

FIRE PERFORMANCE AND TECHNOLOGICAL PROPERTIES OF MEDIUM DENSITY FIBREBOARD MADE FROM RUBBERWOOD TREATED WITH AG[®] PI

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The objectives of this study were to investigate the efficacy of a phosphorous-based fire retardant, AG[®] PI on the fire performance as well as physical and mechanical properties of medium density fibreboard made from rubberwood fibres. The rubberwood fibres were first treated with AG[®] PI at four different concentrations (10%, 15%, 20% and 25%) (w/v) using hot and cold bath processes. The physical and mechanical properties were investigated using the JIS A5906 1983 standard, whereas the fire performance was investigated using an in-house method known as the reaction to fire test. There was significant interaction ($p < 0.05$) between different of AG[®] PI concentrations. Generally, fibreboards treated and manufactured with higher concentrations of AG[®] PI had superior fire performance. The weight loss and burnt area of the fibreboards were found to be reduced with the increase of AG[®] PI concentration. The mechanical properties in terms of modulus of rupture (MOR), modulus of elasticity (MOE) were not significantly affected by the AG[®] PI concentration, even though the values increased with the increase of AG[®] PI concentration up to 20%. However thickness swelling (TS) and internal bond (IB) properties were minimally affected by the increase of AG[®] PI concentration. The values of the two parameters were found decrease when the fire retardant concentration increased.

Keywords: Fire performance; Fire retardant; Concentrations; Hevea brasiliensis; MDF, Technological properties

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INTRODUCTION

Medium density fibreboard (MDF) is an engineered wood product that is formed by breaking down hardwood or softwood residues into fibres, mixing it with wax and a resin, and forming it by applying high temperatures and pressures. MDF is much denser than plywood and particleboard, hence it can be used as a construction material (Anon, 2008). Rubberwood has been documented as a suitable raw material for wood-based panels such as MDF and particleboards (Izran *et al*, in press). Like other wood-based panels, the barrier that may limit the usage of medium density fibreboard, especially for construction is its flammability. Flammability is due not only to the properties of the

fibres, but also dependent on the flammability of the resin. Consequently, fire retardant treatment needs to be performed to reduce the flammability. There are many approaches to accomplish fire retardant treatment. One of the most effective approaches is a heat treatment process. The treatment is done by soaking wood fibres into heated chemical solution (hot soaking) before they are then soaked into chemical solution at ambient temperature (cold soaking). The hot soaking is to swell the wood fibres and to remove extractives in the lumens, whereas the cold soaking is to shrink the wood fibres, hence creating absorption force which encourages the chemical solution to fill in the lumens (Truax 2010; James 2003). Even though the treatment is time consuming and can give adverse effects to wood fibres, it is effective for impregnating the fire retardant deep into wood fibres, thus providing deeper and more durable fire protection. For the treatments, various fire retardants can be utilized. AG[®] PI is one of them.

AG[®] PI is an ammonium polyphosphate (N₄NO₂P)-formulated fire retardant. It is composed of polyphosphoric acid and ammonium salt. This chemical is white in colour, decomposes at 250°C, rapidly decomposes at 300°C, and is considered environmentally friendly. AG[®] PI is commonly used as a flame retardant for plastics, adhesives, elastomers, paints, intumescent coatings, mastics, wood, chipboard as well as paper and textile coatings. It is also used in fertilizers, emulsifiers and stabilizers (Ash and Michael, 2004). There are a number of advantages in using AG[®] PI as a fire retardant. The fire retardant has low solubility, high phosphorous content and is resistant to leaching by water. However, there are limited findings on the efficacy of the fire retardant on wood and wood products (Anon 1999). This study attempted to impregnate different concentrations of AG[®] PI fire retardant into fibreboards made from rubberwood. The efficacy of the treatments was assessed on the resistance of the treated fibreboards to weight loss and area. As additional information, the physical and mechanical properties of the treated fibreboards were also studied.

MATERIALS AND METHOD

Materials

Rubberwood fibres were obtained from Forest Research Institute Malaysia (FRIM). Rubberwood fibres were screened to obtain fibre size between 0.5 to 1 mm. They were oven dried at a temperature of 90°C for approximately 24 hours to achieve a 4% moisture content using an industrial oven for fire retardant treatment. Urea formaldehyde (UF) resin was used as a binder. The properties of the UF resin are as follows: F/U ratio: 2.5, solid content: 55%, viscosity: 45 cps, specific gravity: 1.255 and pH 8.0. It was obtained from Malayan Adhesives and Chemicals Sdn Bhd, Shah Alam, Malaysia. No hardener and wax were mixed with the binder. AG[®] PI was utilized as the treatment chemical, which was sponsored by Koppers Hickson Sdn Bhd.

Fire Retardant Treatment

AG[®] PI was obtained in the form of emulsion. The fire retardant was diluted to different concentrations (10%, 15%, 20% and 25%) with water at a temperature of 80°C as instructed by the supplier. The dried fibres were then dispensed into the solution and

were left for 3h. After that, the solution with fibres was left cooled to ambient temperature. They were then removed from the solution and dried to 4% MC before fabrication of MDF.

Fibreboard Fabrication

The target board size and density were $(340 \times 340 \times 10)$ mm ($l \times w \times t$) and 650 kgm^{-3} respectively. Firstly, the dried fibres were placed in a fibreboard blender which was switched on for five minutes to loosen the fibres in it, so that resin-fibre mixing process will be smoother. After five minutes, with the fibreboard mixer operating, 10% urea formaldehyde resin (based on oven dry weight of the fibres) were sprayed onto the fibres in the mixer via an airless spray gun which was attached on top of the mixer. When the fibres and resin were evenly mixed, they were removed from the mixer and dispersed in a wood former on a caul plate covered with a teflon coated glass fibre. The teflon-coated glass fibre sheet prevents the furnish from sticking on the caul plate during hot pressing process later. A wood block was placed on top of the dispersed mixture and was cold pressed at 35 kg/cm^2 for about 5 min, to form a mat. After the cold press, the wood block was removed and another teflon-coated glassfibre sheet was placed on top of the pressed mixture, before it was pre-pressed at a pressure of 500 psi for 5 min.

The pre-pressed mixture was finally hot pressed at a pressure of 130 kg/m^2 for 12 min. For untreated mixture, the hot pressing time was 7 min. All treated and untreated fibres were pressed to 12mm thickness at a temperature of 170°C to remove excessive moisture from the fibres and resin. Different pressing times were applied for the treated and untreated MDF because the curing rate of the resin was affected by the fire retardant impregnated into the fibres. The effects of fire retardant to curing rate of resin were studied (Izran *et al.*, 2009). A total of 15 fibreboards were manufactured. The fabricated fibreboards were conditioned in a conditioning room ($65 \pm 5 \%$ RH and $27 \pm 2^\circ\text{C}$) for a week before they were trimmed for fire, physical and mechanical testing. Fibreboard size for each test is presented in Table 1.

Table 1. Sample Dimensions for Fire, Physical, and Mechanical Tests of Rubberwood MDF

Name of test	Dimension (mm)
TS and WA	50 x 50
MOR and MOE	20 x thickness + 50 x 50
D	100 x 100
MC	50 x 230
RTF	220 x 220
IB	50 x 50

TS: thickness swelling, WA: water absorption, MOR: modulus of rupture, MOE: modulus of elasticity, D: density, MC: moisture content, RTF: reaction to fire test, IB: internal bond

Fire Performance

Fire performance of the untreated and treated fibreboards was assessed using an in-house method called reaction-to-fire test involving weight loss and burnt area. Sample size for the test was $(220 \times 220 \times 12) \text{ mm}^3$. The samples were first weighed to get their initial weights. A bunsen burner was used as flame source and the distance between the flame and the sample surface was set at 3 cm. The samples were inclined at 45 degrees.

The samples were burnt for two minutes after ignition occurred on the samples. The samples were re-weighed after the test, and the burnt area on the sample surface was determined using graph paper. The weight loss and burnt area were determined using the following equations:

$$\text{Burnt area (\%)} = [Ca / Ta] \times 100\% \quad (1)$$

$$\text{Weight loss (\%)} = [Wi - Wa / Wi] \times 100\% \quad (2)$$

where, Ca is the carbonized area (cm), Ta is the total board area (cm), Wi is the conditioned initial weight (g), and Wa is the conditioned weight after testing (g).

Fibreboard Strength Assessment

The strength of the treated fibreboards was assessed by physical and mechanical test methods outlined in the Japanese Industrial Standard, (JIS A5906, 1983). The moisture content was assessed for two different periods, which were three days and one month after board fabrication to see whether there are any significant differences in moisture content between those two periods. All data were analysed using one-way analysis of variance to determine the differences in properties between fire retardant treatments.

RESULTS AND DISCUSSION

Weight loss and Burnt Area

Lower total weight loss implies higher resistance against thermal degradation of fire, and a smaller burnt area indicates better protection against flame spread. The outcomes of weight loss and burnt area for the treated and untreated fibreboards are given in Table 2. The results indicated that AG[®] PI decreased the weight loss significantly with increasing concentrations from 0% to 25%, whereas it reduced burnt area significantly when its concentration increased to 20%. Increase in the AG[®] PI concentration resulted in decrease of weight loss and burnt area values. The weight loss of the MDF decreased from 7.89% to 2.78%, 1.96%, 1.28% and 1.04%, while the burnt area decreased from 25.76% to 15.84%, 12.19%, 8.33% and 8.13%, as the concentration increased from 0% to 25% respectively.

The active ingredient of AG[®] PI is phosphorous, and like most phosphorous-based fire retardants, AG[®] PI works by enhancing char formation on the sample surface. The protection mechanism of AG[®] PI is as same as other phosphorous-based fire retardants such as monoammonium phosphate and BP[®] which provides a protective layer which subsequently reduces flame spread (Izran et al. 2010b). Previous laboratory tests indicated that AG[®] PI was able to improve flame spread classification to Class 0 (Anon, 1999). The fire retardant successfully reduced flame spread, as indicated by the burnt surface area and impaired thermal degradation of the fibreboards.

Physical and Mechanical Properties

The results of the physical and mechanical properties are presented in Table 3. The treated fibreboards appeared increasingly darker as the treatment concentration increased compared to the untreated. The presence of high concentration of the chemical imparted a dark brown coloration on the boards.

Table 2. Fire Performance of Rubberwood MDF Treated with AG[®] PI

Fire retardant concentrations (%)	^a Weight loss (%)	^a Burnt area (%)
0	7.89 (0.89) ^b	25.76 ^b
10	2.78 (0.29) ^c	15.84 ^c
15	1.96 (0.20) ^d	12.19 ^{cd}
20	1.28 (0.16) ^{de}	8.33 ^d
25	1.04 (0.17) ^f	8.13 ^d

^aMean of three samples

Values in parentheses are standard deviations

¹Means within a column followed by same alphabets are not significantly different (p=0.05)

Physical Properties

Thickness swelling provides a measure of the dimensional stability of the fibreboards. Lower thickness swelling values indicate a more stable board. The effects of moisture on wood-based panels determine their properties and possible uses. Thickness swelling can be affected by many process variables such as wood species, element geometry, board density, resin level, blending efficiency, and pressing conditions (Kojima *et al.*, 2009). For this study, interestingly, the thickness swelling value was found to decrease with increasing AG[®] PI concentrations. The average thickness swelling value for the untreated fibreboards was 26.83%. The values for 10% to 25% concentrations were 26.56%, 23.74%, 23.61% and 20.51% respectively. The effect of AG[®] PI concentration on thickness swelling was not significant (p>0.05). There was also no significant increase in MC between the treated and untreated fibreboards that were conditioned for three days and one month. Generally, fire retardant treatments cause reduction of thickness swelling as they indirectly have an effect on the internal bonds. It has been reported that the internal bond has a direct relationship with thickness swelling (Febrianto *et al.* 2010).

The presence of the AG[®] PI salts in fibreboards prevented strong linking between fibres as the resin is not able to have direct contact with the fibre surfaces, hence reducing the internal bond. Consequently, there was a reduction in thickness swelling. As AG[®] PI is resistant to leaching by water, this advantage makes the chemical acceptable for outdoor use where there is exposure to high humidity (Anon 1999). The water-resistant characteristic also reduces moisture uptake, resulting in dimensionally more stable fibreboards.

Table 3. Physical and mechanical properties of rubberwood fibreboards treated with AG[®] PI

Testing	Fire retardant concentrations (%)				
	0	10	15	20	25
TS (%) ^a	26.83 (4.73)	26.56 (1.28)	23.74 (0.46)	23.61 (1.4)	20.51 (3.32)
MOE (MPa) ^a JIS:2000MPa	953.77 (457.65)	905.17 (343.91)	1184.33 (327.54)	1528.67 (211.39)	790.43 (173.36)
MOR (MPa) ^a JIS: 13.8 MPa	7.98 (3.64)	4.31 (2.17)	5.61 (1.72)	6.43 (1.27)	2.41 (0.56)
D (kgm ⁻³) ^a JIS: 700kgm ⁻³	703.00 (83.34)	625.60 (55.20)	671.10 (21.60)	737.70 (23.40)	723.60 (30.20)
MC1 (%) ^a	12.190	13.16	13.537	13.06	13.25
MC2 (%) ^a	13.25	14.42	14.57	14.41	14.39
IB (kgcm ⁻²) ^a JIS: 3.87 kgcm ⁻²	0.13 (0.04)	0.06 (0.09)	0.05 (0.06)	0.04 (0.02)	0.04 (0.02)

MC1 = MC after three days, TS= thickness swelling, MOE = modulus of elasticity
 MC2 = MC after 1 month, MOR = modulus of rupture, D = density, IB = internal bond
^aMeans of three samples, Values in parentheses are standard deviations.

Table 4. Summary of ANOVA for the Effects of Different Fire Retardant Concentrations on the Properties of Rubberwood Particleboards

Property	TS	MOR	MOE	IB	WL	BA
	Cont. (%)	Cont. (%)	Cont. (%)	Cont. (%)	Cont. (%)	Cont. (%)
F-value	2.68	2.93	2.52	2.54	122.6	19.43
P-value	0.0939	0.08	0.11	0.01	0.01	0.01
Significant level	ns	ns	ns	*	*	*

*=significant at p<0.05; Cont= Concentration; TS=thickness swelling, MOR=modulus of rupture, MOE=modulus of elasticity; IB=internal bond; WL= weight loss; BA= burnt area

Mechanical Properties

Modulus of rupture (MOR) is identified as the force necessary to break a specimen of specific width and thickness. The other definition is the maximum fibre stress at failure. Modulus of rupture is also known as flexural strength or torsional strength (Anon 2010). To establish modulus of rupture, a sample with a specified cross section and length is subjected to increasing force until it breaks. The force at the point of breaking is related to modulus of rupture. Modulus of rupture can only be determined for brittle materials (Anon 2007) such as fibreboard.

For this study, contradictory results were found for MOR and MOE. The fire retardant treatment did not significantly influence the MOR and MOE values. For MOE, the highest mean value was 1528.67 MPa, which was obtained from fibreboards treated at 20% concentration. The lowest mean value was recorded from fibreboards treated at 10% concentration. The original stiffness was 953.77 MPa. MOE mean values for those treated at 15% and 25% concentration were 1184.33 MPa and 790.43 MPa respectively.

As for MOR, the trend of results was almost similar to MOE excluding fibreboards treated at 10% and 25% concentrations. This time, fibreboards treated at 25% concentration showed the lowest MOR mean value of 2.41 MPa. The original strength was 7.98 MPa and this decreased to 4.31 MPa with the 10% treatment. However, at 15% concentration, the MOE increased to 5.61 MPa at 15% concentration. The stiffness increased to 6.43 MPa at 20% concentration, before it declined sharply to 2.41 MPa at 25% concentration. The reductions may have been caused by the heat from the hot-pressing process. Pan et al., (2010) reported that between 120°C and 210°C, the natural fibres tend to be desiccated and produce water vapour and other non-combustible gases and liquids including carbon dioxide, formic acid, acetic acid, glyoxal, and water. Cellulose is often thought to be primarily responsible for the strength of the wood fibre; therefore, reducing the length of the cellulose molecules (degree of polymerization) would cause a reduction in macro-strength properties. This theory of hydrolytic cellulose depolymerization was originally proposed by Ifju (1964) and modified to also include hemicelluloses by Sweet and Winandy (1999). The increase in brittleness of the phosphorous fire retardant treated MDF panels due to chemical action of acid was observed. Brittleness of the panels was probably due to embrittlement of the wood fibers caused by crystal formation within the wood cell-walls or cross-linking between cellulose or hemicellulose molecules.

The internal bond test is commonly used as a fundamental indicator of the adhesive performance in wood composites. This test method covers the determination of tensile strength properties of the tested boards or adhesive bonds in wood. From the results of the study, it was apparent that the internal bond of the treated fibreboards was inferior to the untreated. The original internal bond mean value was 0.13 kgcm⁻². A similar result was found in a previous study on MDF containing mono- and di-ammonium phosphate (Ayrilmis 2007). The IB strength losses noted in the results are probably jointly related to three issues. One is that the fire retardants may be causing chemical and/or mechanical changes in the wood cell-wall structure and chemistry. The second is that some fire retardants may inhibit or accelerate curing of resins by altering the requisite pH of the resin during curing. The third is that contamination of wood fibre surface by the presence of loosely adhering crystalline deposits of fire retardants at the glue-line may interfere with the attainment of intimate fibre-to-fibre contact, which is important to maximum bond strength (Ayrilmis 2007).

The reduction in internal bond value is often due to inadequate curing of the resin. Inadequate curing prevents the resin from creating strong links between fibres in the fibreboards, thus causing swelling. Urea formaldehyde resin is a binder that has high water content of approximately 14.6%. To achieve ample curing of the resin, it is important to remove moisture from the resin during hot pressing. This is why a temperature as high as 170°C was set during hot pressing for this study.

Previous research by Zaidon et al. (1998) reported that, suitable hot pressing temperature for UF resin was 125°C, but for this study, the temperature was increased to 170°C to eliminate excessive moisture in the fibres and resin, thus increasing the efficiency of resin curing. To determine accurate hot pressing time, it is best to conduct a gelation time test as well as buffering capacity test (Izran *et al.* 2009; Izran *et al.* 2010). Determining the hot-pressing time will not only help in accomplishing adequate curing of the resin and producing boards with good mechanical properties, but also prevent surface burns of the boards caused by overexposed to heat. The hot-pressing efficiency can also be increased if different pressing pressures are used (Izran 2009).

CONCLUSIONS

1. AG® PI was successfully impregnated into rubberwood fibres through heat treatment using hot and cold bath processes. The fire retardant was effective in improving fire performance by reducing thermal degradation as well as flame spread of the treated fibreboards.
2. The chemical imparts a darker brown coloration on treated fibreboards. There was slight increase in water absorption and moisture content caused by the fire retardant in the MDF. However, the increase was not statistically significant ($p>0.05$).
3. Inconsistent modulus of rupture and modulus of elasticity results were observed. Decrease of MOR and MOE values happened at the treatment concentrations of 10% and 25%. The strength reductions in the specimens treated with the phosphorous fire retardants were probably caused by a combination of accelerated resin cure and thermal decomposition.
4. The IB was also not significantly affected by the fire retardant, though there was slight decrease with the increase in fire retardant concentrations.
5. The study indicated that AG® PI has good potential for use as a fire retardant treatment chemical for rubberwood MDF due to the improved fire performance attributes without having adverse effects on the physical and mechanical properties.

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