



**UniMAP**

**The Effect of Filler Content and Size, and Different Ratio  
of Isocyanate/Polyol on the Properties of Wood  
Sawdust/Polyurethane Foam Composite**

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by

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A dissertation submitted  
in partial fulfillment of the requirements for the degree of  
Master of Science (Polymer Engineering)

**School of Materials Engineering  
UNIVERSITI MALAYSIA PERLIS**

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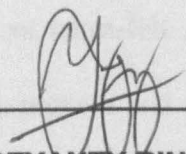
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## LIST OF ABBREVIATIONS

ASTM	American Society and Testing of Materials
CFC	Chlorofluorocarbon
MDI	diphenylmethane diisocyanate
MTIB	Malaysian Timber Industry Board
PEG	Polyethylene glycol
PEO	Polyethylene oxide
POE	Polyoxyethylene
PU	Polyurethane
SEM	Scanning Electron Microscopy
TGA	Thermogravimetric Analysis
F-11	Trichlorofluoromethane
USD	United State Dollar

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## LIST OF SYMBOLS

hr	Hour
kg	Kilogram
<i>m</i>	Mass
m <sup>3</sup>	Meter cubic
μm	Micro meter
μ	Micron
mg	Miligram
ml	Mililiter
mm	Milimeter
min	Minute
php	Parts per hundred polymer
%	Percent
rpm	Rotation per minute
s	Second
wt.%	Weight percent

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**KESAN KANDUNGAN DAN SAIZ ZARAH PENGISI DAN NISBAH  
ISOSIANAT/POLIOL KE ATAS SIFAT-SIFAT BUSA KOMPOSIT SISA  
HABUK KAYU / POLIURETANA**

**ABSTRAK**

Penyelidikan ini difokuskan kepada kajian sifat-sifat busa poliuretana terisi dengan sisa habuk kayu yang berbeza saiz zarah dan nisbah isosianat/poliol [NCO/OH] matriks. Komposit busa sisa habuk kayu (WSD) / poliuretana (PU) telah disediakan secara "free-rise casting" menggunakan kaedah sistem 'satu langkah'. Pengaruh saiz zarah pada pencirian fizikal, tingkah laku mekanikal, terma dan morfologi telah dikaji menggunakan WSD dengan dua saiz zarah yang berbeza (45  $\mu\text{m}$  dan 250  $\mu\text{m}$ ). Bagi kekuatan mampatan dan modulus mampatan, pengisi pada 5 phr untuk saiz zarah 45  $\mu\text{m}$  adalah optimum berbanding saiz zarah 250  $\mu\text{m}$ . Walau bagaimanapun, selepas melebihi nilai 5 phr, nilai kekuatan mampatan, modulus mampatan mula berkurangan. Pola yang sama seperti kekuatan mampatan dan modulus mampatan dapat diperhatikan untuk kekuatan lenturan dan modulus lenturan. Kekerasan komposit busa WSD/PU menurun dengan pertambahan WSD. Kajian pengimbas mikrograf elektron (SEM) menunjukkan bahawa saiz sel yang hampir sama taburannya diperolehi dengan penambahan pengisi (WSD) ke dalam komposit busa PU. Keputusan analisis terma-gravimetri (TGA) menunjukkan komposit busa WSD/PU mempunyai kestabilan terma yang lebih baik berbanding busa PU. Bagi komposit busa WSD/PU dengan perbezaan nisbah [NCO/OH], sifat mampatan, lenturan dan kekerasan menurun dengan penurunan kandungan isosianat (MDI). Kajian pengimbas mikrograf elektron (SEM) menunjukkan bahawa perbezaan nisbah isosianat dalam komposit busa WSD/PU menghasilkan perbezaan saiz struktur sel tertutup busa disebabkan oleh sambung silang yang lebih rendah antara isosianat dan poliol. Keputusan analisis terma-gravimetri (TGA) menunjukkan bahawa walaupun perbezaan nisbah [NCO/OH] penghasilan komposit busa WSD/PU mempunyai kestabilan terma yang lebih baik berbanding busa PU.

**THE EFFECT OF FILLER CONTENT AND SIZE, AND DIFFERENT RATIO  
OF ISOCYANATE/POLYOL ON THE PROPERTIES OF  
SAWDUST/POLYURETHANE FOAM COMPOSITE**

**ABSTRACT**

This research is focused to study the effect of wood sawdust (WSD) filled polyurethane (PU) foam with different particle sizes of WSD and different ratio of isocyanate/polyol [NCO/OH] PU matrix. WSD/PU composite foams were prepared by free-rise casting method using one-shot one-step system. The influence of the particle size on the physical characterization, mechanical behaviour, thermal and morphology has been studied using WSD with two different particle sizes (45 $\mu$ m and 250  $\mu$ m). Composite foams were tested for the evaluation of mechanical properties and cell morphologies as a portion of WSD present. For compression strength and modulus, filler loading at 5 php for 45  $\mu$ m particle size is the optimum compared to 250  $\mu$ m particle sizes. However, after exceeding a 5 php value, the properties started to decrease. The flexural strength and modulus, the trend was similar as observed in compression strength and compression modulus. The hardness of the WSD/PU foam composite decreased with the increasing of WSD. Scanning electron microscopy (SEM) study showed that regular cell size distribution can be obtained in addition of WSD filler into PU foam composite. The results of thermogravimetric analysis (TGA) showed WSD/PU foam composites has higher thermal stability compared with PU foam. For WSD/PU foam composite with different [NCO/OH] ratio, the compression, flexural and hardness properties decreased with the decreasing of isocyanate (MDI) content. Scanning electron microscopy (SEM) study indicated that different ratio of WSD in polyurethane foam led to different sizes of closed cell of foam due to lower degree of crosslinking between isocyanate and polyol. The results of thermogravimetric analysis (TGA) showed that even the ratio of [NCO/OH] is lower, the WSD/PU foam composites has higher thermal stability compared with PU foam.

## CHAPTER 1

### INTRODUCTION

#### 1.1 Research Background

Plastic foams are synthetic polymers that are used widely throughout the world for various applications because of their wide range of physical properties and suitability to most of the commercial processes. They incorporate various amounts of air or gas in the form of voids, cells, and configuration shapes. The resulting, lighter materials are referred to as foam, structural, cellular, blown, expanded, sponge, etc. There are rigid and flexible (elastomeric) foams.

Nowadays, foams have been widely used in many industrial. It has low density properties and made them excellent as thermal insulators and floatation devices. Their properties of lightness and compressibility made them ideal as packing materials and stuffing too. Other applications include filtration, aeration, sensors and sorbents (Ligoure et al., 2005).

Although plastic foams provide numerous benefits, they also have limited degradability when discarded after use and consequently can be an environmental problem. Adding biodegradable crosslink to the plastic foams could resolve the degradation problems.

Nowadays, interest in biodegradable foams is growing. Increasing prices on plastic resins made from oil and natural gas have placed more emphasis on alternative materials. Further, the environmental concern of waste reduction has created a unique

opportunity for the development of renewable polymers and using them for making polymeric foams. It is well known that the landfill problem associated with synthetic plastic products and shrinking space available due to population explosion has placed a greater emphasis on developing new polymeric materials that are either biodegradable or recyclable.

Tropical rainforests in South-East Asia are blessed with more than 15,000 different plant species of which about 3,000 species can be categorized as timber species. Major revenues of most countries in South-East Asia are derived from the exportation of wood products. In fact, Malaysia is a top exporter of wood products with the revenue of over USD50 billion in 2011 (MTIB and Statistical Dept. of Malaysia, 2011).

Wood, a natural cellulose composite material of botanical origin, possesses unique structural and chemical characteristics that render it desirable for a broad variety of end use. The level of suitability for a given end use (i.e. wood quality) is frequently determined by the wood response to imposed physical and chemical treatments. From the chemical perspective, wood tissue (including cells and intercellular substance) is a composite material constructed from a variety of organic polymers, namely cellulose, hemicellulose and lignin (Gu et al., 2012).

Since the early 1980s the interest in composite made from cellulose fibre has been growing. Rozman et al., (2003) have mentioned that the utilization of lignocellulosic composite has been attributed to several advantages such as low density, greater deformability, less abrasiveness to equipment, biodegradability and low cost. In recent years, these cellular materials have been improved by the incorporation of fillers such as fibres and particles. The final properties of these cellular solids depend on foam

density, the type and loading of fibres or particles, fibre length or particle diameter, and the geometrical structure.

The objective of composite development is to produce a product with performance characteristics that combine the positive attributes of each constituent component. Like other lignocellulosic material, wood is strong, lightweight, abundant, nonhazardous, and relatively inexpensive. Any lignocellulosic can be chemically modified to enhance properties such as dimensional stability and resistance to biodeterioration. This provides incentive for producing a variety of value-added products from different raw materials combined to provide improvements in cost or performance, or both (Brent et al., 1994).

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## 1.2 Problem Statements

PU foams are widely used because of their wide range of physical properties and suitability to most of the commercial process such as in furniture industry, packing, coatings, decorating, building construction, insulation, shoe industry and transportation. However, PU foams have limited degradability when discarded after use and consequently can be an environmental problem. By adding degradable filler to the PU foams could resolve the degradation problem in PU foams (Wang et al., 2008).

The increase demand of wood in many applications such as furniture, interior decorating and construction industries will significantly increase the generation of wastes. Among these waste, sanding dust and sawdust deserve special attention, as low density materials require greater storage room and can cause major pollution problems if they are not properly disposed (Fornasieri et al., 2011). These wastes are either being burnt which can lead to environmental problems like air pollution and emission of green houses gases, etc.

Considering the growing interest of natural fibers in the production of composite with different polymer matrices, wood wastes such as sawdust are an alternative source of fibers, thus can reduce the environmental impact of the large amounts of waste produced by the industries (Fornasieri et al., 2011). Therefore, the incorporation of wood sawdust as the filler in PU foams can reduce the magnitude of the environmental problems, enhance the properties in PU foams and reducing the cost of the PU foam. According to Rivera-Armenta et al. (2004), a solution for contamination problems of PUs due to their difficult disintegration to the environment is to include a natural material in the formulation in order to give some properties like biodegradation and avoid contamination.

### 1.3 Objectives

The objective of this research was to develop and characterize natural filler based polymer composite namely wood sawdust filled polyurethane (PU) foam composite. The primary objectives of the study are listed as:

1. To investigate the effect of wood sawdust filler size and wood sawdust filler loading in PU foam composite.
2. To study the PU foam composite with different ratio of isocyanate/hydroxyl group [NCO/OH].
3. To evaluate the mechanical properties of WSD filled PU foam composite by compressive, flexural and hardness testing and characterize the WSD filled PU foam composite with FTIR, thermal, and morphological properties.

#### 1.4 Scope of Study

This study aimed to develop a degradable foam using WSD as filler in PU matrix. WSD have the advantage that they are renewable resource and have marketing appeal. The fabrication of WSD/PU foam composite was carried out in three steps; the first was the WSD filler preparation, foam composite fabrication and foam composite testing and characterization. The foam composite was fabricated in a laboratory scale using one shot-one step process. The mechanical properties for WSD/PU foam composite were tested by following ASTM, including compressive, flexural and hardness test. The morphologies of WSD/PU foam composite were characterized by scanning electron microscopy (SEM) and an optical image analyzer. The thermal properties such as thermal stability were measured by thermal gravity analyzer (TGA). In addition, the chemical and structural changes of WSD/PU foam composite were determined by infra-red spectroscopy (FTIR).

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Introduction to Polyurethane

##### 2.1.1 Basic Polyurethane Chemistry

Polyurethanes are part of a very versatile group of materials that find uses in a wide range of applications, both domestic and industrial. Polyurethanes are widely used in many applications such as paints and lacquers, foam mattresses, medical implants, and industrial applications such as rollers, electrical encapsulation, engineering components, shoe soles, seals, and in the mining industry (Clemitson, 2008).

Polyurethanes were first investigated by Otto Bayer and his collaborators at I.G. Farben industrie A.G. in Germany and independently by Hoshino and Iwakura of the Tokyo Institute of Technology in Japan, as well as a research group at E.I. DuPont de Nemours Co. in the United States (Kaneyoshi, 2004).

Polyurethane is one of the thermoset materials. It is named because of the presence of the urethane linkage ( $-\text{NHCO}-\text{O}-$ ) which also known as carbamate in organic chemistry. Intensive research discovered that there were many modifications that could be made to the chemistry surrounding the urethane linkage (Clemitson, 2008).