



**POLYURETHANE FILLED GRAPHENE NANOPATELETS FOAM  
NANOCOMPOSITES FOR SOUND ABSORPTION AND HEAT  
RESISTANCE**

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

by

**AHMAD ZULFADHLI BIN MOHD SAPELI**

**B051410250**

**950702-11-5179**

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Date :

## **APPROVAL**

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Engineering Materials) (Hons). The member of the supervisory committee are as follow:

.....  
**(Dr. Jeefferie bin Abd Razak)**

## ABSTRAK

Buih poliuretana (PU) dihasilkan oleh campuran Polyoil (bahagian A) dan isosianat (MDI) (bahagian B) dalam nisbah 1: 0.25. Tujuan penyelidikan adalah memperkenalkan cara mengembangkan buih PU yang diperkuat dengan graphene nanoplatelets (GNP). GNP sendiri mampu menyediakan dan memperbaiki sifat fizikal dan terma dari sampel yang dihasilkan. Penyediaan PU/GNP yang melibatkan kaedah ultrasonifikasi dan penyelesaian pengkompaunan. Prosedur penyediaan yang melibatkan beban GNP yang berlainan (0.00% berat, 0.25% berat, 0.50% berat, 0.75% berat, 1.00% berat). Komposisi optimum yang terbaik akan menjadi kritikal dan penting dalam menentukan sifat mekanik dan terma haba nanokomposit PU / GNP dengan berkaitan cara penyerapan bunyi dan ujian rintangan haba. Akhir sekali, morfologi permukaan PU / GNP akan dianalisis lagi dan terperinci dengan menggunakan alat pemerhatian SEM. Ujian ini adalah penting untuk mencari komposisi mana yang paling sesuai untuk meningkatkan penyerapan bunyi dan aplikasi rintangan haba dari nanokomposit PU / GNPs . Hasil daripada ujian penyerapan bunyi menunjukkan bahawa 0.75% berat daripada penambahan GNP menunjukkan hasil penyerapan terbaik ke atas buih nanokomposit PU / GNP yang hasil penyerapan menunjukkan 0.7610 daripada 1.000. Penyelidikan ini juga digunakan untuk mengawal pembaikan terma haba / nanokomposit PU / GNP. Selain itu, penambahan GNP ke dalam sampel akan meningkatkan rintangan haba dengan ketara kerana ia akan menyerap kira-kira 38% dengan 1.00% berat GNP dan aliran haba juga akan meningkat serta peningkatan nilai GNP. Ini menunjukkan bahawa GNP boleh menjadi bahan berskala nano untuk bahan maju yang terbaru dan meningkatkan sifat nanokomposit berbuih.

## ABSTRACT

Polyurethane (PU) foams are producing by the mixture of Polyoil (part A) and an isocyanate (MDI) (part B) in the ratio of 1: 0.25. The purpose of the research is to introducing the way of developing PU foams reinforced with graphene nanoplatelets (GNPs). GNPs itself is capable to provide and improve the physical and thermal properties of the produced sample. The preparation of PU/GNPs involving ultrasonication and solution compounding methods. The procedures of the preparation involving different GNPs loading (0.00 wt%, 0.25 wt%, 0.50 wt%, 0.75 wt%, 1.00 wt%). The best optimum composition will be critical and important in determine the mechanical and thermal properties of PU/GNPs nanocomposites foams by relate to the sound absorption and heat resistance testing. Lastly, surface morphologies of PU/GNPs will be analysed further and detail by using SEM observation tools. The testing is important to find at which composition is the most suitable to improve the sound absorption and heat resistance application of the PU/GNPs nanocomposites foams. The results from sound absorption test show that 0.75 wt% of GNPs addition show the best absorption coefficient towards the PU/GNPs nanocomposites foams which the absorption coefficient show 0.7610 out of 1.000. The research also applicable to control thermal improvement of the PU/GNPs nanocomposites foams. Besides, addition of GNPs into the sample will significantly improve the heat resistance as it will residue about 38 % with the 1.00 wt% of GNPs and heat flow also will increase as well as with the increasing of GNPs values. This show that GNPs can be new advance nanomaterial and improve properties of the nanocomposites foams.

## **DEDICATION**

Much love for

My late father, Mohd Sapeli bin Abdullah

My lovely mother, Hanita binti Mohd

My dear brother and sister, Qusyairi, Aina Marha, Lutfil hadi and Ariff Adli

And to all my friends for giving me moral support, money, cooperation, encouragement  
and also understandings

Thank You So Much & Love You All Forever

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## LIST OF ABBREVIATIONS

PU	-	Polyurethane
GNPs	-	Graphene Nanoplatelets
SEM	-	Scanning Electron Microscope
TGA	-	Thermogravimetric Analysis
DSC	-	Differential scanning calorimetry
ASTM	-	American Society for Testing and Materials
MDI	-	Methyl diphenyl diisocyanate
XRD	-	X-ray Diffraction
OH	-	Hydroxyl grup
PNC	-	Polymer Nanocomposites
CNT	-	Carbon Nanotube
HDI	-	Hexamethylene diisocyanate
TODI	-	3, 3' – dimethyl - 4-4' - biphenyl diisocyanate
AIBN	-	2-2' azobisisobutyronitrile
PBC	-	Polyurethane Bone Cements
HTPB	-	hydroxyl – terminated polybutadiene
GIC	-	Graphene intercalated
RGO	-	Reduced graphene oxide
CB	-	Carbon Black
MWCNT	-	Multiwall Carbon Nanotube
SOP	-	Standard Operation Procedure
SWNT	-	Single wall Nanotube



## LIST OF SYMBOLS

Mm	-	Millimetre
m	-	Meter
$\text{g/cm}^3$	-	Gram per centimetre cube
wt%	-	Weight Percentage
$^{\circ}\text{C}$	-	Degree Celsius
Nm	-	Nanometre
2D	-	Two Dimension
%	-	Percentage
MPa	-	Mega Pascal
$\text{kg/gm}$	-	Kilogram per gram
mL	-	Mililitre
$\text{m}^2/\text{g}$	-	Metre square per grams
$\text{g/mol}$	-	Grams per mol
mmol	-	Milimol
g	-	Grams
cm	-	Centimetre
mg	-	Miligrams
ml/min	-	Mililitre per minutes
$^{\circ}\text{C}/\text{min}$	-	Degree Celsius per minute
$^{\circ}$	-	Angle of degree
Hz	-	Hertz

# CHAPTER 1

## INTRODUCTION

This chapter provides full explanation about background of study, problem statement, research objectives, scope of study, significance of study, thesis organization and overall summary. The relevancy of conducting this research is comprehensively justified and accomplished. Furthermore, in this chapter depth of investigation are covered in the research scopes.

### 1.1. Research Background

First of all, polymeric foams is a leading member of wide range and highly diverse family of polymers or plastics. Polymers in the form of plastics are the most abundant products that are used in our daily life. (Salleh *et al.*, 2017) has portrayed that bio-based polyurethane (PU) was combined from squander cooking oil-based polyol for application as host in strong polymer electrolyte. According to (Deguchi *et al.*, 2017), a majority of consuming industries nowadays had used polymers that are synthesized from petroleum sources. There are around 80% of polymer these days are shaped utilizing petrochemical assets. Therefore diminishment of oil based feedstock will be the primary issue for polymer based industry so as to keep up the generation of PU based item later on.

Nowadays, polymeric foam have been widely used because of their user-friendly and outstanding mechanical, thermal and electrical properties. There are two kinds of foams that are known as unbending and adaptable foams. There are two different kinds of polymer foams with more particular utilize which are microcellular foams and elastomeric foams (Moll & Kumar, 2012). This research has selected polyurethane to be produced from waste cooking oil as it recently has attracted various application based on its properties. These polyurethane foams, all with to a great degree distinctive physical properties, are made by differing a fundamental expansion polymerization response including a diol or polyol, a diisocyanate, and water.

Recently, Polyurethane foam products are everywhere. The polyurethanes (PU) foams are generally utilized as protecting and center materials for furniture, cooling and solidifying frameworks, in housebuilding, and shipbuilding (Witkiewicz & Zielinski, 2006). Theoretically, PU foams is a mixture from the reaction between polyisocyanate and polyol matrix. From the reaction urethane bonds will be produced. The utilization of unbending foams is come about because of their low warmth conduction coefficient, low thickness, low water ingestion, moderately great mechanical quality. The utilization of unbending foams is come about because of their low warmth conduction coefficient, low thickness, low water ingestion, and generally great mechanical quality (Witkiewicz & Zielinski, 2006).

As a step to reduce the occurrence of error and failure, the properties of polymer foams are needs to be improved further. Improvement of polymeric foams can be done by adding filler materials, to produce a nanocomposite. Nanocomposite is a multiphase strong material where one of the stages has one, a few measurements of under 100 nanometers (Thomas *et al.*, 2016).

Nanocomposite are plan and make new materials with uncommon adaptability and change in their physical properties. Inclusion of nanofillerencounter will make polymeric foam structure become weak (De Oliveira *et al.*, 2013). Introducing nanocomposite means that the polymeric foams have through a new advance technology to improve the properties engineering materials.

Carbon based nanomaterials are among the filler that progressively becoming trending as its superior function meets the requirement strong and super lightweight material. Examples of carbon based nanomaterials are carbon nanofibers, carbon nanotubes, graphene nanoplatelets and many more. For this research, the scope will be limited into effects of graphene nanoplatelets addition to polyurethane (PU).

Foaming process could reasonably be expected include in methodology to expand the effectiveness of using graphene nanoplatelets over polymers and enhancing its scattering for those polymer grid (Antunes *et al.*, 2016). Scientist nowadays, have found that graphene nanoplatelets are claimed to have an extraordinary thermal stability, thermal conductivity, and high resistance towards heat degradation and sound absorption. Adding GNPs will make PU based nanocomposites become ten times greater than original PU sample. Thermal properties are vary with different mixture ratios. GNPs as nano-additives were added for thermal properties enhancement and also increase thermal enthalpy. GNPs are the promising material that have higher thermal conductivity and thermal enthalpy. Secondary, heat conductivity will have the ability will accelerate warm vitality stockpiling. thus improving energy efficiency. Heat conductivity of unfoamed nanocomposite expanded for two endless supply of 5wt% graphene (Gedler *et al.*, 2016). High thermal conductivity were obtained by increasing relative density and cell size. Expansion of graphite or graphene-like nanoplatelets to epoxy brings about direct increased of heat conductivity with expanding of nanoparticles fixation, achieving four times upgrades of heat conductivity for unfilled epoxy with addition of 5 wt% graphene loading by arrangement of solid interfacial communication amongst lattice and nanoparticle, for example it can be done through silane-crosslinking. Sound absorption was demonstrate by relationship between cell openness of polyurethane (PU) foams and sound absorption behaviour. Microcellular structure of fabricated PU foams were observe and sound ingestion coefficients were measured utilizing a Brüel and Kjaer impedance tube. Fabricated PU foams show good sound absorption compare to foam double mass density (Hyuk Park *et al.*, 2017).

## 1.2 Problem Statement

Recently, there are a lot of interest in environmental issues that lead to developing of new advanced material with multifunctional and eco-friendly. Demands for polyol have been increased rapidly and as we know a large portion of existing PU foams is creating by using petrochemical assets, for example, unrefined petroleum and characteristic gases.

However, hydrocarbon based assets are lacking and will be utilized until it reach the time limits. This type of assets isn't reasonable and won't not have the capacity to further support the the need of future age in the aspect of technology. As we can see and compare for renewable resources, petrochemical base hydrocarbon are more toxic and utilize high amount of energy that may effect and give problems to the greenhouse pollution. All of these problems will harm the condition, wellbeing and influencing the economy at a long run.

Polyoil is characterized as oligomeric that must contain at least two hydroxyl gathering (Sonnenschein & Wendt, 2013). In order to introduce an hydroxyl group in the structure of PU, the transesterification process are need to be done.

Transesterification will introduce vital element of hydroxyl group for PU production. Additionally, there will be an issue of monomer heterogeneity by the presence of double bond in triglyceride cross link structure that tend to minimize the PU properties. Synthesizing natural based polyol will minimizing the properties of PU foams.

Polyurethane are quite difficult to achieve simultaneously adaptability and branch of useful gatherings in a similar polymer. Thus, it may be lacking in advance applications, for example, adaptable hardware and adaptable sensors. Polyurethanes also exhibit hazards for the most part because of the nearness of destructive forerunners, for example, isocyanates and phosgene.

In order to gain the properties, carbon based nanomaterials need to be considered thus graphene nanoplatelets was selected based on the characteristic that is meets the requirement to improve the properties of polyurethane. Graphene nanoplatelets are the nanomaterials that have a range of thickness from 0.3 up to 100 nm and density of  $2 \text{ gcm}^{-3}$ . Homogenous dispersion of GNPs into polymer matrix could generate interesting phenomenon due to tremendous potential of nanofiller. It also have maximum surface contact area with polyurethane as it contribute to improve the thermal stability.

Satisfactory sound absorption and heat resistance using a polyurethane foam is an intriguing desire for achieving high fuel efficiency of vehicles. It has been demonstrate with a microcellular geometry manipulation. Sound protection properties of those composites will improve with expanding filler heaps of nanoclay (Bahrambeygi *et al.*, 2013). GNPs which are short heaps of 2D graphene sheets, have pulled in an extraordinary measure of consideration as a potential nanoreinforcement. Nanocomposites in light of GNPs have demonstrated aggressive practical properties similar to those of materials in view of CNTs. To improve the physical properties of nanocomposites, homogeneous dispersion of GNPs and ideal interfacial bond inside the polymer framework must be reacts to each other. Therefore, to attain potential of sound absorption and heat resistance, there must be an interaction within streamlining scattering and interfacial holding between the polymer network. Mechanical properties is related to the sound absorption properties based on condition of the sample, which as the amount of GNPs combine with PU increase then the sound absorption properties will improve that the material produce will absorb more sound that react to the material.

### **1.3 Objectives**

Before start the research, there must be consist of an objective that need to be fulfil to lead as a guide for a good result in the research

1. To prepare PU/GNPs nanocomposites foams with various GNPs loadings for sound absorption and heat resistance application.
2. To characterize and optimize the sound absorption and heat resistance behaviour for PU/GNPs nanocomposite
3. To relate the resulted performance of sound absorption and heat resistance of PU/GNPs nanocomposite with the fracture surface morphologies via SEM observation.

#### **1.4 Scope of Study**

This research scopes are as follow:

- a) Use as purchased GNPs – without surface treatment; and commercial part A and part B for PU development (1 : 0.25)
- b) Incorporate nanomaterials graphene nanoplatelets (GNPs) to be filled into polyurethane (PU) for improving the properties of PU based polymeric foams.
- c) Characterize the properties of PU/GNPs based nanocomposites that could improving the strength, sound absorption, and also heat resistance of PU based polymeric foams.

## **1.5 Noteworthy of Study**

Producing PU from polyol (part A) and an isocyanate(MDI) (part B) by adding GNPs will significantly improve the properties of polymer foams nanocomposite. Higher strength will be upgraded due to inclusion of GNPs. This study will focus on how GNPs enhance the nanocomposite foams strength when it is incorporated with polyurethane foams.

This study also focus on how GNPs will affect the heat resistance and sound absorption. Streamlining scattering and interfacial holding between the polymer grid and the nanofiller is critical for achieving the maximum capacity of the subsequent composite for sound absorption. PU/GNPs also lead to have a good heat resistance. The mechanism of how an study focus on how inclusion of smaller amount of GNPs might enhanced the properties of polyurethane foams will be studied. The result obtained are done by some testing to find its characterization.

## **1.6 Thesis Outline**

This report is mainly consisted of five major chapters to be discussed which are an presentation, writing survey, approach, result and discourse and the conclusion and suggestion. Firstly, the content of Chapter 1 was elaborate about an overview and details background studies of PU filled GNPs, problem statement of study, objectives, research scope, the significant of study, and summary of overall findings. Next, Chapter 2 will cover about literature review and related theory on PU based polyol, GNPs, properties of each material, polymer nanocomposite system, processing methods, and also methods that are used to characterize and optimize sound and heat resistance behaviour. Furthermore, Chapter 3 will detail the explanation about the preparation of samples, testing method and morphological observation of each samples. Then, the result and discussion for every test including the physical testing, mechanical, thermal testing and morphological characteristic were discussed thoroughly in the Chapter 4. Finally, Chapter 5 were concludes an overall research which later offering suggestion and recommendation in order to improve this research for further investigation.