PREPARATION AND CHARACTERIZATION OF POLYURETHANE FILLED BENTONITE COMPOSITES FOAMS

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

by

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APPROVAL

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

………………………………….

(DR. JEEFFERIE BIN ABD. RAZAK)

ABSTRACT

The bentonite mineral clay has its special characteristic as it is derived from the natural source. Previous researchers had incorporated the bentonite clay with polyurethane foams in order to produce the composites foams. Although there are previous research that utilized various types of clay in their study, there are still limited researches that combine bentonite with polyurethane foams in fabricating the functional high performance composites. The purpose of this study is to prepare and characterize the performance of bentonite at various loading by incorporating it with PU foams. The PU/Bentonite composites foams were produced by mechanical mixing, followed by foam growth and curing of the foams. The selection of optimum bentonite filler formulation were based on the polyol and isocyanate ratio that are fixed into 1:14 ratio. This study had utilized different weight percentages of filler loadings which are 0 wt. %, 0.50 wt. %, 0.75 wt. %, 1.00 wt. %, 3.00 wt. % and 5.00 wt % as to evaluate and observe the resulted physical, mechanical and thermal properties of PU/Bentonite composites foams. Next, the fracture surface morphology of the selected sample were analysed by scanning electron microscopy (SEM) observations. At the end of this study, mechanical and thermal performance of the produced composites were succeeded in certain remarkable improvement. At 1.00 wt. % of bentonite addition, it successfully improved the fracture toughness strength at about 120 % enhancement while at 0.50 wt. % of bentonite addition, about 77% of compressive strength were improved. Besides, the thermal stability of PU/Bentonite composites foams were achieved at 5 wt. % of bentonite addition as proven by the TGA analysis. This research is significantly important to be carried out as it could provide another alternative for advanced materials that utilizing the advantage of naturally derived material for many possible high performance applications.

ABSTRAK

Bentonite mempunyai ciri-ciri istimewanya tersendiri kerana berasal dari sumber semulajadi. Para pengkaji terdahulu telah menggunakan lempung di dalam kajian mereka untuk menghasilkan komposit poliuritana. Akan tetapi, penggunaan bentonite di dalam kajian yang sedia ada amatlah sedikit untuk menghasilkan komposit poliuritana. Kajian ini dijalankan untuk menghasilkan komposit poliuritana bersama bentonite sebagai pengisi di dalam komposit tersebut. Komposit PU/Bentonite dihasilkan menggunakan pengacau mekanikal untuk campuran bentonite bersama poliuritana diikuti dengan pengembangan span dan diletakkan di dalam oven untuk proses pengawetan. Pemilihan kadar campuran bentonite bergantung kepad campuran polyol dan isocyanate yang mana kadarnya telah ditetapkan iaitu 1:14. Kajian ini menggunakan berat yang berbeza untuk setiap penghasilan komposit span iaitu 0 wt. %, 0.50 wt. %, 0.75 wt. %, 1.00 wt. %, 3.00 wt. % dan 5.00 wt % untuk memerhati dan menilai tahap mekanikal dan haba komposit tersebut. Seterusnya, permukaan patah telah dilihat melalui SEM. Di akhir kajian ini, prestasi mekanikal dan juga haba PU/Bentonite komposit telah berjaya ditingkatkan. Penambahan bentonite pada berat 1.00 wt. % telah menunjukkan peningkatan prestasi kadar keliatan patah pad 120 %. Di smaping itu, kadar mampatan juga meningkat pada 0.5 wt. % penambahan bentonite sebanyak 77 %. Selain itu, kestabilan haba PU/Bentonite komposit telah dilihat pada 5 wt. % penambahan bentonite melalui penilaian TGA. Kajian ini adalah penting untuk menyediakan satu lagi bahan temaju menggunakan kelebihan bahan semulajadi sebagai bahan komposit aplikasi.

DEDICATION

I hereby dedicate this to
my beloved mother, Jamilah Ahmad
my late father, Shahrum Amlee Mohamed
my appreciated siblings, Syuhadatul Nabila, Syuhadatul Nadira and Mohamad Arief Akram.
my beloved friends, The Joyah squad and 4BMFB 13/17
Last but not least my Supervisor, Dr. Jeefferie Abd Razak who had given me guidance, moral support, encouragement and cooperation in completing this research.
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LIST OF ABBREVIATIONS

PU	-	Polyurethane
PMC	-	Polymer Matrix Composites
TDI	-	Toluene diisocyanate
TDA	-	Toluene diamine
ASTM	-	American Society for Testing and Materials
SEM	-	Scanning Electron Microscope
TGA	-	Thermogravimetric Analysis
DSC	-	Differential Scanning Calorimeter
LOI	-	Limiting Oxygen Index
K	-	Thermal Conductivity
XRD	-	X-ray Diffraction
PSA	-	Particle Size Analyzer

LIST OF SYMBOL

℃	-	Degree Celcius
wt. %	-	Weight Percentage
php	-	Parts by weight
μ	-	Micro
hrs	-	Hours
min	-	Minutes
λ	-	Wavelength
a	-	Alpha
Å	-	Angstrom
ml	-	millilitre
g	-	grams
cm	-	centimetre
mm	-	millimetre
kN	-	kilo Newton
ρ	-	Density
m	-	Mass
v	-	Velocity

CHAPTER 1

INTRODUCTION

1.1 Background

Polymer matrix composites (PMC) has been described as the combinations of polymer resin and reinforcement filler. There are many materials related to PMC including polyurethane (PU) as their matrix. Polyurethane is a generic name for compounds which obtained by step-growth polymerization. The reaction of polyurethane between two monomers occurs when polyols and isocyanate took place. In the year of 1983, polyurethane were first developed by Otto Bayer in Germany. The usage of polyurethane were expanded after that and are commonly used in industries. Nowadays, polyurethane have been used in wide range of applications such as adhesive, foams and coatings. The polyurethane based products are generally used and performed their functions in various service condition and environment. Some of the environment are not suitable to let the PU being exposed at longer exposure time, since it will cause major destruction to the structural integrity or impede their performances and properties. Towards the end, these may limits their failure life-time due to early failure. As for example, it is well known that the PU are not possessed good resistance towards heat. Hence, this situation may limits their usage under the elevated or higher service temperature.

The purpose of this study is to develop a new generation of PU based composites foams with enhance thermal properties by conducting the modification into their formulation through addition of bentonite clay filler. In this research, polyols used are palm oil based polyols, while the toluene diisocyanate is used as their isocyanate part, to ensure the production of PU based composites foams filled with bentonite mineral filler could be achieved. Palm oil was chosen as the green polyols alternative based on their accessibility, low cost and potential of renewability. In addition, bentonite based mineral filler were selected since it has the miracle characteristic of anti-flammable, higher resistance towards heat, cheap and able to perform as thermal or heat barrier for outstanding insulation purposes.

The demand in advanced materials with good thermal insulation properties had increased accordingly. One of the material that could exhibits good thermal insulation characteristic is foam type materials. As prior mentioned above, this research is to develop the polyurethane filled bentonite composite foams for thermal insulation purposes. Hence, one way to improve the thermal insulation behaviour of PU is by adding bentonite clay as filler. According to the previous study which using clay as filler, the research state that the addition of clay were acted as pores nucleation agent and at the same time was increased the matrix viscosity thus, reducing the rate of pore growth (Pravakar Mondal, 2006). This could give the possibility of creating the higher content of small size of pore with small mean average of closed cell in the making of PU/Bentonite composites foams. This could be an ideal conditions for foams structure that are specifically developed for the usage of good thermal insulation of PU based products. This morphological structure of foams are required as to provide an air gaps that will dissipate the heat in the event of thermal exposure. The heat entrapment situation could be avoided in this cases. Since PU foams is a thermoset polymer, it contains certain degree of thermoplasticity and able to fall off at elevated temperature (Fuest, 1996). The presence of bentonite will further enhance the insulation behaviour of this PU composites foams since it will destruct the growth of pores or cell size. The reduction of cell size yields smaller radioactive contribution and thus reduction in total thermal conductivity (Biedermann *et al.,* 2001).

In this research, the potential of bentonite to enhance the insulation behaviour of PU polymer based foams were investigated. The effects of bentonite filler loadings and the free rise molding technique towards the growth of PU foams pores or cell were further evaluated through the observation by scanning electron microscope (SEM) and optical microscopy (OM). The characteristics of produced foams will be correlated in this study with the thermal insulation performances and other supporting testing components, which are mechanical, and physical testing. For thermal insulation properties evaluation, various thermal testing were conducted, which are thermogravimetric analysis (TGA) to understand their heat degradation behaviour, differential scanning calorimeter (DSC) for understanding of heat flow characteristic, thermal conductivity testing by using simple heat transfer set-up to understand the heat flow efficiency and last but not least the limiting oxygen index (LOI) and UL 94 flammability testing to investigate the fire resistance characteristic of produced PU/Bentonite composites foams.

1.2 Problem Statement

There are many research about thermal conductivity of polyurethane based composite. However, there is too little and no specific study has been dedicated on thermal insulation for polyurethane based composite foams. Therefore, this study was aimed is to develop a polyurethane filled bentonite composite foams for thermal insulation purposes. Bentonite which acted as a filler will be mixed together with polyurethane to produce the composite foams. By adding the filler, the properties or performance of produced composite foams may be enhanced. According to Petrucci and Torre (2017), the usage of filler could improve the performance, saving cost and controlling the processability of produced composites materials.

In the era of globalization, researcher are trying to cultivate ecologically friendly materials from renewable resources (Patnaik *et al.,* 2015). Polymers has been widely used for various industrial applications. The development of roof as insulating material which used recycled polyester and sheep wool is one of the good example of bio-based materials utilization for insulation application. This action could minimize the carbon foot print while sustaining the environment (Patnaik *et al.,* 2015). The utilization of polyurethane as insulating material is limited due to the thermal degradation of PU which start flaming at the temperature above 250°C. When the PU materials undergone the thermal degradation, several hazardous chemical and gaseous might be emitted and evolved. This chemical might not be seen and it is not in the form of smoke (American Chemistry Council, 2014). This situation could harms the environment and people at surroundings.

Polymer foams possesses many application as they inherit a good mechanical, physical and thermal properties. As we know, polymer has a lower service temperature compared than other materials such as ceramic which is known as a refractory material. The service temperature of polymer is limited and cannot be as high as most off the polymer. It will be degraded or burnt when it reach their degradation point temperature. In order to increase its thermal insulation properties, mineral clay is introduced as a filler in the polymer foam. Polyurethane (PU) based foams are thermally unstable. In the event of heat exposure, the foams experience heat degradation which collapsing their mechanical and physical structure integrity. Hence, PU foams properties are need to be improved with the addition of mineral based filler that possessed good resistance towards heat.

In polymer foam the formation of pore structure is very important. To achieve a desired pore structure, the homogeneity of the pore structure must be controlled with careful. Presence of bentonite will be acted as anti-flammability agent which helps to stop the fire, but due to higher density of bentonite, it tends to precipitate within less density of PU foams matrix. This situation limits the dispersion of filler added which later could diminish the resulted performances of PU/Bentonite composites foams.

When dealing with the presence of bentonite filler within PU based composite foams, the problem in controlling the homogeneity of pore structure was really challenging. Foams with good thermal insulation behavior must possess closed cell morphological structure. This to ensure lower thermal conductivity (K) value in the event of heat transfer. However, presence of filler creates uncertainties for the structure of foams. Hence, this study will further investigates the effect of filler loadings on these issues. It is important to achieve closed pore structure in enhancing the thermal insulation properties and it is known that the pore structure are efficient in trapping the gas inside the pore and avoid the heat transfer by exothermic reaction. By controlling the amount of blowing agent and distribution in the foam, the closed pore structure can be obtained as to achieve lower value of thermal conductivity. Lower value of thermal conductivity indicates good thermal insulation characteristic. Blowing agent played a major role in controlling the foam density. The usage of higher percentage of blowing agent could result in the formation of gas bubbles and less dense foam. As the bubbles grow, it became unstable and one way to control it by usage of surfactant. The expansion of bubbles will determine the open or closed pore structure, pore size, shape and distribution. However, this situation is not that easy to control as the presence of filler could destruct the growth of cell. Hence, this study take an initiative to investigate the effect of bentonite filler loadings in promoting and controlling the quality and quantity of pores or cell formation in the structure of produced foams.

In conclusion, by taking this matter into consideration, the motivation in completing this research was fully justified. The presence of bentonite into the matrix of polyurethane could solve the issue of thermal or heat resistance of foams whereby by tackling this issue, it will certainly able to enhance the other resulted properties such as the mechanical and structural integrity PU/Bentonite composites foams.

1.3 Objectives

The main aim of this research is to study the engineering properties of polyurethane filled bentonite composite foam which includes mechanical, physical, thermal and their morphological properties. The objectives of the research are as follows:

a) To prepare PU/Bentonite composite foams by varying filler loadings.
b) To characterize the physical, mechanical and thermal behaviour of PU/Bentonite composites foams.
c) To observe the fracture surface morphologies of PU/Bentonite composites foams using SEM observation.

1.4 Research Scope

In this research, polyurethane were used as a matrix and bentonite clay as a filler. The palm oil based polyol and toluene diisocyanate were used as polyols and isocyanates respectively. The different loadings of bentonite filler which are 0 wt. %, 0.50 wt. %, 0.75 wt. %, 1.00 wt. %, 3.00 wt. % and 5.00 wt. % were used in the composites foams preparation. The polyurethane composites foams filled with bentonite were prepared via mechanical stirring method and hand stirring which involved a free rise molding procedure. This method was chosen because of its simplicity, lower cost, safe operation and faster manufacturing cycle. Then, the PU/Bentonite composites were heat treated in the oven for 30 mins duration.

Apart from that, the PU/Bentonite composite foam were characterize by mechanical properties, physical properties and thermal properties. The properties that will be evaluated are compression strength, fracture-toughness, water absorption characteristic, density, thermogravimetric analysis (TGA) and UL-94 flame retardant test. Besides, the fracture surface of polyurethane filled bentonite composite foam were observed by using scanning electron microscope (SEM) observation. At last, the correlation between the thermal insulation performance with foams morphologies were established with other supporting results from various mechanical, physical and thermal analysis.

1.5 Project Significance

The significance of the research are to gain knowledge about polyurethane composite foam by carrying out some related testing and characterization. Besides, this research was carried out to develop more information and also to understand about the role of bentonite clay when it incorporating to the polyurethane foams, as to improve their thermal insulation behaviour of produced polyurethane composite foams. In addition, higher strength of composites foams product will be developed from this research.

1.6 Organization of Thesis

This research is organized into several sub-topic and main chapters. In the FIRST Chapter, it provide the concise background of research, problem statement, scope, project significance, summary of methodology and thesis organization. The following are Chapter TWO briefly discussed about the literature review of the research which includes the related theories and past investigation on polyurethane, bentonite, polyurethane based composites foams, thermal insulation behaviour of polymer foams and materials characterization and properties evaluation. Chapter THREE were discussed about the methodological flow involved, the method chosen and the characterization procedure of produced PU/Bentonite composites foams. The flowchart of overall research starting from the beginning until the end of characterization process was also included in this chapter. Result and discussion obtained from this research will be included in Chapter FOUR. All the results or related figures from the testing and characterization of produced materials were placed and comprehensively discussed in this chapter. Last but not least, Chapter FIVE will conclude the entire research findings. The entire discussion from the beginning of the chapter which is the introduction until the result and discussion obtained will be summarized and concluded in this final chapter.