UNIVERSITI SAINS MALAYSIA

LAPORAN AKHIR PROJEK PENYELIDIKAN JANGKA *PENDEK*

FINAL REPORT OF SHORT TERM RESEARCH PROJECT

Sila kemukakan laporan akhir ini melalui Jawatankuasa Penyelidikan di Pusat Pengajian dan Dekan/Pengarah/Ketua Jabatan kepada Pejabat Pelantar Penyelidikan

1. Nama Ketua Penyelidik: Tay Guan Seng
Name of Research Leader

☐ Profesor Madya/ *Assoc. Prof.*   ☑ Dr./ *Dr.*   ☐ Encik/Puan/Cik *Mr/Mrs/Ms*

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2. Pusat Tanggungjawab (PTJ): Pusat Pengajian Teknologi Industri
School/Department

3. Nama Penyelidik Bersama: Prof. Rozman Hj. Din
Name of Co-Researcher

4. Tajuk Projek: Preparation and Characterization of Polyurethane Foam from Biomass Material
Title of Project

5. Ringkasan Penilaian/*Summary of Assessment*:	Tidak Mencukupi *Inadequate* 1	2	Boleh Diterima *Acceptable* 3	Sangat Baik *Very Good* 4	5
i) Pencapaian objektif projek: *Achievement of project objectives*				√	
ii) Kualiti output: *Quality of outputs*				√	
iii) Kualiti impak: *Quality of impacts*			√		
iv) Pemindahan teknologi/potensi pengkomersialan: *Technology transfer/commercialization potential*			√		
v) Kualiti dan usahasama : *Quality and intensity of collaboration*				√	
vi) Penilaian kepentingan secara keseluruhan: *Overall assessment of benefits*				√	

6. Abstrak Penyelidikan
(Perlu disediakan di antara 100 - 200 perkataan di dalam **Bahasa Malaysia dan juga Bahasa Inggeris**. Abstrak ini akan dimuatkan dalam Laporan Tahunan Bahagian Penyelidikan & Inovasi sebagai satu cara untuk menyampaikan dapatan projek tuan/puan kepada pihak Universiti & masyarakat luar).

Abstract of Research
(An abstract of between 100 and 200 words must be prepared in Bahasa Malaysia and in English). This abstract will be included in the Annual Report of the Research and Innovation Section at a later date as a means of presenting the project findings of the researcher/s to the University and the community at large)

Please refer to attachment

7. Sila sediakan laporan teknikal lengkap yang menerangkan keseluruhan projek ini. [Sila gunakan kertas berasingan]
Applicant are required to prepare a Comprehensive Technical Report explaning the project. (This report must be appended separately)

Please refer to attachment

Senaraikan kata kunci yang mencerminkan penyelidikan anda:
List the key words that reflects your research:

Bahasa Malaysia	*Bahasa Inggeris*
Getah berbusa polyuretana	Polyurethane foam
Indek had oksigen	Limited oxygen index
Bahan lignoselulosa	Lignocellulosic material

8. Output dan Faedah Projek
Output and Benefits of Project

(a) * **Penerbitan Jurnal**
Publication of Journals
(Sila nyatakan jenis, tajuk, pengarang/editor, tahun terbitan dan di mana telah diterbit/diserahkan)
(State type, title, author/editor, publication year and where it has been published/submitted)

1. Research paper, ISI citation, G.S.Tay, L.N. Ong & H.D. Rozman (2011) Mechanical Properties and Fire Retardant Behaviour of Polyurethane Foam Reinforced with Oil Palm Empty Fruit Bunch. (Accepted to be published in Journal of Applied Polymer Science)

(b) **Faedah-faedah lain seperti perkembangan produk, pengkomersialan produk/pendaftaran paten atau impak kepada dasar dan masyarakat.**
State other benefits such as product development, product commercialisation/patent registration or impact on source and society.

This research work is in the preliminary study and the output of this research work can be used to develop a product in our future research works.

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(c) **Latihan Sumber Manusia**
Training in Human Resources

i) Pelajar Sarjana:
Graduates Students
(Perincikan nama, ijazah dan status)
(Provide names, degrees and status)

ii) Lain-lain: Ong Lu Nee (undergraduate) (Graduated)
Others

9. Peralatan yang Telah Dibeli:
Equipment that has been purchased

None

Dr. Tay Guan Seng
Pensyarah
Rancangan Teknologi Bio-sumber, Kertas dan Penglitup
Pusat Pengajian Teknologi Industri
Universiti Sains Malaysia
11800 USM, Pulau Pinang, Malaysia

Tandatangan Penyelidik
Signature of Researcher

8/9/2011

Tarikh
Date

Komen Jawatankuasa Penyelidikan Pusat Pengajian/Pusat
Comments by the Research Committees of Schools/Centres

This short term grant has been executed very well. Overall the school is satisfied with the output of the grant : 1 ISI publication with impact factor.

Nevertheless it is expected that the researchers will strive for more outputs in their future research grant projects.

Professor Azhar Mat Easa
Deputy Dean
(Graduate Studies & Research)
School of Industrial Technology
Universiti Sains Malaysia
11800 USM, Penang

9/9/2011

TANDATANGAN PENGERUSI JAWATANKUASA PENYELIDIKAN PUSAT PENGAJIAN/PUSAT
Signature of Chairman [Research Committee of School/Centre]

Tarikh
Date

Abstrak

Dalam Kajian ini, busa poliuretana (PU) telah disediakan oleh metilena difenil diisosianat (MDI) dengan polietilena glikol yang bermolekular 200 (PEG200) dan tandan buah kelapa sawit kosong (EFB). Sebelum MDI dicampurkan, EFB bercampur dengan PEG200 yang mengandungi pemangkin, semangkin, bahan permukaan dan agen peniup yang ditentukan. Selepas itu, MDI ditambahkan ke dalam campuran dan dikacau dengan pengakau mekanikal selama 30 saat sebelum busa PU ditubuhkan. Busa PU yang matang kemudian menjalankan pelbagai ujian mekanikal and ujian indeks oksigen terhad. Daripada keputusan yang diperoleh, modulus mampatan dan kekuatan mampatan meningkat apabila amaun EFB dan nisbah NCO/OH meningkat. Ini adalah diakibatkan oleh penurunan segmen-segmen lembut apabila jumlah pengisi EFB bertambah dan pertambahan segmen-segmen keras apabila nisbah NCO/OH bertambah. Akan tetapi, kekuatan mampatan menurun apabila kandungan EFB ditambahkan. Ini disebabkan oleh pengurangan PU matriks yang dibentukkan dan pertambahan titik penumpuan tegasan menyebabkan kegagalan berlaku. Bagi kekuatan impak, pertambahan amaun EFB dan nisbah NCO/OH menambahkan ketegaran busa PU, dan menyebabkan penurunan kekuatan impak. Selain itu, seperti yang dijangkakan, ujian indeks oksigen terhad menunjukkan peningkatan dalam nilai indeks oksigen (OI) apabila kandungan bahan pencegah kebakaran amonium polifosfat (APP) dan nisbah NCO/OH dinaikkan dan menunjukkan penurunan apabila kandungan EFB dinaikkan. Tambahan pula, kandungan APP juga dapat memberi kesan yang sedikit ke atas sifat-sifat mekanikal busa PU.

Abstract

In this study, Polyurethane (PU) foam was prepared using polymeric methylene diphenyl diisocyanate (MDI), polyethylene glycol with molecular weight 200 (PEG200) and oil palm empty fruit bunch (EFB), with one shot process. First, EFB was blended with PEG200 consist of various types of additives, such as catalyst, co-catalyst, surfactant and blowing agent. Then, MDI was added to the mixture and stirred vigorously for 30 seconds before PU foam could be formed. The PU foam was then subjected various testing such as compression test, impact test and limited oxygen index testing. From the results obtained, it can be seen that compression modulus and compression strength was increased as EFB and NCO/OH content increased. This was due to the decreasing of soft segments when EFB filler loading increased and increasing of hard segments as NCO/OH ratio increased. Except for compression strength which was reduced as EFB content increased, due to the reduction of PU matrix formation, increasing of stress concentration point where failure occurred. For impact strength, increasing of EFB and NCO/OH content increased the stiffness of PU foam therefore caused decreasing on impact strength. Besides, limited oxygen index testing showed that oxygen index (OI) value was increased as content of ammonium polyphosphate (APP) fire retardant and NCO/OH content was increased; and decreased as content of EFB increased, which was expected. Content of APP also gave slightly effect on the mechanical properties of PU foams made.

Technical report

Grant title: Preparation and Characterization of Polyurethane Foam from Biomass Material

Grant number: 304/PTEKIND/639002

The objective of this study was to investigate the effects of isocyanate/hydroxyl ratio and ammonium polyphosphate (APP) content on the properties of polyurethane foam. Polyurethane (PU) foam was prepared from polymeric diphenylmethane diisocyanate and polyethylene glycol with molecular weight of 200, reinforced with oil palm empty fruit bunch (EFB) using one shot process. Basically, this research work was separated into 3 phases. In phase 1, various types of raw materials were prepared and a preliminary study was carried out. From the preliminary study result, it was noticed that various types of additives include surfactant, catalysts and blowing agents must be added at the accurate amount. It was also revealed that various types of catalyst must be added in order to control the blowing rate of the polyurethane foam. Hence, in the second phase, many attempts had been conducted to determine the optimization of preparation condition and PU foam preparation. After obtaining the optimize condition, various parameters were studied and measurements were also carried out to determine the properties of the PU foam prepared (Phase 3).From the results, it was noticed that EFB enhanced the properties of the PU foam which is believed that this phenomenon was due to EFB acting as hard segment in PU foam system. The NCO/OH ratio played an important role in determining the properties of the PU foam produced. However, since EFB is a highly flammable material, APP was introduced to the PU foam system. From the results, APP improved the fire retardant behavior of the PU foam.

Tay Guan-Seng

From: onbehalfof+exb6+case.edu@manuscriptcentral.com on behalf of exb6@case.edu
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To: Tay Guan Seng
Subject: Journal of Applied Polymer Science - Decision on Manuscript # APP-2010-04-1219.R3

Attachments: CTA--2-.pdf

CTA--2-.pdf (29 KB)

23-Jul-2011

Dear Dr. Tay:

Thank you for submitting your revised manuscript entitled "Mechanical Properties and Fire Retardant Behaviour of Polyurethane Foam Reinforced with Oil Palm Empty Fruit Bunch." to the Journal of Applied Polymer Science. It is a pleasure to accept your manuscript in its current form for publication. Please address the final comments of the reviewer(s) who evaluated your manuscript are included at the bottom of this letter.

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May I take this opportunity to thank you for contributing your work to our Journal.

Sincerely,

Prof. Eric Baer
Editor-in-Chief, Journal of Applied Polymer Science exb6@case.edu

Reviewer(s)' Comments to Author:

Reviewer: 1
Comments to the Author
Dear authors
It is accepted with minor revision.
In table 5, it should be added the char residue content and the legend of degradation temperature should be corrected.
In other word, it should be rewritten TGA section.
Sincerely

Mechanical Properties and Fire Retardant Behaviour of Polyurethane Foam Reinforced with Oil Palm Empty Fruit Bunch.

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Abstract:

The objective of this study was to investigate the effects of isocyanate/hydroxyl ratio and ammonium polyphosphate (APP) content on the properties of polyurethane foam. Polyurethane (PU) foam was prepared from polymeric diphenylmethane diisocyanate and polyethylene glycol with molecular weight of 200, reinforced with oil palm empty fruit bunch (EFB) using one shot process. The effect of EFB content on the properties of PU foam was also studied. It was noticed that EFB enhanced the properties of the PU foam. This was due to EFB acting as hard segment in PU foam system. The NCO/OH ratio played an important role in determining the properties of the PU foam produced. However, since EFB is a highly flammable material, APP was introduced to the PU foam system. From the results, APP improved the fire retardant behaviour of the PU foam.

Keywords: Polyurethane, limited oxygen index, fire retardancy, oil palm empty fruit bunch.

Introduction

The use of natural fiber has been the interest of many scientific studies due to vast advantages offered, such as lower production cost and less abrasive to equipment. Natural fiber or lignocellulosic materials based plastic composite is among the most rapidly growing markets in this decade. According to Rossi (2002)[1], the demand for these products in North America and Western Europe amounted to about 1.3 billion pounds valued at roughly US$ 900 million, which represents almost 20% growth from the year 2001 level. In North America, building products made from lignocellulosic materials, especially decking, account for two-thirds of the market due to its cost effectiveness as compared to natural wood. In the western countries, automotive products such as interior door panels, exterior skirting and underbody panels were produced from long natural fiber, such as flax, hemp, jute or kenaf, reinforcing a variety of thermosets and thermoplastics. These applications of lignocellulosic materials are principally replacing glass fiber in reinforced plastic.

However, in producing a good lignocellulose-based composite, the mechanism and efficiency of stress transfer between the components often determine the performance of the composite, and this transition region is called the interface. With possible chemical reactions between the components at the interface, the performance of the composites will be improved. One of the examples of these composites is lignocellulosic fiber based-polyurethanes (PU). Lignocellulosic fibers are classified as hydroxyl-rich materials that may react with the isocyanate groups to form an interfacial region, which is a urethane linkage [2].

Polyurethane (PU) is one of the environmental compatible polymers which has been extensively prepared using different types of natural resources. The is due to the fact that natural resources (e.g. lignocellulosic materials) possess hydroxyl groups that can interact chemically with isocyanate compound to form urethane linkage. According to Hatakeyama et al. (1993), lignocellulosic material consists of two or more hydroxy (OH) groups per molecule and it can be used as polyol in the preparation of PU [3]. Recently, various studies have been carried out to utilize lignocellulosic materials to produce PU. Desai et al. (2003) prepared polyurethane adhesive from potato starch and natural oils. They found that the adhesive produced was superior as compared to the commercially available adhesives [4]. Wang and Chen (2007) prepared PU foam using liquefied wheat straw. Based on the results, PU foam prepared from liquefied wheat straw showed good mechanical properties and comparable with commercial PU foam [5]. On the other hand, Wang et al. (2008) prepared PU foam from liquefied corn stover and the results demonstrated that the properties of the PU foam could be adjusted by varying NCO/OH ratio [6]. Ferrer et al. (2008) compared PU produced from vegetable derived polyol and synthetic polyol. They found that vegetable derived polyol produced PU with compact network as compared to the one prepared from synthetic polyol [7]. Rozman et al. prepared PU composite using oil palm empty fruit bunch (EFB) and they found that EFB could act as a reactive component in the preparation of PU composite [8-10].

Many attempts have been carried out using natural resources in the preparation of PU foam. Yao et al. (1996) had prepared PU foam from liquefied starch as a polyol. From the results, they found that liquefied starch prepared has feasible viscosity and hydroxyl number for the preparation of PU foam. [11] Kwon et al. (2007) had also prepared PU foam from starch with different NCO/OH ratio. It was found that the cell size and compression strength of the PU foam increased as the NCO/OH ratio was increased. [12] Yan et al (2008) had prepared PU foam from liquefied cornstalk. They found that PU foam prepared from liquefied cornstalk showed excellent mechanical properties and thermal properties. [13] Chen and Lu (2009) had prepared PU foam from liquefied wheat straw and the result showed that thermal stability of the PU foam prepared was improved with the presence of liquefied wheat straw. [14]

Malaysia has a lot of lignocellulosic materials available and most of them are industrial waste and agricultural by-products which are under utilized. Among others, saw dust from wood industry, risk husk from rice mill, coir from coconut industry and empty fruit bunches, fronds and trunks from oil palm industry are promising resources of lignocellulosic materials for preparation of value-added products. Currently, there is no specific application of lignocellulosic materials produced in Malaysia due to various reasons and flammability of the product could be one of the reasons. In this study, PU foam incorporated with oil palm empty fruit bunch (EFB) was prepared and the effects of NCO/OH, EFB content and the amount of fire retardant additive added on the compression properties and fire retardancy of PU foam were investigated.

Experimental

Materials

Oil palm empty fruit bunch in fiber form was supplied by Ecofuture Bhd., Malaysia and polyethylene glycol with molecular of 200 (PEG 200) was supplied by Fluka Chemika. Polymeric diphenylmethane diisocyanate (MDI) was obtained from Maskimi Polyol (M) Sdn. Bhd. Malaysia, whereas ammonium polyphosphate (APP) was purchased from Clariant (M) Sdn. Bhd.

Preparation of EFB powder

The EFB fiber was washed with commercial detergent in order to remove the impurities such as oil residue on fiber surface before it was ground. Then, the EFB powder was sieved using Retch siever to separate the powder into different particle size. In this study, EFB particle with the size of 53μm-180μm was used.

Preparation of PU foam

The PU foams were prepared by one-shot process, where all the ingredients were mixed simultaneously and allowed to cure at room temperature. First, small amount of silicone oil (surfactant), dibutyltin dilaurate (catalyst), triethyamine (TEA) and water were mixed with PEG 200 and stirred with propeller stirrer for 5 minutes at approximately 500 rpm to ensure a homogeneous mix, followed by addition of MDI to the mixture. The mixture was stirred for 30s, before it was poured into a larger container and allowed to rise at room temperature. The obtained PU foams were left for complete cure for 24 hours at room temperature before cutting into specific dimensions for different types of testing. Preparation of foam was repeated for different NCO/OH ratio (0.7, 0.9, 1.1 and 1.3 respectively), filler contents (5%, 10%, 15% and 20%) and different fire retardant content (1%, 3%, 5%).

PU foam measurements

Compression test

Compressive strength and modulus of foams was determined by Universal Testing Machine, GOTECH GT-TGS-2000 with a load cell of 20kN. The test was performed according to ASTM D1621 with a crosshead speed of 5mm/min. Size of the specimens were 60 x 60 x 50mm (width x length x thickness), The compressive stress at 10% deformation of its original thickness was calculated. Compression modulus was then calculated according to formula as follows:

$$E_c = WH / AD$$

Where,

E_c : Compression modulus, Pa (or Psi)

W : Load, N (or lbf)

H : Initial specimen height, m (or in)

A : Initial horizontal cross-sectional area, m^2 (or in^2)

D : Deformation, m (or in)

Impact Testing

Impact tests were conducted using Ray-Ran Pendulum Impact Tester. The Charpy method was carried out using samples with dimensions of 65 x 12 x 10mm (length x width x thickness).

Scanning electron microscopy (SEM) study

PU foams were investigated with a field-emission scanning electron microscope (FE-SEM) Leo Supra 50VP. The specimens were mounted on an aluminium stub and sputter coated with a thin layer of gold to avoid electrostatic charging during examination.

Fire Retardancy Test

Limiting Oxygen Index (LOI) test was carried out according to the British Standard BS2782, to determine the relative flammability of foam. LOI is defined as the minimum concentration of oxygen in an oxygen–nitrogen mixture, required to just support downward burning of a vertically mounted test specimen. Hence, higher LOI values represent better flame retardancy. Test specimen dimensions used were 100 x 10 x 4mm (length x width x thickness).

Thermogravimetry

Thermogravimetry analysis was carried out using Perkin Elmer Pyris 6. Small amount of sample (approximately 7mg) was placed in the platinum pan before it was put in the furnace. Then, the sample was heated from 30°C to 800°C with a heating rate of 10°C/min.

Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR analysis was done using a Nicolet Avatar FTIR Spectrophotometer. Approximately 1mg of EFBPU foam was mixed with dried Potassium Bromide (KBr). Then, the mixture was grinded into fine powder using a mortar and pressed into a disc-like shape and finally used for the FTIR test in the range 4000–400 cm^{-1}, with 64 scans

Results and Discussion

Effect of EFB content on cell size

Figure 1(a) depicts the SEM micrograph for PU foam prepared from MDI and PEG 200 without EFB, while Fig. 1(b) and Fig. 1(c) show the SEM micrograph for PU foam filled with 5% and 20% EFB, respectively. By comparing the SEM micrograph, it can be seen that the size of the PU foam cell decreases when EFB is incorporated. This could be attributed to the presence of EFB which is believed to fill up the space in PU foam. In addition, the formation of PU cell could be interrupted by the presence of EFB and thus resulting in irregular size of the cell. This is evident in Fig. 1(d) and Fig. 1(e), in which EFB is seen embedded between two cells. This phenomenon could have reduced the size of PU foam cell.

Effect of EFB content on mechanical properties

In the preparation of PU foam reinforced with EFB, a preliminary study was carried out in order to determine the appropriate EFB content for obtaining the optimum strength of PU foam. Table 1 depicts the effect of EFB loading on compression strength and compression modulus of PU foam prepared with NCO/OH of 1.1. As can be observed, the compression strength increases as the EFB fiber content is increased. This is similar to the previous study carried out by Hatakeyama et al. [3] and Chang et al. [15]. According to Chang et al. [15], the compression strength, glass transition temperature (T_g) and thermal conductivity of PU foam could be enhanced by increasing the soy flour content. Meanwhile, Hatakeyama et al. [3] observed similar trend and they attributed this phenomenon to lignocellulosic material which acts as a hard segment in the PU foam. According to previous study by Rozman et al. [10], EFB was found interacting with PU matrix and enhancing the interfacial properties between PU matrix and EFB. This is evident in SEM micrographs (Fig. 1(d) and Fig. 1(e)) where EFB was well embedded within the PU matrix. Consequently, the efficiency of the stress transfer from PU matrix to EFB is enhanced. This explanation also applied to the observation on compression modulus (Table 1), as the EFB content increases, the compression modulus is increased. Hence, it can be said that EFB could act as a hard segment in the PU foam system and its inherent stiffness has produced PU foam with higher modulus as compared to the one without EFB.

Table 2 depicts the effect of EFB content on impact strength of the PU foam. It can be seen that impact strength decreases as the EFB content is increased. According to Strong [16], impact strength is strongly dependence on the ability of the material to move or deform to accommodate impact, whereas Folkes [17], stated that major energy absorbing mechanism in a composite material is referring to the energy required in deforming the fiber-matrix bonding and pulls it out completely. Generally, polymer with high strength and stiffness would result in low elongation to failure. Therefore, the decreasing of PU foam impact strength is believed to be due to the increment of hard segment in PU foam and the increase of stiffness of PU foam as the EFB content is increased.

From this study, it is noticed that the optimum EFB content is about 20%. Further increase in EFB content results in difficulties in mixing due to the increase in viscosity of polyol and EFB mixture. Hence, in the subsequent study, the effects of NCO/OH ratio and APP content are based on PU foam prepared from 20% EFB.

Effect of NCO/OH ratio and APP content on PU foam mechanical properties

Fig. 2 shows the effect of NCO/OH ratio and APP content on compression strength at 10% strain. Generally, compression strength of PU foam increases from 0.9 to 1.1, after which no significant difference is observed, regardless of APP content. This is probably due to the interaction between MDI and hydroxyl group from EFB and PEG 200. According to Cheradame et al. (1989) [18], when NCO/OH ratio was lower than unity, the PU obtained was mostly from the condensation of isocyanate and glycol. For NCO/OH value closes to 1, lignocelluslioc material (lignin) would participate in the polycondensation reaction that resulted in an improvement in the mechanical properties. When NCO/OH value was greater than 1.2, PU terminated with –NCO would be formed, which could further react with other urethane groups leading to a change in the morphology [5]. From previous study [10], unreacted OH in PU system could act as a stress concentration point which reduced the strength of the PU system. Hence, it can be said that the hydroxyl group from EFB or polyol in PU system has reacted with isocyante group from MDI when increase in NCO/OH ratio up to 1.1. This is in line with the previous investigation [19] where the increment of NCO/OH ratio resulted in the increase of intermolecular cross-linking.

It is observed that there is a tendency for compression strength to be decreased as the APP content is increased. This could be attributed by two factors; (i) the possibility that unreacted APP presented in the foam serves as stress concentration points, which reduce the strength and (ii) the brittleness of the PU foam which is due to the inherent rigidness of APP. As can be seen in Fig. 3, when the APP content increases, the stiffness (compression modulus) of the PU foam is increased.

Compression modulus of PU foam can also be affected by NCO/OH ratio as observed in Fig. 3. However, no significant difference is observed between those with NCO/OH of 1.1 and 1.3. The increase in the modulus may be attributed to two benzyl rings of MDI which can restrict the molecular movement in PU system. The increase of NCO/OH ratio increases the amount of MDI used and subsequently increase the number of benzyl ring the PU system. Tanaka et al. (2008) stated that when a pressure is applied, the sample containing more soft segments were easier to be compressed [20]. Thus, it can be said that a stiffer PU can be obtained when higher NCO/OH ratio is used.

The ratio of NCO/OH and APP content can also play a crucial role in determining the impact properties of PU foam reinforced by EFB. From Fig. 4, the impact strength decreases as NCO/OH ratio and APP fire retardant content are increased. As aforementioned, increasing of NCO/OH ratio results in molecular restriction in PU foams and therefore, the ability of foams to absorb energy decreases. According to the study carried out by Stael et al. (2001), composites with low impact strength could be attributed to the inhibition of deformation capacity of matrix due to the presence of a stiffer material [21]. As for APP content, it is in agreement with the result obtained in compression modulus. As mentioned earlier, PU with higher stiffness possesses lower impact properties. Hence, it is clear that APP has positive effect on the stiffness of the PU foam.

Limited Oxygen Index

Limited oxygen index (LOI) is used as a qualitative method to evaluate the fire retardancy of polymeric materials. Table 3 shows the relationship of oxygen index (OI) value of PU foams with different EFB content. As expected, OI value decreases as EFB content increased. This is due to EFB being a lignocellulosic filler which is a highly flammable material. However, there is no significant difference on OI value as EFB filler loading is further increased (10%, 15% and 20%). This indicates that the EFB content of 10% is the minimum amount of EFB where significant change in OI is observed.

The effect of NCO/OH ratio and APP content on OI value is shown in Fig. 5. OI value increases when NCO/OH ratio is increased. According to Lanrock (1995) higher NCO/OH ratio gave higher isocyanurate linkages which were found to be thermally stable, and produce less combustible gasses in PU foam [22]. According to Bhattacharjee & Engineer (1996), the presence of isocyanurate can be determined using FTIR spectroscopy, which absorbance of 1415 cm^{-1} will be shown due to C-N vibration in the 6-membered heterocycle. Meanwhile, absorbance of 1220 cm^{-1} also can be observed which is attributed to the presence of urethane group [23]. By looking at the FTIR spectrum depicted in Fig. 6, it can be seen that the transmittance of 1415 cm^{-1} and 1220 cm^{-1} are evidence. In addition, no isocyanate transmittance is observed which is in the region of 2260-2280 cm^{-1}. Hence, it is believed that the urethane linkages had been produced from the interaction of isocyanate and hydroxyl group. The excess of isocyanate group added (due to higher NCO/OH) is expected to react with urethane linkages (trimerization) to produce isocyanurate. With this regards, it is believed that the increase in OI value when NCO/OH ratio is increased could be due to higher isocyanurate linkages present in the foam especially for the ones produced with NCO/OH of 1.3.

The results also show that the OI value of PU foam increases as APP content is increased. According to Duquesne et al. (2001), the addition of APP to PU accelerates the decomposition of matrix which leads to an increase in the amount of high temperature residue, under an oxidative or inert atmosphere. The stabilized residue acts as a protective thermal barrier during the intumescent-fire retardancy process. Intumescent, is a process when combustion occurs, a cellular charred layer on the surface of material protects the underlying materials from the action of heat flux or flame and acts as a physical barrier limiting the diffusion of combustible volatile products towards the flame and oxygen towards the polymer [24]. Therefore, the increase in APP addition level results in an increase in OI value due to the increase in char formation. This result is supported with the result obtained in TGA (Table 4). It can be seen that the degradation temperature shows slight increase as the amount of APP is increased. It also can be said that increment of APP amount in PU foam has resulted in the increase of mass residue.

Conclusion

It can be concluded that the addition of EFB and APP at different percentages and NCO/OH ratios showed significant influence on the properties of PU foams. The amount of EFB incorporated in PU foam was found to affect the compression properties. Since impact strength was reciprocal to the stiffness of the PU foam, progressive increase in EFB content resulted in a reduction in impact strength. It was evident that inherent stiffness of EFB has contributed to the PU foam. The amount of MDI (from various NCO/OH ratios) added to the formulation influenced the properties of PU foam since benzyl ring could also act as a hard segment and thus restricted the molecular movement in PU system. Meanwhile, the addition of APP in formulation had increased the fire retardancy, compression and impact properties of the PU foam.

ACKNOWLEDGMENTS
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References

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Table 1: Compression properties of PU foam with different EFB content (NCO/OH = 1.1)

EFB content (%)	Compression strength (MPa)	Compression modulus (GPa)
5	5.07 (±0.23)	2.35 (±0.31)
10	5.55 (±0.16)	2.91 (±0.27)
15	6.19 (±0.37)	3.33 (±0.25)
20	6.54 (±0.24)	3.45 (±0.15)

Table 2: Impact strength of PU foam with different EFB content (NCO/OH = 1.1)

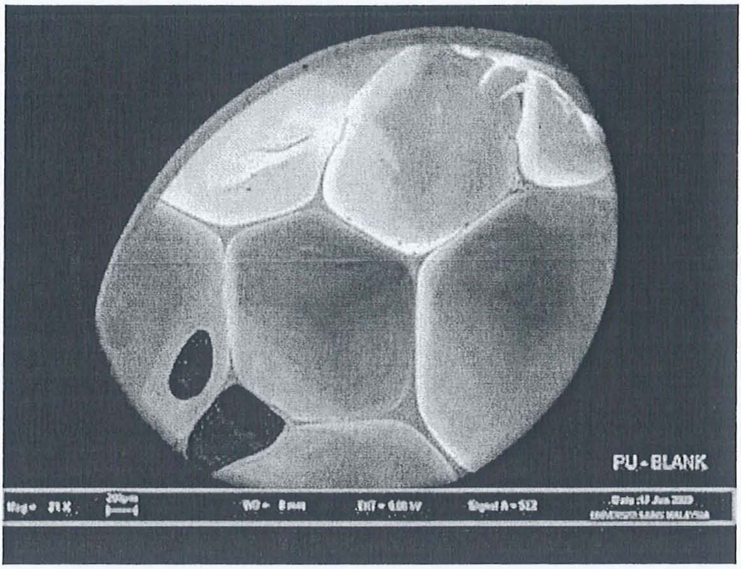
EFB content (%)	Impact strength (kJ/m)
5	16.75 (±0.14)
10	15.27 (±0.15)
15	11.86 (±0.21)
20	11.01 (±0.43)

Table 3: Oxygen index value of PU foam with different EFB content (NCO/OH = 1.1)

EFB content (%)	Oxygen index
5	19.95 ($\pm$0.04)
10	19.37 ($\pm$0.09)
15	19.37 ($\pm$0.11)
20	19.36 ($\pm$0.07)

Table 4: Thermal degradation temperature and mass residue at 800°C of PU foam with different APP content. (NCO/OH = 1.1, 20% of EFB)

APP content (%)	Thermal degradation temperature (°C)	Mass residue (%)
0	285.73 (±1.75)	21.22 (±0.68)
1	285.69 (±1.31)	20.73 (±1.12)
3	289.09 (±1.62)	21.49 (±0.83)
5	296.64 (±0.84)	30.76 (±1.55)



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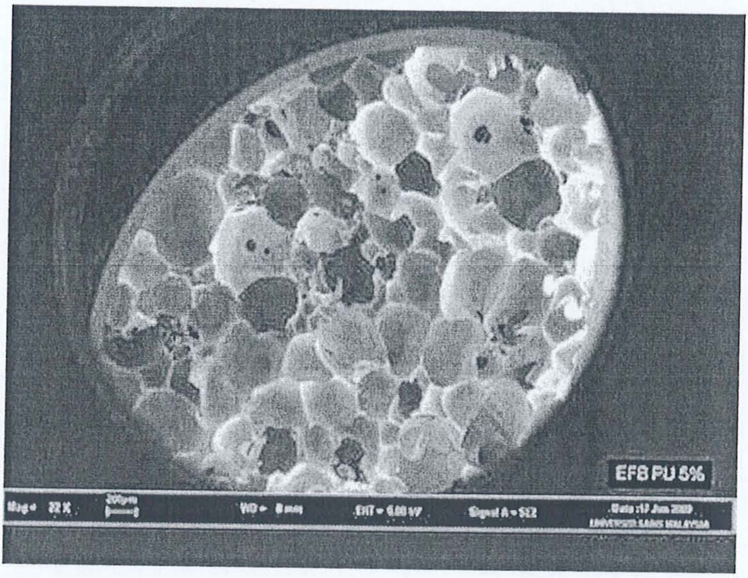

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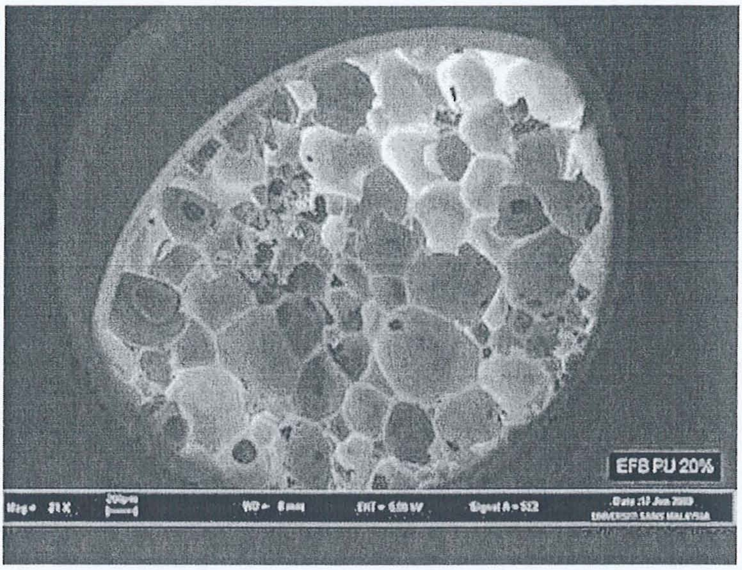

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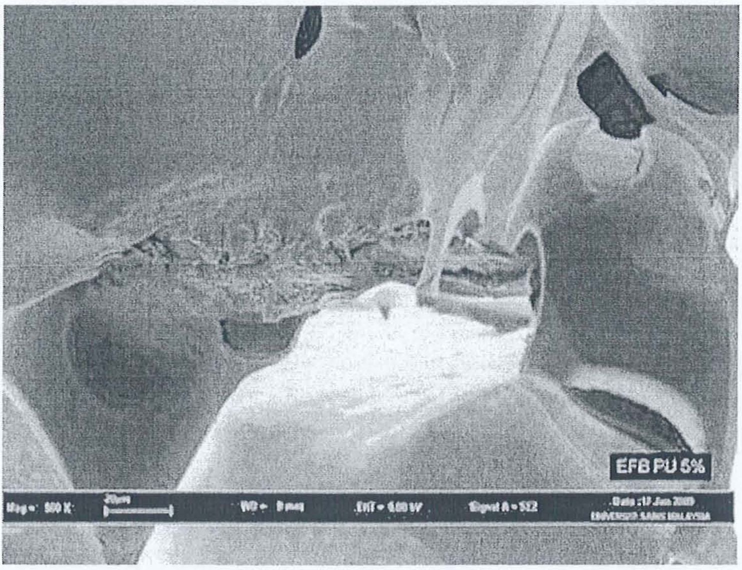

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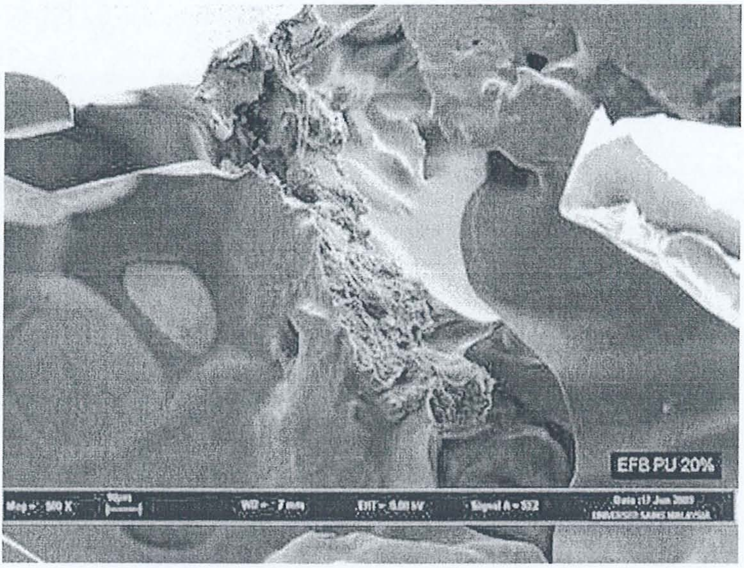

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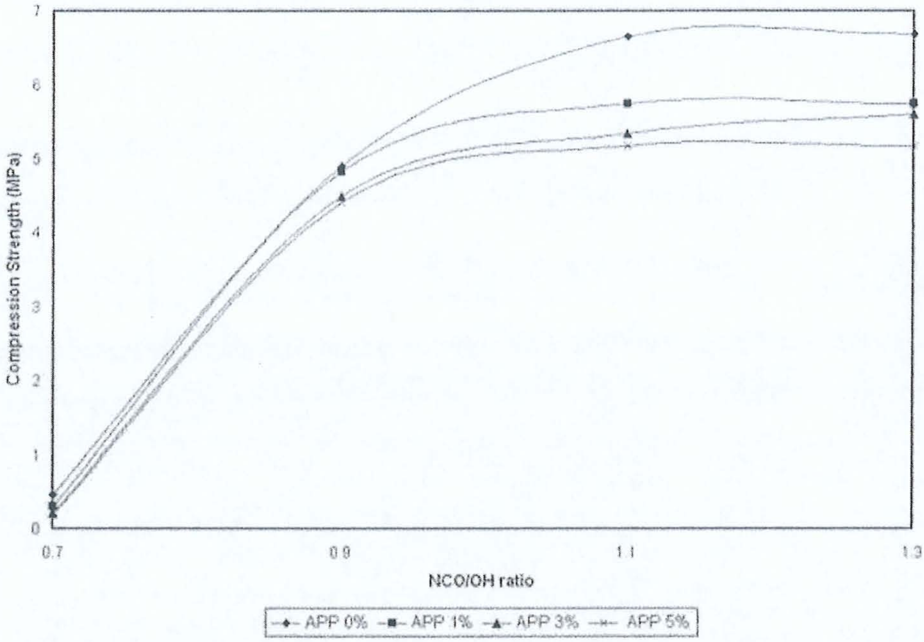

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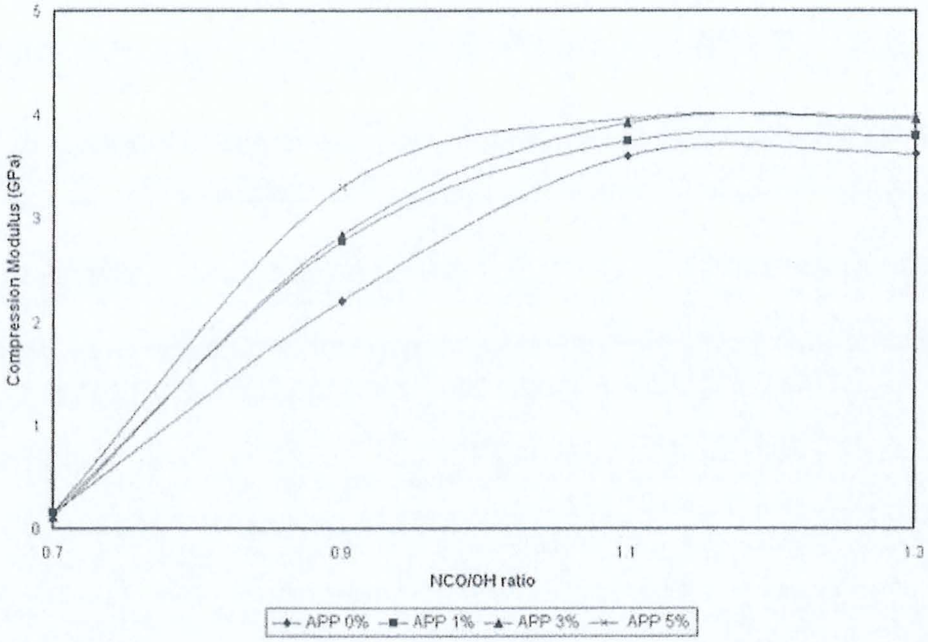

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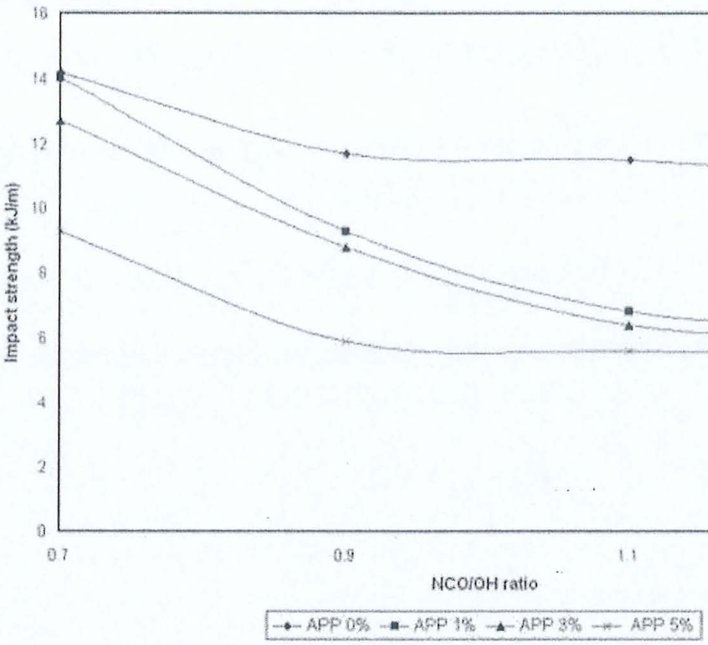

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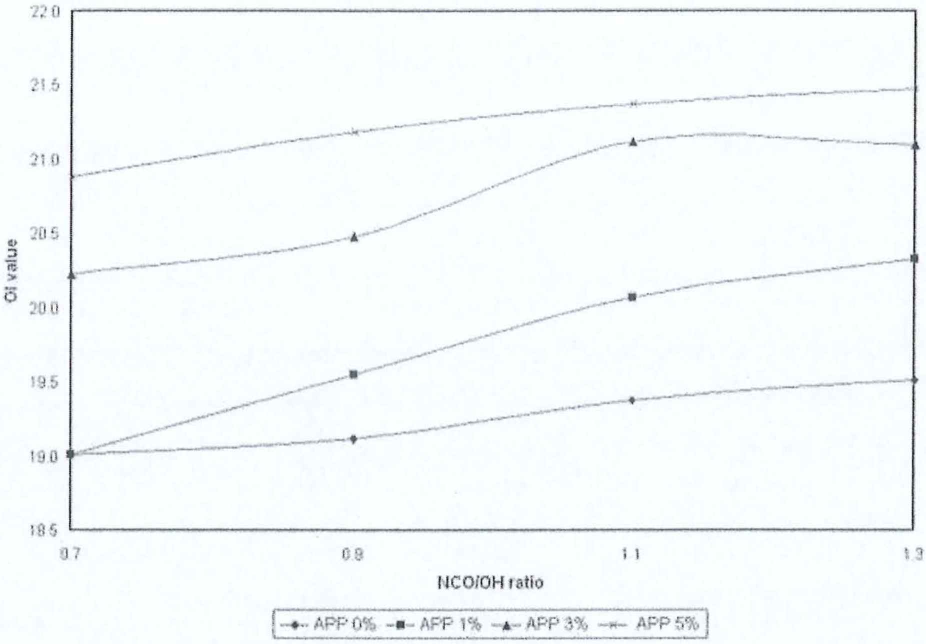

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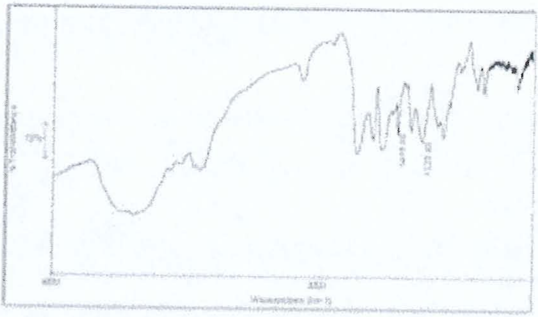

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Abstrak

Dalam Kajian ini, busa poliuretana (PU) telah disediakan oleh metilena difenil diisosianat (MDI) dengan polietilena glikol yang bermolekular 200 (PEG200) dan tandan buah kelapa sawit kosong (EFB). Sebelum MDI dicampurkan, EFB bercampur dengan PEG200 yang mengandungi pemangkin, semangkin, bahan permukaan dan agen peniup yang ditentukan. Selepas itu, MDI ditambahkan ke dalam campuran dan dikacau dengan pengakau mekanikal selama 30 saat sebelum busa PU ditubuhkan. Busa PU yang matang kemudian menjalankan pelbagai ujian mekanikal and ujian indeks oksigen terhad. Daripada keputusan yang diperoleh, modulus mampatan dan kekuatan mampatan meningkat apabila amaun EFB dan nisbah NCO/OH meningkat. Ini adalah diakibatkan oleh penurunan segmen-segmen lembut apabila jumlah pengisi EFB bertambah dan pertambahan segmen-segmen keras apabila nisbah NCO/OH bertambah. Akan tetapi, kekuatan mampatan menurun apabila kandungan EFB ditambahkan. Ini disebabkan oleh pengurangan PU matriks yang dibentukkan dan pertambahan titik penumpuan tegasan menyebabkan kegagalan berlaku. Bagi kekuatan impak, pertambahan amaun EFB dan nisbah NCO/OH menambahkan ketegaran busa PU, dan menyebabkan penurunan kekuatan impak. Selain itu, seperti yang dijangkakan, ujian indeks oksigen terhad menunjukkan peningkatan dalam nilai indeks oksigen (OI) apabila kandungan bahan pencegah kebakaran amonium polifosfat (APP) dan nisbah NCO/OH dinaikkan dan menunjukkan penurunan apabila kandungan EFB dinaikkan. Tambahan pula, kandungan APP juga dapat memberi kesan yang sedikit ke atas sifat-sifat mekanikal busa PU.

Abstract

In this study, Polyurethane (PU) foam was prepared using polymeric methylene diphenyl diisocyanate (MDI), polyethylene glycol with molecular weight 200 (PEG200) and oil palm empty fruit bunch (EFB), with one shot process. First, EFB was blended with PEG200 consist of various types of additives, such as catalyst, co-catalyst, surfactant and blowing agent. Then, MDI was added to the mixture and stirred vigorously for 30 seconds before PU foam could be formed. The PU foam was then subjected various testing such as compression test, impact test and limited oxygen index testing. From the results obtained, it can be seen that compression modulus and compression strength was increased as EFB and NCO/OH content increased. This was due to the decreasing of soft segments when EFB filler loading increased and increasing of hard segments as NCO/OH ratio increased. Except for compression strength which was reduced as EFB content increased, due to the reduction of PU matrix formation, increasing of stress concentration point where failure occurred. For impact strength, increasing of EFB and NCO/OH content increased the stiffness of PU foam therefore caused decreasing on impact strength. Besides, limited oxygen index testing showed that oxygen index (OI) value was increased as content of ammonium polyphosphate (APP) fire retardant and NCO/OH content was increased; and decreased as content of EFB increased, which was expected. Content of APP also gave slightly effect on the mechanical properties of PU foams made.

Tay Guan-Seng

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To: Tay Guan Seng
Subject: Journal of Applied Polymer Science - Decision on Manuscript # APP-2010-04-1219.R3

Attachments: CTA--2-.pdf

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23-Jul-2011

Dear Dr. Tay:

Thank you for submitting your revised manuscript entitled "Mechanical Properties and Fire Retardant Behaviour of Polyurethane Foam Reinforced with Oil Palm Empty Fruit Bunch." to the Journal of Applied Polymer Science. It is a pleasure to accept your manuscript in its current form for publication. Please address the final comments of the reviewer(s) who evaluated your manuscript are included at the bottom of this letter.

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May I take this opportunity to thank you for contributing your work to our Journal.

Sincerely,

Prof. Eric Baer
Editor-in-Chief, Journal of Applied Polymer Science exb6@case.edu

Reviewer(s)' Comments to Author:

Reviewer: 1
Comments to the Author
Dear authors
It is accepted with minor revision.
In table 5, it should be added the char residue content and the legend of degradation temperature should be corrected.
In other word, it should be rewritten TGA section.
Sincerely