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Effect of Phosphite Loading on the Mechanical and Fire Properties of Palm-Based Polyurethane

(Kesan Penambahan Fosfit ke Atas Sifat Mekanik dan Pembakaran Poliuretana Asas Sawit)

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ABSTRACT

Fire-retarding polyurethane (PU) composite was produced by adding 2,4-ditert-butylphenyl phosphite (FR) to palm-based monoester resin with loading percentage of 0, 2, 4, and 6 wt%. The Shore D hardness index increased marginally with increasing FR content. However, the impact and flexural strengths decreased with increasing FR loading attributed to the weak interfacial bonding between FR and PU matrix. The fire test indicated lowering of burning rate (from 5.30 mm·s^{-1} to 2.80 mm·s^{-1}) as the loading percentage of FR increased. The combustion enthalpy of the composites also decreased with higher loading percentage of FR.

Keywords: Burning rate; fire-retarding; palm-based polyurethane; phosphite

ABSTRAK

Komposit poliuretana (PU) berperencat api telah dihasilkan dengan menambahkan 2,4- ditertbutilfenil fosfit (FR) ke dalam resin monoester asas sawit dengan penambahan 0, 2, 4 dan 6%bt. Indeks kekerasan Shore D meningkat dengan peningkatan komposisi FR. Walau bagaimanapun, kekuatan hentaman dan fleksuralnya menurun dengan penambahan FR akibat terhasilnya lekatan antaramuka yang lemah antara FR dan matrik PU. Ujian pembakaran menunjukkan kadar pembakaran menurun (daripada 5.30 mm·s^{-1} ke 2.80 mm·s^{-1}) apabila penambahan FR ditingkatkan. Entalpi pembakaran komposit ini menurun dengan peratusan FR yang tinggi.

Kata kunci: Fosfit; kadar pembakaran; perencat api; poliuretana asas sawit

INTRODUCTION

The most important commercial polyurethane products are foams that are commonly classified as either flexible or rigid depending on their mechanical performance and cross-link densities. Rigid polyurethane foams are widely used in building insulation and domestic appliances, due to their superior mechanical properties and low density. However, the polyurethane industry is facing environmental challenges due to the type of auxiliary blowing agents used during the polymerization process. Due to that, n-pentane and HCFC141b are of a better choice compared to chlorofluorocarbon (CFC). The cost and availability of HFCs has led the polyurethane foam industry to focus on pentane as the primary blowing agent. Unfortunately, the inherent high flammability of pentane has resulted in polyurethane foams that fail to meet the required regulatory fire tests. Therefore, the polyurethane industry has to respond to these challenges by designing additive packages (catalysts, surfactants, cross-linkers and flame retardants) that can overcome these flammability shortcomings, while providing physical and mechanical properties comparable to those of rigid polyurethane foams blown with CFCs and HCFCs (Song et al. 2005).

Several research works have been carried out to the identify parameters that govern mechanical behavior of particulate composites. Generally, it has been found that the reinforcement effect increased with decreasing particle size and increasing adhesion to the matrix. The mechanical and physical properties of PU composites based on rice husk and polyethylene glycol have been studied by Rozman et al. (2001). From the results of flexural, tensile and impact tests, it is clearly demonstrated that the incorporation of rice husk as an OH group provider in a PU system has increased strength of the composites. Rozman et al. (2001) and Mohd Ishak et al. (1998) have used oil palm empty fruit bunch fiber (EFB) as filler in their plastic composites. EFB and glass fiber were blended with polypropylene (PP) as the matrix to form a hybrid composite with improved strength by the usage of coupling agents. All these studies were only focusing on improving mechanical strength without further investigation on the flame retardancy of the composites utilizing lignocellulosic materials and other bioresources which are easily ignited.

To limit the flammability of polymer materials, it is necessary to incorporate fire retardant (FR) in the polymer. Molecules containing phosphorous groups can be

considered as fire retardants allowing improved material in term of thermal and fire properties. Phosphorus, silicon and nitrogen compounds are the agents mostly used for replacing halogenated compounds in flame retarding resins, especially epoxy. The halogen-free products are attractive and being used at large globally (Spirckel et al. 2002). Flame retardant can be used as either reactive or additive chemicals. The reactive flame retardants are incorporated into the polymeric materials by covalent bonding between the polymer and the flame retardant, whereas the additive types are dissolved in the polymer (Dvir et al. 2003; Frank et al. 2001; Jang et al. 1998; Lewin 2001; Liu & Wang 2006; Reed et al. 2000). The mechanical properties, thermal stability, and flammability of flame retarded PU/ organo clay nancomposite were investigated by tensile, thermogravimetric and cone calorimetry experiments. It is shown that the flame retarded nanocomposite has enhanced tensile strength, capacity of carbonaceous char formation and better flame retardant properties compared to control PU (Song et al. 2005). The focus of the present study is to enhance the fire resistivity of palm-based PU by using phosphite as the fire retardant without sacrificing its mechanical and thermal properties. Moreover, study on this field has not been widely carried out focusing on vegetable oil-based polymer, namely the palm-based PUs.

Experimental Procedures

Material

The palm-based ester was synthesized via esterification and polycondensation process as suggested by Badri et al. (2000). Palm kernel oil was reacted with diethanolamine catalyzed with sodium ethanoate at 180-190°C for 20 minutes. The synthesized esteramide has a hydroxyl value of 350-370 mg KOH/g. Its viscosity is 380-400 cps with moisture content of about 0.05%. The hardener was 2, 4-diphenylmethane diisocyanate (MDI), (Cosmo-polyurethane, Port Klang, Malaysia); the catalysts; tetramethylhexanediamine (TMHDA) and pentamethyldiethylenetriamine (PMDETA) (Cosmo Polyurethane (M) Sdn Bhd, Port Klang, Malaysia) and the silicone surfactant (Niax L5404, Witco Ltd, Singapore) were used without any further treatment. The flame retardant is Irgafos 168 (2, 4-ditert-butylphenyl phosphite) supplied by Asahi (M) Sdn. Bhd. Irgafos 186 used in this experiment is a white powder of 18 μm of average size, insoluble in water with the moisture content of 0.30% and melting point of 182°C

Preparation of Palm -Based Polyurethane and Its Composites

The palm-based PU was prepared by reacting 100 g of crude MDI with 100 g of palm-based resin (mixture of 100 parts by weight (pbw) palm-based esteramide, 2.0 pbw Niax L5404 as surfactant, 0.30 pbw TMHDA as curing catalyst and 0.15 pbw PMDETA as blowing catalyst). The mixture was agitated vigorously using a standard propeller at a speed of 2000 rpm for 60 seconds at ambient temperature (~25°C). The reaction time: cream time - initial reaction time (CT), fiber time – liquid to solid phase transition time (FT), tack-free time – procuring time (TFT) and the rise time- end of reaction time (RT) were noted. The free-rise density (FRD) was determined by dividing the mass of the foam in the cup with the capacity of the cup as shown in the following equation (Badri et al. 2000):

$$\text{Free-rise density, FRD} = \frac{\text{(mass of foam and cup-mass of cup), kg}}{\text{capacity of cup, m}^3}.$$

Characterization of the Polyurethane and Its Composites

Mechanical characterizations A Portable shore D hardness tester (Durometer Affri system Series 3300 MRS, Affri Cee-Versea, Italy) was used with measurement being conducted according to ASTM D2240 standard to determine the hardness of the sample. The boards produced were cut to samples with dimensions of 63 mm × 63 mm × 3 mm. The values of the shore D hardness index obtained represent the mean of five specimens. Impact testing was conducted according to ASTM D256-88 standard. The Izod method was employed, using unnotched samples with dimensions of 63 mm × 13 mm × 3 mm on a Zwick impact tester (Model 5101, Zwick Roell Group, Georgia, USA), with a pendulum energy of 2 J. The impact strengths were calculated by dividing the energies (J) recorded on the tester by the cross-sectional areas (mm^2) of the specimens. The values of the impact strengths represent the mean of five specimens. Three-point bending method in the determination of the flexural strength was conducted according to ASTM D790-86 standard. The PU composites produced were cut to test samples with dimensions of 120 mm × 12 mm × 3 mm. The flexural test was carried out using universal test machine (Model 5525, Instron Corporation, Massachusetts, USA) at a cross-head speed of 3.1 $mm \cdot min^{-1}$. The values obtained represent the mean of five specimens.

Determination of Combustion Entalphy Bomb calorimeter model IKA C 4000 was used to determine the initial enthalpy of the composites. About 1.00 g of PU composites was put in the sample cup. The bomb was prepared by putting a wire (5 cm length) between two electrodes. A thread was also tied on the divider of the two wires touching the sample. The bomb was placed inside the chamber and was close tightly with purging oxygen at a pressure of 30 bar into the chamber. The chamber was transferred to a container where 1.8 L of distilled water was poured into the container. Monitoring of the process was carried out using the calorimeter IKA system (Oelke & Zuehlke 1969; Arthur 1986).

Fire Testing This test method was carried out to determine the relative burning characteristics and flame resistance properties. It measured the properties of the composites

in reactions to heat and flame under controlled laboratory conditions. The test results represented flaming plus glowing time in seconds under the conditions of the test. ASTM D 5048-90 (Procedure B- test of plaque Specimens) standard was followed.

Scanning Electron Microscope (SEM) Analysis A Leo VP SEM-1450 SEM was used to study the interaction and distribution of FR in the polyurethane composites by observation made onto the impact fractured samples. The samples were coated with gold of thickness 20Å with Bio-rad microscience division-SC 500 to prevent electrical charging during examination. The observation was made at 250 times magnification on the control polyurethane (0% FR) and the polyurethane composites with 2%, 4% and 6% FR.

Results and Discussions

MECHANICAL PROPERTIES

The results of the mechanical testing obtained are summarized in Table 1. The hardness index may give an initial indicator to physical strength of the PU composites. The hardness index increased with increasing amount of FR. This result is in close agreement with research carried out by Khairiah Haji Badri et al. (2006), where composite with filler produced higher hardness than that without filler. The hardness index increased by 27, 32% and 38% with 2, 4 and 6 wt% of FR, respectively, giving a linear relationship between both parameters.

The impact strength of materials reflected its ability to resist high-speed fracture. The impact strength of the PU composites decreased with increasing FR content. This may be attributed to the weak interfacial bonding between the filler and the PU matrix. The highest impact strength was observed for the control PU compared to the PU filled with FR (6.9 $kJ \cdot m^{-2}$). The PU composite with 6 wt% FR has the lowest impact strength compared to others, in agreement with research carried out by Sumaila et al. (2006). The impact strength decreased by 25%, 24% and 23% with loading of 2, 4 and 6 wt% FR, respectively.

Both the flexural strength and modulus decreased as the amount of FR increased as summarized in Table 1. The flexural strength of the control PU was reduced by 28% compared to PU composites with the addition of 2 wt% FR. Further reduction was observed with increasing loading amount of FR from 2 to 4 wt% (reduction of 22%). The highest amount of FR gave the lowest flexural strength as shown in Figure 4 (reduction of 20%). The same trend was observed for the modulus of the PU composites. The modulus decreased by 26%, 23% and 22% when 2, 4 and 6 wt% of FR was added to the PU matrix, respectively. The lowest modulus was observed at the addition of 6 wt% FR. Higher loading of FR decreased the wetting properties of the matrix and reduced the degree of encapsulation of matrix around the FR. As a result, the FR was exposed greatly to direct stress and thus, low stress transfer was experienced.

HEAT AND FIRE PROPERTIES

The effect of FR on the network structure of the control PU and the PU composites can be determined by measuring the heat release. The heat released from combustion or the enthalpy of combustion was determined with a bomb calorimeter. Figure 1 shows the enthalpy of the control PU and the PU composites. It decreased by increasing amount of FR. This trend is in good agreement with most of the results reported in the literature (Jang et al. 1998; Lewin 2001) where they used organophosphorus compounds and combined them with montmorillonite nanoparticles and used it to retard flame and incorporate them in epoxy resin.

The highest enthalpy was observed on the control PU as shown in Figure 1. The enthalpy of control PU was 308.6 $MJ \cdot kg^{-1}$. When 2 wt % FR was added to the PU matrix the enthalpy decreased by 25%. PU composite with 4 wt% FR showed enthalpy of 303.4 $MJ \cdot kg^{-1}$. The lowest enthalpy was observed for PU composite with 6 wt% FR where the reduction was 24.7%. This is attributed to the fact that less energy was required to break the bond in PU composites because of the presence of FR which played an important role in the combustion process where it released free radicals which bond to the oxygen during combustion process (Twarowski 1993). This reduced the hydrogen atoms and breaks the organic chain and resulted in lower burning speed of the materials (Twarowski 1993; Korobeinichev et al. 1999; Korobeinichev et al. 2004).

The burning process of polymers is generally described in three stages: fuel production, ignition and then burning. When a solid polymer is heated with rising temperature, eventually the polymer started to degrade.

TABLE 1. The mechanical properties of the PU composites with 0, 2, 4 and 6 wt% FR

FR content (wt %)	Shore D Hardness (arbitrary)	Impact Strength ($kJ \cdot m^{-2}$)	Flexural Strength (MPa)	Flexural modulus (MPa)
0	37	7.0	33.07	1026
2	40	6.4	31.18	972
4	47	6.1	24.64	860
6	55	5.8	22.02	827

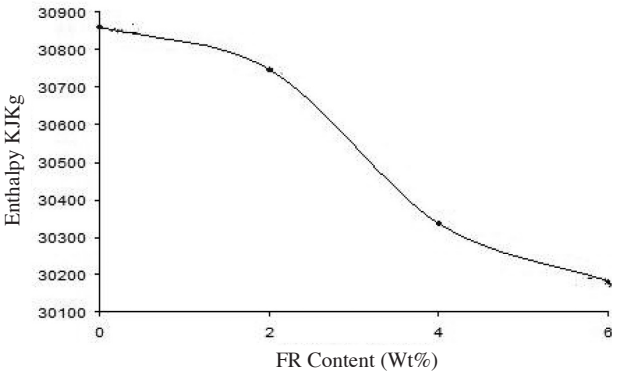

FIGURE 1. Heat released (combustion enthalpy) of the control PU and PU composites

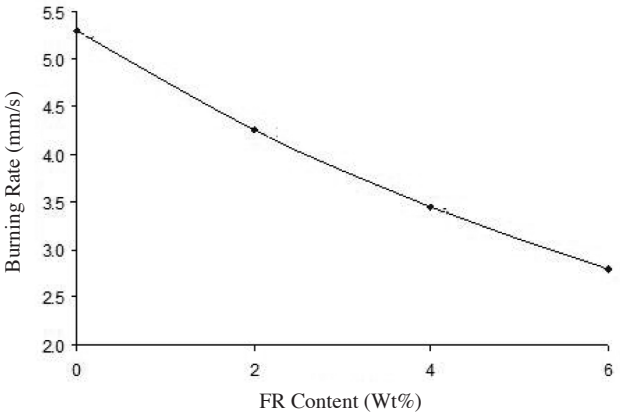

FIGURE 2. Burning rate of the control PU and the PU composites

During degradation, small molecules were produced in which the combustible compounds were evaporated and mixed with air, forming a flammable mixture. When this mixture is concentrated and the temperature also reached flammability limits, it started to burn. The exothermic heat from the burning process fed back to the condensed phase, causing further degradation of the polymer. In order to stop or retard the burning process, flame retardants are generally added to the polymers. A variety of mechanisms such as gas-phase retardation, condensed-phase retardation and dissipation of combustion heat can fulfill the retardation (Wang et al. 1999). To end the combustion, there is need to interrupt the burning cycle. Once a fire has already begun, flame retardants can reduce the tendency of the fire to spread by reacting with the product and producing a less flammable char or noncombustible gaseous layer along the boundary of the fire (Kirk Othmer 2001).

Flammability tests are classified based on various characteristics of fire response. In various flammability tests, the fire test is carried out to determine the relative burning characteristic and flame resistance properties. Phosphite used in this study has good flame retardancy and good thermal properties because the hydroperoxide decomposers prevent the split of hydroperoxides into highly reactive alkoxy and hydroxyl radicals (Schwetlick 1990). Figure 2 shows the results from the fire test for the control PU and the PU composites with varying amount of FR.

The PU burnt rapidly after exposure to the flame and was totally consumed. The rate of burning of the control PU was higher than the PU composites. This is attributed to the high flammability of PU which is highly combustible in the absence of flame retardant (Reed et al. 2000) as supported by Figure 2. The incorporation of FR into the PU system resulted in significant decrease in the burning rate. At 2 wt% of FR, the burning rate of PU composites decreased by 26.6%. Same trend in the reduction of the burning rate was observed for the PU composite with 4 wt% FR. It was decreased by 21.5%. The PU composite with 6 wt% FR has the lowest burning rate with a reduction of 17.7%. The FR has a good flame retardant effect on the PU composites as reported in previous work (Shelton 1997) where the FR presenting high fire-retardancy property. It has been reported that phosphorus-oxygen species (PO) in the vapor phase, which will react with hydrogen radicals, can act as an active flame poison (Spirckel et al. 2002).

The P–O–C bonds break more easily than the C–O–C bonds, forming P–O–P bonds. The presence of phosphorus promotes the formation of compact char, which protects the underlying polymeric materials from further attack from flame or heating. Generally, phosphites react with hydroperoxides to yield inactive products (ROH) according to the following general reaction:

$$(RO)_3 - P + ROOH \rightarrow (RO)_3 - P = O + ROH.$$

Alkyl radical, R is very unstable because it tends to be involved in oxygen activity. The possibility for the radical to react with FR is very low. Therefore, most FR will react with peroxide free radical and hindering alkyl radical formation (Shelton 1997) as shown below where A representing the FR:

$$\begin{array}{llllll} ROO\bullet & + & AH & \rightarrow & ROO\bullet & + \ A\,(FR) \\ 2\,ROO\bullet & + & AH & \rightarrow & ROOH & + \ ROOA \\ \hline ROO\bullet & + & A & \rightarrow & ROOA & \end{array}.$$

The FR cut the chain by changing R. radical in the propagation step with aromatic A radical. This process only gives yield if A does not participate in radical transferring reaction with the polymer chain. Electron transfer might occur as well. Some FR or its reaction products can act as alkyl radical capture (Quan 1973).

SEM micrographs were used to observe the adhesion and distribution of FR in PU composites using the impact fractured samples. The observation was made on impact fractured samples of the control PU (0% flame retardant) and polyurethane composites with 2%, 4% and 6% FR as shown in Figure 3, 4, 5 and 6. They support the findings in the analysis of mechanical properties. Figure 3 shows SEM micrographs for fractured surface of control PU. The control PU has uniform structure with homogeneity in the

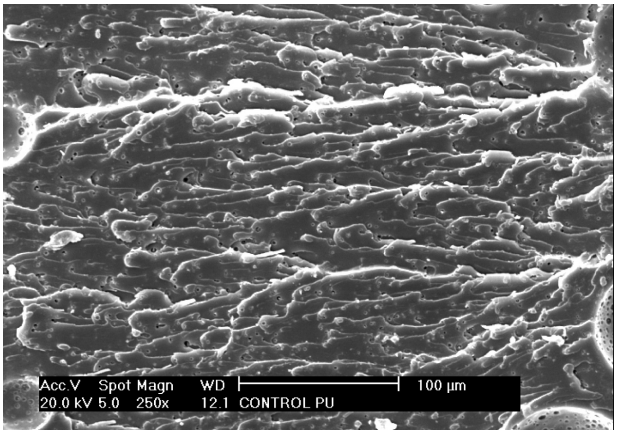

FIGURE 3. SEM micrograph of PU control

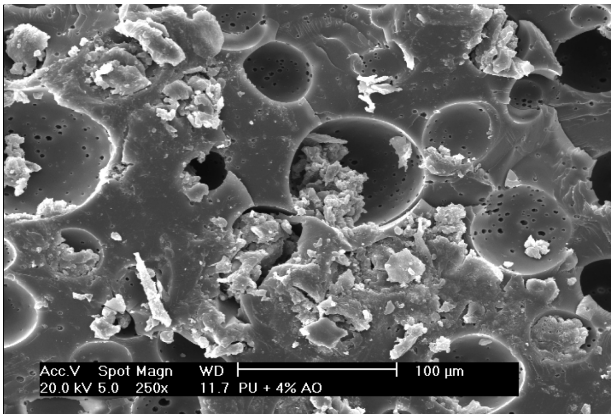

FIGURE 5. SEM micrograph of PU composite with 4 wt% FR

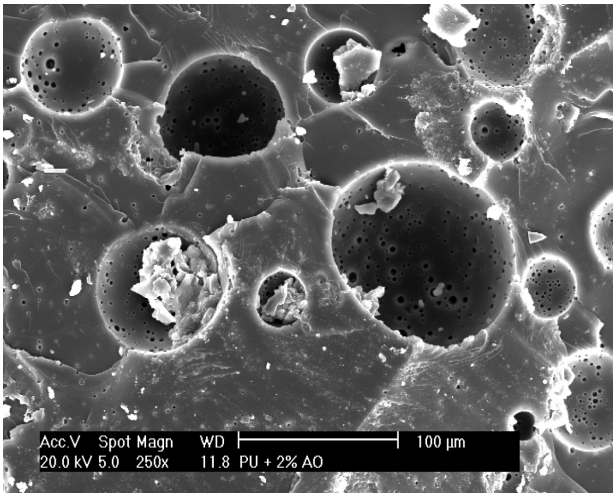

FIGURE 4. SEM micrograph of PU composite with 2 wt% FR

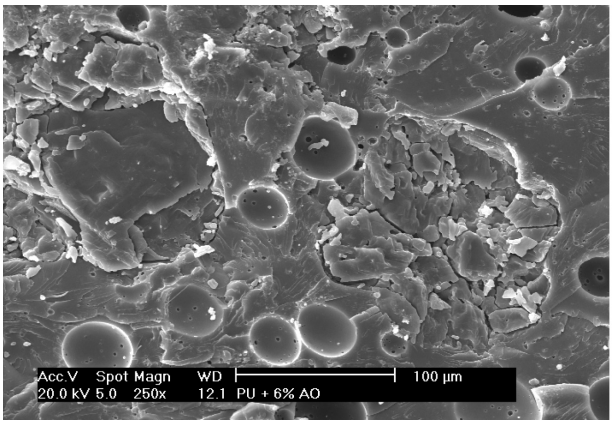

FIGURE 6. SEM micrograph of PU composite with 6 wt% FR

matrix. The SEM micrograph for PU composite with 2% of FR indicated that the FR particles are not evenly dispersed throughout the PU matrix as shown in Figure 4. This uneven distribution between FR particles and PU matrix could be the reason to the weak interaction between FR particles and PU matrix, indicated by the poor mechanical properties of the PUcomposites. Increase of FR content in the PU matrix by 4% causes initial agglomeration of particles, affecting adversely on dispersion and distribution of FR particles in the PU matrix. This led to further reduction in the mechanical properties of PU composites; due to the poor adhesion between FR and PU matrix as shown in Figure 5. Agglomeration was obvious and was observed for PU composite with 6% FR as shown in Figure 6. The agglomeration became dominant at higher FR content. Since agglomeration of FR increase, the uniform distribution of PU composites decreased thus directly affecting the mechanical properties of PU composite (Sumaila et al. 2006). The occurrence of particles agglomeration is responsible for the FR – FR particle contacts instead of PU – FR particle contacts. This shows that there is only physical interaction between FR particles and polyurethane matrix.

CONCLUSION

This study shows the effect of 2, 4-ditert-butylphenyl phosphite (FR) as a fire-retardant on the mechanical and thermal properties of palm-based polyurethane. The FR content was varied at 2, 4 and 6 wt%. The investigation on the mechanical properties showed that the hardness index was obviously increased with increasing amount of FR. The impact strength of the FR-filled PU composites decreased with increasing FR content. This could be linked to weaken interfacial bonding between the filler and the PU matrix with higher amount of FR. The PU composite with 6 wt% FR was observed to have the lowest impact strength compared to others. The impact strength decreased with increasing FR. The optimum loading was observed at 2% FR. The presence of higher loading of FR embedded in the polymer matrix results in reduced ability to absorb impact energy. The FR created matrix discontinuity, and each particle acts as site of stress concentration, and leads to micro-crack. Higher amount of FR gave the lowest flexural strength and modulus. Higher loading of FR decreased the wetting properties of the matrix and reduced the degree of encapsulation of matrix around the flame retardant. As such, the flame retardant was exposed to direct stress and low stress transfer. This may also lead to weak interfacial

bonding between the FR and the PU matrix. The burning rate of control PU was higher than FR-filled PU composites due to the high flammability of control PU. The fire test indicated lower burning rate as the percentage loading of FR increased. The enthalpy determined by bomb calorimetry supported the burning test results. The combustion enthalpy of the control PU was 7371 cal/g and dropped 25% when 2 wt% FR was added to the PU matrix. The lowest enthalpy was observed for PU composite with 6 wt% FR.

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