THE EFFECT OF FLAME RETARDANT ADDITIVE ON THE PROPERTIES OF KENAF/POLYURETHANE FOAM COMPOSITE

by

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LIST OF ABBREVIATIONS

ASTM	American society for testing and materials
ATH	Aluminum hydroxide
Br	Bromine
CO	Carbon monoxide
EFB	Empty fruit bunch
EG	Expandable graphite
F	Fluorine
FLO	Florean
FPF	Flexible polyurethane foam
FPNX	Fyrol PNX
FRIM	Forest Research Institute Malaysia
FRs	Flame retardants
FTIR	Fourier Transforms Infrared Spectroscopy
HCN	Hydrogen cyanide
H_2SO_4	Sulfuric acid
I	Iodine
IB	Internal bond
IFRs	Intumescent flame retardants
KCF	Kenaf core fiber
KF	Kenaf fiber
KFRC	Kenaf fiber reinforced composites
KFPU	Kenaf fiber polyurehane
KM_nO_4	Potassium permanganate
KOH	Potassium hydroxide
KRPU	Kenaf rubber polyurethane
LDPE	Low density polyethylene
LKTN	Lembaga Kenaf Tembakau Negara
LOI	Limiting oxygen index
MARDI	Malaysian Agricultural Research and Development Institute
MDI	Methylene diphenyl diisocyanate
MH	Magnesium hydroxide
MMT	Montmorillonite

MOE	Modulus of elasticity
MOR	Modulus of rupture
MSDS	Material safety data sheet
NFC	Natural fiber composite
PO	Polyolefin
PS	Polystyrene
PU	Polyurethane
PUF	Polyurethane foam
PVC	Polyvinyl chloride
SEM	Scanning electron microscope
TCEP	Tris (2-chloroethyl) phosphate
TCPP	Tris (chloropropyl) phosphate
TDCPP	Tris (1,3-dichloro-2-propyl) phosphate
TGA	Thermogravimetric analysis
TPSS	Thermoplastic sago starch
TRPU	Tire rubber polyurethane
ZnB	Zinc borate

LIST OF SYMBOLS

α	Alpha
%	Percent
°C	Degree celcius
cps	Centipoise
g/cm^3	Gram per cubic centimeter
GPa	Gigapascal
KPa	Kilopascal
MPa	Megapascal
mm/s	Millimeter per second
ml/min	Milliliter per minutes
N	Newton
nm	Nanometer
Pa.s	Pascal-second
phr	Part per hundred resin
rpm	Rotation per minutes
s	Seconds
T_{max}	Maximum temperature
$T_{5\%}$	Temperature at 5 weight percent loss

Kesan Bahan Tambah Perencat Nyalaan keatas Sifat-sifat Kenaf /Komposit Busa Poliuretana

ABSTRAK

Kajian ini tertumpu kepada penyediaan dan pencirian komposit busa poliuretana (PUF) dengan penambahan gentian kenaf dan perencat nyalaan, iaitu grafit boleh kembang (EG) dan aluminium hidroksida (ATH). Serat teras kenaf telah dikisar kepada saiz 63 μm digunakan sebagai pengisi semula jadi di dalam PUF pada jumlah yang berbeza iaitu 1, 3, 5 dan 7 wt%. EG (5, 10 dan 15 wt%) dan ATH (2, 4 dan 6 wt%) dimasukkan ke dalam PUF. EG dikisar selama 1 jam dan 30 minit dalam mesin bola gilingan yang menghasilkan kepingan EG dengan saiz 100 μm. Komposit ini disediakan dengan menggunakan poliol berasaskan campuran minyak sawit dan isosianat dengan nisbah 1: 1.1. Komposit PUF disediakan melalui proses pencampuran dengan menggunakan pengaduk mekanikal dengan kelajuan 2000 rpm. Kesan pembebanan pengisi dan perencat nyalaan dengan jumlah yang berbeza keatas PUF dikaji untuk menentukan masa kenaikan, ketumpatan, sifat mampatan, tingkah laku keboleh bakaran, analisis terma dan kajian morfologi. Hasil keputusan yang diperolehi menunjukkan bahawa PUF terisi gentian kenaf mengambil masa kenaikan lebih pendek berbanding dengan PUF terisi perencat nyalaan. Sementara itu, nilai ketumpatan meningkat dengan peningkatan jumlah pengisi dan perencat nyalaan ke dalam PUF. Untuk kajian mekanikal, kekuatan mampatan dan modulus komposit PUF berkurang kerana jumlah gentian kenaf dan perencat nyalaan meningkat. Kekuatan mekanikal meningkat untuk PUF/15EG dan PUF/KF/15EG disebabkan oleh luas permukaan yang besar yang disediakan oleh partikel-partikel EG yang dihancurkan. Berdasarkan ujian LOI dan keboleh bakaran, PUF dengan jumlah perencat nyalaan yang paling tinggi dan dengan kehadiran gentian kenaf (PUF/KF/6ATH/15EG) menunjukkan ciri-ciri perencat nyalaan yang cemerlang. Nilai LOI meningkat daripada 18.3 hingga 24.55 manakala bagi ujian keboleh bakaran pula menunjukkan bahawa komposit PUF dalam kedudukan mendatar memberikan kadar pembakaran yang rendah berbanding dengan kedudukan menegak, yang menunjukkan kepekaan yang rendah terhadap api. Kajian kestabilan terma komposit PUF mempamerkan tiga peringkat penguraian berlaku. Keluk TGA menunjukkan kestabilan terma meningkat dengan penambahan gentian kenaf dan perencat nyalaan ke dalam matrik PUF. Kajian morfologi mendedahkan bahawa struktur sel PUF kurang teratur kerana jumlah bahan tambah dan pengisi yang tinggi ditambah ke dalamnya. Mikrograf SEM juga mempamerkan bahawa partikel EG terletak di strut sel busa yang membawa kepada peningkatan sifat mekanikal komposit PUF. Sementara untuk sampel yang terbakar, mikrograf SEM mempamerkan kehadiran "struktur seperti cacing" yang telah terhasil dari pengembangan EG. Oleh itu, dapat disimpulkan bahawa gentian kenaf dan perencat nyalaan memainkan peranan penting dalam menentukan sifat komposit PUF.

The Effect of Flame Retardant Additive on The Properties of Kenaf/Polyurethane Foam Composite

ABSTRACT

This research is mainly focused on the preparation and characterization of polyurethane foam (PUF) with the addition of kenaf fiber and flame retardants, which are expandable graphite (EG) and aluminum hydroxide (ATH). Kenaf core fiber was ground into 63 µm in size and was used as the natural filler in the PUF at different amount which are 1, 3, 5 and 7 wt%. Various amount of EG (5, 10 and 15 wt%) and ATH (2, 4 and 6 wt%) was incorporated into the PUF. EG was milled for 1 hour and 30 minutes in a ball mill machine which in turn produces EG particles with 100 µm in size. The composites were prepared by the mixture of palm oil based polyol and isocyanate with the ratio of 1:1.1. The PUF composites were prepared through mixing process by using a mechanical stirrer with 2000 rpm speed. The effect of different amount of filler loading and flame retardant on the PUF was studied to determine the rising time, density, compression properties, flammability behavior, thermal analysis and morphological studies. From the results obtained, it showed that PUF filled with kenaf fiber takes shorter rise time compared to the PUF filled with flame retardants. While, density values were increased with the increasing amount of filler and flame retardant into the PUF matrix. For mechanical study, compression strength and modulus of the PUF composites was reduced as the amount of kenaf fiber and flame retardants were increased. The mechanical strength was improved for PUF/15EG and PUF/KF/15EG due to the large surface area provided by the pulverized EG particles. Based on LOI and flammability test, it was recorded that PUF with the highest amount of flame retardants and with the presence of kenaf fiber (PUF/KF/6ATH/15EG) shows excellent flame retardancy properties. LOI value was increased from 18.3 to 24.55 while for flammability test it shows that PUF composites in horizontal position give low burning rate compared to in vertical position, which represent low sensitivity to flame. Thermal stability study of the PUF composites display three decomposition stage occurred. The TGA curves indicate that the thermal stability was increased with the incorporation of kenaf fiber and flame retardants into the PUF matrix. Morphological studies revealed that the cell structure of the PUF was less irregular as higher amounts of additives and filler was added into it. The SEM micrograph also displays that EG particle was located on the foam cell strut which leads to improve the mechanical properties of the PUF composites. While, for burned sample, SEM micrograph displays the presence of "worm like structure" that was developed from the expansion of EG. Thus, it can be concluded that kenaf fiber and flame retardants plays crucial role in determining the properties of the PUF composites.

CHAPTER 1 : INTRODUCTION

1.1 Research Background

Composites define as a combination of two or more materials mixed together to obtain new material with different properties from the separate components (El-Shekeil et al., 2013). A polymer composite material consists of polymer as matrix while fiber acts as the reinforcement. Nowadays natural fiber reinforced composites have a growing demand due to its attractive properties over traditional fiber reinforced composites such as lower material cost, reduce energy consumption, renewability and ease of gaining (Azmi et al., 2012).

Recently, study on natural fibers has become an interesting topic among the researcher. The utilization of natural fiber especially lignocellulosic materials in polymer composites are increasing due to low cost, less abrasiveness to equipment, less irritation to skin and respiratory and acceptable specific strength and stiffness (Rozyanty et al., 2013). Lignocellulosic materials including kenaf, wood, rice husk and many others have been applied as reinforcing material or filler in the thermoplastic or thermoset composites.

Kenaf (*Hibiscus cannabinus, L.)* is a herbaceous annual plant that contain high-quality of cellulose (Hadi et al., 2014). Among lignocellulosic materials, kenaf is attractive due to its ability to grow quickly, abundance in supply, light in weight and low in cost. In Malaysia, the National Kenaf Research and Development Program has been formed to develop kenaf as a new industrial crop in Malaysia (Khalil & Suraya, 2011). In addition, kenaf plantations in Malaysia can be harvested three times in a year, which can be considered as the renewable source. Kenaf was widely applied in the production of paper, absorbent, vehicles, building materials (Webber III et al., 2002) and as fire retardant materials.

Polyurethane (PU) is a thermoset polymer that formed by the addition polymerization between polyisocyanate and polyfunctional alcohols (polyol polyether or polyol polyester) to form urethane linkages (Xue & Wen, 2014). Extensive studies have been focused on developing of bio-based polyols due to awareness on the environmental issues and depletion of petroleum resources (Yebo, 2016). In this research, palm oil based polyol has been used to replace petroleum-based polyol. PU products can be classified into various major groups which are rigid foams, flexible foams, elastomers, coatings and insulations (Sangeetha *et al.*, 2014).

Among various kinds of PU products, rigid polyurethane foam (PUF) was widely been used due to high mechanical properties with lighter density (Frances & Banon, 2014) and good resistance to solvent and chemicals. For many years, PUF has been applied in a large number of applications such as automotive interiors, packaging (Gaan et al., 2015), constructions (Sripathy & Sharma, 2013) and thus play important role in industry and daily life. However, the major drawbacks of PUF are easy to ignite and the flame spread quite fast if exposes to fire (Jiao et al., 2013). During fire, polymer loss its weight, damage costly equipment and chokes people by releasing toxic and acidic gases like carbon monoxide (CO) and hydrogen cyanide (HCN) (Modesti & Lorenzetti, 2002).

The use of flame retardants as the additive to minimize fire hazards in polymeric materials is highly efficient. The common flame retardants used in polymeric materials are inorganic, phosphorus and halogenated compounds (Sain & Panthapulakkal, 2004). Generally, halogen flame retardant was used due to its effectiveness in controlling the burning. However, the gas emissions from the burning of polymer with halogen flame retardants are hazardous and toxic to humanity (Onuegbu et al., 2012). Regarding to the bad health and environmental problems, several brominated and chlorinated phosphorus flame retardant has been banned in the past (Betts, 2008).

Nowadays, much attention has been given to halogen free flame retardant as it able to improve the flammability characteristics which delay or slowing down the rate of burning and reduce the toxic gas releases from the burning of polymer. The results from extensive studies showed that expandable graphite (EG) and aluminum hydroxide (ATH) is classified as halogen free flame retardant. Therefore, in this work the researcher attempt to study on the influence of various types of halogen free flame retardant system which is EG and ATH in kenaf/PUF composites on its physical, mechanical and thermal properties.

Expandable graphite is an intercalated graphite compound in some oxidants such as potassium permanganate and sulfuric acid which are inserted between the carbon layers of graphite (Jin et al., 2014). On the other hand, it is also classified into intumescent flame retardants (IFRs) system that works by expansion and generation of voluminous insulating layers (Chen et al., 2010), improving flame retardant properties of polymeric matrix and leads to the formation of char layer. The EG expands hundred times from its original size and covers the entire burning surface by its worm like structure. In addition, EG reduces the mass loss, heat releases, anti-dripping properties, smoke and toxic gas released (Jin et al., 2014).

Research on incorporating of inorganic flame retardant with EG to enhance the flame retardant efficiency in polymer has been done since last 10 years. Combination of additive and reactive flame retardant can lead to additive, synergistic and antagonistic effect. In addition, synergistic effect can be produce when the specific flame retardants were combined together (Lomakin & Zaikof, 2013). ATH is nontoxic, low cost, environmental friendly and smoke suppressing halogen-free flame retardant additives (Jin et al., 2014). Furthermore, incorporation of ATH as the flame retardant able to improve the formation of char in polymer. ATH works by releasing significant amount of water content and metal oxides at high temperature and absorbing heat from combustion zone (Sain & Panthapulakkal, 2004). In addition, metal oxide produce acts as a protective layer on polymer surface that shielding the polymer from further decomposition (Rakotomalala et al., 2010).

1.2 Problem Statement

Depletion of petroleum resources and escalating in price of petrochemical based materials is inducing to the production of polymeric materials by using natural resources. In this research, kenaf fiber was used as the filler while palm oil based polyol was used to replace petroleum based polyol in producing PUF. In addition, the incorporation of natural fiber gives less sensitivity effect to the environment and human. Furthermore, natural fibers can decrease the landfill disposal problems because it can be degraded easily. While palm oil is the most common economical oil crops which can be used as an alternative way for the production of PU products.

Recently, the concern of researchers, manufacturers and consumers on fire safety has increased due to much loss of life and great economic lost that are caused by fire hazards. Flame retardant has been used as the additive to minimize fire hazards in polymeric materials. Among various types of flame retardants, phosphorus and halogenated compound was the most favorable in the past due to its efficiency to reduce flammability. However, those flame retardants producing dense and toxic smokes during combustion. This research will focus on the halogen free flame retardant which is EG and ATH to modify the flammability characteristics of PUF composites for thermal insulating purposes.

1.3 Research Objectives

The objectives of this research are as follows:

1. To investigate the optimum loading of kenaf fiber on physical and mechanical properties of kenaf/PUF composites.
2. To determine the effect of kenaf fiber and flame retardant additive on rise time, cell size and cell distribution of PUF composites.
3. To characterize the effect of kenaf fiber and flame retardant additive on physical, thermal, flame and mechanical properties of kenaf/flame retardant/PUF composites.

1.4 Scope of Studies

In this research, PUF were prepared by the mixture of palm oil based polyol and isocyanate with the ratio of 1:1.1. While, kenaf core fiber at 63 μm has been used as the filler in the PUF composites. The amount of filler used was varied from 1 wt%, 3 wt%, 5 wt% and 7 wt%. The mixture of polyol, isocyanate and kenaf fiber was stirred by using a high speed mechanical stirrer at 2000 rpm speed. The dimension of the closed mold used to obtain PUF bulk is 25 cm x 20 cm x 8 cm. The PUF composites were heated in a conventional oven at 80 °C for 2 hours to ensure the foam composites has been fully cured. Flame retardants, which are EG and ATH were used as the additive in the PUF composites. EG was milled by using a planetary ball mill machine with 450 rpm speed for 1 hour and 30 minutes to obtain the small particulate size of EG.

Physical properties of the foam composites were determined by using density test. While, compression test was conducted to determine on the mechanical properties of the PUF composites. For the morphological study of the composites, Scanning Electron Microscope (SEM) analysis was conducted to observe on the cell size and cell distribution of the foam composites. Thermogravimetric Analysis (TGA) was performed to study on the thermal properties of the foam composites. Limiting oxygen index (LOI) and flammability test were also conducted on the PUF filled with flame retardants to investigate on the flame retardancy properties of the PUF composites. Free rise method was conducted to determine on the rise time taken for the PUF composites. For this purpose, PUF composites were prepared in an open container with dimension of 11.50 cm x 6 cm.