The following Figure 1.1 depict the example of metal EMI shielding material in an electronic product casing.

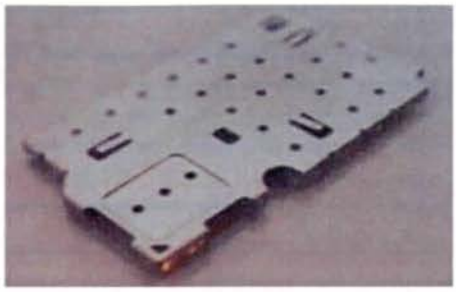

Figure 1. 1: Metal EMI Shielding material

The purpose of this study to develop a new generation of EMI shielding material by using nylon66 with the addition of conductive nanofiller material. Nylon66 is produced from combining of two monomers each containing six carbon atom, hexamethylenediamine and adipic acid. In this research, nylon66 are used because it possessed good wear resistance, higher tensile stress and good resistant to alkali. Besides, nylon66 also has longer molecular chains resulting in more hydrogen bonds formation. Hence, many researchers has investigated about nylon66 in recent years (Abdelbary *et al.*, 2013). Good thermal stability and mechanical strength of nylon 66 is discussed as an important engineering thermoplastic (An *et al.*, 2017). Nylon has been used because of their combination of toughness, stiffness, high melting point and chemical resistance attributes. However, Figure 1.3 depicts the molecular structure of nylon that was synthesized from the condensation polymerization.

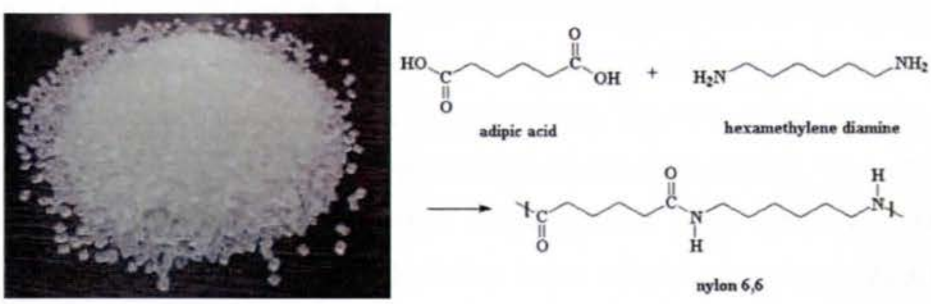

Figure 1. 2: Microstructure of nylon66

Graphene nanoplatelets (GNPs) is another important material phase that are used in this research. The properties structure of GNP attracts researcher because its arrangement of carbon atom was in honeycomb network structure had displayed great level of stiffness and strength, close to the theoretical limit (Ahmad *et al.*, 2017). Besides, the addition of GNPs into polymer could improving the mechanical properties of resulted nanocomposites. It also shows that, crystal nucleation and the microstructure of the nanocomposite can influenced by adding GNPs (Ahmad *et al.*, 2017). Next, the monolayer of GNPs is 100 times stronger than steel, so it can conduct electricity better than copper and improve the surface barrier properties because of it platelet like morphology. Nevertheless, process of polymer that containing GNP are still difficult because it uses melt compounding technique. Generally, the mixing process involved with low loading addition of GNPs and it was very difficult to guaranteeing it mixed properly (Antunes *et al.*, 2016).

Figure 1. 3: Graphene nanoplatelets and its structure

The sample of EMI shielding material was fabricated using nylon 66 by adding a filler material of GNPs. Both of these material will be mixed by using the high speed mixture. After that, the sample will be prepared by using melt blending twin screw co-rotating compounder to produce nanocomposite in pellets form. The twin screw extruder machine are consisted of two co-penetrating and self-cleaning identical screws. Thus, the shaft is rotated in the similar direction. The benefit of using a twin screw extruder is remarkable mixing ability, high level of process flexibility, better control of process parameter, high productivity and high economic opportunities. Injection moulding is processed for producing parts by injecting the material into a closed mould. Besides, injection moulding can performed by using any host material such as metal and glasses. The pellets will be gone through injection moulded into standard testing specimens via Ray-Ran test sample injection moulding press. The barrel temperature and mould were set up to 295°C and 95°C respectively. But, the sample was preheated in the barrel 10 to 15 minutes before to injection moulding process. Next, the sample will be exposed to the electron beam radiation to improve the agglomeration of single layer graphene and maintain the inherent properties of graphene, for optimum EMI shielding performances. The image of common agglomeration condition in some of polymeric matrices was stated in Figure 1.5.

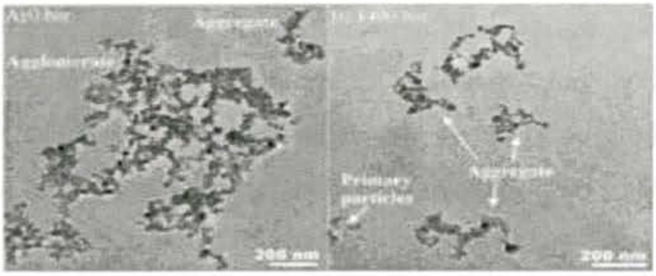

Figure 1. 4: Common agglomeration condition in some of polymeric matrices

In this study, the effect of electron beam radiation on the dispersion of graphene nanoplatelets in nylon66 composite were carefully investigated. Electron beam radiation is applied to improve the dispersion of GNPs within the nanocomposites. The correlation between the mechanical properties and electrical conductivity to the effect of electron beam radiation towards nylon66/ GNPs based nanocomposite were observed. The EMI test samples were characterize by physical, thermal, mechanical and morphological observation. The electrical conductivity will be tested to determine the suitability of polymer as an EMI shielding material, which is the reciprocal of electrical resistivity. Next, EMI shielding effectiveness was determined by a combination of two mechanisms reflection and absorption. Shielding effectiveness will be measured by using the Vector Network Analyser (VNA) instrument to the sample. Final analysis to the sample is a gel content. This testing is performed to determine the percentage of gel content by verifying the percentage of crosslink density of polymer in the presence of high energy radiation beam.

1.2 Problem Statement

There are many researches about the EMI shielding material either by using metal or polymer based composite. However, there is too little and no specific study has been performed specifically by using nylon 66 based polymer composite. Therefore, this study was aimed to investigate about the effect of electron beam radiation on the dispersion of graphene nanoplatelets in nylon 66 for the EMI shielding material application. GNPs were used as a nanofiller and will be mixed together with nylon 66 to produce EMI shielding material. By adding the nanofiller, the properties of nylon 66 may be enhanced and could increase the electrical conductivity of resulted nylon66/GNPs nanocomposites.

The addition of GNPs could enhance the mechanical properties of produced nanocomposites, particularly in their stiffness and strength characteristic. Besides, GNPs also has become famous as cost effective nanofiller for polymer matrix nanocomposites (Ahmad *et al.*, 2017). Properties of nylon 66 such as very lightweight and easy to shape also has attracted researchers to explore about this polymer based composite. In addition, nylon 66 is has higher strength and good wear resistance.

Generally, polymer matrix has an inclination to cause agglomeration with graphene nanoplatelets while mixing. The effect of agglomeration has an impact on percolation threshold and the effectiveness of electrical conductivity of graphene based polymer nanocomposites. Besides, agglomeration also decreases mechanical and conductivity of graphene due to particle does not disperse in mixing of nylon 66. In addition, strength between nylon 66 and graphene nanoplatelets also lesser.

1.3 Objectives

The main aim of this research is to study the effect of electron beam radiation on the dispersion of graphene nanoplatelets in nylon 66 based polymer nanocomposites which includes mechanical, physical, thermal and their morphological behaviours. The objectives of this research are as follows

1. To prepare nylon66/ GNPs nanocomposites by melt blending process using twin screw co-rotating compounder route
2. To evaluate the electrical conductivity, EMI shielding effectiveness, dynamic mechanical analysis and related physical properties of nylon 66/ GNPs nanocomposites with regards to the effect of electron beam radiation
3. To investigate the correlation between GNPs dispersion in nylon66 matrix with Electron Beam Radiation dosage through electron microscopy morphological observation

1.4 Research Scope

In this research, nylon66 was used as a polymer composite and graphene nanoplatelets as a nanofiller. For samples preparation, 100% and 99.7% of nylon 66 were used, while 0% and 0.3% graphene nanoplatelets were added. There are 15 samples that were produced which five sample only for unfilled nylon 66, five sample of added non-functionalised GNP and another five samples of optimum functionalised GNP with different application of EB dosage. After that, the testing was followed adding by formulation of VTMS at about 15% that is used to optimize the functionalised GNPs. The amount of EB dosage used are 0, 50, 100, 150, 200 kGy.

Firstly, the samples were prepared by mixing both of the GNPs and nylon66 in high speed mixer. After that, the mixed formulations will be mixed in the melt blending using twin screw co-rotating compounded to produce nanocomposite pellets. This method was chosen because of their mixing capability in achieving improved dispersion of the filler. Next, the pellet will undergoes injection moulding machine by injection molded into the standard testing specimen using Ray-Ran test sample injection moulding. Besides, electron beam radiation will be applied at each of the samples to possibly increase the dispersion and reduce the agglomeration of nylon 66/GNPs nanocomposite to enhance the electrical conductivity of the test sample for EMI shielding applications.

Apart from that, the EMI test samples were characterized by physical, thermal, mechanical and morphological observation. Besides, the electrical properties and shielding effectiveness also investigated. For morphological analysis, scanning electron microscopy (SEM) was utilized. In addition, Fourier Transform Infrared spectroscopy (FTIR), melt flow index (MFI) and X-ray diffraction (XRD) were used to investigate the physical analysis. Thermal analysis will be performed by using the dynamic mechanical analysis (DMA). At last, the electrical properties were calculated by measuring the electrical conductivity. Shielding effectiveness has also become the main objective of this experiment. The electromagnetic interference shielding effectiveness (SE) of conducting samples can be influenced by many aspects like electrical conducting, dielectric constant and thickness of material. Shielding effectiveness will be measured by using Vector Network Analyzer (VNA) instrumentation.

1.5 Organization of Thesis

The organization of the thesis are consisted of five (5) chapter and each of the chapter will explain information related to the research interest on the effect of electron beam radiation to the dispersion of graphene nanoplatelets (GNPs) in nylon 66 based polymer nanocomposites.

- Chapter 1 is about the introduction of this research. It consist of background of study, problem statement, objective, scope and organization of the thesis. The reader will be informed about material used, method utilized and the analysis that will be applied in this research.
- Chapter 2 consist the literature review of this project. It covers the explanation about the basic of nylon 66 and GNPs. Besides, it will explain about the process selected to perform the EMI shielding and process needed to enhance the dispersion of nylon 66/GNPs.
- Chapter 3 will discuss about methodological flow involved, the method chosen and characterization procedure on EMI shielding. The flowchart of overall research also are included in this chapter.