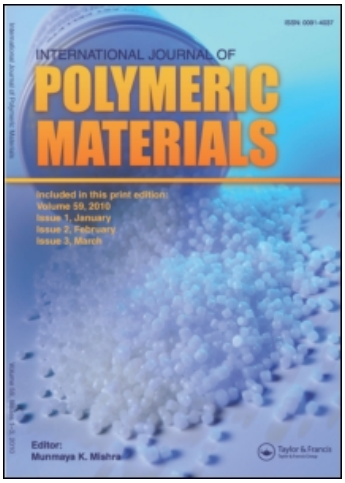

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Fire Resistant Sheet Moulding Composites from Hybrid Reinforcements of Oil Palm-Fibres and Glass Fibre

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Fire resistant sheet moulding compounds based on biofibre-glass hybrid reinforcements were developed and the efficacy of the fire retardancy was determined by the measurement of the limiting oxygen index and the results were correlated with the differential scanning colorimetric data.

Keywords: Bio-fibres; oil palm trunk; fronds; empty fruit bunch; unsaturated polyester; limiting oxygen index; DSC thermograms

INTRODUCTION

Thermoplastic and thermosetting polymers are generally reinforced with fibrous materials to improve their physical and mechanical properties and are combined with fillers to further improve the properties but more specifically to reduce the final cost. Sheet Moulding Compounds or Sheet Moulding Composites (SMC) represent such reinforced and filled polymers [1]. Sheet Moulding compounds are versatile reinforced composites which can be made from indefinite number of possible formulations ideally suited for an extensive range of application areas such as in transportation, appliances, construction, electrical and

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chemical industries. Of these, automotive industry has the highest level of consumption for SMC. SMC technology comprises two distinct steps: compounding and moulding. In the compounding operations all ingredients except fibres are mixed together to form the paste. The fibres are then wetted with this paste to form the final compound. The basic resins are thermosetting type such as unsaturated polyester, epoxy resins or phenolic resins. There are a number of additives which are incorporated into the resin paste formulations for specific functions namely latent catalysts, internal releasing agents, low profile additives, thickening agents, rheological modifiers, fire retardant additives, inhibitors, etc. Fillers and fibres constitute major components which impart the desired physical and mechanical properties. In short the SMC technology provides ample opportunities for engineers to develop composite materials with engineered performance characteristics and durability.

The fibre reinforcement employed for the production of SMC is glass fibre which are in the form of chopped strands and are randomly and uniformly distributed in the entire composite. Natural fibres which have gained significant importance in recent times as reinforcing fibres in synthetic polymers, offer excellent potential to replace partically or completely glass fibres in the SMC. Natural fibrous biomass which can be renewed in perpetuity can be utilized as the component of hybrid reinforcements together with synthetic fibres such as glass fibre in the composite materials. Opportunities that such composites offer in terms of optimum performance, minimized weight, cost effectiveness and controlled biodegradability among many other advantages are available for a wide range of application areas. When biofibres are considered for reinforcement in composites, two broad categories have been identified [2].

1. Price-driven composites for which cost dictates the market.
2. Performance-driven composites for which properties dictate the market.

In general bio-fibre based composites are mainly price-driven commodity composites which will have just adequate properties for the desired performance at relatively low cost. Glass fibres are still the most widely used reinforcement in the SMC. The fibre-glass is obtained from non-renewable resources by processes which are capital

and energy intensive. Fibre-glass further causes substantial damage to expensive moulds due to severe abrasive actions. If organic fibres are employed either exclusively or in hybrid combination with glass-fibre in the manufacture of reinforced composites, one would expect not only substantial reductions in the cost of production of the product, but also convert the otherwise un- and underutilized bio-fibres into assets.

A wide variety and grade of natural fibres are available. Different types of fibres can be obtained from different parts of the same plant. For instance different grades of fibres can be obtained from oil palm tree such as oil palm trunk (OPT), fronds, empty fruit bunches, palm fruit pressed fibres and shells. An earlier paper [3] described the use of fruit pressed fibre for the production of SMC. Malaysia has over 2–5 million hectors under palm oil tree cultivation. There are over 300 palm oil mills operating in Malaysia crushing palm fruit into crude oil. The by-products of the mill are Empty Palm Oil Fruit Bunches (EFB)* and palm fruit pressed fibres. They have no commercial value**. At the moment they are either burnt as fuel for the boiler or used for mulching purposes in the fields. In both the above disposal the fibre nature of the material is not utilized.

Development of composite materials from hybrid reinforcement consisting of bio-fibres and glass appears to be challenging. Besides the physical and mechanical properties, fire resistance of the composites is extremely important. Investigations were therefore taken up to develop matrix compositions to impart fire-resistance to the final composite. This paper presents results on the development of fire resistance SMC bases on hybrid reinforcement consisting of Empty Fruit Bunch Fibres and glass fibre.

MATERIALS AND METHODS

Unsaturated polyester resin Revesol P9728 from M/s Revertex (M) Sdn Bhd., Johor Baru, Malaysia was used. Empty fruit bunch fibres were obtained from Malpom Industries Bhd, Sungai Bakap, Malaysia.

*EFB waste generated by the oil palm industry in Malaysia is estimated to be about 8 million tonnes per year.

**EFB is readily available at a very low cost at a typical token price of RM 10.00 per tonne.

Fibreglass chopped strands were cut from E-glass ravings. Calcium carbonate used as fillers were OMYA BLR-3, Hydrocarb and Millicarb. Tetrabromophosphate ester used was Reoflam PB 460 from FMC. Zinc Borate empolyed in the present work was Fibrebrake 290 from US Borax. Aluminium Trihydrate was obtained from Alcan chemicals.

Silicone fluid used was SFR 100 obtained from GE silicones. Magnesium carbonate was obtained from Morton International and was of particle size less than 10 micron. Other chemicals were used as obtained from laboratory chemical suppliers.

Methods

1. Preparation of Non-Woven Mat

Both the biofibre mats as well as the hybrid mats were made by dispersing the fibres in water and poured into a 'deckle box' provided with a sieve at the bottom. Water drained through the sieve and mat of uniform composition was formed on the sieve. The water was squeezed out from the mat by hydraulic press and the mat was dried for about 3 hours at 100–105°C. Throughout the experiments a ratio of biofibre to glass was maintained at 7:3 by weight based on the results reported earlier [3].

2. SMC Paste Formulations

Formulation ingredients such as unsaturated polyester, inorganic fillers (OMYA BLR 3, Millicarb, Hydrocarb etc) fire retardant additives, internal release agents (Zinc stearate), catalyst (*t*-butyl perbenzoate), thickener (MgO) were mixed together thoroughly for about 20 minutes to obtain formulations ready for impregnation with reinforcement mat.

The quantities of the ingredients (other than filler and fire retardant chemicals) in the paste formulations are given below.

Unsaturated polyester resin	1.0 part
Thickening agent	0.012 part
Internal mould releasing agent (Zinc stearate)	0.010 part
Styrene	0.050 part
Catalyst (*t*-butyl perbenzoate)	0.040 part

The quantities of inert and fire retardant fillers/additives added to the above mix are given in Table I. The formulations FC1 and FC2 in the column 1 of the Table I are the controls and contain only the calcium carbonate filler. The formulations F1 to F6 contain different fire retardant fillers and additives as described in the table.

3. Mat Impregnation

The requisite amount of paste as prepared above was applied on two carrier films (HDPE) and the hybrid mat comprising of glassfibre and biofibre (in the weight ratio 3:7) was placed sandwiched between the resin paste and squeezed by a hand roller to promote good impregnation of the mat with the resin paste. The resin paste content was 70% in the SMC by weight. The compounded sheet was then stored to mature in controlled environment until tackfree and leathery in consistency.

TABLE I Quantities of filler, fire retardant fillers/additive

Sample	*Filler*	*Reinforcement*	*Fire Retardant Additive and Remarks*
FC1	Omya HydroCarb $CaCO_3$	Empty Fruit Bunch Palm Fibre	No fire retardant additive or fillers (Control)
FC2	Omya HydroCarb $CaCO_3$	Empty Fruit Bunch Palm Fibre + Glass Fibre	No fire retardant additive or fillers (Control)
F1	Aluminium Trihydroxide	Empty Fruit Bunch Palm Fibre + Glass Fibre	Aluminium Trihydroxide Filler cum for retardant
F2	50% Aluminium Trihydroxide + 50% Zinc Borate	Empty Fruit Bunch Palm Fibre + Glass Fibre	50% Aluminium Trihydroxide + 50% Zinc Borate Filler cum fire retardant
F3	Aluminium Trihydroxide	Empty Fruit Bunch Palm Fibre + Glass Fibre	Brominated Phosphate Ester (6 phr) Fire retardant additive
F4	Magnesium Carbonate	Empty Fruit Bunch Palm Fibre + Glass Fibre	Magnesium Carbonate filler cum fire retardant
F5	Aluminium Trihydroxide	Empty Fruit Bunch Palm Fibre + Glass Fibre	Silicone Fluid (6 phr) Fire retardant additive
F6	Zinc Borate	Empty Fruit Bunch Palm Fibre + Glass Fibre	Zinc Borate Fire retardant additive

4. Pressing of the SMC

The tack-free SMC sheets of dimension 17.5 cm by 17.5 cm were pressed in a hydraulic hot press at a temperature of 145–150°C and at pressure of 12 Kg/cm^3 in a closed mould. The time of cure depended on the thickness and as a thumb rule, one minute per mm of final thickness of the moulded sheet was adopted.

5. Determination of the Limiting Oxygen Index

The Limiting Oxygen Index (LOI) was determined in accordance with the ASTM D 2863-77 method. Specimens of dimension 70 mm by 150 mm and thickness 6.5 0.5 mm were employed for the test.

6. DSC Measurements

The endothermic effects accompanying the heating of samples (F1 and F2) between room temperature and 300°C were measured in the DSC scan in a Du Pont Instruments Thermal Analyst 2000 at a heating rate of 10 deg. per minute.

RESULTS AND DISCUSSION

The results of the limiting oxygen index (LOI) of the with and without the incorporation of the fire retardant additives are shown in Figure 1. FC1 and FC2 in the figure represent the control and have the LOI values of 23.8 and 24.1 respectively. The LOI values of the samples (F1 and F6) treated with fire retardant additives are also shown in the same figure. It can be observed that all the values are above 30 thus indicating that all the treatments employed in the present investigation produced composites with fire ratings superior to the control. Samples which contained aluminium trihydrate gave LOI values exceeding 40 thus constitutes the most efficacious of the various fire retardant additives employed in the investigation. The aluminium trihydrate used in the fire retardant SMC formulations was ultrafine type. Release of chemically bound water from aluminium trihydrate occurred under the conditions of ignition and this produced an endothermic effect. The moisture evolution could have further reduced the oxygen level at the

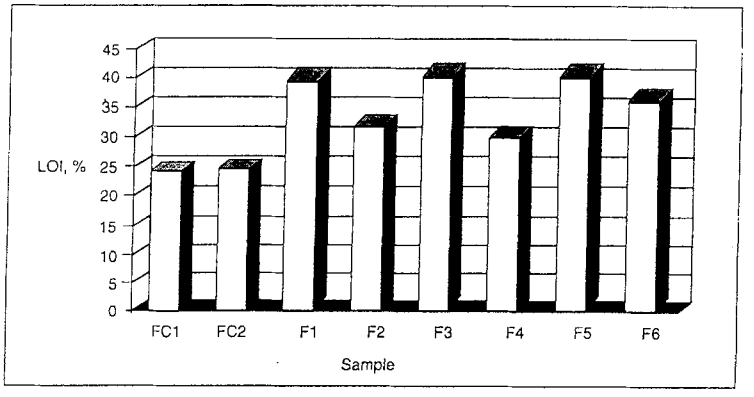

FIGURE 1 Effect of Fire Retardants on the Limiting Oxygen index.

surface and resulted in the self-extinguishing of the flame. With the aluminium trihydrate, relatively much lower viscosity of the resin mix was possible even at higher filler loadings. This in turn not only reduces the cost but also facilitates efficient impregnation of the hybrid reinforcing fibre mat. Besides the above advantages, the material is halogen free and has low toxic hazard in handling. Further the material is cost effective and is reported to give good electrical insulation.

There is a marginal improvement in the LOI values when brominated phosphate ester is added together with the aluminium trihydrate. The brominated phosphate ester was empolyed in the formulation due to its effectiveness as fire retardant by virtue of the presence of both phosphorus and bromine in the same molecule and thus synergistically provide enhanced flame retardancy. Similarly the addition of silicone fluid to the formulations containing aluminium trihydrate improves the LOI values slightly. Addition of zinc borate as filler in the SMC matrix formulation increased the LOI significantly when compared with the control although the values were less than those obtained for samples containing aluminium trihydrate. Much higher values (LOI 44) have been reported [4] for the SMC (based on fibreglass) formulations containing a mixture of zinc borate and halogenated polyester and antimony oxide. However such materials are expensive and will not be justified to be incorporated in formulations meant for price driven bio-fibre composites as in the present case.

Magnesium carbonate gave moderate fire retardancy with the LOI value of above 30. Combination with aluminium trihydroxide may improve performance.

Mechanical properties such as modulus of rupture and impact strength of samples incorporating fire retardant additives are given in Figures 2 and 3. It can be seen that the samples containing aluminium trihydrate gave high values of modulus of rupture and impact strength. The values of water absorption of samples are given in Figure 4. It is to be noted that the water absorption of the samples are quite low despite the presence of bio-fibres as the reinforcement in the composites.

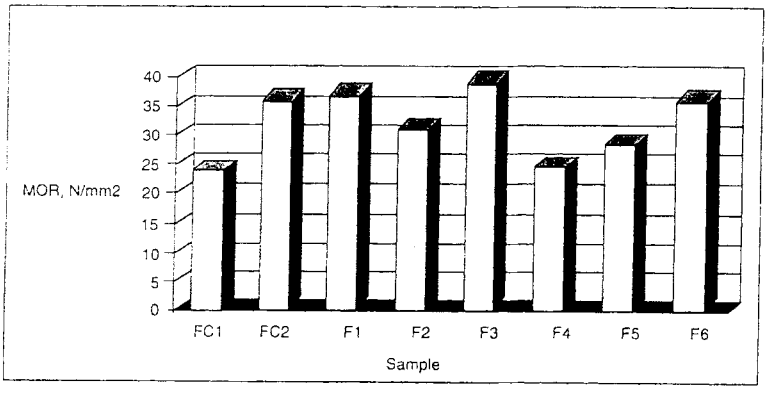

FIGURE 2 Effects of Fire Retardants on MOR.

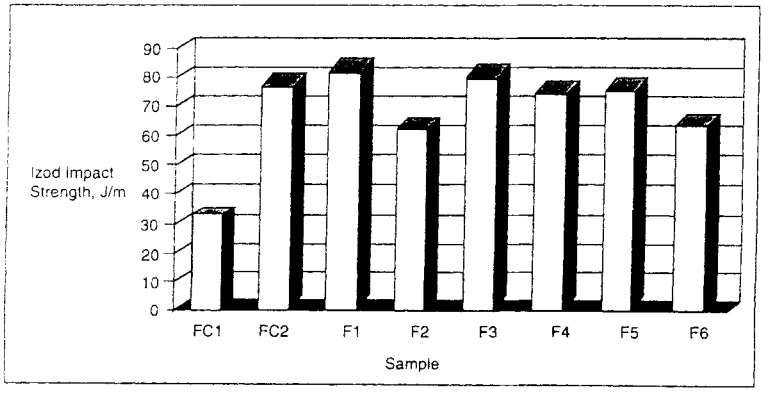

FIGURE 3 Effects of Fire Retardants on Izod Impact Strength.

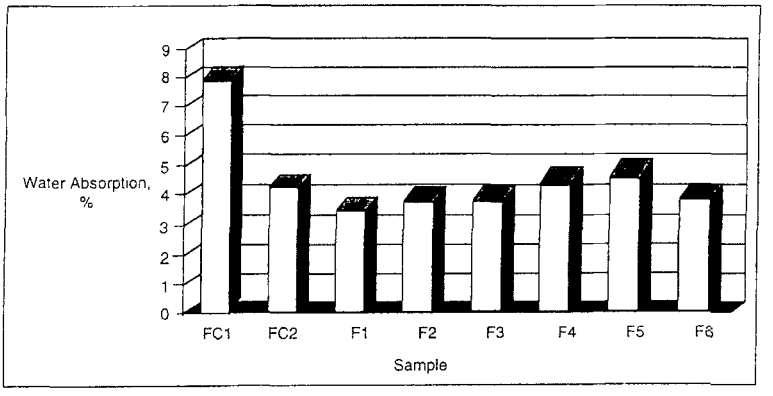

FIGURE 4 Effect of Fire Retardants on the Water Absorption.

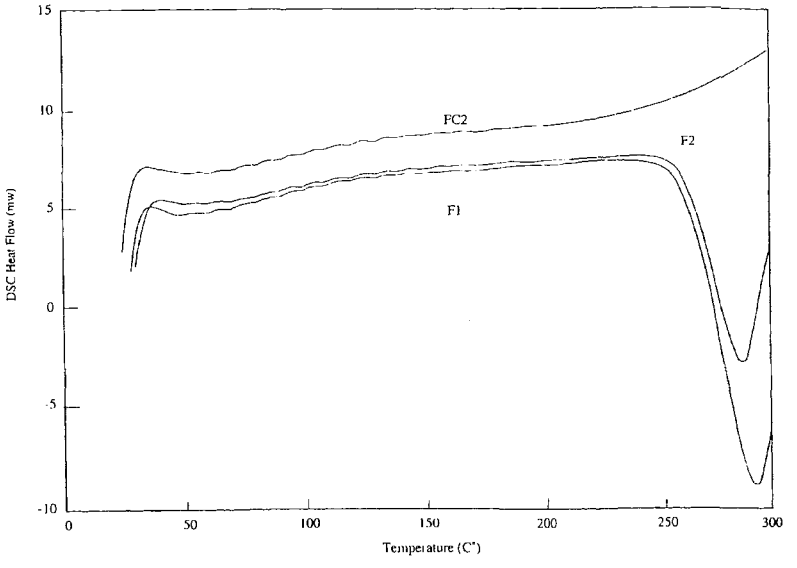

FIGURE 5 DSC Scan of Fire Retardant Treated Bio-composites. FC2 Control, F1 Aluminium Trihydrate, F2 Aluminium Trihydrate and Zinc Borate.

DSC thermograms of the control and samples containing fire retardant additives were obtained from the Du Pont Instruments Thermal Analyst 2000 at a heating rate of 10 deg. per minute in the temperature range 30 to 300°C. The thermograms are given in Figure 5. It can

be seen that the endothermic effects are absent in the case of control. On the other hand aluminium trihydrate and a mixture of aluminium trihydrate and zinc borate produced significant endothermic effect. The endothermic effect of aluminium trihydrate alone is better than the formulations containing the equal percentages of aluminium trihydrate and zinc borate. These results collaborate excellently with LOI values reported earlier in that the LOI values of samples containing aluminium trihydroxide alone is superior to the samples containing a mixture of aluminium trihydrate and zinc borate.

CONCLUSION

It is possible to produce a fire resistant reinforced composite materials from hybrid reinforcement consisting of oil palm tree bio-fibres and glass fibres. The fire resistance ratings as indicated by the Limiting Oxygen Index values are quite high when fire retardant fillers such as aluminium trihydrate, zinc borate, magnesium carbonate and brominated phosphate ester are incorporated in the formulations. The aluminium trihydrate appears to provide the best performance at perhaps the least cost. The inclusion of biofibres as the reinforcing material in the composite does not appear to detract from the fire retardant ratings of the composite.

Acknowledgement

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