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Effect of Halogen Free-flame Retardants on The Mechanical and Morphological Properties of Kenaf/Polyurethane Foam Composites

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Abstract. Kenaf/polyurethane foam (PUF) was synthesized form PUF and kenaf core fiber with expandable graphite (EG) and aluminum hydroxide (ATH) at varying amounts. In the production of polymer foam composites, the processing methods determine the properties of the final products. In this work, the potential of high energy milled on EG has reduced the EG platelet size to improve its dispersion in PUF matrix. From the results obtained, PU/KF/6ATH/10EG shows the highest mechanical properties, i.e. modulus and compression strength, while PU/KF/2ATH/5EG shows the lowest mechanical properties. Scanning Electron Microscope (SEM) shows the effect of adding kenaf fiber, EG and ATH in PUF to the foam cell structure and size.

1. Introduction

Nowadays, studies on natural fiber/ PUF composites are widely exploited among the researcher. PUF have been commercially used in variety of applications, especially for rigid PUF that widely applied as thermal insulating materials [1] due to low thermal conductivity and good mechanical properties. PUF was produced from the complex generation of cellular structure that is made up of solid and gaseous phases [2]. In some cases, more than one solid component is added as fillers or additives in order to improve the physical, mechanical or thermal properties of the PUF. Generally, PUF are produced from the reaction between polyol and isocyanate via addition polymerization [3]. Presently, most of the polyol used in the production of PUF was synthesized from petroleum sources.

However, depletion of petroleum sources and high cost of petroleum based materials has induced to the production of natural based polyol. Based on several researches done, palm oil was extensively used as the polyol in the production of PUF due to its price and production efficiency compare to any other commercial oils [4].

The addition of natural fibers as the filler in PUF has been extensively used due to their characteristics such as economical and environmental friendly [5]. Moreover, addition of kenaf (Hibiscus cannabinus, L.) offers various advantages such as enhancing on mechanical and thermal properties of PUF. In addition, the existence of lignin in kenaf fiber can provide free OH groups which can contribute to polarization between the fiber and matrix [6]. It is well known that the addition of filler or additives in PUF leads to increases in foam density and decreases the foam cell size [7]. In general, the filler should be uniformly dispersed in the matrix to obtain excellent mechanical properties [7] [8].

Natural fiber/ PUF composite is well known with its favorable properties such as light weight, low production cost [9] and excellent hardness. However, the poor fire resistance of PUF caused by its porosity, low density and the nature characteristics of organic cellular materials itself has restricted its application especially as fire resistance materials [10]. For this purpose, flame retardant was added into the PUF to minimize and improve flame retardancy of PUF. In recent years, there have been worldwide demands on halogen free flame retardants such as intumescent system and mineral flame retardants, as they are quite effective in flame protection.

Recently, aluminum hydroxide (ATH) was commonly used as mineral flame retardants, which can contribute to low production of smoke released. Besides non-toxic, ATH is low cost [11], odorless and easy to handle. However, high load of ATH is required to achieve good flame retardancy properties, which sacrifices the mechanical performances of the polymer. Thus, ATH was combined with expandable graphite (EG) to improve the flammability properties and lower the ATH loading in polymer matrix which improved the mechanical properties of the polymer [12]. EG is a graphite intercalation compound in which sulfuric acid (H_2SO_4) is inserted between the carbon layers of the graphite [13]. Extensive studies proved that incorporation of EG into PUF able to improve its flame retardancy properties. However, the inclusion of EG could disrupt the mechanical properties of the PUF composites. This is due to the bigger size of EG has deteriorate the cell structure of PUF as EG locates on the cell strut which leads to weaken the mechanical properties of PUF [14]. Therefore, EG particles have been pulverized to achieve fine size to obtain smaller particle size and thus improve the dispersion quality of EG in PUF [13].

Therefore, in this study, rigid PUF was prepared by replacing petroleum based polyol with palm oil based polyol. Herein, we reported the effect of kenaf fiber with halogen-free flame retardants on the mechanical properties and morphology of PUF.

2. Experimental

2.1 Materials

Polyether polyol with amine catalyst (Maskimifoam 9935B/35) is palm oil based polyol while isocyanate (Maskiminate 80) contains the mixture of Polymeric MDI with a concentration of 55% and Methylene Diphenyldiisocyanate with 45% concentration. Both materials were supplied by Maskimi Polyol Sdn. Bhd. Additives used for polymerization of PUF are aluminum hydroxide (ATH) and expandable graphite (EG). Both of the additives were manufactured by Sigma-Aldrich. Kenaf core fiber (*Hibiscus cannabinus L*) was supplied from Lembaga Kenaf Tembakau Negara (LKTN).

2.2 Preparation of filler

Kenaf core fiber was dried naturally under the sunlight to remove its moisture content. Then, the fiber was ground using a grinder and sieved with 63 μm sieve.

2.3 Preparation of additives

EG was pulverized in a high energy mill (planetary type), with 450 rpm speed for 1 hour and 30 minutes to obtain small particulate size of EG. The EG content was varied from 5, 10 and 15 wt%. While, ATH content was varied at 2, 4 and 6 wt%.

2.4 Preparation of composites

Kenaf/PUF composites were prepared according to the formulation in Table.1. The palm oil based polyol was stirred at 2000 rpm with a high speed mechanical stirrer and allowed to degass for 2 minutes. Then, kenaf fiber was added at a fixed amount which is 1 wt%. While, the amount of EG and ATH were varied as shown in the Table.1. All the particles were added and wetted in the polyol and was stirred continuously until a homogenous mixture was obtained. Lastly, the mixture was reacted with isocyanate and continuously stirred. The mixture was cast into a mold, covered and allowed to cure. The cured foam was removed from the mold after 24 hours. Then, the samples were placed in the oven for 2 hours at 80°C for post cure.

Table 1. Formulation for polyurethane foam compsites based on ratio 1:1.1.

Kenaf (wt%)	Expandable graphite (EG) (wt%)	Aluminum hydroxide (ATH) (wt%)				Polyol (g)	Isocyanate (g)
		1st series	2nd series	3rd series	4th series		
1	5	0	2	4	6	23.66	26.03
1	10	0	2	4	6	23.66	26.03
1	15	0	2	4	6	23.66	26.03

2.5 Mechanical test

Compression test for rigid cellular plastic was conducted under ASTM D1621-10 by using Instron 4206 machine. The cure PUF composites were cut by using a band saw machine with dimensions of 50 mm x 50 mm x 50 mm (length x width x thickness). Compression test was performed at room temperature with a constant crosshead speed at 10mm/min and the load was applied until the foam was compressed to approximately 80% of its original thickness (height). The samples were compressed in the direction parallel to foam rise. The strength and modulus of five specimens for a sample were measured and averaged.

2.6 Morphology

Scanning electron microscope (SEM) was conducted using JEOL JSM 6460 model. This analysis was done to determine the effect of additives and filler loading on foam cell size and distribution. It also reveals information about the sample surface morphology and mechanical properties. The samples were cut into 5 mm x 5 mm x 5 mm (length x width x thickness) and were coated with a very thin layer of platinum by using Auto Fined Coater to attain observation on the samples. Observation was made at 100 x magnifications.

3. Results and discussion

Figure 1 and Figure 2 represent the compression strength and modulus of PUF composites with varying amount of ATH. Generally, the achievement of good mechanical properties in filled PUF system was determined by the distribution and dispersion of filler in the polymer matrix [15]. Based on the results obtained, incorporation of EG into PUF matrix generates to a significant increase in the compression strength and modulus of the pure PUF. The improvement in mechanical properties of PU/15EG was due to the large surface area provided by the pulverized EG. In addition, EG is located on the foam cell struts as shown in Figure 3 (b) which can contribute to increases the strength and modulus of PUF composites.

Addition of ATH into PUF composites was drastically reduced the compression strength and modulus. This is due to disruption in the PUF system that was caused by rigidity of ATH instead the presence of kenaf and EG particles. However, the compression strength and modulus was increased at 4 wt% of ATH and further increases at 6 wt% of ATH. But, it decreased slightly at 15 wt% of EG for both compositions. Thus, it can be concluded that ATH did not able to show excellent mechanical properties at low amount. Whereas, it tends to show significant increases as EG was incorporated in the PUF composites. Based on the results obtained, it is observed that, PU/KF/6ATH/10EG gives the highest value for compression strength and modulus, while the optimum amount of EG incorporated in PUF is10 wt%. This is because during the processing of PUF composites, the addition of EG, ATH and kenaf fiber has resulted in great viscosity increases, especially for high EG and ATH content. This will lead to produce poor dispersion of filler and additives in the PUF matrix.

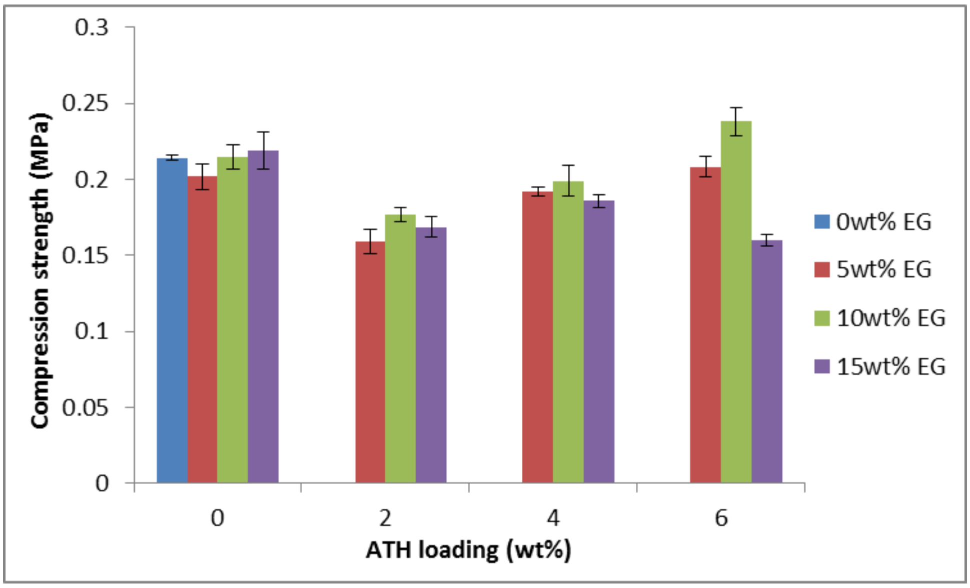

Figure 1. Compression strength of PUF composites.

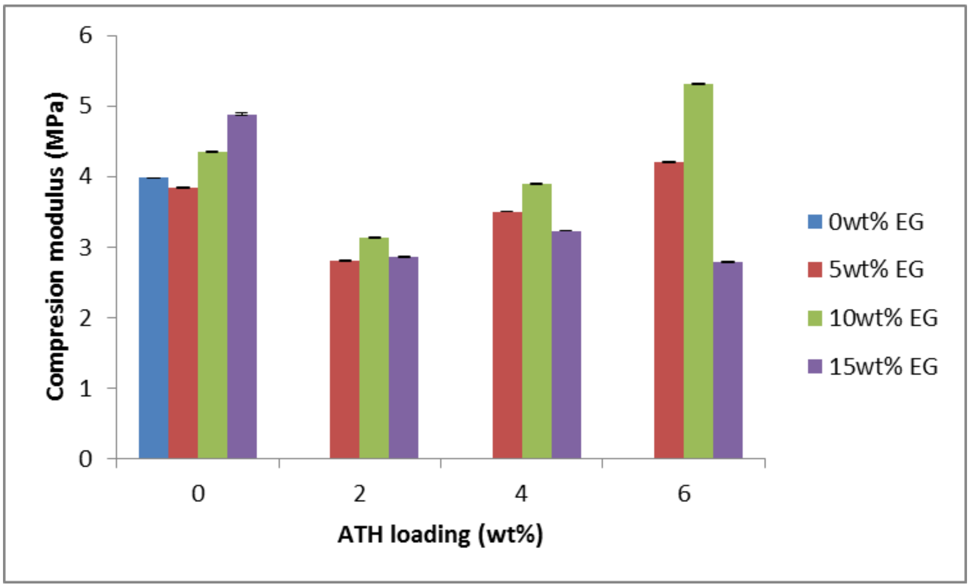

Figure 2. Compression modulus of PUF composites.

SEM was conducted in order to study on the morphology and cell structure of PUF composites. Figure 3 displays the SEM micrograph of rigid PUF composites. Based on Figure 3 (a), it can be seen that the cell shape of the pure PUF is approximately spherical. Besides that, the pure PUF cell size and cell distribution are nearly uniform with closed cell foam structure [6]. Figure 3 (b) represents PUF with 15 wt% of EG particles. According to Shi and his co-workers study [14], EG particles do not locate in the cell struts but between the cell walls due to their bigger size. Thus, in this work, EG particles have been pulverized to improve the distribution of EG in PUF matrix. From the image, it can be seen that EG particles was randomly distributed at the PUF cell struts which resulted to higher mechanical strength and modulus of the PUF composites. As shown in Figure 3 (c), the ruptured cell wall of PUF composites was mainly due to the rigidity of ATH particles. While, Figure 3 (d) shows the PUF structure becomes smaller and regular as 6 wt% of ATH and 10 wt% of EG was added into the PUF matrix. Generally, foam with regular and smaller cell size will possess better mechanical performance as proven in Figure 1 and Figure 2. Thus, the combination of EG and ATH could give synergistic effects in mechanical properties of PUF composites.

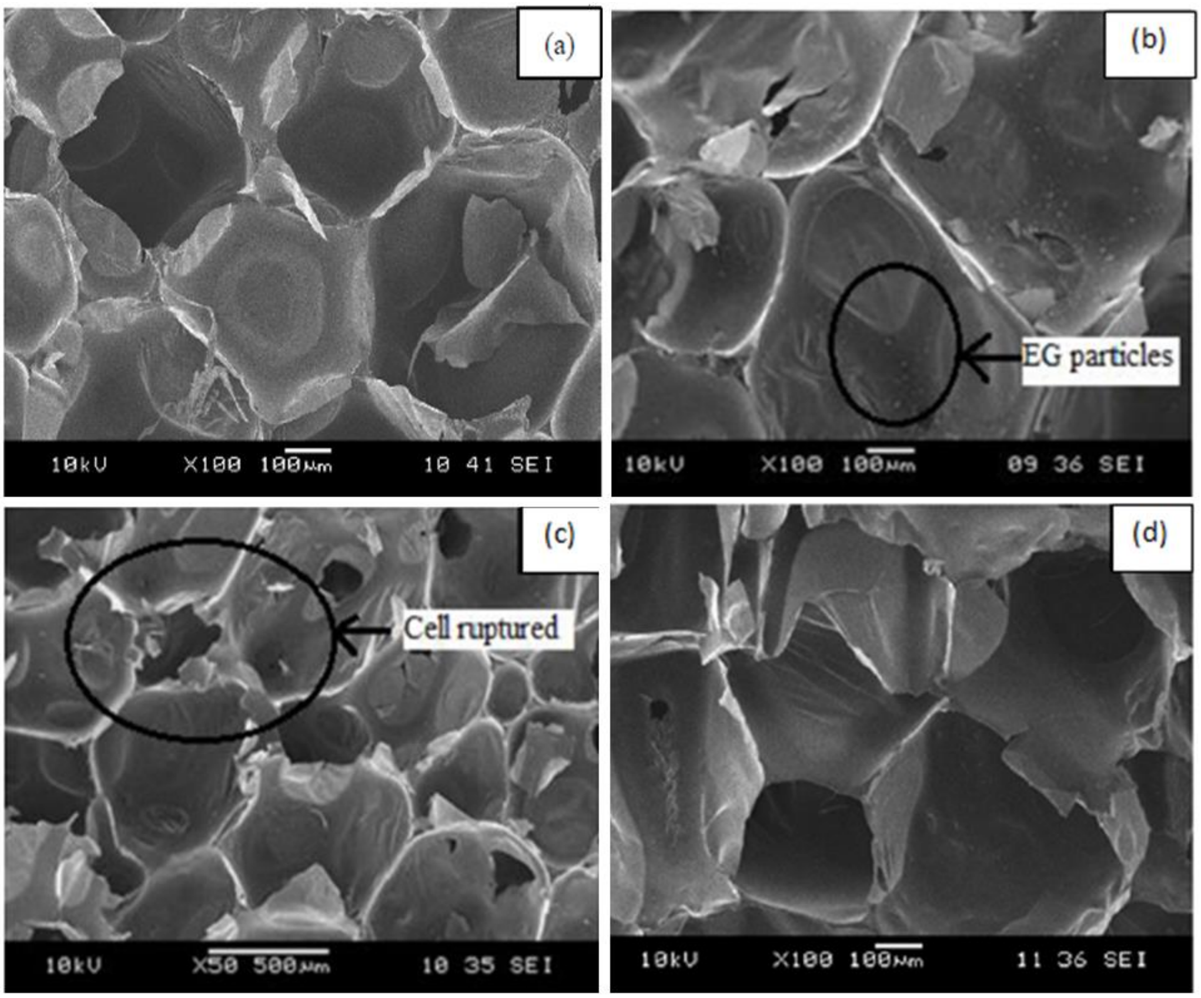

Figure 3. SEM micrograph of (a) PUF (b) PU/KF/15EG (c) PU/KF/2ATH/5EG (d) PU/KF/6ATH/10EG.

4. Conclusion

The incorporation of kenaf fiber, EG and ATH into palm oil based polyurethane foam has been carried out. Compression test shows that PU/KF/5EG shows the lowest compression strength and modulus while PU/KF/6ATH/10EG shows the highest. The incorporation of ATH at lower amount in the PUF has deteriorate the cell structure, thus lowering the compression strength and modulus of the composites. However, the compression strength and modulus was increased at higher ATH loading.

Addition of EG at 10 wt% shows good mechanical performance of the PUF composites. In conclusion, the combination of EG and ATH could produce synergistic effect on the mechanical properties of PUF composites.

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