

Development and Evaluation on the Performance of Fire Retardant Fibreboards

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IRPA Project Number : 03-02-05-2087 EA 002

Acknowledgement

We would like to sincerely acknowledge the MOSTE for financial support from the IRPA grant; Borden Chemical (M) Sdn. Bhd for providing the resin samples, Merbok MDF (Sdn) Bhd for supplying the refined rubberwood fibres, Borax USA/Singapore for providing the zinc borates, Dr. Richard Murphy Imperial College, UK for the collaboration work and providing samples of ATH and Puan Zalila Ali from the School of Mathematics USM for her helping hand in the statistical analysis.

Introduction

Medium density fibreboard (MDF) is a widely used wood composite in furniture manufacture and building construction. In such application resistance to flame spread and fire is an advantage. The properties of MDF can be improved with the use of flame retardant chemicals and several studies have investigated the fire retardant treatment of wood based composites (Hashim et al. 2005; Hashim et al. 2002; Grexa, et al. 2003; Kozlowski et al. 1999). There are three main groups of flame retardant chemicals; a) inorganic eg. metal hydroxides, antimony compounds, boron compounds and phosphorus compounds; b) halogenated products based primarily on chlorine and bromine; c; organophosphorus products which primarily rely upon phosphate esters (Anon., 1997). For wood and wood products, the most commonly used fire retardant chemicals include borax-boric acid, zinc borates, chromated zinc chloride, ammonium sulphate, and nitrogen-phosphate mixtures (Holmes, 1977; Myers and Holmes, 1975; Winandy et al. 1988; White and Sweet, 1992; Sain, et al., 2004). The main ways of application of flame retardant to wood and wood products are by impregnation of wood with flame retardant materials, incorporation of the flame retardant into the glue system or surface treatment of the products. Continued interest have been shown on the need of flame retardant treatment of wood based materials. This is shown by the studies conducted from researchers around the world (White and Sweet, 1992, Le Van and Winandy, 1989; Le Van et al., 1996 and Grexa and Lubke , 2001).

Experimental

In this study undertaken, the development of the flame retardant treatment using bio resources available in Malaysia was focused. Refined Rubberwood fibres and refined empty fruit bunch fibres (EFB) were used to manufacture experimental MDF panels using dry process.

Experimental MDF of dimension 21.2 x 21.2 x 0.5 cm and target density of 0.7gm/cm³ were made using a small scale laboratory press. The boards were made either from thermo mechanical processed rubberwood (*Hevea brasiliensis*) fibers free from resin or refined empty fruit bunch fibres obtained from MDF mill in Malaysia. The adhesive used were phenol formaldehyde and urea formaldehyde at 15% resin level based on oven dry weight of the boards.

Flame retardant chemicals used were sodium aluminate, zinc borate, and aluminum trihydrate. Four concentrations of flame retardant treatment were used; 10%, 15%, 20% and 30% based on the oven dried weight of fibers along with control without flame retardant chemicals. For zinc borate and sodium aluminate, they were incorporated in the resin mix during blending. For ATH application, the chemicals were scattered evenly onto the fibres manually. A resin was then incorporated with the treated fibres in the blender. The press time was 10 minutes at 180°C with a pressure of 12N/mm². A total of 4 panels were made for each concentration along with the control. The boards were then conditioned to standard procedure of 65% relative humidity with temperature of 20°C prior to testing. Figure 1 and 2 shows an overview of the experimental procedures.

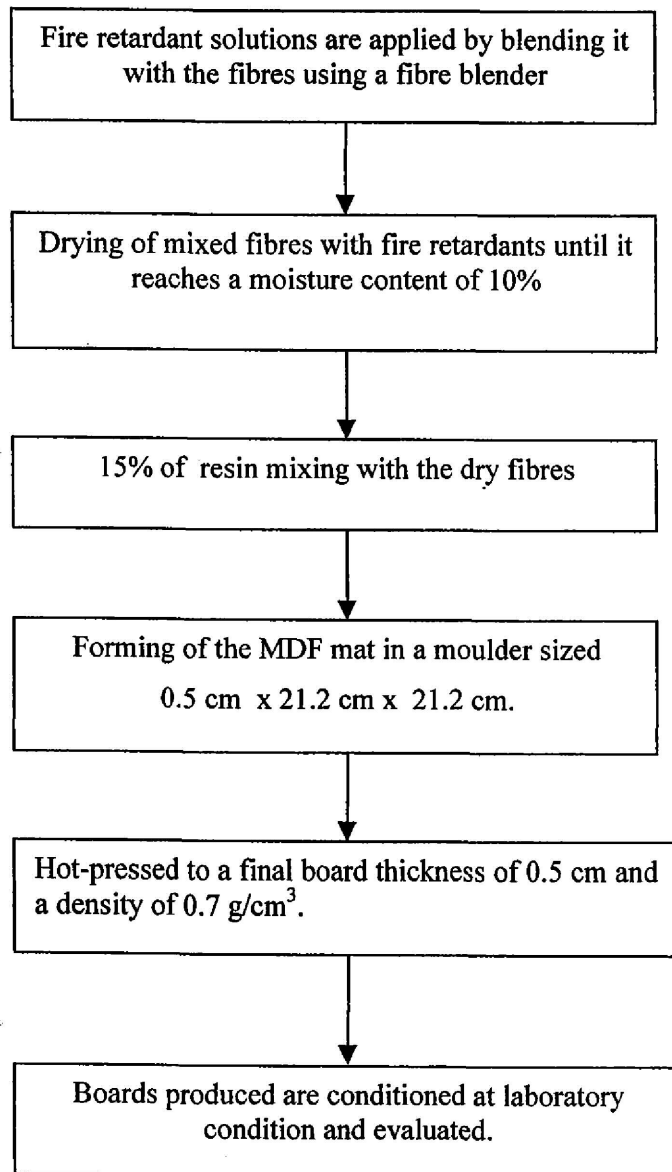


Figure 1- Production of FRTMDF using sodium aluminate and zinc borate

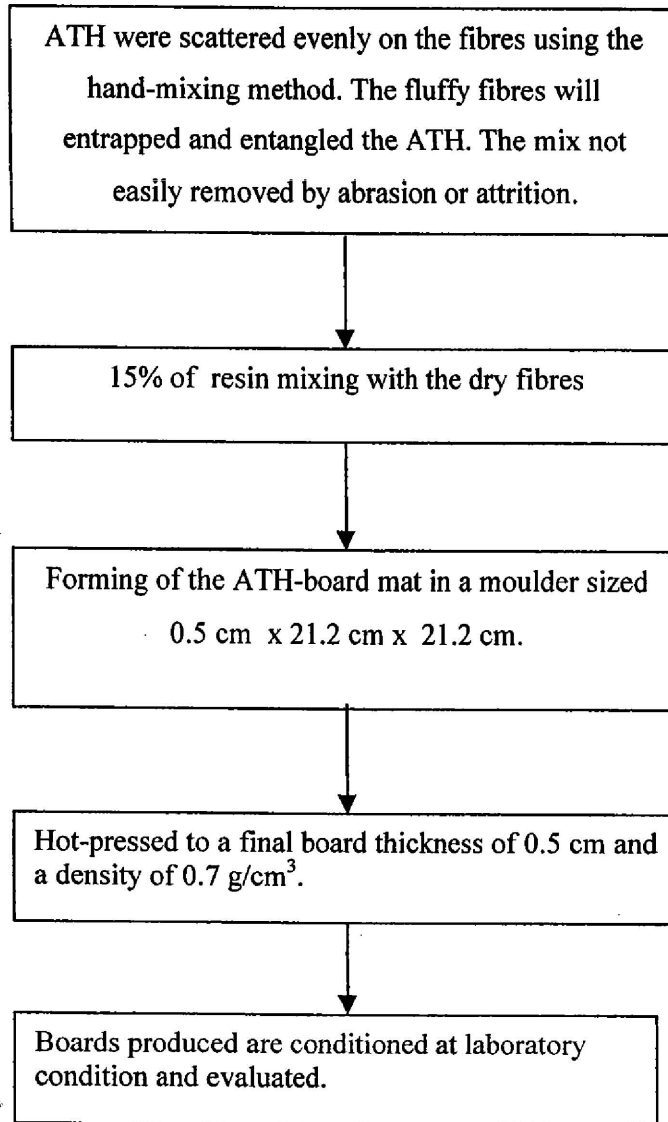


Figure 2 - Production of FRTMDF using of ATH

Evaluation

Mechanical testing

Evaluation on the mechanical properties and physical properties of the boards were conducted. Bending strength (MOR) was carried out according to ISO 16978(2002) with modification of size use 120 mm x 20 mm x 5 mm. The evaluation of internal bond strength (IB) was carried out in accordance with ISO 16984 (2002). Thickness swelling and water absorption of MDF were done in accordance with ISO 16983 (2002). For each test, there were 8 replicates and 3 samples from each panel. The cyclic test was carried out in accordance with ISO 16987 (2002). The boil test was carried out in accordance with ISO 16998 (2002).

A gelling time of the mixture of fire retardant chemical and UF resin was carried out to investigate whether the fire retardant chemicals interfere with the resin in 100°C distilled water. The formation of adhesive film and its cure was further conducted using phenol formaldehyde resin. Phenol formaldehyde resin and flame retardant chemicals were coated on the same glass plate and cure at a temperature of 50°C for 2 minutes. The cured film was examined by light microscope and image analyzer to find out the nature of the film cured.

Fire testing

Fire tests used to evaluate the samples include Thermogravimetry (TG), Differential Scanning Calorimeter (DSC), and cabinet test (ASTM D 1360-79).

DSC is a well established method particularly in evaluating the heat pyrolysis of each samples. This analysis was carried out using Perkin-Elmer Pyris-1 DSC, equipped with an internal Cooler 2P-cooling accessory and calibrated using n-decane and indium. Samples (5-10mg) were examined within an atmosphere of dry N₂ gas maintained at a flow rate of 20mL/min. Samples were encapsulated in standard aluminium pans and an empty aluminium pan were used as a reference. All samples were annealed at heating rate of 20°C/min.

Thermal weight loss measurements were made using Perkin-Elmer TGA-7 thermogravimetric analyzer (TGA). Testing were carried out under a stream of dry N₂ gas/O₂ gas (flow rate = 30mL/min) at a temperature range of 30-300°C with heating rate of 20°C/min. Weight of samples used was within 4-10mg.

Char index, weight loss and area of ellipse were obtained from the cabinet method ASTM D 1360-79 with some modifications. It is originally a standard test method for fire retardancy of paints where it determines the fire retardant properties of a coating system on wood surface. The modifications were ;

- The area of ellipse formed was determined and measured after the procedure and then compared between the chemicals.
- It was immediately tested and not coated as referred to the test method.
- Leaching test was not included.

SEM/EDX

Scanning Electron Microscope (SEM) Leica Cambridge S-360 was used to study the surface morphology of samples. The samples were coated with gold using sputter Polaron Sc 515, with thickness $\approx 30\text{nm}$.

Energy Dispersive X-ray (EDX) micro analyzer (Cam Scam Editor) was used to obtain qualitative and semi qualitative info of the elements present in the specimen. Beam voltage of 15 kV was used for all specimens.

Results and discussion

Properties of rubberwood fibres and EFB

Table 1 shows the chemical composition of rubberwood and EFB fibres used in this study.

Table 1: Chemical composition of rubberwood fibres and EFB

	Lignin	Cellulosa	Alpha-cellulosa	Holocellulosa	Extractives
Rubberwood fibres	30%	31.58%	91.4%	92.9%	2%
EFB	20.4%	29.91%	39.82%	75.6%	1.5%

In comparison with Ramli et al. (2002), the average of fresh EFB from mills contained 30.5% lignocellulose, cellulose (45%), hemicellulose (32.8%) and lignin at about 20.5%.

Moisture content is one of the critical factor to produce an MDF with excellent performance. Table 2 shows the moisture content of the rubberwood fibres and EFB fibres used.

Table 2. Moisture content of rubberwood and EFB fibres.

Raw material	Moisture content (%)
Rubberwood fibres	11.46 (0.26)
EFB fibres	12.53 (0.38)

The production of MDF

MDF properties

From the study, the MDF that were produced from rubberwood fibres bonded with UF has shown a light colour. It is usually white to nearly colourless, and this results in creating no colour to the resulting panel (Moslemi, 1974). In contrast, the addition of PF resin gives a darker appearance of MDF. MDF made from EFB fibres on the other hand gives a much darker and despite its natural brownish colour and texture of EFB fibers. It is also observed that the presence of ATH and zinc borate gives out a whiter surface on the MDF.

In general, both types of MDF apparently appear smooth and a solid edge. The smooth surface might be because of the application of water spray on the top surfaces of the mat. As mentioned by Pizzi (1994), in order to shorten the pressing time water spray is applied on the surfaces of the mat. It also prevents the adhesive from precuring on the surface during closure of the press before contact with the hot top caul sheet. On the other hand, it gives a much faster increase in temperature and cure. With this water spray covering the surface enables it to vaporize while in contact with the hot caul sheet of the press, and migrates from the surfaces to the core of the panel. With the mass transfers from the surface to the core cause density distribution through the mat. Not only that it adds mass to the core but heat are transferred and as a sorbed material it plasticize core fibers and a greater density is created (Irving, 1976). The solid edge on the other hand which is known as one of the attractive property is believed to result from uniform density over the cross section (Suchsland and Woodson, 1991)

From observation it takes 1 minute to make contact with the gauge bars from the start of the press closure. As soon as contact were made with the gauge bars steam escapes between 1-4 minutes. It was then after 2 minutes the pressure was slowly decreased from maximum pressure until the final press. As mentioned by Vick (1999), pressure is one of the crucial factors in manufacturing MDF. It acts by bringing the molecular contact with the wood surfaces and this eventually the resin were forced to penetrate into the wood structure for more effective mechanical interlocking.

MDF is a very suitable product in evaluating fire retardant chemicals towards mechanical properties and fire properties. This is because it is a more uniform product, easily available in large production lots and handling panel-size material,

Table 3: Moisture content and specific gravity of flame retardant MDF using phenol formaldehyde ^a

Type of fire retardant	%	Moisture content (%)	Specific gravity
		Mean	
Control	0	7.15	0.94
		(0.95)	(0.02)
ZB	10	6.88	0.95
		(0.08)	(0.02)
	15	7.05	0.95
		(0.99)	(0.01)
	20	8.12	0.95
(0.38)		(0.02)	
30	8.64	0.93	
		(0.34)	(0.01)
SA	10	9.64	0.73
		(0.05)	(0.02)
	15	10.08	0.95
		(0.24)	(0.02)
	20	10.38	0.97
(0.27)		(0.01)	
30	11.04	0.93	
		(0.31)	(0.02)
ATH	10	8.05	0.96
		(0.26)	(0.01)
	15	9.48	0.94
		(0.05)	(0.02)
	20	9.68	0.95
(0.05)		(0.02)	
30	10.07	0.97	
		(0.24)	(0.02)

^a Figures in parentheses are standard deviations

Table 4: Moisture content and specific gravity of flame retardant MDF using urea formaldehyde ^a

Type of fire retardant	%	Moisture content (%)		Specific gravity
		Mean		
Control	0	5.26	0.95	
		(0.07)	(0.01)	
ZB	10	4.92	0.95	
		(0.08)	(0.01)	
	15	8.72	0.95	
		(0.25)	(0.02)	
	20	8.96	0.94	
(0.21)		(0.02)		
30	9.06	0.93		
		(0.23)	(0.02)	
SA	10	9.85	0.95	
		(0.05)	(0.01)	
	15	7.43	0.92	
		(0.94)	(0.02)	
	20	8.31	0.95	
(0.98)		(0.01)		
30	8.63	0.96		
		(0.32)	(0.01)	
ATH	10	8.61	0.95	
		(0.25)	(0.02)	
	15	9.61	0.96	
		(0.05)	(0.01)	
	20	9.87	0.97	
(0.08)		(0.02)		
30	10.09	0.92		
		(0.31)	(0.01)	

^a Figures in parentheses are standard deviations

and despite its low cost acceptable variability limits were done to give uniform fire performance (Levan, 1985). The performance of wood materials in fire depends on density and moisture content (Winandy *et al.*, 1991).

Overall the density of MDF samples made in this study obtained were in the range between 0.7 to 0.8 g/cm³. It is known that density is a critical factor in determining the mechanical performance of the MDF.

Adhesive properties

Table 5 : Summary of the resins properties

Resin	Solid content (%)	pH	Viscosity (mPa.s/cps/25°C)
Phenol formaldehyde	55.71	13	85.42
Urea formaldehyde	55.60	12	199.9

All resin applications were based on resin solids content and percentage of total board moisture content. In this study about 15% of PF and UF resin were used based on oven-dried weight of fibres used. This amount of percentage was chosen in order to produce satisfactory board properties similar to commercial MDFs, and eventually focusing more in optimizing the most suitable and effective flame retardant in the MDF produced. The PF resin used in this study was a resole type.

The Production of Flame Retardant MDF

ATH formulation

This equation was based on earlier study on the production of wet-process fibreboard (Lim, 2002).



Alum sodium hydroxide sodium aluminate

Molecular weight:

$$\text{K}_2\text{SO}_4\text{Al}_2(\text{SO}_4)_3 \cdot 24\text{H}_2\text{O} = 884.4$$

$$\text{NaOH} = 39.99$$

$$\text{NaAlO}_2 = 81.97$$

100g fibers → 10g ATH was used.

The objective is to determine the quantity of each chemical used to obtain 10 g of ATH.

81.97 g sodium aluminate comes from 884.4 g of alum

$$\frac{10 \times 884.4}{81.97} \text{ g alum} = 107.9 \text{ g of alum}$$

81.97 g sodium aluminate comes from 39.99 g of NaOH

$$\frac{10 \times 39.99}{81.97} \text{ g NaOH} = 4.88 \text{ g of NaOH}$$

30 g alum needs 300 mL H₂O

107.9 g alum needs 300×107.9 g H₂O = 1079 mL H₂O for dissolving 107.9 g alum

30

10.5 g NaOH needs 210 mL H₂O

4.88 g NaOH needs $\frac{4.88 \times 210}{10.5}$ g H₂O = 97.6 mL H₂O to dissolve in 4.88 g NaOH.

Solution 1:

Take 107.9 g of alum dissolve in 1079 g of water

Solution 2:

Take 4.88 g of NaOH dissolve in 97 g of water

Following the equation above, it was concluded that a lot of water will be consumed, time and energy was wasted. It was therefore not applicable and practical in producing an MDF. Instead, a commercial ATH was used in the current research.

First analysis from the first trial

Commercial ATH was used in this research and because of its solubility, NaOH was used with a certain amount. After blended with fibres it was then blended together with alum enough to neutralize the fibres to pH 7. Lastly, it was blended with UF and NH₄Cl as the hardener. With PF resin, alum was not needed. Nevertheless, the boards failed and not cured inspite of the resin, both UF and PF flows out while heat from the hot press was applied. This was caused by the corresponding OH present from the NaOH and alum which interferes with the UF and personally OH from NaOH on the PF resin.

Fig. 3 shows for boards treated with ATH bonded with the UF resin has the most amount of Na and the least amount of Al. It shows that the objective of producing a flame retardant board has not been reached whereby only a small percentage of Al was present in the board and a high percentage of NaOH in the ATH board. Moreover, using this method makes the content of ATH insignificant.

Analysis from the Second trial

Second trial had succeeded because it comply the standard of commercial boards. This was proved by the results of mechanical properties. By using the hand-mixing method, the fluffy fibres acts by entrapping and entangling the ATH. It was also not easily removed by abrasion or attrition.

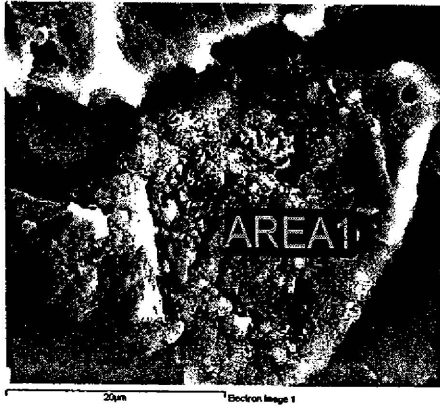
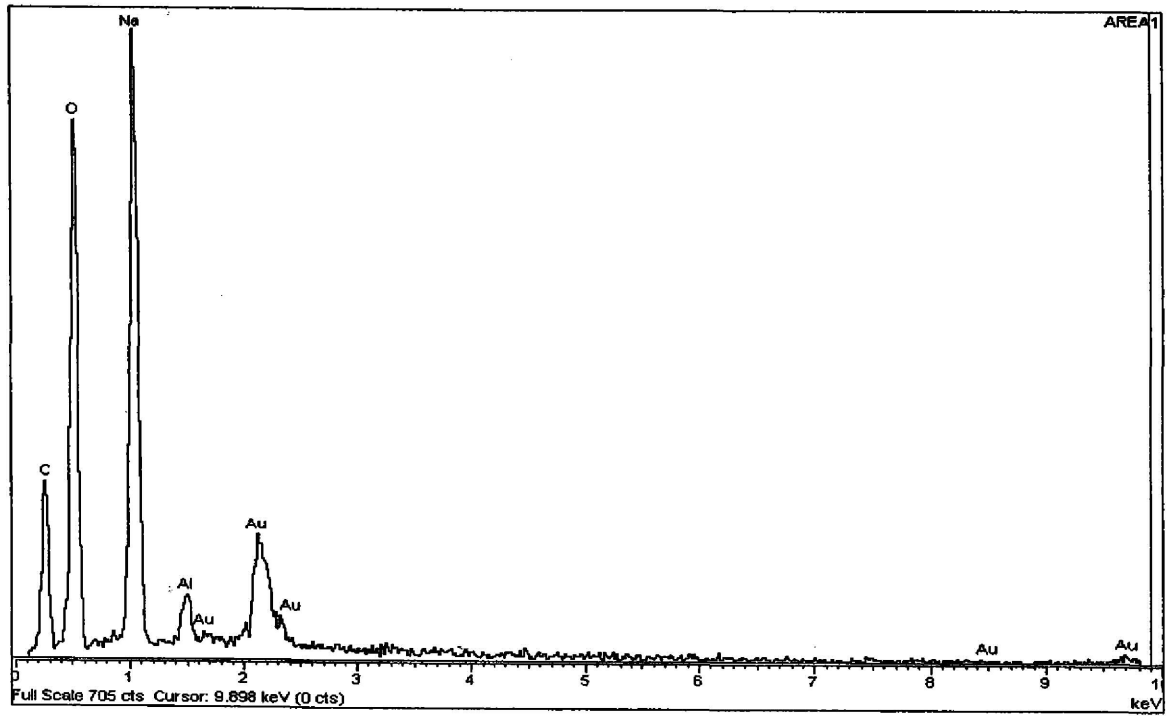


Figure 3. SEM-EDX showing FRTMDF treated with ATH bonded with UF resin

Mechanical and physical properties

Mean values of mechanical properties of fire retardant-treated MDF using rubber wood fibres

The effect of fire retardant chemicals on mechanical strength was done by comparing the data for static bending modulus of rupture (MOR), endurance/cyclic MOR test (ENDMOR), internal bond (IB), endurance/cyclic IB test (ENDIB), thickness swelling (TS), endurance/cyclic TS (ENDTS), water absorption (WA), endurance/cyclic WA (ENDWA), and boil test between each chemicals at each type of resin. Table 6 and 7 summarize the mechanical and physical properties using both UF and PF resin. As the density of MDF was above the recommended minimum, all the samples easily met the minimum strength requirements by the ISO.

Table 8 and 9 however illustrate the mechanical and physical properties after the endurance/cyclic test using PF and UF resin. MDF panels made from rubberwood fibres and bonded with PF are exposed to three cycles, each comprising immersion in water, freezing, and drying at elevated temperature. After cyclic treatment, the samples were reconditioned and their swelling in thickness and residual strength determined. Mechanical properties of cyclic and boil test after conditioned can be used to determine the relative effect of difference of fire retardant chemical and levels. According to ISO standard, the cyclic exposure results for all samples was substandard. Cyclic exposure appears to be more severe. MDF after cyclic test appears brash and brittle, crumbles easily and darkens.

In MOR properties, the mean values increased gradually with the addition of PF resin on the chemicals whereby it reaches the highest mean at 30% level. Using UF resin though shows a negative trend where 10% level shows the highest mean value. Other mechanical properties; ENDMOR, IB, ENDIB and BOIL shows relatively small variation from the mean values. MOR and IB strength were reduced compared to that of control panels, except for MOR with PF resin where it shows a positive trend. Overall, the treated samples showed a greater tendency for degradations than the untreated samples. At 15% level, all fire retardant types exhibited a reduction in strength at higher levels were it is lower than the minimum

requirement by ISO standard. The data from the tables revealed a pattern of decreasing TS and WA mean values with the increasing of chemical levels up to 30%. Conversely, it is shown that lower strength boards may result at high levels of fire retardant.

Table 6

Mechanical properties of flame retardant MDF using rubberwood fibres and PF^a

Type of fire retardant	%	MOR (N/mm ²)	IB (N/mm ²)	TS (%)	WA (%)
Mean					
Control	0	11.56 (4.11)	0.4 (0.01)	35.55 (16.60)	54.29 (24.98)
Zinc borate	10	23.23 (3.89)	0.44 (0.04)	27.22 (2.06)	96.14 (15.79)
	15	25.05 (2.19)	0.35 (0.02)	26.35 (3.81)	48.78 (3.97)
	20	28.14 (2.62)	0.33 (0.01)	22.82 (3.11)	38.85 (9.89)
	30	33.59 (2.80)	0.26 (0.02)	13.06 (8.05)	27.46 (3.06)
Sodium Aluminate	10	15.05 (2.98)	0.38 (0.02)	27.68 (3.75)	95.11 (16.92)
	15	21.48 (6.86)	0.34 (0.01)	24.98 (4.34)	48.61 (11.56)
	20	25.19 (1.76)	0.31 (0.02)	19.50 (3.57)	40.74 (5.39)
	30	29.55 (4.11)	0.26 (0.02)	13.87 (8.09)	33.02 (9.07)
Aluminum Trihydrate	10	14.91 (4.13)	0.36 (0.02)	31.05 (7.33)	95.68 (25.97)
	15	31.18 (8.3)	0.35 (0.01)	28.52 (6.08)	54.57 (6.09)
	20	36.34 (1.85)	0.32 (0.01)	24.92 (4.86)	30.61 (10.14)
	30	39.77 (1.91)	0.25 (0.05)	19.40 (5.27)	23.49 (10.20)

Table 7

Mechanical properties of flame retardant MDF using rubberwood fibres and urea formaldehyde ^a

Type of fire retardant	%	MOR (N/mm ²)	IB (N/mm ²)	TS (%)	WA (%)
Mean					
Control	0	20.32 (6.16)	0.65 (0.02)	33.84 (1.75)	58.46 (15.78)
Zinc borate	10	20.99 (3.2)	0.57 (0.02)	28.98 (3.41)	77.78 (7.80)
	15	18.17 (6.71)	0.54 (0.02)	16.68 (0.95)	69.23 (15.62)
	20	17.36 (0.8)	0.52 (0.02)	14.08 (8.05)	67.74 (9.36)
	30	15.73 (0.7)	0.47 (0.04)	11.95 (6.95)	50.52 (13.62)
Sodium aluminate	10	25.67 (4.13)	0.61 (0.02)	19.10 (0.69)	91.50 (19.15)
	15	18.43 (5.00)	0.54 (0.02)	17.14 (1.53)	73.93 (22.02)
	20	15.51 (1.05)	0.52 (0.02)	16.96 (10.15)	60.30 (14.78)
	30	13.6 (0.75)	0.48 (0.01)	13.78 (13.89)	32.11 (12.80)
Aluminium Trihydrate	10	32.05 (9.63)	0.67 (0.02)	26.50 (5.00)	86.74 (7.79)
	15	28.05 (7.54)	0.6 (0.02)	24.45 (4.64)	52.92 (5.90)
	20	27.39 (1.63)	0.57 (0.03)	22.11 (7.56)	40.61 (7.46)
	30	22.48 (2.22)	0.51 (0.03)	19.45 (4.20)	33.20 (7.72)

Table 8

Mechanical properties after cyclic test ISO/DIS 16987 and boil test ISO/DIS 16987 using rubberwood fibres and phenol formaldehyde ^a

Type of fire retardant	%	MOR	IB	TS	WA	IB
		(N/mm ²) (cyclic)	(N/mm ²) (cyclic)	(%) (cyclic)	(%) (cyclic)	(N/mm ²) (boil)
Mean						
Control	0	20.55 (5.00)	0.27 (0.01)	21.48 (4.36)	130.33 (28.75)	0.16 (0.02)
Zinc borate	10	9.52 (2.98)	0.26 (0.02)	47.96 (7.96)	110.11 (13.82)	0.17 (0.01)
	15	9.01 (1.02)	0.21 (0.01)	68.24 (7.65)	158.52 (25.33)	0.15 (0.02)
	20	8.74 (1.68)	0.18 (0.01)	81.27 (12.56)	202.24 (15.65)	0.13 (0)
	30	7.76 (1.85)	0.15 (0.02)	90.39 (10.39)	216.78 (40.70)	0.10 (0)
Sodium Aluminate	10	6.56 (1.20)	0.25 (0.01)	37.67 (8.46)	121.36 (23.68)	0.17 (0.01)
	15	6.17 (1.03)	0.24 (0.01)	44.64 (15.98)	121.60 (36.93)	0.14 (0.02)
	20	4.45 (1.52)	0.22 (0.01)	65.07 (10.75)	148.52 (19.43)	0.12 (0.02)
	30	3.05 (0.57)	0.17 (0.01)	99.37 (13.25)	160.94 (15.41)	0.08 (0.01)
Aluminum Trihydrate	10	18.16 (1.6)	0.27 (0.01)	10.37 (4.62)	144.16 (30.47)	0.17 (0.01)
	15	16.61 (1.21)	0.21 (0.01)	20.05 (8)	150.59 (19.90)	0.14 (0)
	20	15.34 (1.52)	0.17 (0.04)	47.53 (9.79)	148.52 (19.43)	0.12 (0.01)
	30	13.87 (1.28)	0.13 (0.01)	70.79 (16.25)	160.94 (15.41)	0.10 (0.01)

Table 9

Mechanical properties after cyclic test ISO/DIS 16987 and boil test ISO/DIS 16987 using rubberwood fibres and urea formaldehyde ^a

Type of fire retardant	%	MOR (N/mm ²) (cyclic)	IB (N/mm ²) (cyclic)	TS (%) (cyclic)	WA (%) (cyclic)	IB (N/mm ²) (boil)
Mean						
Control	0	6.67 (0.6)	0.27 (0.01)	95.22 (18.81)	137.07 (31.92)	0.17 (0.01)
Zinc borate	10	7.34 (1.14)	0.24 (0.01)	50.28 (26.21)	112.71 (30.63)	0.17 (0.01)
	15	5.95 (1.18)	0.23 (0.02)	24.83 (12.16)	75.30 (12.77)	0.14 (0.01)
	20	5.08 (0.36)	0.2 (0.01)	17.54 (4.57)	54.46 (9.76)	0.11 (0.02)
	30	4.12 (0.5)	0.17 (0.02)	14.29 (6.88)	48.49 (8.33)	0.09 (0.01)
Sodium Aluminate	10	4.23 (0.78)	0.25 (0.01)	36.17 (5.99)	127.45 (30.64)	0.17 (0.01)
	15	3.45 (2.20)	0.22 (0.02)	22.73 (8.81)	61.86 (9.70)	0.15 (0.01)
	20	3.24 (1.24)	0.19 (0.01)	21.35 (8.03)	56.83 (3.76)	0.11 (0.01)
	30	2.37 (0.6)	0.14 (0.02)	19.32 (6.32)	61.86 (9.70)	0.08 (0.02)
Aluminum Trihydrate	10	8.34 (3.86)	0.27 (0.01)	33.15 (7.77)	116.29 (16.78)	0.16 (0.01)
	15	6.91 (5)	0.24 (0.02)	22.90 (5.16)	103.15 (15.52)	0.14 (0.01)
	20	4.50 (1.36)	0.19 (0)	20.59 (3.41)	95.60 (10.50)	0.12 (0.01)
	30	3.74 (0.93)	0.16 (0.03)	15.34 (5.07)	89.46 (17.12)	0.10 (0.01)

Normalized values of mechanical and physical properties in comparison with control

The results of mean MOR values of MDF bonded with PF are summarized in Table 6 and after being normalized are shown as a percentage of control values in Figure 1 using PF resin. Overall the normalized values of levels of chemicals are much higher than the control. It is shown that each levels of fire retardant influence the treatment on MOR and it depends on the type of resin. Referred to Figure 1, 30% MDF treated with ATH has the highest normalized value compared to control boards whereas boards treated with 10% ATH shows the least normalized value. Mean MOR values are illustrated in Table 6-7 and normalized values in Figure 4 and 5 on MDF bonded with UF. A general reduction in MOR properties for UF bonded was found. The MOR strength was least affected by the addition of fire retardant. Higher levels gave great reduction in MOR properties.

Figure 6 and Table 6-9 illustrates the effect of fire retardant treatment on IB using PF resin. The IB results are shown in Table 6-9 and Figure 7 using UF resin. Both shows slight reduction in IB strength.

Table 6-9 , Figure 5 and 6 shows TS and WA of MDF samples made from PF resin for three types of fire retardant and 4 different percentages. In TS with PF bonded samples, all normalized values are least affected by the chemicals in comparison to control. WA however has high normalized values compared to control at 10% level. At 15% and above it begins to show reduction of water absorption.

The TS and WA data of UF bonded samples are summarized in Table 6-9, and normalized values are showed graphically in Figure 10 and 11 using UF resin. The TS and WA strength is lower in comparison to control and the decrease is up to 30% level.

The ENDMOR mean values are summarized in Table 6-9 and normalized value in Figure 9 using PF resin. A small reduction was found as levels increase and was least affected by the ENDMOR. MDF panels are made from rubberwood bonded with UF after cyclic test are shown in Table 6-9 and Figure 12-13. The ENDMOR showed a decreased trend compared to control, but both ZB and ATH has higher normalized value in comparison to control at 10% level.

Table 6-9 and Figure 14 shows IB cyclic strength of MDF panels made from rubberwood and PF. The results of IB strength after cyclic test are shown at Table 86-9 and Figure 115 using UF resin. Increasing the levels gave a lower ENDIB strength value for these samples compared to control. This is similar using UF resin.

Results of ENDTS and ENDWA of MDF panels bonded with PF after going through the cyclic test and soaking in cold water for 24 hours are shown in Table 7, Figure 16 and Figure 17. Both TS and WA of MDF panels bonded with UF after cyclic test and after soaking in cold water for 24 hours are summarized in Table 8, Figure 15 and Figure 16. General reduction in both ENDTS and ENDWA for UF-bonded samples was found whereas an increase of mean values for samples made from PF resin.

In principle, IB strength is determined using samples which have been immersed in water to a boiling point of ($\approx 100^{\circ}\text{C}$) for (120 ± 5) minutes and a pH of $7 \pm 0,5$. The IB strength from the boil test are presented in Table 7 and Figure 17 using PF resin. Table 8 and Figure 18 shows IB strength in the boil test of MDF panels bonded with UF depending on fire retardant type and level. The IB strength in boil test showed a decrease compared to control for both UF and PF bonded resin. Figure 1-18 illustrates the normalized value in comparison to control for all mechanical properties.

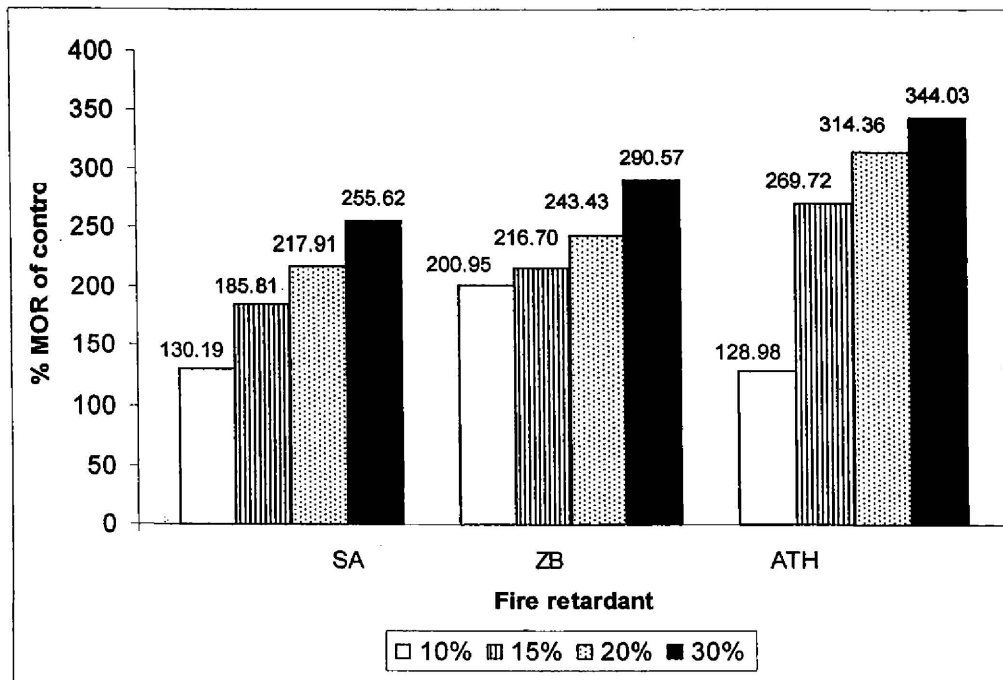


Figure 4 : Modulus of rupture of MDF made from rubberwood fibres and PF

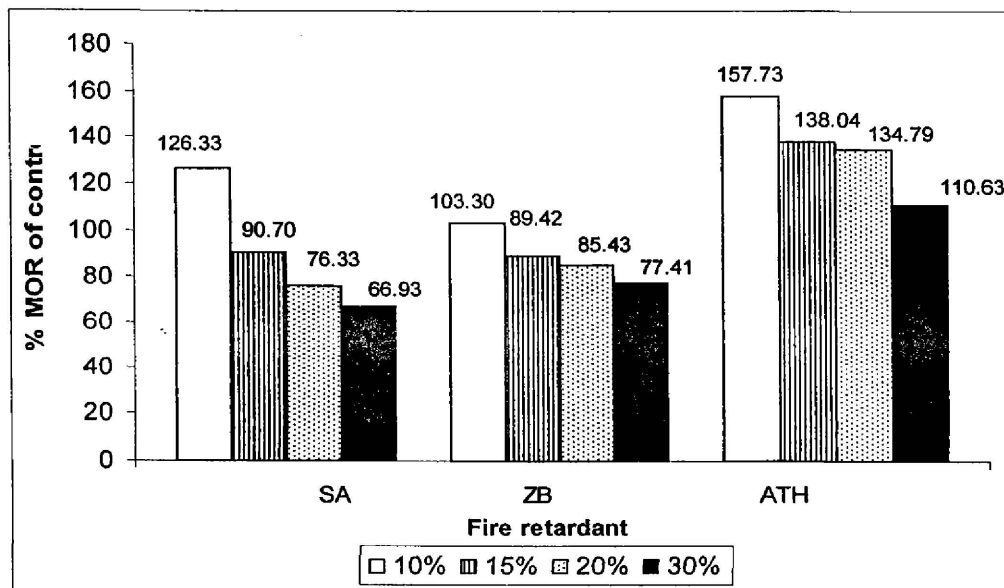


Figure 5 : Modulus of rupture of MDF made from rubberwood fibres and UF

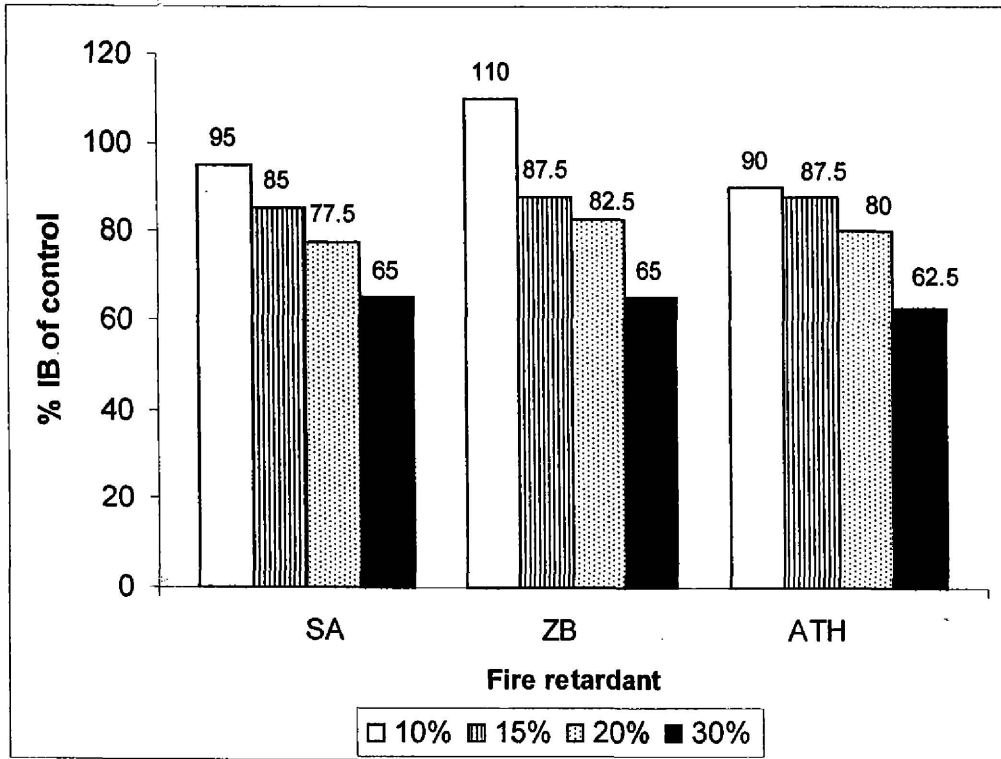


Figure 6 : Internal bond of MDF made from rubberwood and PF

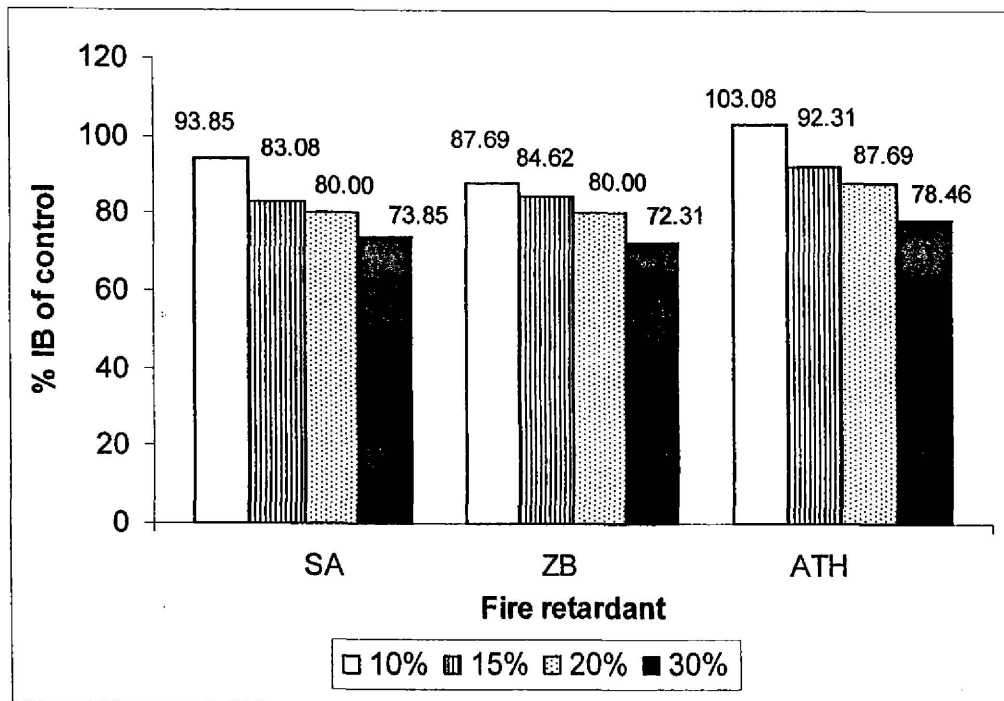


Figure 7: Internal bond of MDF made from rubberwood and UF

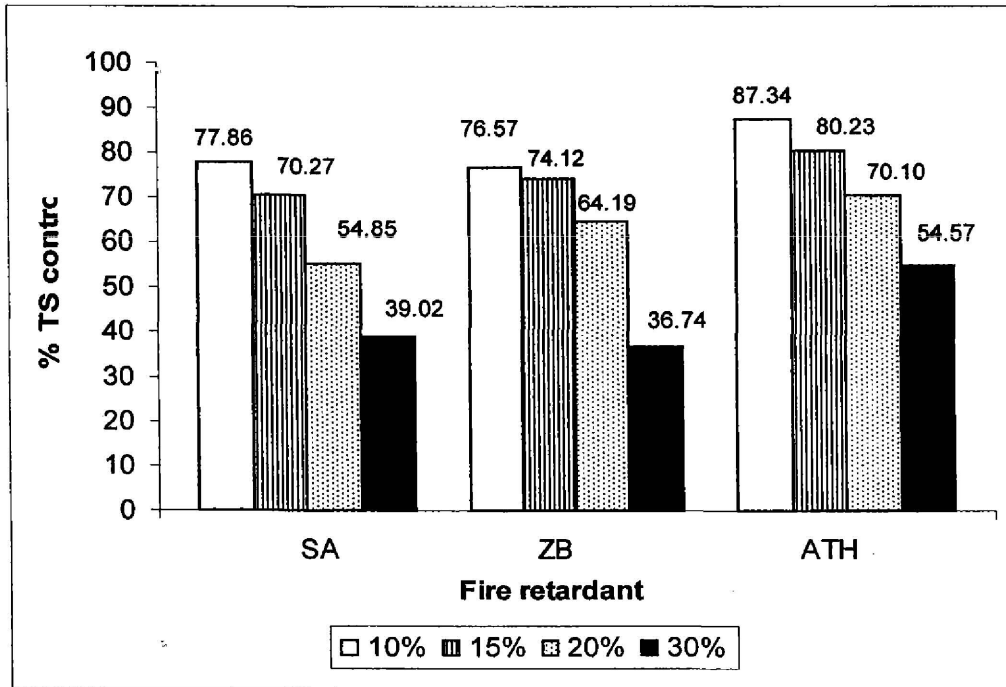


Figure 8 : Thickness swelling of MDF made from rubberwood and PF

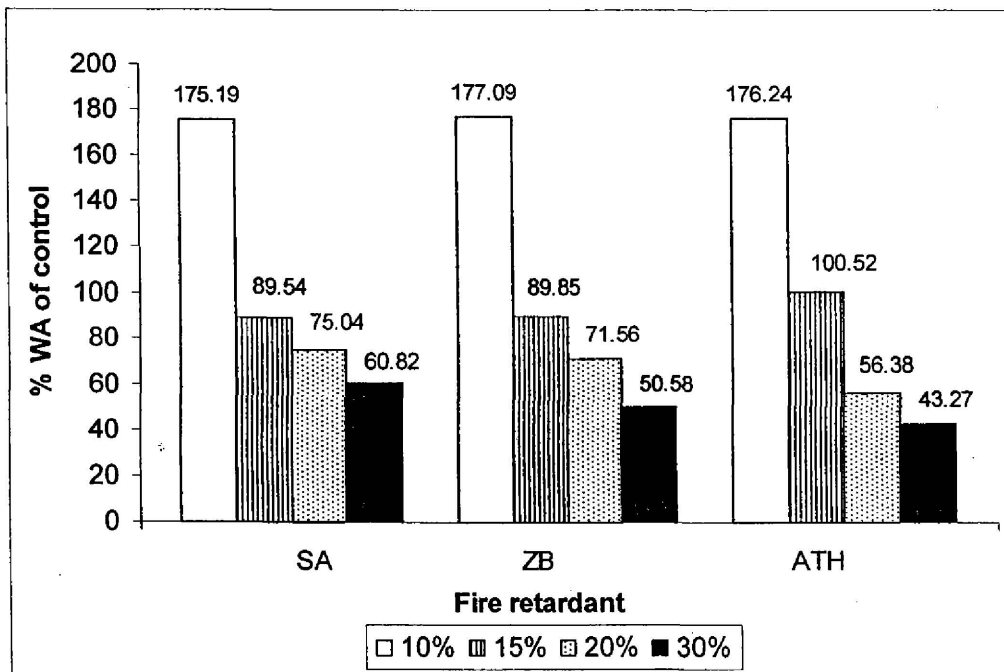


Figure 9 : Water Absorption of MDF made from rubberwood and PF

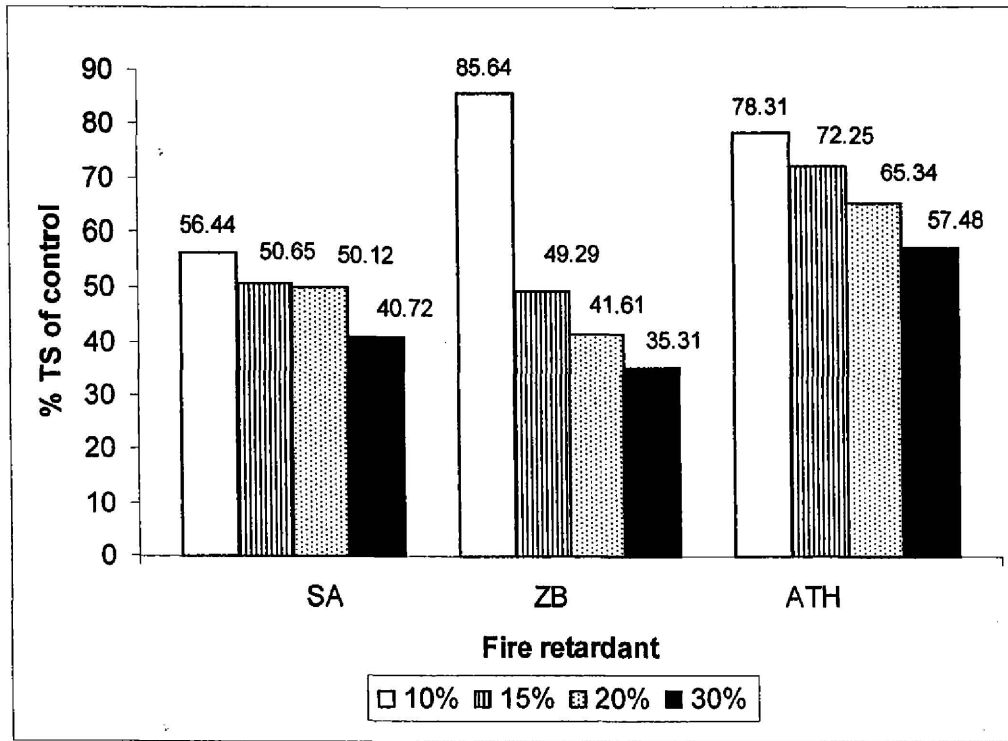


Figure 10 : Thickness swelling of MDF made from rubberwood and UF

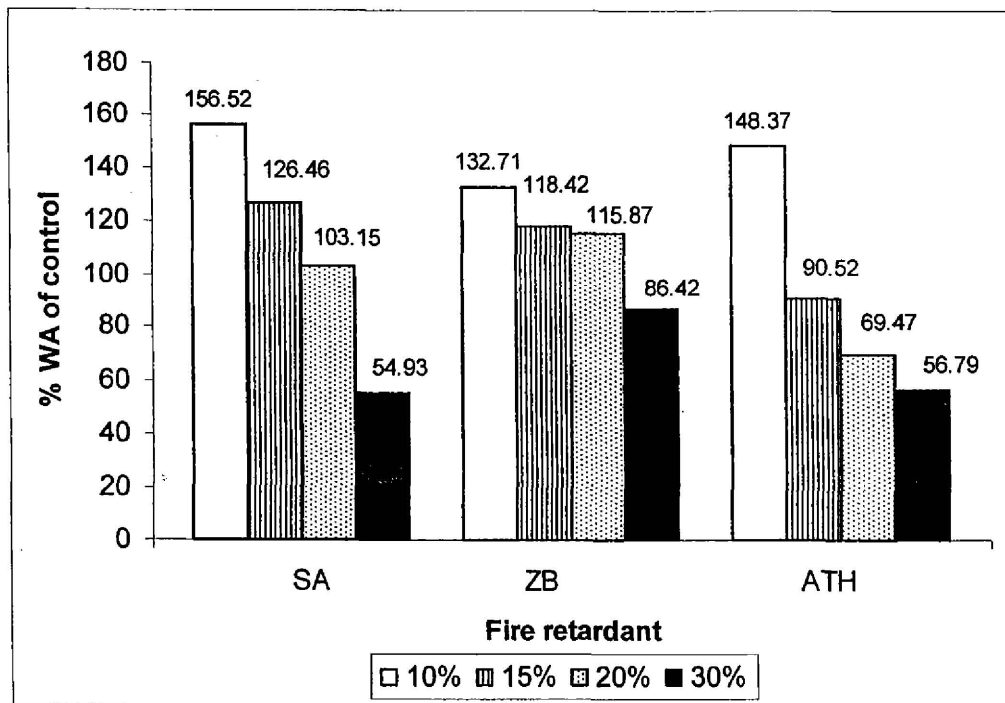


Figure 11: Water absorption of MDF made from rubberwood and UF

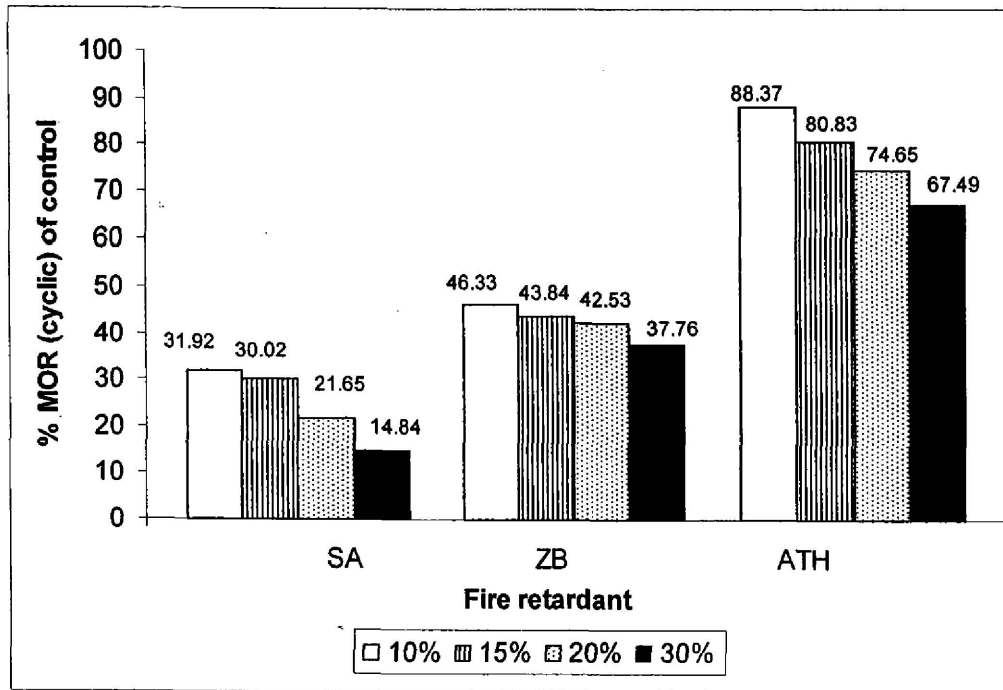


Figure 12: Modulus of rupture (after cyclic test) MDF made from rubberwood fibres and PF

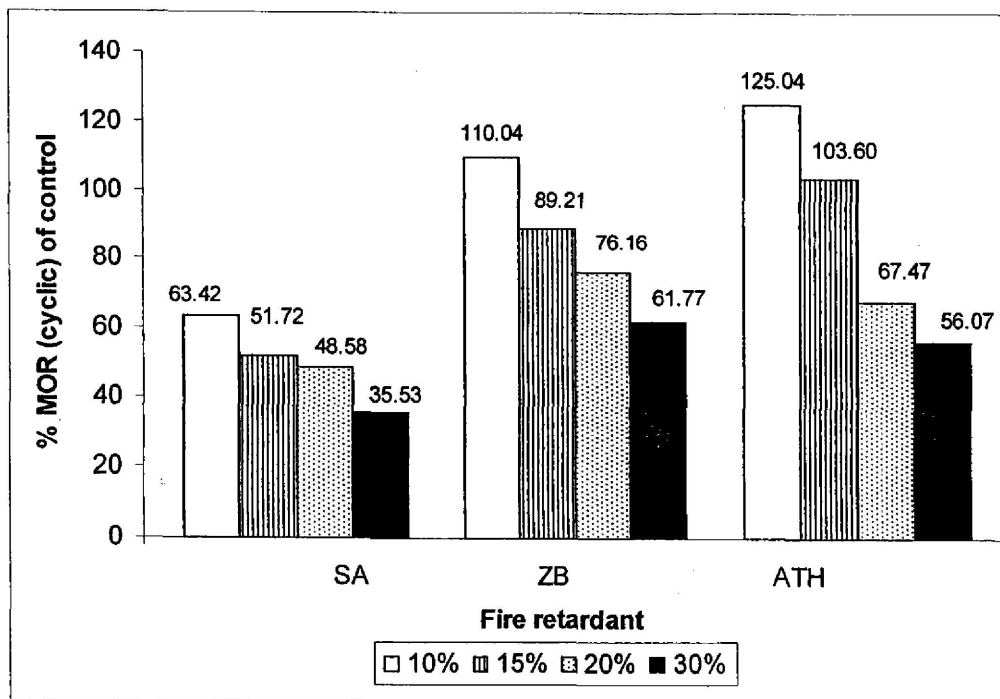


Figure 13: Modulus of rupture (after cyclic test) of MDF made from rubberwood fibres and UF

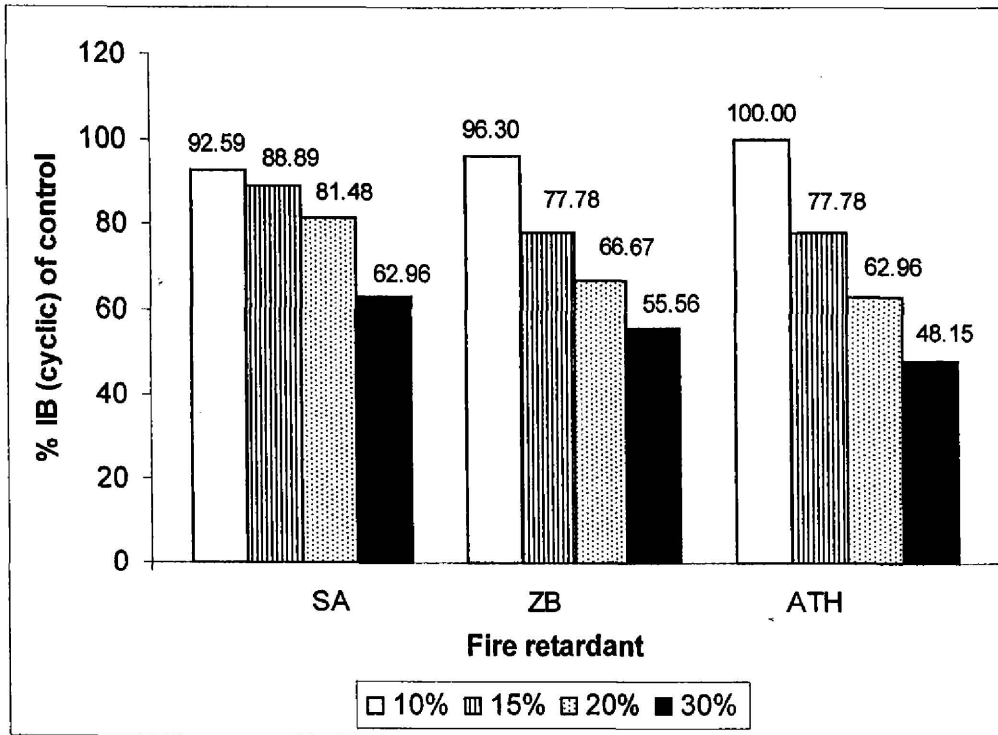


Figure 14: Internal bond (after cyclic test) of MDF made from rubberwood and PF

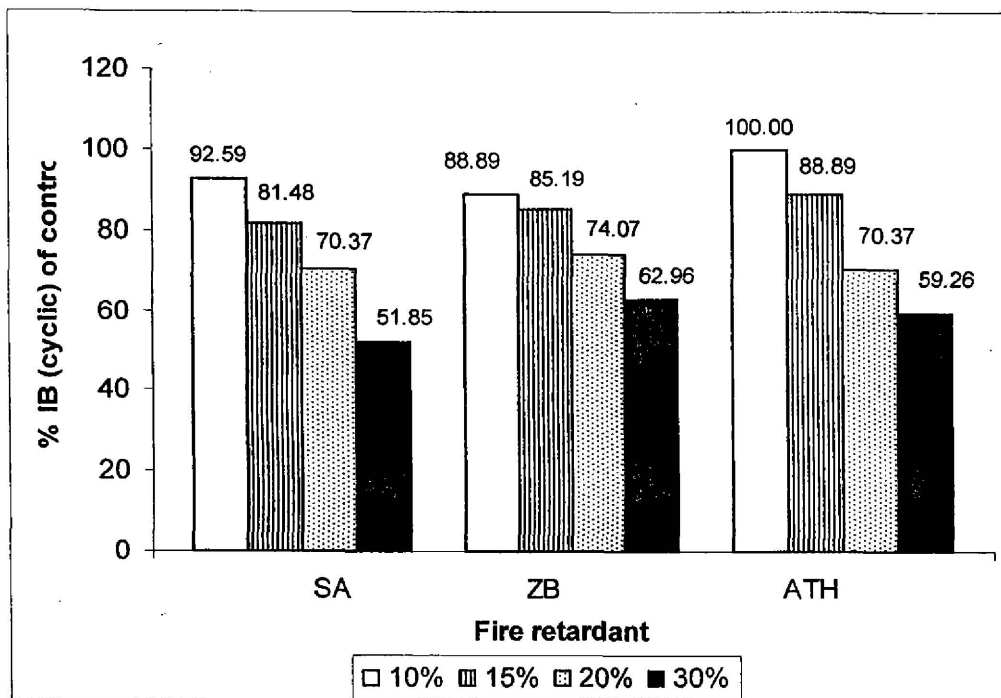


Figure 15: Internal bond (after cyclic test) of MDF made from rubberwood and UF

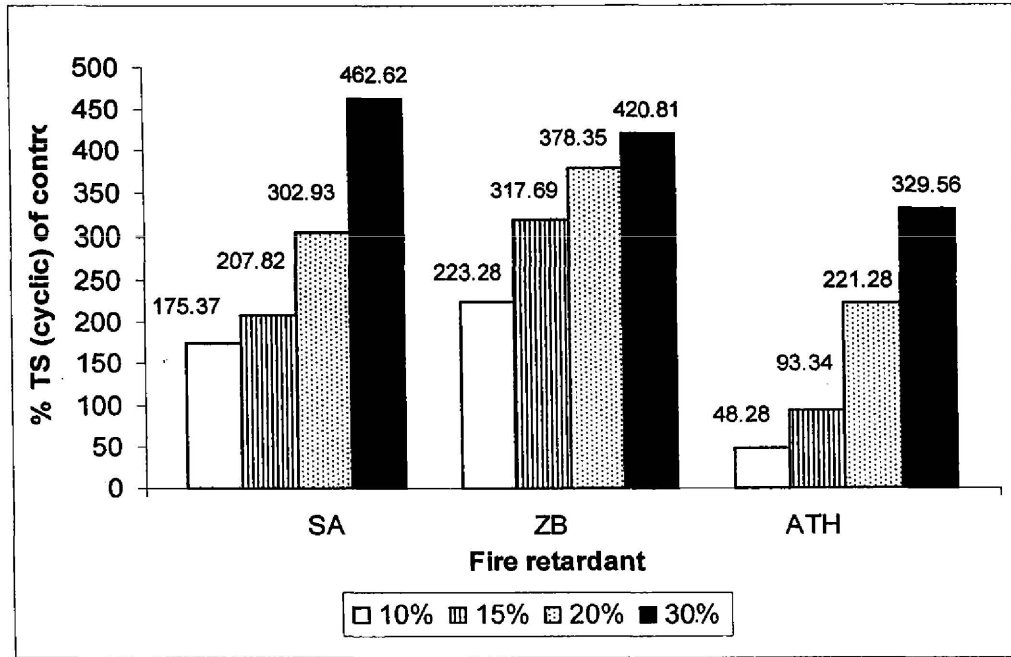


Figure 16: Thickness swelling (after cyclic test) of MDF made from rubberwood and PF

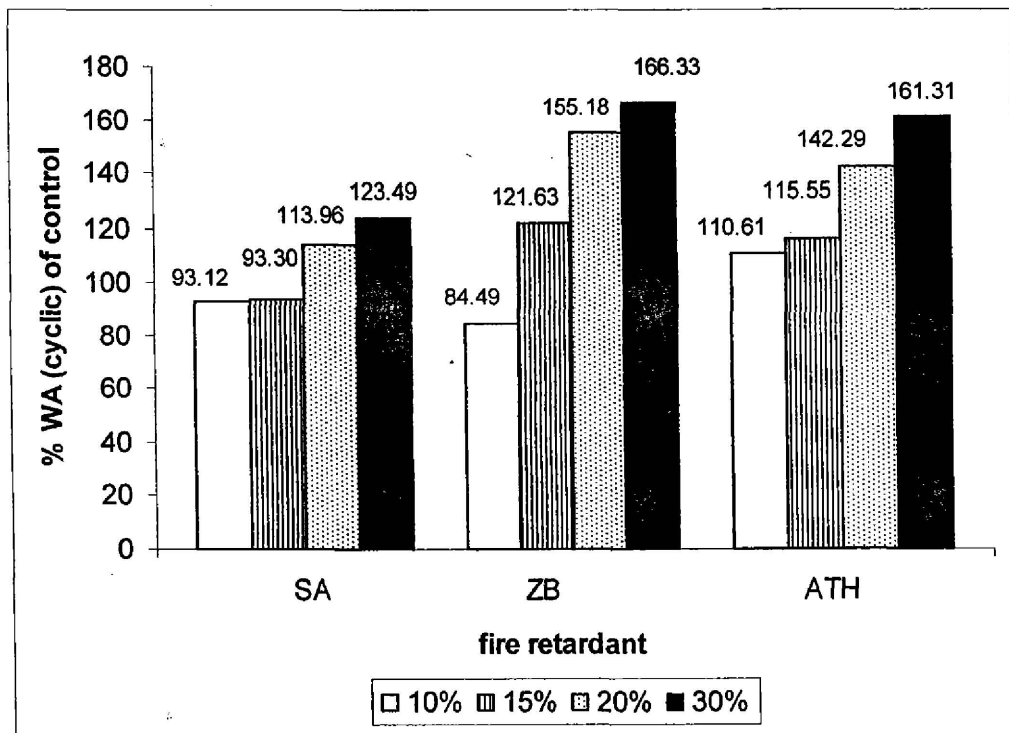


Figure 17: Water Absorption (after cyclic test) of MDF made from rubberwood and PF

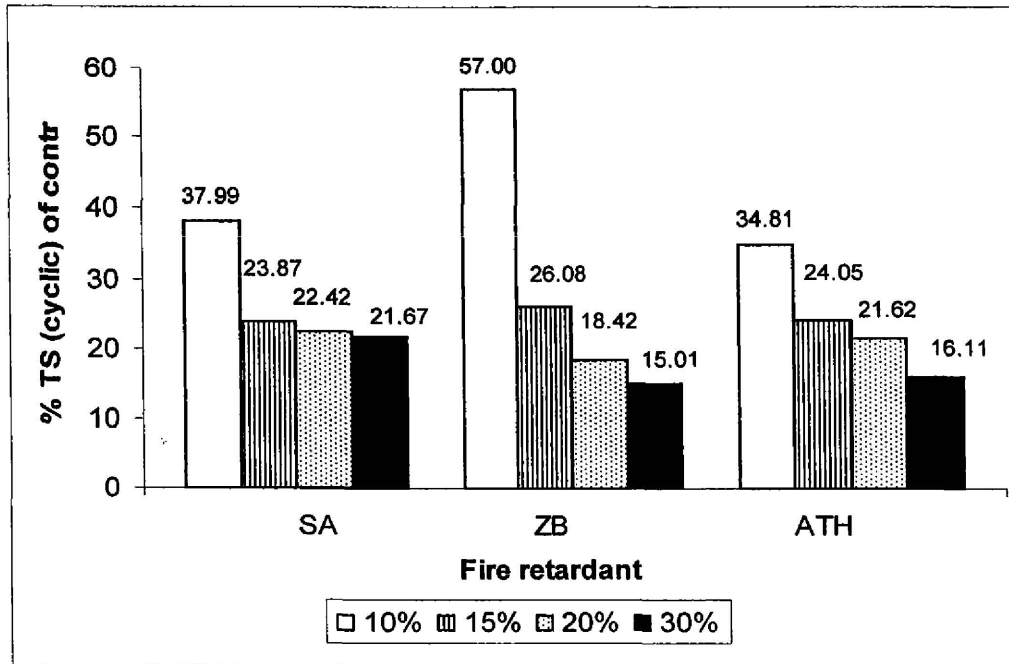


Figure 18: Thickness swelling (after cyclic test) of MDF made from rubberwood and UF

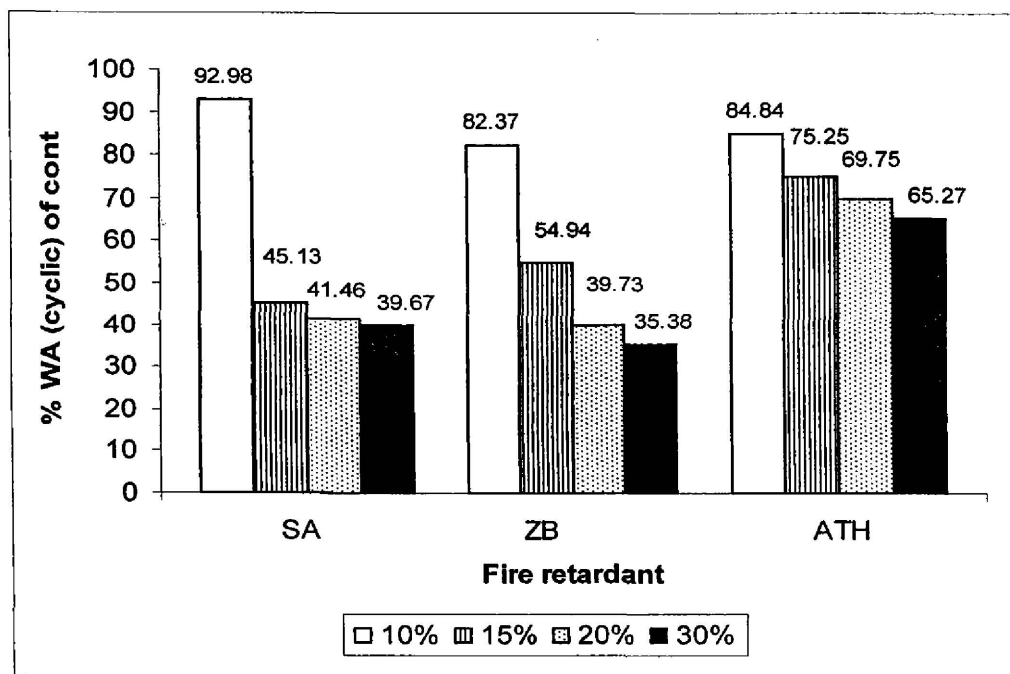


Figure 19: Water Absorption of MDF made from rubberwood and UF

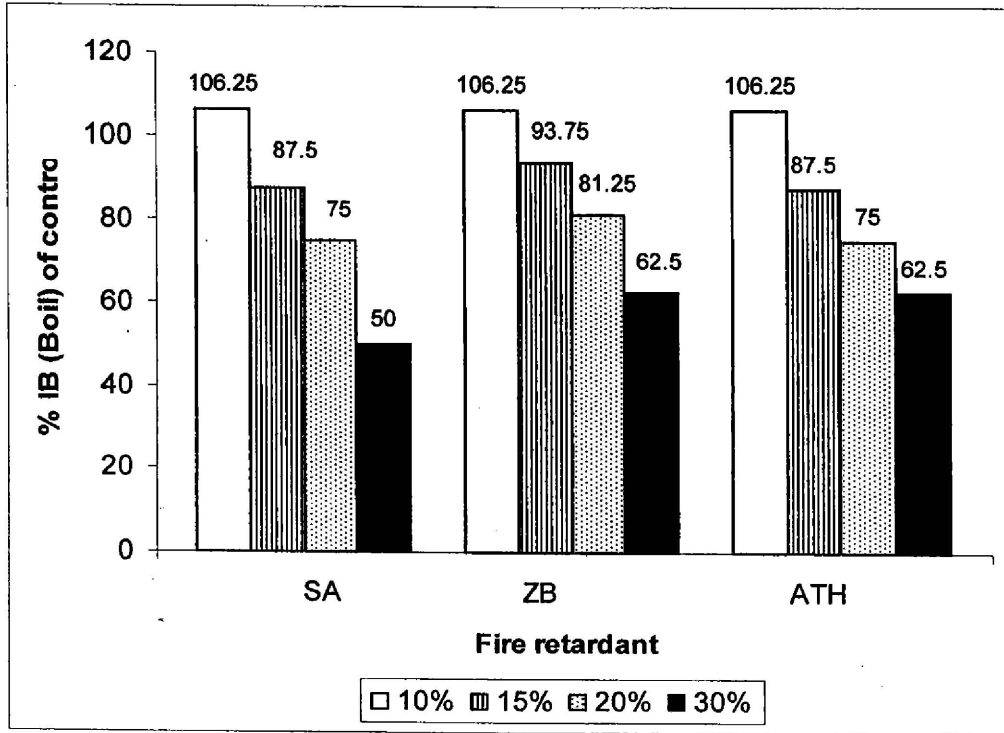


Figure 20: Internal bond (after boil) of MDF made from rubberwood and PF

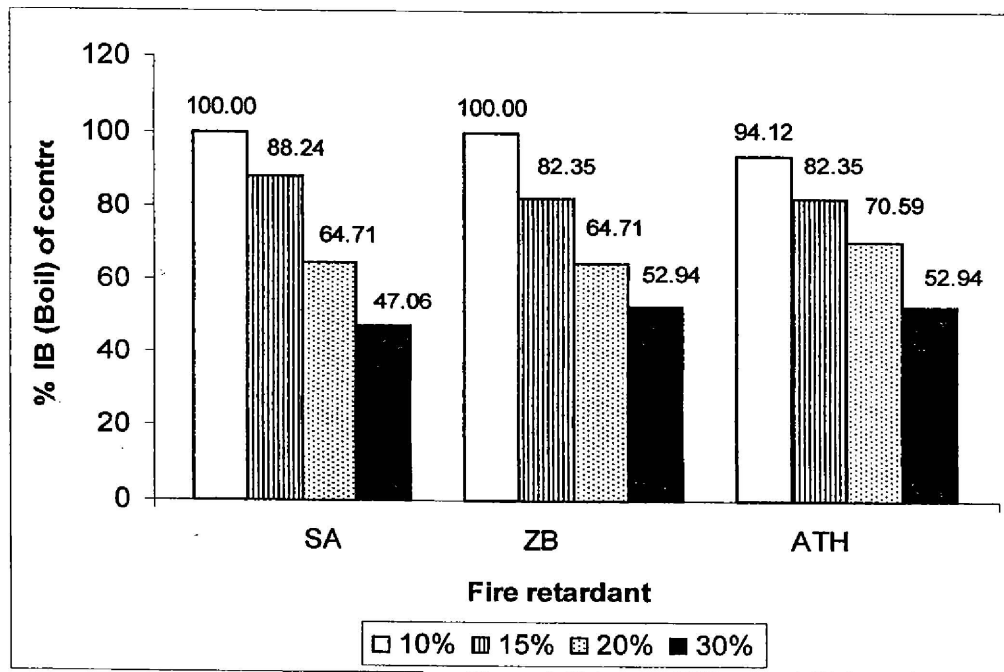


Figure 21: Internal bond (after boil) of MDF made from rubberwood and UF

Multiple comparison analysis

ANOVA showed that the interaction between fire retardant chemicals and resins is significant (p -value < 0.000) for all the properties evaluated.

It is concluded that the mean value of each level of fire retardant type depends on the type of resin applied. A multiple comparison procedure was done to investigate the nature of interaction effects. This is done by estimating the differences in mean values between the levels of fire retardant chemicals using each type of resin.

Mechanical properties

The MOR of MDF made using PF-resin, control, 15% SA, 10% ATH, 10% SA shows low mean MOR values and these chemicals do not differ with each other. The highest mean MOR value is 30% ATH at 39.77N/mm^2 . It differs significantly from the control which has the lowest mean MOR value. For UF-bonded samples, 10% ATH shows the highest mean MOR value and it performs equally with 15% ATH. 30% SA shows the lowest mean and perform equally with 20% SA. The highest and lowest mean MOR values do not significantly differ from the control sample. Overall, 30% ATH shows the best performance in PF resin and 10% ATH shows the best performance using UF resin.

Under dry and wet cyclic conditioning, 30% SA, 20% SA shows low mean ENDMOR values using PF resin and performs equally with each other. The high mean ENDMOR however are control, 10% ATH and 15% ATH. These chemicals perform equally with each other. All chemicals differ significantly from control except 10%, 15% and 20% ATH. Using UF resin, 10% ATH, 10% ZB, 15% ATH and control have high mean ENDMOR value but do not significantly differ from one another. The lowest ENDMOR value however are 30% SA, 20% SA and 15% SA and performs equally with each other. In comparison to control sample, all chemicals significantly differ with each other except the highest mean values and 15% SA and 20% ATH. 10% ATH shows the best performance using PF and UF resin.

For IB strength, 30% ATH indicates the lowest mean IB value using PF resin. It performs similarly with 30% SA and ZB. Control however shows high mean IB values and the fire retardant chemicals do not differ with each other in this respect of 10% ATH and 10% SA. Samples using UF resin on the other hand show low mean

IB values for 30% ZB, SA and ATH and these chemicals do not differ significantly with each other. Control, 10% ATH and SA have high mean IB value irrespective of fire retardant chemicals. 10% SA shows the best performance for IB strength with both resins.

Among the PF-bonded samples after the cyclic conditioning, 30% ATH, 30% ZB and 20% ATH have low mean ENDIB values and do not significantly differ with each other. 10% ATH and control shows high mean ENDIB value and do not differ significantly with each other. UF-bonded samples shows that 30% ATH has the lowest mean ENDIB value and performs equally with 30% SA. 10% ZB, ATH and SA, 15% ATH and control have high mean ENDIB value and performs equally with each other. 10% ATH shows the best performance using PF and UF resin.

In boil test with PF resin, control, 10% ZB, SA and ATH shows high mean IB values from boil test and these levels of chemicals do not differ significantly with each other. 30% SA, ZB and ATH shows low mean boil values and perform equally with each other.

UF-bonded samples however show 30% SA as the lowest mean boil value. 10% ZB and 10% SA have high mean boil values and perform equally with 15% SA and control. 10% SA and 10% ZB shows the best performance using both UF and PF resin.

Physical properties

After the thickness swelling test using MDF bonded with PF resin, it is shown that 30% ZB has the lowest mean TS value and it perform equally with 30% SA and ATH and 20% ATH and ZB. 15% ATH, 10% ATH and control show high TS values and these chemicals do not differ with each other. UF-bonded samples however show the lowest mean TS value at 30% ZB. It performs equally with 30% SA and ATH, 20% SA and ZB, 15% SA and ZB, and 10% SA. High mean TS values are 10% and 15% ATH, 10% ZB and control and these chemicals has no significant difference with each other. 30% ZB shows the best performance using both PF and UF resin.

In ENDTS with PF-bonded samples, 10% ATH indicates the lowest mean ENDTS values whereas 20% ZB, 30% ZB and 30% SA and control shows high mean ENDTS values. Control sample significantly differ with both high and low mean ENDTS values. However with UF-bonded samples, 30% ZB, ATH, SA and 20% ZB,

ATH, SA have low mean ENDTS values and do not differ significantly with each other. 10% ZB is the highest mean ENDTS value and perform equally with control. Overall, 10% ATH shows the best performance in PF resin whereas 30% ZB in UF resin.

In terms of water absorption using PF resin, 30% ATH shows the least water absorption and perform equally with 30% ZB and SA, 20% ATH and ZB. High mean water absorption values are 15% ATH and 10% SA, ATH and ZB and do not differ with each other. There is no significant difference between the control sample and high and low mean values. In respect to UF bonded samples, 30% SA and ATH and 20% ATH have low mean WA values with control and these chemicals do not differ with each other. High mean WA values are 10% of ZB, ATH and SA, 15% of SA and ZB, and 20% ZB. These levels of chemicals do not differ with each other. 30% ATH shows the best performance using PF and UF resin.

10% ZB shows the lowest mean ENDWA value using MDF bonded with PF and perform equally with 10% and 15% SA, 10% ATH and control. The high mean ENDWA values are 20% and 30% ZB, and 30% ATH. These levels of chemicals do not differ significantly with each other. With UF resin however, the highest level of chemicals shows the lowest mean ENDWA value and do not differ significantly with each other. 10% SA however has the highest mean ENDWA value and performs equally with 10% ZB, all three levels of ATH and control. Overall, 10% ZB indicates the best performance using PF resin whereas 30% ZB using UF resin.

Discussion of the Fire Retardant treated board on mechanical properties

Relationship of chemical properties to strength properties of fire retardant treated MDF

The stiffness and strength of MDF depends upon the properties of the individual wood components and how these are combined. Factors that gives satisfactory properties are additives, sizing agents, pressing variables and others (Groom et al, 1999). As recently reported, fire retardant chemicals and high temperature environments can degrade the strength properties of wood (Le Van and Winandy, 1990). The magnitude of wood degradation depends on the particular levels of fire retardant used and the resins which may produce unwanted secondary side effects, such as increased moisture content (Le Van and Winandy, 1990). It was found that fire retardant treatments using both UF and PF resin can affect the strength of the MDF samples. As quantity of fire retardants increased, mechanical properties decreased.

It was observed from the tables and graphs above negative trends of mechanical properties are formed as higher levels of fire retardant chemicals increases to 30%. After 24 hours, MDF showed a high value of swell for the low retention compared to control and a decrease in swell for the high retention level. It is known from the method of the production of fire retardant-treated MDF in this project, certain levels of fire retardant chemicals are blended and it penetrates into each fibers. This is followed by the addition of PF or UF resin. Therefore, the swelling and water absorption of the samples decrease with increasing levels of chemicals. This might possibly be caused by the fibres and chemicals which are saturated enough to avoid water from entering and cause the samples to swell. Therefore the presence of these chemicals in the MDF give rise to the reversible component of the swell in the boards.

When undergoing the cyclic exposures on the samples, the mean values of mechanical properties both using PF and UF resins continue to decrease and gives further degradation. In terms of results on the cyclic test for thickness swelling and

water absorption, the following observations has been made. The control of MDF made from UF resin adhesive without any fire retardant gave a value of 95.22% thickness swelling and a value of 137.07% water absorption as referred to Table 8.

From table 7, PF without the fire retardant gave a swelling of 21.48% and an absorption of 130.33% much lower than UF. These results are expected considering the effect of the fire retardant chemicals on the results of the cyclic test. The following observations have been made. UF resin performs equally well with all the fire retardant chemicals with the swelling results after the cyclic test much better than without fire retardant. This shows a strong synergistic effect between the urea and the fire retardant chemicals in improving the performance in the cyclic test.

The role of ZB, SA and ATH is quite interesting. When this fire retardant chemicals are used in conjunction with NH_4Cl , the function has delayed-action hardener thereby preventing the pre-cure which is normally accounted during the hot pressing of UF based MDF. Gelling time measurements made with UF and NH_4Cl and fire retardant chemicals has shown in the table below have also confirmed independently the delayed action. Because of this, IB of MDF is well established to resist the effect of moisture during the cyclic test. IB results are indicated better mean values using UF than with PF bonded.

It is crucial to determine the suitable fire retardant chemical which gives the overall satisfactory strength results when bonded with UF resin. From table 2, both zinc borate and SA-treated has better physical properties than ATH. In conclusion, ATH has the best performance using UF resin.

Table 10: Fire retardants and levels which has the highest mean values in mechanical and physical properties using UF resin.

Properties	ATH-treated (%)	SA-treated (%)	ZB-treated (%)
Mechanical properties			
Modulus of Rupture (MOR)	*10,15		
Endurance MOR (ENDMOR)	*10,15		10
Internal bond (IB)	*10	10	
Endurance IB (ENDIB)	*10,15	10	10
BOIL	10	10	*10
Physical properties			
Thickness swelling (TS)	30	10,15,20,30	15,20,*30
Water absorption (WA)	20,30	*30	
Endurance TS (ENDTS)	20,30	20,30	20,*30
Endurance WA (ENDWA)		15,20,30	20,*30

* Level of fire retardant chemical which has the highest mean value

Table 11: Gelling time fire retardant chemicals and UF resin

<i>Chemical</i>	<i>Curing time (s)</i>
NH ₄ Cl+UF	2.15
NH ₄ Cl + UF + ATH	3.30
NH ₄ Cl+UF+SA	4.03
NH ₄ Cl+UF+ZB	6.48

A gelling time experiment was done to investigate whether the fire retardant chemicals interfere to the resin in 100°C distilled water. Mixing with UF under distilled water and record the gelling time (Table 11).

Simultaneously in evaluating the optimum amount of NH_4Cl in order to similarize the curing time of UF, it shows that ATH is the ideal curing time which leads to the highest strength properties above all. It has the closest curing time of 1.15s to NH_4Cl and UF which has a curing time of 2.15s. It promotes optimum cure characteristics to protect the system from risk of precure. If the precure is short, the resin will have the tendency to cure before the application of pressure. Sodium Aluminate is less ideal followed by ZB. ZB has the longest curing time, caused by the action of interference with the cure of UF resin. This leads to low strength properties compared to SA and ATH. It is suspected that the loss of mechanical strength is also caused by the rate of acid hydrolysis in the wood as acidic of boric acid and zinc and levels and resins increases (Le Van and Winandy, 1990). As the fire retardant chemical levels increases, the cellulosic chains will be hydrolyzed where it is often thought to be primarily responsible for the strength of the wood fiber. The length of cell molecules (DP) would be reduce which eventually reduce the macro-strength properties (Sweet and Winandy,1999). This mechanisms is initiated by the addition of proton in oxygen in the hydroxyl group and this gives an unstable carbonium ion. This ion will rearranges and regenerates proton and the process of dehydration of glucose by the acids are propagated. Another way is by the attack of proton on the glycosidic linkages that will form a depolymerization of polysaccharide chains (Le Van and Winandy, 1990). It is also mentioned by Vick (1999) that a few salts release acids at temperatures lower than fire conditions, and this will lead to destructive hydrolysis of wood. By priming treated-wood surfaces with certain alkaline aqueous solutions before bonding and selecting resins of appropriate molecular-size distribution, strong and durable bonds can be made to certain fire retardant treated woods (Vick,1999).

In contrast to the performance of UF in the cyclic test, the PF bonded MDF boards have much inferior performance. In the case of SA, there is considerable increase in the alkalinity of the binder fire retardant system. This in turn degrades the fibre particularly under the higher temperature conditions employed during the hot pressing thus resulting in much higher swelling compared to the control.

In order to examine the manner in which of the fire retardants can interfere with the formation of the adhesive film and its cure, experiment were conducted to cast films made from PF and the fire retardant chemicals, coat the same on a glass plate and cure at a temperature of 50°C for 2 minutes. The cured film was examined by light microscope and image analyser to find out the nature of the cured film. If the compatibility between the PF and fire retardant chemical is good, a continuous film with good cohesion strength will be formed. If the two materials are incompatible, phase separation will occur and a continuous film cannot form. This will in turn adversely alter the performance of the adhesion.

Micrographs cured PF films together with those containing different fire retardants are given in figures 22-25.

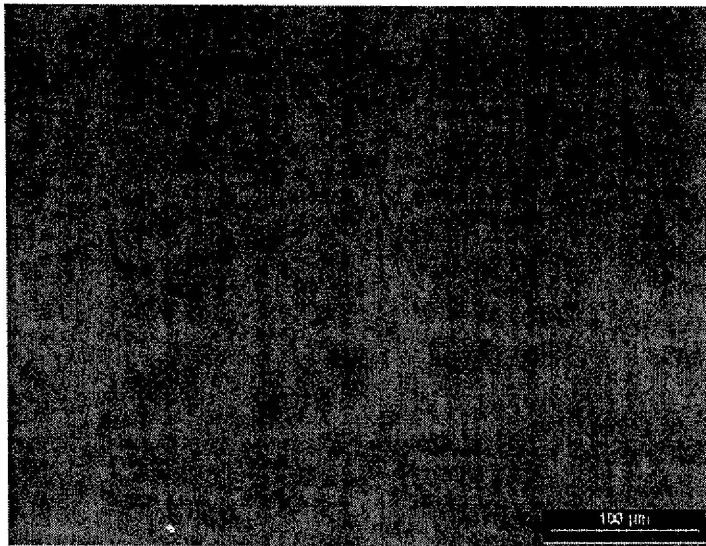


Figure 22 : Cured film of phenol formaldehyde

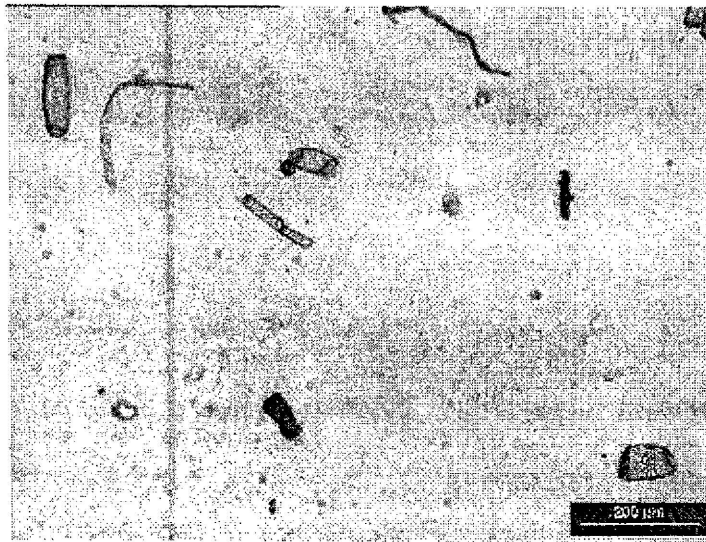


Figure 23: Cured film of phenol formaldehyde and ATH

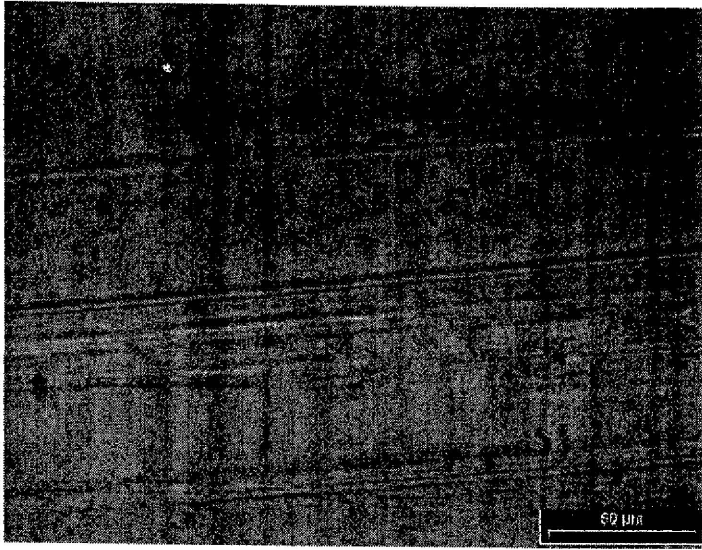


Figure 24: Cured film of phenol formaldehyde and SA

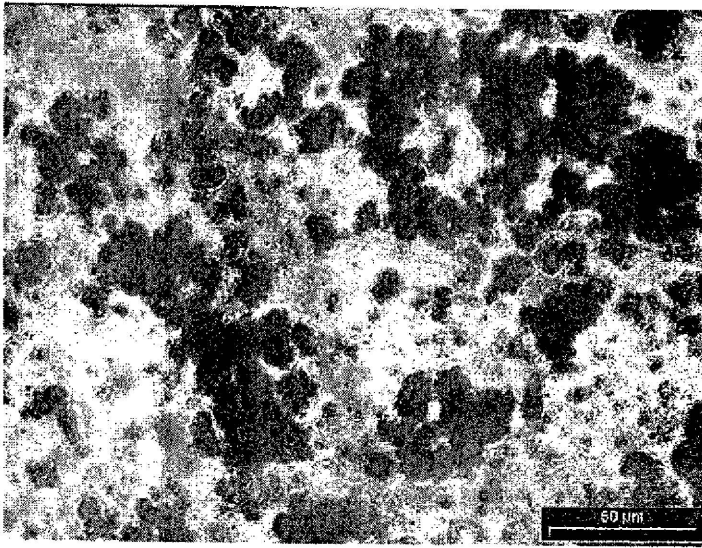


Figure 25: Cured film of phenol Formaldehyde and zinc borate

It can be seen that films made from phenolic resins and those made from the mixture of phenolic resin with sodium aluminate have given fairly uniformly cured film without any sign of phase separation. On the other hand, ATH causes a slight phase separation as evidenced by the microscopic studies. The resin remains fairly homogenous given after curing. On the other hand, the films made from a mixture of phenol formaldehyde and zinc borate particularly have shown visible sign of phase separation and cluster formation leading to discontinuity in the film. This process can cause localized stress concentration.

This phase separation results in discontinuity of the adhesive film thereby impairing the cohesive strength. Such clusters will also interfere with the establishment of a sound adhesive bond between the fibres. The MDF boards made under this conditions are therefore susceptible to interaction with moisture during cyclic test resulting in the poor performance of the PF bonded MDF. As fire retardant chemicals used are inorganic salts, they tend to spread widely throughout the MDF samples with moisture movement. Creep will occur owing from the cyclic moisture contents. Furthermore with the presence of these chemicals, creep are accelerated and significant mechanical properties are degraded (Le Van and Winandy, 1990).

It is vital to determine the suitable fire retardant chemical which gives the overall satisfactory strength results when bonded with PF resin. Among the three chemicals, ATH shows the best performance in all mechanical and physical properties with PF as the binder followed by SA and ZB. 10% ATH is found to be suitable in producing good mechanical and physical properties.

This above observation account for the fact why the ATH and sodium aluminate gives good internal bond strength, including in boil and cyclic test in contrast to the poor IB in the case of zinc borate, as referred in the table below. The highest IB strengths generally having the most uniform resin distribution (Butterfield et al, 1992).

Table 12 : Fire retardants and levels which has the highest mean values in mechanical and physical properties using PF resin.

Properties	ATH-treated (%)	SA-treated (%)	ZB-treated (%)
Mechanical properties			
Modulus of Rupture (MOR)	*30		
Endurance MOR (ENDMOR)	*10		
Internal bond (IB)	10	*10	
Endurance IB (ENDIB)	*10		
Boil	*10	10	10
Physical properties			
Thickness swelling (TS)	30	30	20,*30
Water absorption (WA)	20,*30	30	20,30
Endurance TS (ENDTS)	*10		
Endurance WA (ENDWA)	10	10,15	*10

* Level of fire retardant chemical which has the highest mean value

Table 3 shows the summarize of Table 1 and 2 of fire retardants and levels which has the highest mean values in mechanical and physical properties. Overall, ATH has the greatest effect among UF and PF resin in overall responses. The chemical that contribute to most wood degradation is zinc borate-treated.

Table 13 : Flame retardants and levels which has the highest mean values in mechanical and physical properties using both PF and UF resin.

Properties	ATH-treated (%)	SA-treated (%)	ZB-treated (%)
Mechanical properties			
Modulus of Rupture (MOR)	10,15,30		
Endurance MOR (ENDMOR)	10		
Internal bond (IB)		10	
Endurance IB (ENDIB)	10		
BOIL	10	10	10
Physical properties			
Thickness swelling (TS)	30	30	20,30
Water absorption (WA)	20,30	30	
Endurance TS (ENDTS)	10,20,30	20,30	20,30
Endurance WA (ENDWA)		15	

After observing both in fire performance and mechanical and physical properties on the fire retardant chemicals, it clearly shows boards treated with SA gives the best flame retardance performance followed by boards treated with ATH and ZB. Mechanical and physical properties on the other has a strong superiority towards boards treated with ATH which gives overall the best performance. This was followed by boards treated with SA and ZB. To conclude, in this study we found that SA was the best flame retardant chemical among ATH and ZB in reducing flame spread and increasing char, and has excellent mechanical and physical properties bonded with the adhesive UF resin.

Performance of flame retardants for MDF from EFB fibres

The results from the table 14-15 show that the MDF boards made from EFB fibres both with UF and PF resin show poor performance. It exhibits lower strength properties and does not meet the requirement stated in ISO standard. The ISO standard for a 7 mm thickness of MDF requires a minimum bending strength (MOR) of 23MPa and a 0.6 MPa of internal bond (IB). In particular bending strength and 24 hour water swelling and absorption properties were very much less than those made from rubberwood MDF. As reported by Maylor et al (1999), MDF made from EFB fibre was significantly inferior to MDF made from rubberwood fibres. Some of the theories are problems with fibre preparation and subsequent gluing difficulties. Oil palm residues give short fibres with a chemical composition more similar to straws and grasses than wood.

Table 14 : Mechanical properties of flame retardant boards using empty fruit bunch and urea formaldehyde ^a

Type of fire retardants	%	MOR	IB	TS	WA
		(N/mm ²)	(N/mm ²)	(%)	(%)
		Mean			
Control	0	19.39	0.48	30.78	90.06
		(4.85)	(0.02)	(1.04)	(44.24)
Zinc borate	10	12.07	0.5	34.51	85.67
		(2.56)	(0.03)	(3.29)	(7.42)
	15	10.58	0.49	39.88	83.34
		(4.63)	(0.02)	(2.55)	(5.36)
20	9.77	0.47	33.39	76.12	
	(0.59)	(0.03)	(8.85)	(10.20)	
30	6.99	0.42	25.85	64.12	
	(1.95)	(0.02)	(3.34)	(6.63)	
Sodium Aluminate	10	1.46	0.44	78.77	122.16
		(0.88)	(0.02)	(9.77)	(19.08)
	15	1.18	0.42	55.57	116.45
		(0.38)	(0.01)	(4.35)	(11.22)
20	1.27	0.39	53.10	99.63	
	(0.27)	(0.01)	(4.78)	(10.55)	
30	1.17	0.36	44.76	86.58	
	(0.12)	(0.02)	(7.77)	(11.32)	
Aluminum Trihydrate	10	19.40	0.48	57.31	83.12
		(1.98)	(0.06)	(4.11)	(7.53)
	15	13.85	0.42	36.04	71.10
		(4.46)	(0.02)	(5.19)	(5.92)
20	13.32	0.38	25.95	68.61	
	(1.17)	(0.01)	(6.36)	(3.50)	
30	12.44	0.33	14.36	49.44	
	(0.89)	(0.02)	(5.28)	(6.91)	

Table 15 : Mechanical properties of flame retardant board using empty fruit bunch and phenol formaldehyde ^a

Type of fire retardants	%	MOR	IB	TS	WA
		(N/mm ²)	(N/mm ²)	(%)	(%)
Mean					
Control	0	8.47	0.53	19.94	59.67
		(1.15)	(0.03)	(2.46)	(11.83)
Zinc borate	10	5.86	0.61	53.61	100.69
		(1.21)	(0.02)	(4.09)	(10.25)
	15	2.91	0.55	35.42	82.23
		(1.01)	(0.01)	(3.99)	(3.93)
	20	2.57	0.49	25.73	78.19
		(0.38)	(0.02)	(5.27)	(7.65)
	30	1.89	0.41	18.75	54.63
		(0.66)	(0.02)	(10.25)	(8.96)
Sodium Aluminate	10	4.9	0.62	20.33	105.41
		(2.21)	(0.03)	(1.41)	(10.86)
	15	3.77	0.54	20.42	81.04
		(1.22)	(0.03)	(1.13)	(7.94)
	20	3.36	0.5	17.18	73.71
		(1.2)	(0.01)	(4.14)	(12.89)
	30	2.94	0.45	15.47	65.07
		(0.32)	(0.04)	(4.43)	(10.55)
Aluminum Trihydrate	10	8.45	0.54	32.90	111.72
		(1.96)	(0.03)	(2.57)	(19.43)
	15	7.83	0.49	18.17	74.17
		(1.84)	(0.01)	(3.78)	(29.75)
	20	7.20	0.46	16.94	66.87
		(1.43)	(0.01)	(2.30)	(7.86)
	30	5.80	0.43	13.63	50.37
		(0.71)	(0.04)	(7.39)	(8.19)

Table 16 : Curing time between raw materials and adhesive

<i>Chemical</i>	<i>Curing time (s)</i>
NH ₄ Cl (1%) + UF + EFB	5.30
NH ₄ Cl (2.0%) + UF + EFB	4.03
NH ₄ Cl (1%)+ UF + RW	3.58

Table 17 : Mechanical and physical properties of EFB boards with the addition of 2% NH₄Cl ^a

Mechanical and physical properties	MOR (N/mm ²)	IB (N/mm ²)	TS (%)	WA (%)
EFB / UF / C / 1% NH ₄ Cl	19.39 (4.85)	0.48 (0.02)	30.78 (1.04)	90.06 (44.24)
EFB / UF / C / 2% NH ₄ Cl	20.19 (0.55)	0.54 (0.01)	28.02 (3.19)	84.81 (3.35)
EFB / UF / C / 4% NH ₄ Cl	22.51 (0.77)	0.60 (0.01)	27.14 (3.52)	83.54 (6.96)

^a Figures in parentheses are standard deviations

Nevertheless, MOR results in general show better strength using UF resin in comparison to PF. This might be possible due to better penetration and wetting properties for the EFB (Nor and Koh, 1992). Yet, there is no improvement in board physical properties and IB strength. Extractives and chemicals presents in the oil palm (particularly on the fibre surface) can slow down the cure of UF resins and hence influence the glue bond strength (Nor and Koh, 1992). Therefore a comparison of curing time between both raw materials, rubberwood fibres and EFB and the adhesive, urea formaldehyde is made.

From the results in Table 16, it can be seen that EFB took a longer time to cure the MDF with UF. Clearly the mechanical properties of EFB is lower than the boards by rubberwood fibres. It is mainly because of the properties of EFB which easily absorbs moisture especially in a slight low relative humidity.

With the absorption of moisture, dimensional stability tend to decline. In order to overcome this problem, it might be necessary to develop a glue formulation for improved penetration and better surface wettability properties for the EFB. Production conditions on UF such as pH, temperature, degree of polymerisation, reaction time and order of addition of reactants could be varied to formulate a more tolerant of higher fibre moisture content (Maylor *et al*, 1999). Improved gluing properties enhances strength properties particularly in terms of internal bond strength. Furthermore, addition of rubberwood fibres might increase the strength properties of MDF inspite of its compatibility with EFB (Anis et al, 2002). Leaching also occurred during the water absorption and thickness swelling test. Therefore, to decrease water absorption to a certain extent an inclusion of hydrophilic substance is needed (Anis, 2002).

Therefore, the amount of hardener was increased to improve the MDF properties to meet the specifications (Table 17). By increasing NH_4Cl to 2.0% with the mixture of UF and EFB, the gelling time is almost similar with the rubberwood fibres. Subsequently, the mechanical properties of EFB boards increases. The experimental board apparently marginally complies with the ISO standard quality specifications. This indicates that NH_4Cl is influencing the strength with MDF from EFB in the performance of the UF resin.

Thermal Analysis on the Flame Retardant MDF

Thermal analysis was intended mainly to study both physical and chemical changes occurring in the treated and control samples on heating. These tests nevertheless were conducted only on fire retardant-treated MDF made from rubber wood fibres where it fits the minimum requirement in the ISO standard, in comparison to MDF made from EFB fibres. The amounts of resin and hardener added (viz 15% and 1%) based on the oven dry weight of fibres used to produce MDF were similar to a commercial MDF. The fire tests conducted on the sample indicated only trends in fire performance and include characterization of thermal stability such as mass loss rate (T_p) and char fraction (μ) in TG, specific heat in DSC, and weight loss, area of ellipse and char index in cabinet test.

The chemical treatment for imparting flame retardants used were Aluminum Trihydrate, Zinc Borate and Sodium Aluminate.

ATH [$Al(OH)_3$] functions as fire retardant due to endothermic effect caused during the high temperature exposure. Zinc borate $xZnO.yB_2O_3.zH_2O$ (Anonymous ⁷) on the other hand enabled the inhibition of mass transfer of combustible vapours. Boron compounds act as buffer whereby acidity and alkalinity are balanced. At temperatures of 290-300C they retain their water of hydration and promotes char (Anonymous ⁷). Sodium Aluminate ($NaAlO_2$) is composed of 50-56% aluminum in Al_2O_3 , 0.05% Fe in Fe_2O_3 and about 40-45% Na in Na_2O (Anonymous ⁶).

The influence of flame retardant MDF towards cabinet test

The cabinet test results (ASTM D 1360-79) are summarized in Table 18 and Table 19. Figure 25 to 27 were plotted as normalized percentage values of untreated control. These values were obtained after each sample sized 0.5 cm x 21.2 cm x 21.2 cm was subjected to flame. Values of char index, area of ellipse and weight loss of samples were determined. Visual observations indicated that the size of ellipse was not the same for various levels of fire retardants bonded with PF and UF resin.

When each fire retardant chemicals and levels were exposed to heat, it still underwent pyrolysis and chars in much the same manner as control panels. Nonetheless, tar and sufficient quantities of combustible gases were not released while surface flame occurred (Abdul Rashid and Murphy, 1993).

There is a direct relationship between the effects of chemicals on the fire performance as referred to table 18.

Apparently zinc borate treated samples have the highest value of char index, area of ellipse and the highest weight loss. Specifically, char index, as referred to the weight of board was less due to the efficiency of the fire retardant chemicals. This study showed that zinc borate functioned far less efficiently in reducing flame propagation compared to sodium aluminate samples and aluminum trihydrate. Aluminum trihydroxide functioned most efficiency as flame retardant.

Table 18 : Cabinet test results (ASTM D 1360-79) on MDF using phenol formaldehyde

Type of fire retardant	%	Char index	Area of Ellipse	Weight loss (%)
Control	0	0.005	10279.24	21.72
SA	10	0.003	5262.04	12.57
	15	0.003	5074.64	10.57
	20	0.003	3145.01	8.00
	30	0.001	1972.16	1.16
ZB	10	0.018	9830.93	18.58
	15	0.010	6961.21	14.85
	20	0.008	6473.26	11.25
	30	0.005	5130.24	7.48
ATH	10	0.002	7999.87	14.14
	15	0.002	4029.46	11.31
	20	0.001	3861.28	9.41
	30	0.000	1664.93	4.18

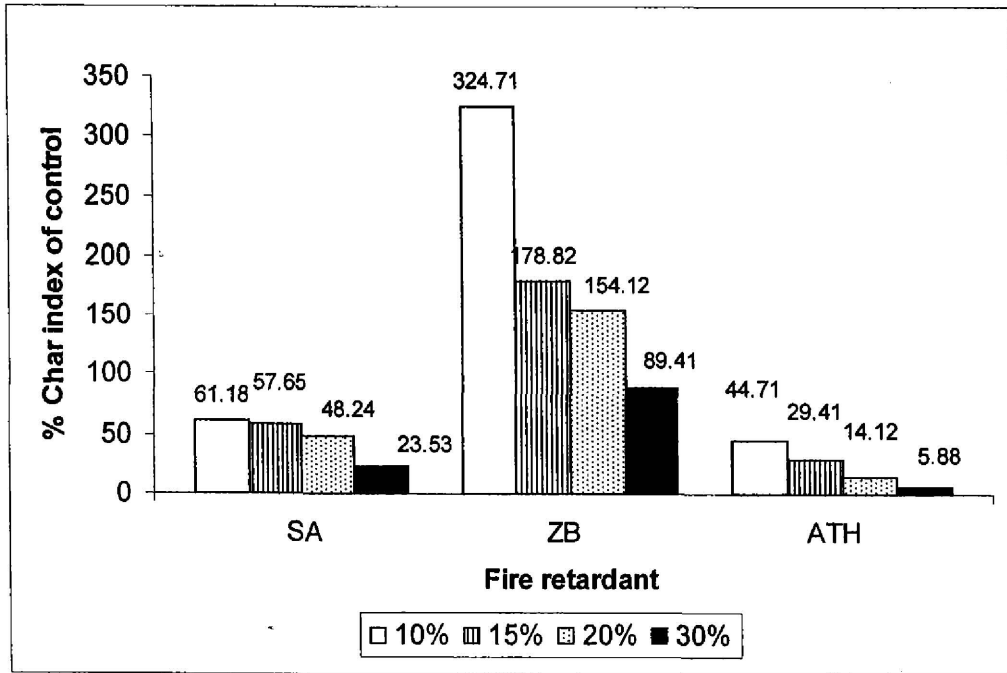


Figure 25 : Char Index of MDF made from rubberwood and PF

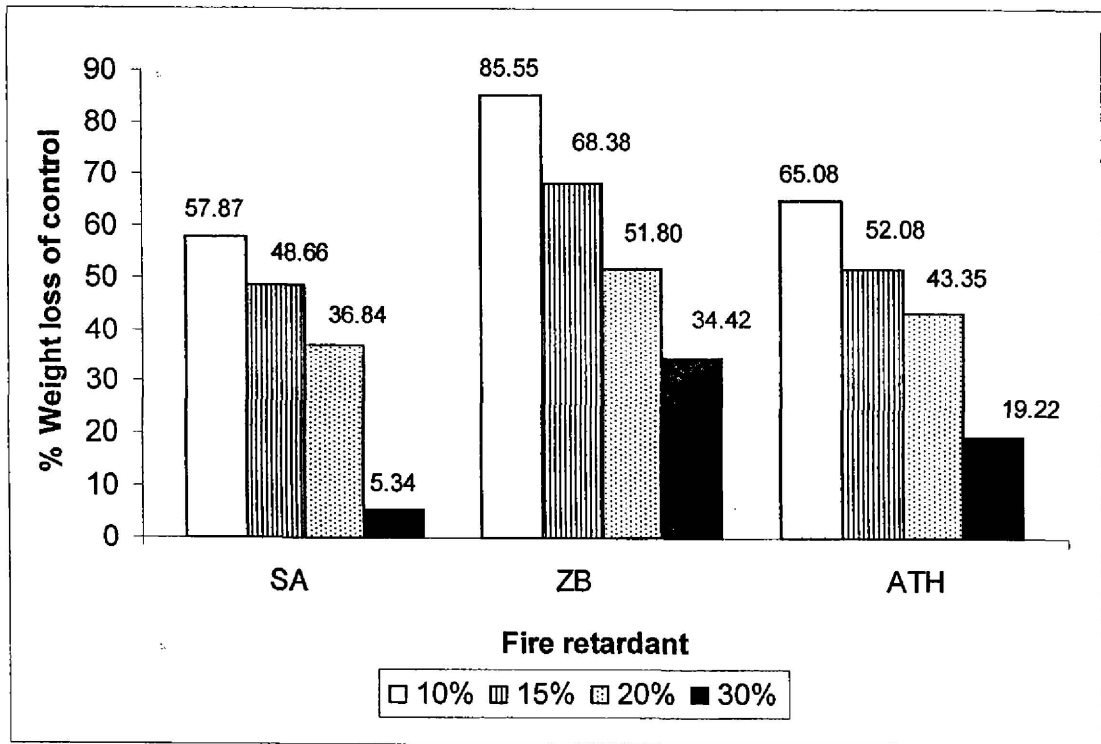


Figure 26 : Weight loss of MDF made from rubberwood and PF

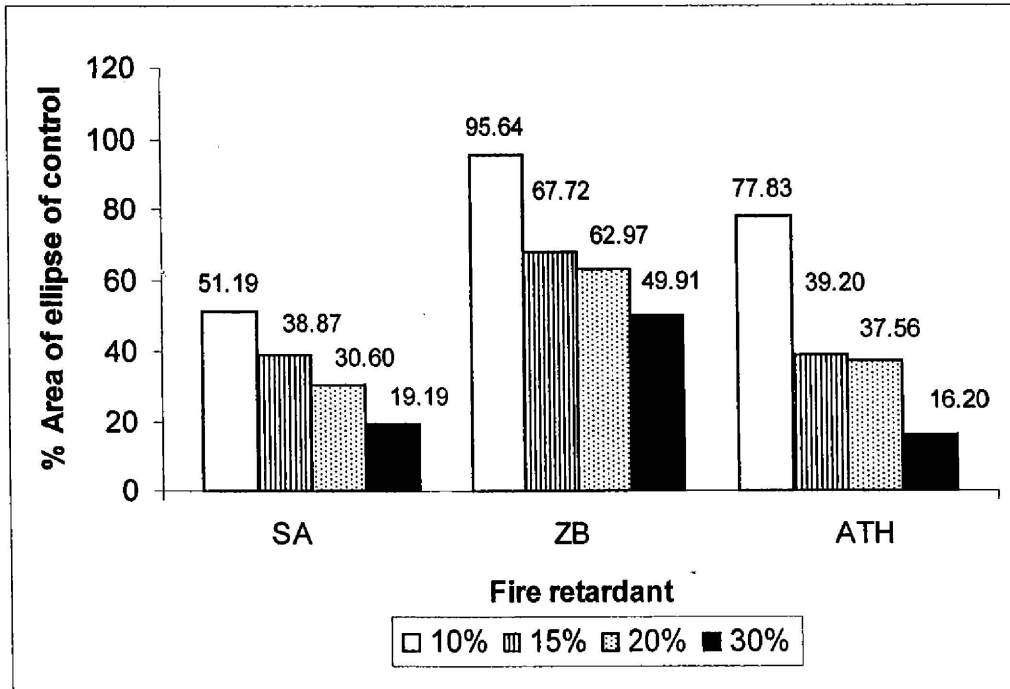


Figure 27: Area of Ellipse of MDF made from rubberwood and PF

Figures 25-27 illustrate the results of fire performance of MDF specimens bonded with phenol formaldehyde. As expected, the char index of samples decreased as the levels of loading of flame retardant chemical increased up to 30%. As compared to urea formaldehyde, the char index values were higher. Zinc borate has the highest amount of char index of control using phenol formaldehyde with a value of 324.71%. The weight loss of sample during the cabinet test in Figure 3 indicated that sodium aluminate showed the best performance with the least weight loss even though the other two fire retardants chemicals also imparted good flame retardancy. Zinc borate on the other hand has the highest value of weight loss. As referred to the above was the area of ellipse formed when the samples were subjected to the impingement of flame at 45° to each sample. Figure 5 shows that zinc borate samples bonded with PF has the largest area of ellipse of 95.64 cm² followed by ATH and sodium aluminate of value of 77.83 cm² and 51.19 cm² respectively. As the loading of fire retardant chemicals increased, the area of ellipse decreased in every case. This clearly showed that the presence of PF resin has a potential of rapid burning which eventually enables all fire retardant types contribute to greater thermal degradation.

As referred to Table 28-30, as expected, overall result shows a negative trend as fire retardant chemicals levels increases. In general boards made from rubber wood fibres bonded with urea formaldehyde have lesser values of char weight, area of ellipse and weight loss when compared with those boards made using phenol formaldehyde resin. This maybe due to exothermal combustion during the thermal decomposition of phenol formaldehyde.

Table 19 Cabinet test results (ASTM D 1360-79) of MDF using urea formaldehyde

Type of fire retardant	%	Char index (g)	Area of Ellipse	Weight loss (%)
Control	0	0.007	5402.30	19.88
SA	10	0.002	3343.65	11.62
	15	0.001	3220.14	7.98
	20	0.001	3102.42	5.46
	30	0.000	1436.57	1.83
ZB	10	0.007	5358.96	25.43
	15	0.006	5211.33	22.34
	20	0.003	4716.92	18.19
	30	0.001	3083.00	10.41
ATH	10	0.001	3966.82	18.13
	15	0.001	3307.62	10.61
	20	0.001	3045.28	6.77
	30	0.000	2084.34	3.83

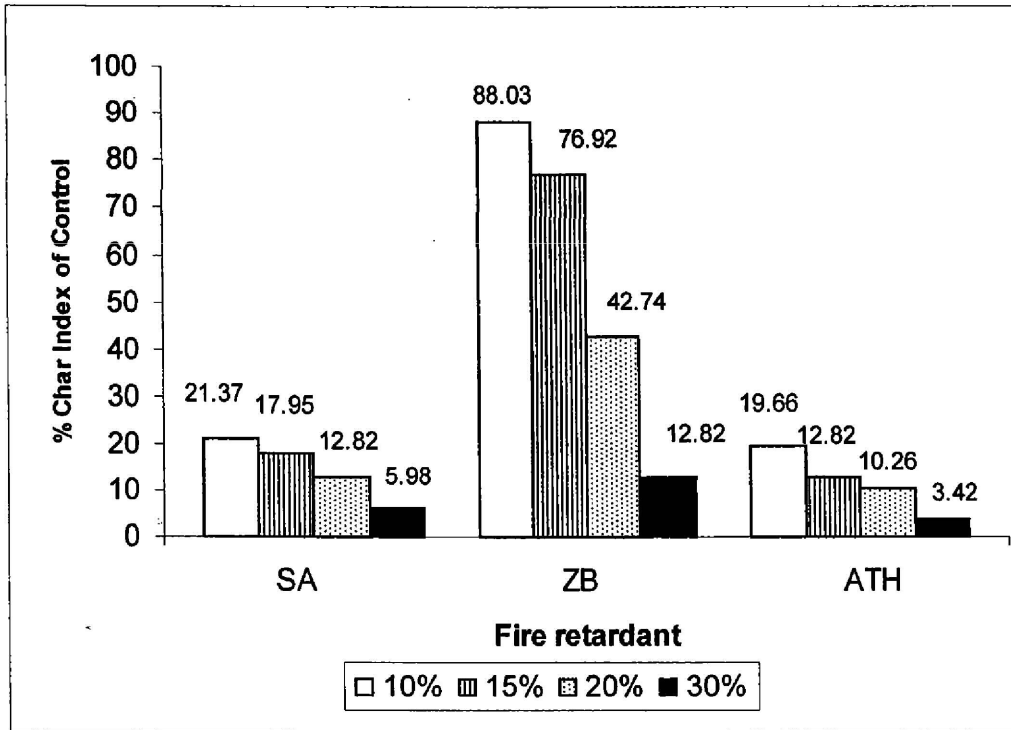


Figure 28 : Char Index of MDF made from rubberwood and UF

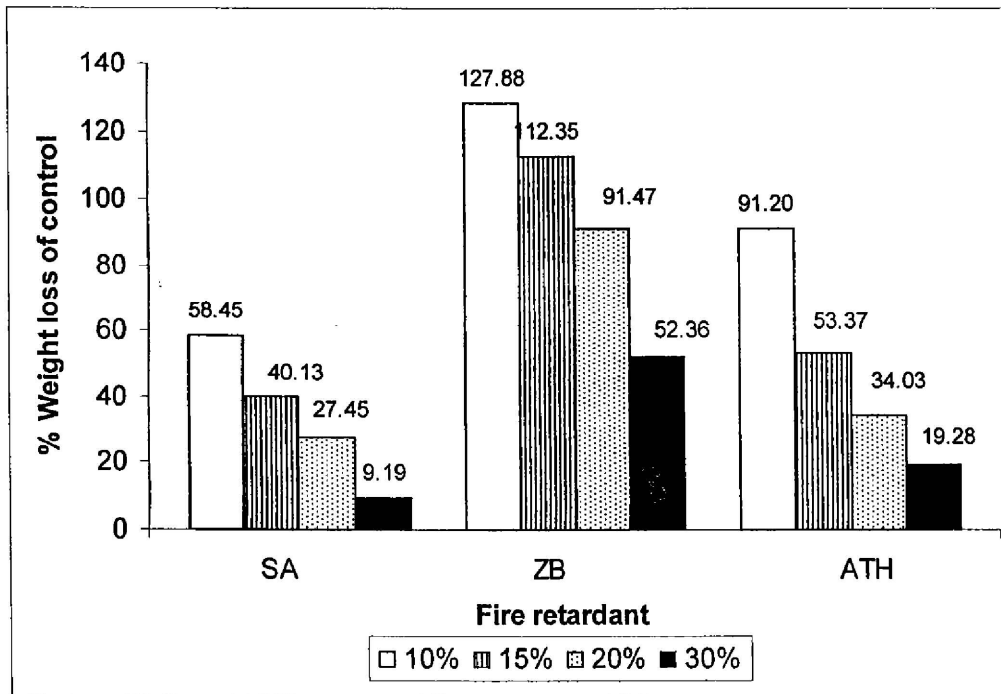


Figure 29: Weight loss of MDF made from rubberwood and UF

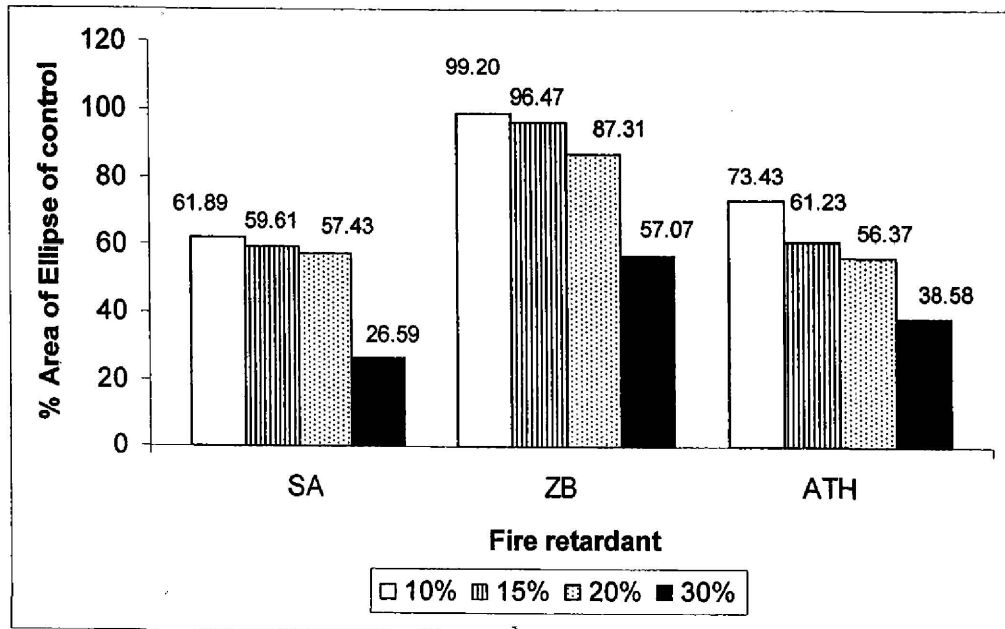


Figure 30: Area of Ellipse of MDF made from rubberwood and UF

In Figure 29, samples bonded with urea formaldehyde indicated a huge reduction of char weight when treated with sodium aluminate and ATH chemicals. Zinc borate has the maximum weight of char of 88.03% compared to control, yet decreased to 12.82 up to 30%.

A pattern of decreasing weight loss is also shown in Figure 29. However overall values were quite similar with MDF samples bonded with phenol formaldehyde. On the other hand, Figure 30 shows a higher efficiency of boards treated with SA with a decreasing trend of area of ellipse to a value of 26.59% in comparison to control samples. This area was much lower than those obtained with other fire retardant chemicals. To conclude, the most pronounced change was shown in boards treated with sodium aluminate and boards treated with zinc borate was indicated as the least effective flame retardant chemical.

The influence of flame retardant MDF towards TG

This thermal analysis techniques are known to have a long history in the study of wood pyrolysis and combustion. Variables like sample size, heating rate, atmosphere and gas flow rate are held constant to minimize any shift variables that could occur on the temperature axis position of the weight loss pattern.

Percentage of weight loss were plotted as a function of temperature for the flame retardant treated MDF. A set of typical degradation patterns for treated and untreated MDF are shown in Figure 31 and 32. The results of measurements on 3 different fire retardants and levels were summarized in Table 20.

The influence of fire retardant on the thermal decomposition can easily be observed. Furthermore, char fraction (μ) were also measured in TG. It is used as a relative measure of the fire retardant effectiveness. Char fraction indicates the percentage of char that remains at 550°C. This was because as char was formed, the heat and the amount of burning volatile product are drastically reduced. Therefore it is pertinent to realize the amount of residual char formed during pyrolysis of flame retardant treated MDF is influenced by the degradation reactions.

Control samples of both type of resins showed considerable weight loss of 74.08% and 73.67% compared to samples treated with fire retardants. High lignin content of 30% in rubberwood and 20.4% of EFB were found from this study. It was mentioned by Irving (1976) that lignin-rich materials were susceptible to pyrolysis which eventually leads to higher char. As temperature reaches 180°C, lignin began to break down and produced volatiles more readily than cellulose.

Table 20 : Thermogravimetry (TG) on MDF using rubber wood fibres and phenol formaldehyde ^a

Type of fire retardant	%	Mean weight loss (%) at 500°C	Carbon (%) at 500°C
Control	0	73.67 (0.83)	26.33
SA	10	58.54 (0.25)	41.46
	30	56.29 (2.20)	43.71
ATH	10	65.78 (2.74)	34.22
	30	65.13 (3.55)	34.87
ZB	10	67.07 (2.76)	32.93
	30	64.35 (1.78)	35.65

(^a Figures in parentheses are standard deviations)

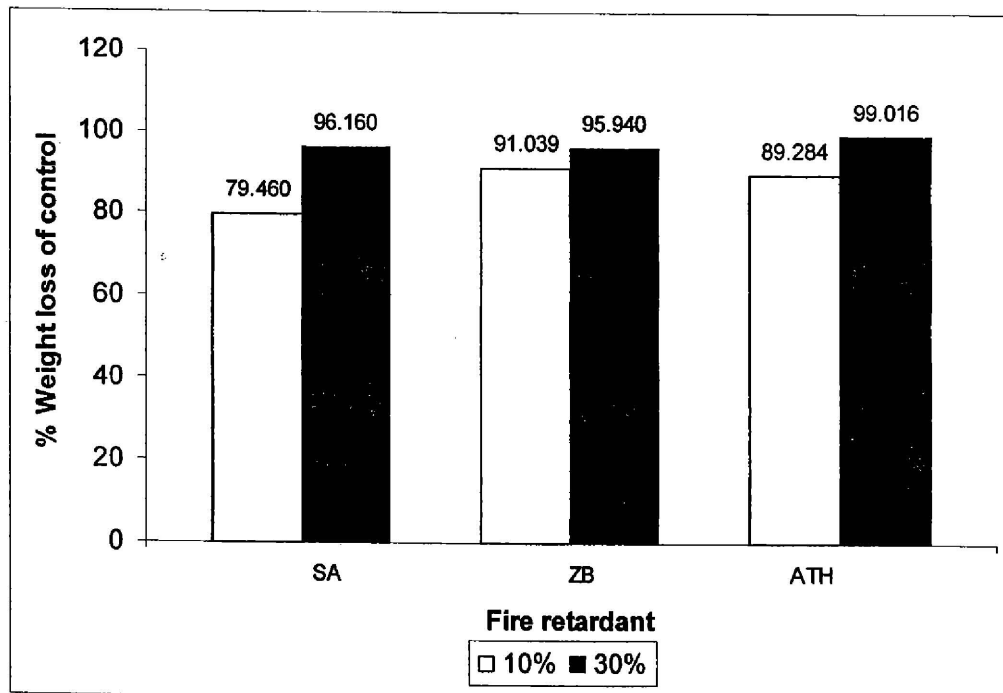


Figure 31: Weight loss of MDF made from rubberwood and PF

As can be seen in Table 21 and Figure 32, char and mean weight loss values vary greatly from boards treated with sodium aluminate to boards treated zinc borate for boards that were bonded with PF resin. The percentage of weight loss of MDF was influenced by the type of fire retardant chemicals and levels whereby the higher the percentage, the lower the percentage of weight loss.

The lowest mass loss (T_p) and highest char yield which represent the amount of carbon were characteristic of boards treated with sodium aluminate. High char yield are usually associated with thermally stable structures in the backbone. As an inorganic chemical, it generally acts simultaneously on the MDF surface by cooling it through the endothermic process and thus pyrolysis products formed were reduced (Troitzsch,1998).

IA remarkable char residue at 550°C of 43.71% was seen in boards treated 30% sodium aluminate. This means the amount of char formed during pyrolysis increased considerably ranging from 41.46 to 43.71% by reacting MDF samples with sodium aluminate. It is known that wood is almost 50% carbon, and this means any char values approaching 50% would give out minimal production of flammable volatiles or smoke (Abdul Rashid and Murphy,1993). Boards treated with sodium aluminate has almost reached the 50% production of char to the maximum of 43.71%. As referred to Bowyer and Haygreen (1989) the reason char was produced from boards treated with sodium aluminate creating minimal volatiles was that as it begins to form char on the surface of MDF which functions as a screen against conduction of radiated heat, which will eventually retard diffusion of inflammable gases from within.

As the layer of char thickens, the burning will become too slow to continue the decomposition until the fire stop. As the amount of sodium aluminate increased to 30%, char increases to large amounts. This was followed by boards treated ATH and zinc borate. Char and mean weight loss values of boards treated with zinc borate were slightly similar with boards treated with ATH both at 10% and 30% level ranging from 64% to 67%.

Table 21: Thermogravimetry (TG) of MDF using rubber wood fibres and urea formaldehyde ^a

Type of fire retardants	%	Mean weight loss (%) at 500°C	Carbon at 500°C
Control	0	74.08 (1.33)	25.925
SA	10	63.21 (4.93)	36.785
	30	54.95 (0.28)	45.049
ATH	10	64.021 (0.65)	35.979
	30	60.298 (3.28)	39.702
ZB	10	72.30 (1.34)	27.702
	30	70.35 (0.16)	29.650

(^a Figures in parentheses are standard deviations)

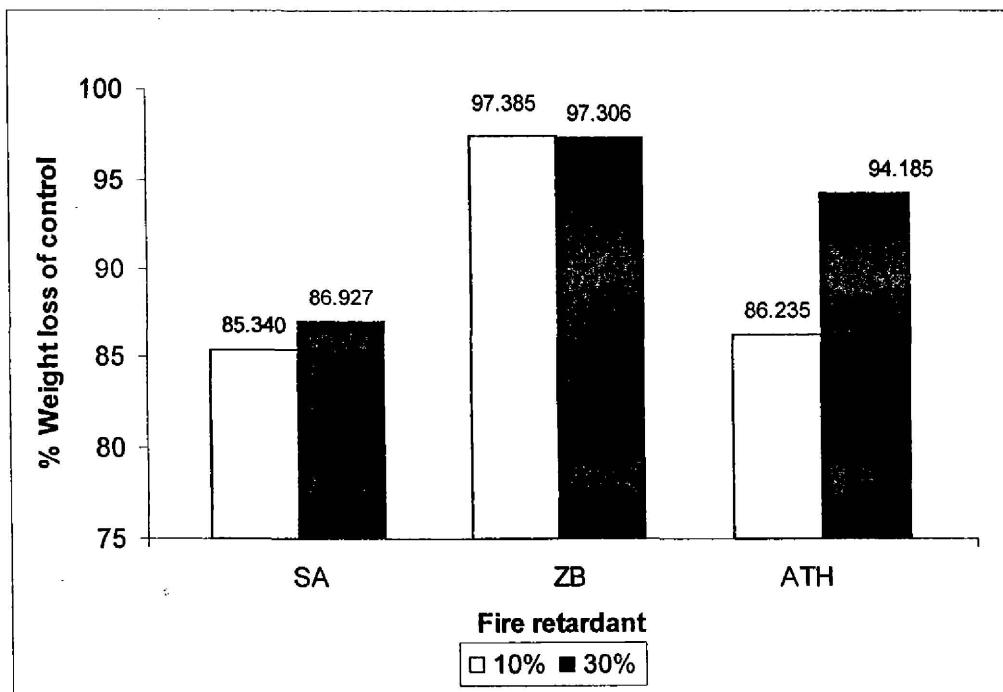


Figure 32: Weight loss of MDF made from rubberwood and UF

Table 21 and Figure 32 give TGA data of the MDF treated with various fire retardants bonded with UF resin under highly pure N₂. As the level of all chemicals increased, mean weight loss decreased and char thickened. It indicated a close mean weight loss value between boards treated with sodium aluminate and boards treated with ATH of 63.215% and 64.021% at 10% level. For boards treated with sodium aluminate remains the lowest mean weight loss followed by samples of boards treated with ATH and zinc borate treated as the highest value of mean weight loss but lowest char fraction. Eventhough all three chemicals are categorized as inorganic fire retardants and has similar modes of actions, yet sodium aluminate and ATH showed to be better chemicals.

Boards treated ATH and bonded with UF resin showed to be a more effective fire retardant whereby a much lower mean weight loss and higher char weight were obtained in comparison to MDF treated ATH bonded with PF resin. This indicated UF resin contributes better interaction with ATH in producing effective fire retardant MDF. Known as a hydrated alumina, it is generally applied in the polymer industry. Yet it contributes high char values of 39.702% after sodium aluminate samples. Overall, samples bonded with UF obtained a higher mean weight loss and lower amount char than PF resin. This means PF resin has better influence on the fire retardants in the TGA evaluation.

The graphs 33-40 were extrapolated from 0°C- 550°C in order to get the behavioural pattern in the loss in weight. TG curves showed an initial decrease in the weight of samples between 100°C to 150°C due to the release of moisture remaining in the samples. From the graphs shown it was observed that fire start and end the chemical decomposition at lower temperature. Therefore, the objective has been reached whereby heat of combustion of the volatiles was reduced (Holmes,1977). As mentioned by Kollman and Wilfred (1968), all known fire retardant increased the charring of wood at relatively low temperature, thus improving thermal insulation.

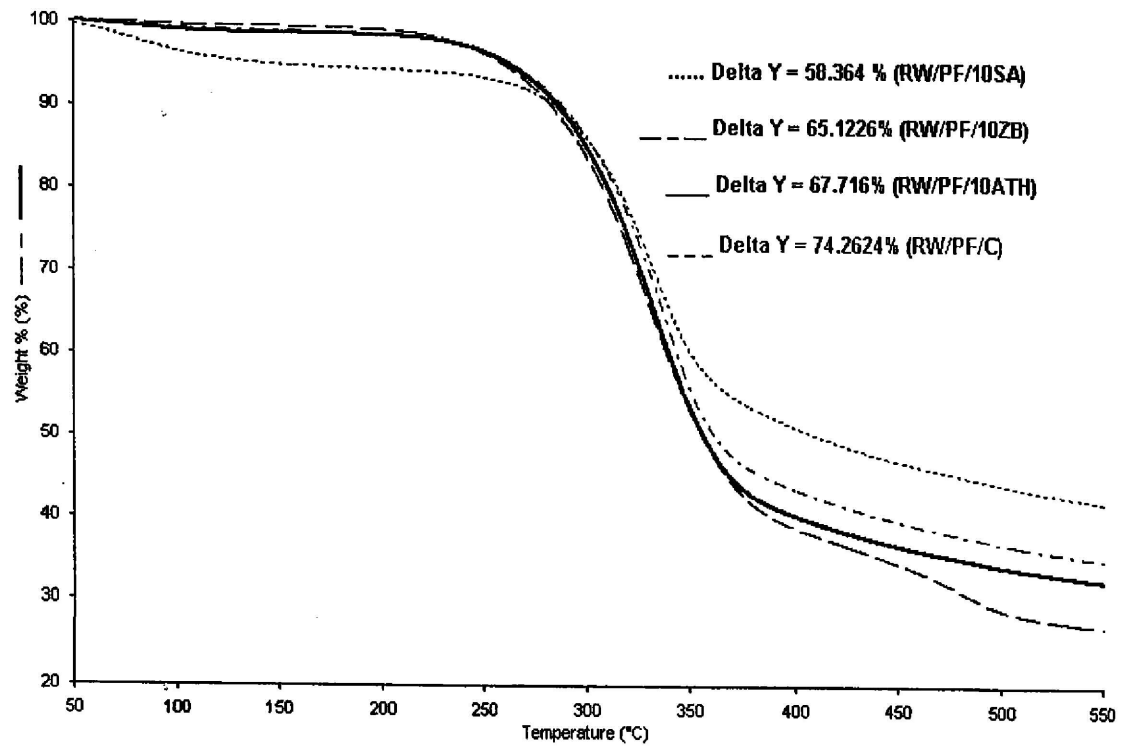
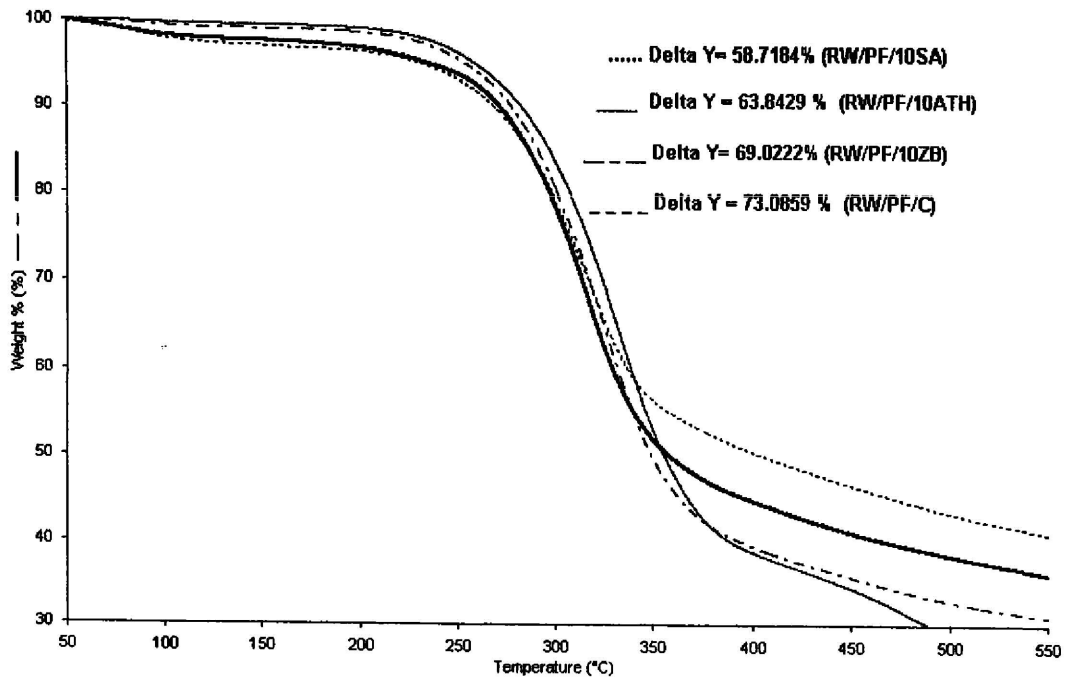


Figure 33 and Figure 34 : RW/PF/10% FR

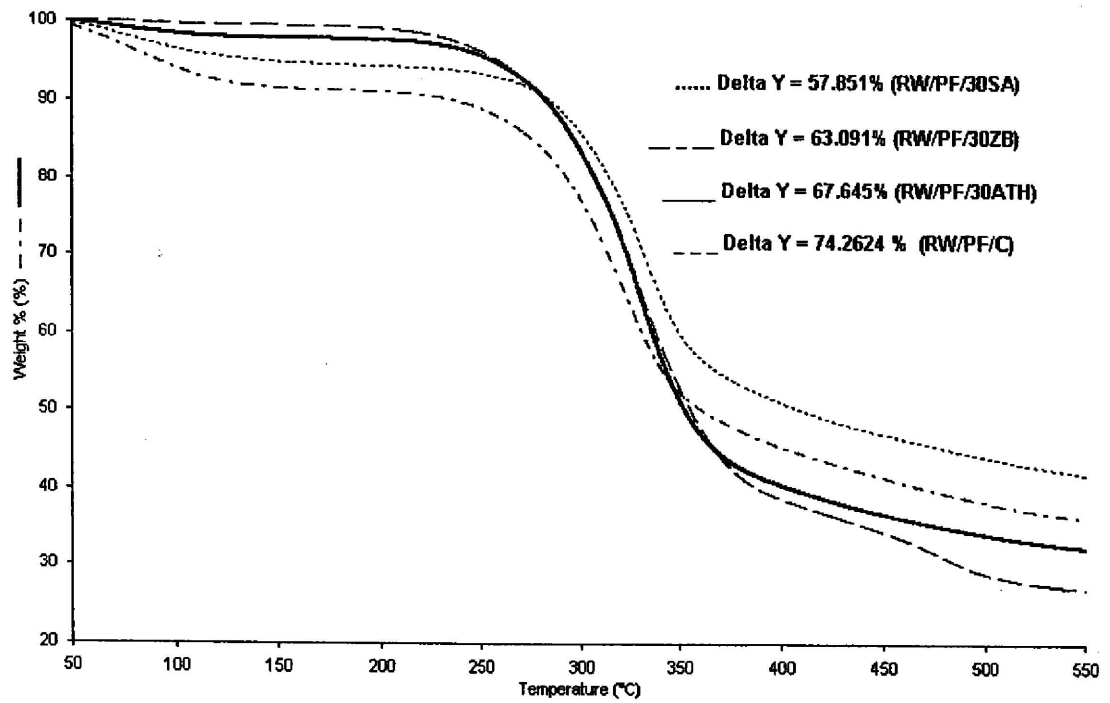
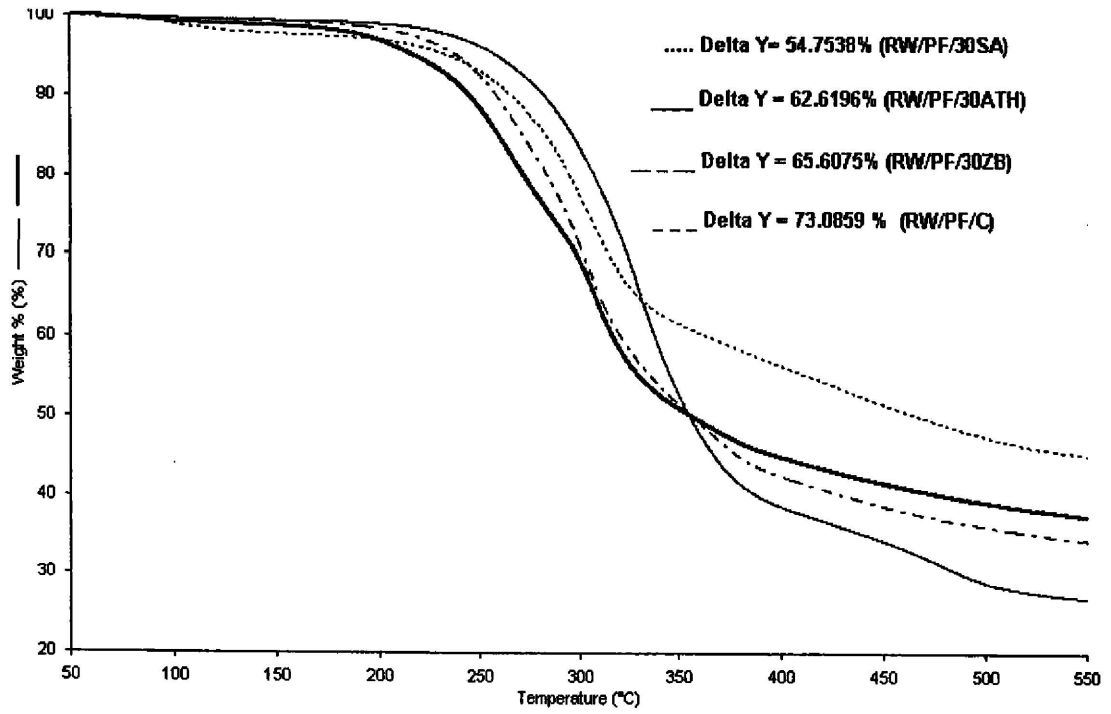


Figure 35 and Figure 36 : RW/PF/30% FR

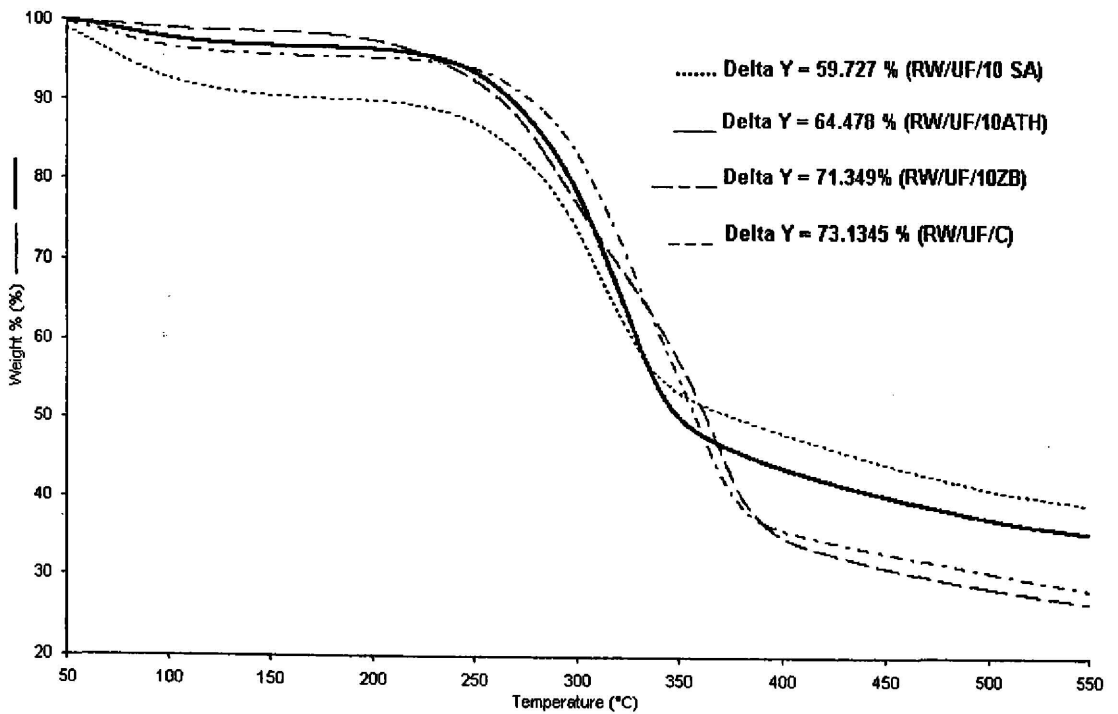
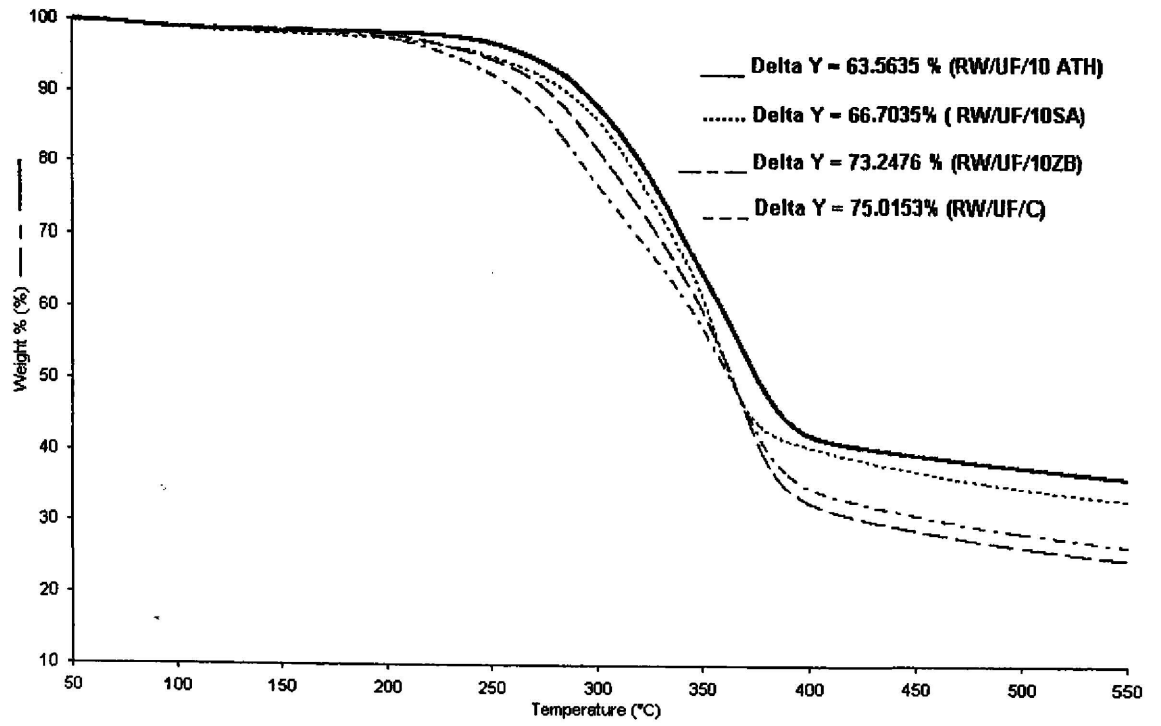


Figure 37 and Figure 38 : RW/UF/ 10% FR

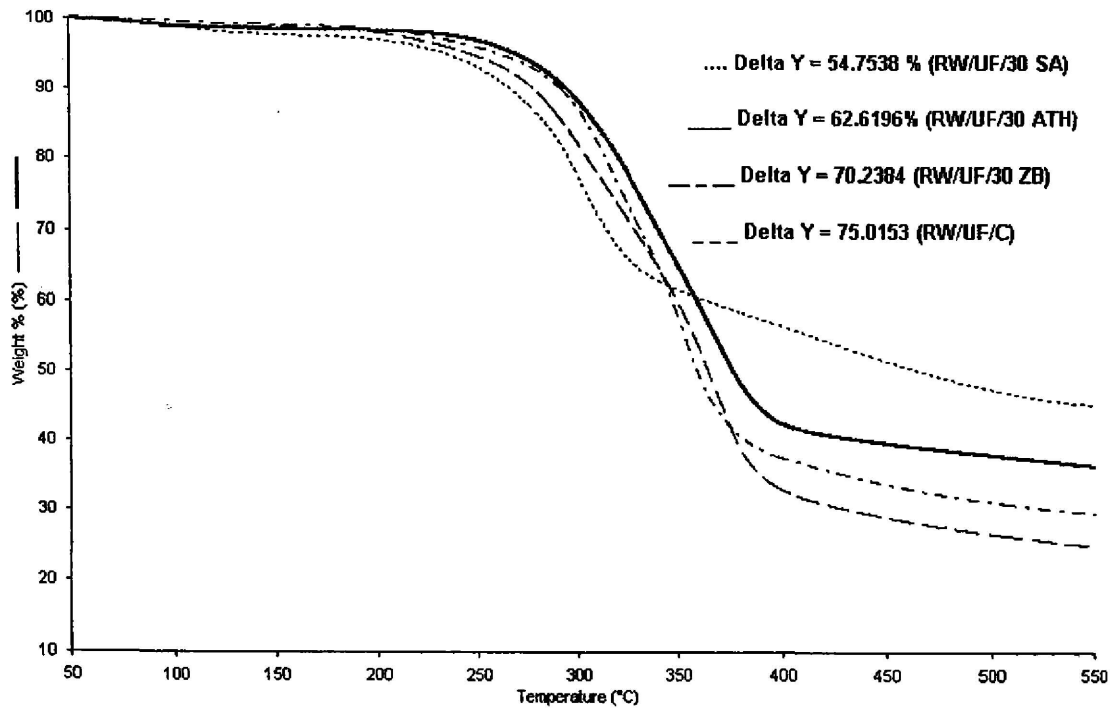
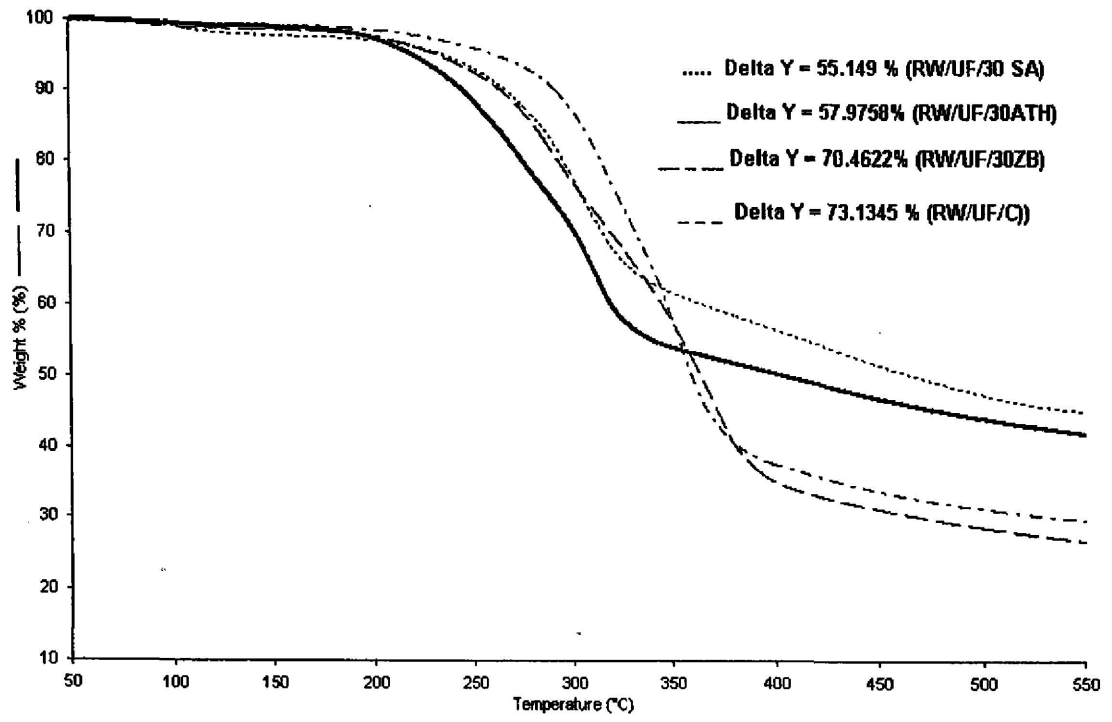
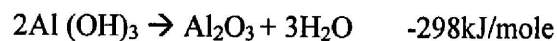


Figure 39 and Figure 40: RW/UF/ 30% FR

According to Figure 33 to 40, in boards treated with sodium aluminate samples obviously changed the thermal degradation behaviour by reducing the initial temperature of thermal degradation. It was at 100°C primary degradation occur towards the acidified MDF samples. At 180°C pyrolysis began to start and became exothermic at 240-260°C whereby condensible vapors were produced such as acetic acid, furfural, and methanol. By comparing the TGA data of fire retardant treated samples and control, the pyrolysis reaction were obviously altered in the presence of chemicals by creating lesser flammable gases and more chars and H₂O. This eventually lead to dehydration and charring of cellulose (Holmes, 1977).

As sodium aluminate-treated boards was indicated as the most effective, high percentage of char was formed from samples with as little as boards treated with 10% sodium aluminate. This was followed by boards treated with ATH. The efficiency of ATH takes place as it breaks down from the temperature 180C-200°C, aluminium oxide and H₂O vapour are released in an endothermic reaction, similarly to this reaction below;



Similar effects as sodium aluminate as an inorganic chemical will occur. In addition, the aluminium oxide will form an insulating protective layer on the substrate and H₂O vapour will act as a diluting agent in the gas phase and forms an O₂ displacing protective layer over the condensed layer (Troitzsch, 1998).

On the other hand, at low temperature the thermal degradation of boards treated with ZB was closely similar to that of the untreated fire retardant. Zinc borate basically functions by forming a glassy protective layer which gives out the effects of O₂ and heat (Troitzsch,1998), from this study zinc borate was obviously the most least effective fire retardant, the maximum amount of char from this treatment being 35.65% at 30% level.

The lower pyrolysis temperature increased amounts of char formed in MDF treated with sodium aluminate and ATH showed that both chemicals could proved to be the most effective fire retardant.

The influence of fire retardant MDF towards DSC

Analyses using DSC were conducted in open aluminium pans under N₂ atmosphere. The calorimetric enthalpy (ΔH cal) is defined as a measurement of the heat energy uptake, given by the area under the transition peak. (Figure 19 and 20).

In the figures, the samples has been heated up by a constant temperature rise and the melting of the crystalline phase will appear as an endothermic process. It also shows Cp data for each fire retardant involved is showing that the amount of energy required to heat this material increases with temperature and goes through a peak during the crystalline melt. In general, pyrolysis of wood components and wood in N₂ showed that active pyrolysis took place after 100C.

Specific heat capacity (Cp) are known as a fundamental thermodynamic property. It is measured as an indicator of changes on structure. Cp increases whenever a material is heated through the glass transition region and eventually starts to mobilize. With that Cp indicates changes in structure. This signifies quantity of heat be delivered to a substance to heat it over a temperature range (Cassel, 2001).

Sodium aluminate is shown to be the effective fire retardant among a ATH and zinc borate inspite of its ability to highest heat absorption during decomposition. ATH is also quite similar to sodium aluminate whereby a large amount of heat of absorption occur. As mentioned by Jian Li et al (2001), heat absorption occur when a large volume of H₂O are liberated. Thus O₂ content is reduced due to the gas phase dilution by the water vapour produced. In short, the mechanism of ATH cause dehydration. This dehydration reaction will lead to the higher levels of char and a dwindling amount of volatiles. Strong endothermic decomposition in the DSC will dilute combustible gases and prevent access of the surface to O₂ of the substrate are prevented. Subsequently, further ignition are suppressed (Suchsland and Woodson, 1991).

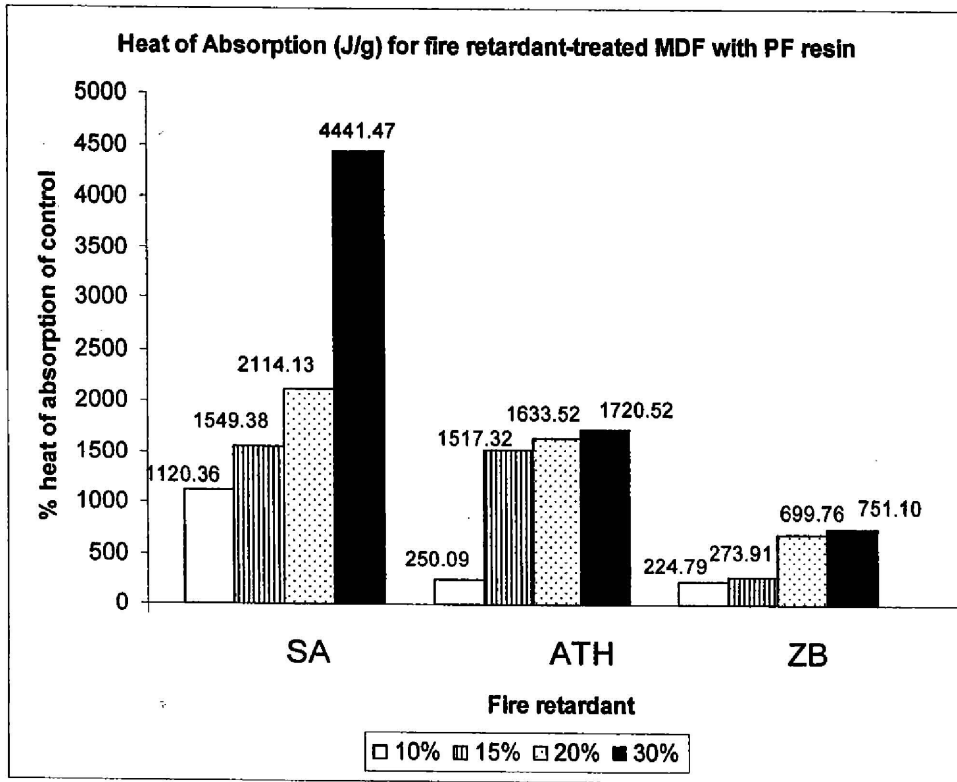


Figure 41: Heat absorption (J/g) of MDF with PF resin

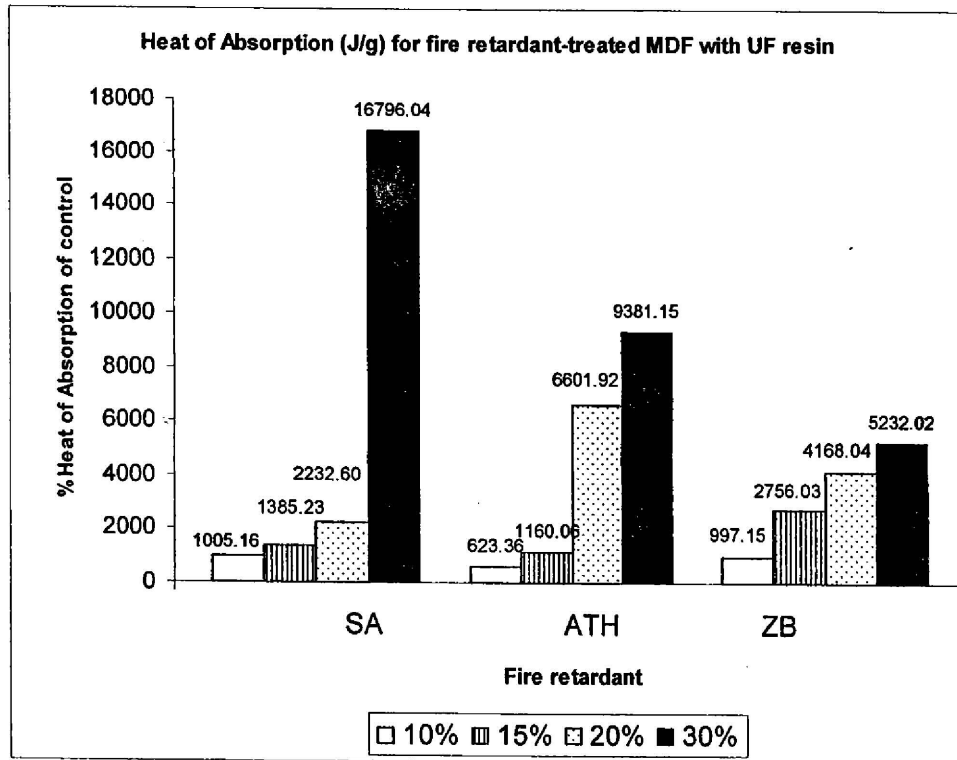


Figure 42: Heat of absorption (J/g) of MDF with UF resin

Figures that 41- 42 a set of DSC heating thermograms were collected at a heating rate of 20°C per minute. As the amount of each chemical reacted with MDF samples increased to 30% level, the temperature of pyrolysis decreased, similar to the TGA results. Figures bonded with PF resin and using sodium aluminate were pyrolyzed at lower temperature of 116°C to 160°C at 10% sodium aluminate followed by a steep increase and an endothermic reaction at a peak of 139°C which was attributed to flaming combustion of volatiles and tars, and the char oxidation was decreased gradually from a range of 200°C to 550°C.

ATH on the other hand at a temperature of 148°C to a maximum of 190°C at a level of 10% were pyrolyzed and similar to sodium aluminate thermogram it has a steep increase and an endothermic reaction at a peak of 159°C. At a temperature of more than 200°C to 550°C, char oxidation occur gradually.

Figures samples treated with zinc borate were pyrolyzed at a temperature of 193°C to 207°C and followed by a steep increase and reaches a peak of 196°C. The char oxidation however starts at a range of 200 to 550°C.

All fire retardant-treated MDF bonded with PF resin has a general gradual increase of temperature of pyrolysis as the level of fire retardant increases. To conclude, Cp is a fundamental, thermodynamic property of material and is the best way to compare samples (Cassel, 2001).

Conclusions

To conclude, there exists 2 type of mechanisms involved in this study where the flame retarding chemicals are directing the pyrolysis reactions to produce more char, H₂O and CO₂ replacing flammable volatiles; and preventing the flaming combustion of these volatiles. As mentioned by Le Van and Collet (1989),

Fire retardant should be designed to increase the char and reduce the production of flammable volatiles by lowering the temperature at which thermal degradation occurs. From this study, it has been shown that the application of these chemicals does qualify the latter. Even low loadings, can have a measurable effect on the distribution of the fire retardant bonded with resins of rubberwood fibres.

By evaluating all three fire tests, it is possible to establish the 'good' and 'bad' fire retardance between all three fire retardant chemicals. It is also allows one to evaluate the prima facie efficiency of fire retardant chemicals in the absence of large samples. As mentioned by Abdul Rashid and Murphy (1993), good fire retardant is typified by high char values (approaching 50%) in nitrogen and additive activities or catalytic dehydration activities in the range of 150-300°C. On the other hand, low char values and additive activities outside the useful temperature range are classified as poor fire retardant.

As summarized, sodium aluminate treated is indicated as the most effective flame retardant, especially for boards bonded with UF-resin. This was followed by boards treated ATH and zinc borate. All fire retardant on the hand reached an excellent retardant from fire at a level of 30%.

Overall discussion

Overall, with the increasing amount of SA, ATH and ZB leads to increasing fire retardant on the thermal analysis done which are cabinet test, TG and DSC. In the cabinet test, overall SA bonded with UF resin is the most effective fire retardant. It has the least weight loss, char index and area of ellipse. In TG at a maximum of 500C, ATH and SA presents a remarkable amount of char both using PF and UF resin. Zinc borate on the other hand has the most weight loss and the least char at 500C similar to that of the untreated fire retardant. Strong endothermic decomposition in DSC are strongly superior towards ATH and SA. They are shown to be effective fire retardants in its ability to highest absorption during decomposition. As summarized, sodium aluminate is established as “good” fire retardant which is typified by high char values (approaching 50%) in nitrogen and additive activities dehydration activities in the range of 150C – 300C at 30% level. This is especially when bonded with UF resin.

On the other hand, flame retardant MDF made from rubberwood fibres which has a profound effect on the mechanical and physical properties is ATH at a level of 10%. This was followed by boards treated SA and ZB. The boards treated with ATH bonded with UF resin gives better strength properties in comparison to PF based. Flame retardant MDF made using EFB however showed substandard mean mechanical and physical properties and do not meet the minimum requirements of the ISO standard. Therefore, only fire retardant MDF samples made from rubberwood fibres proceeded in the thermal analysis inspite of its satisfactory mean values in mechanical and physical properties and easily met the minimum requirement of the ISO standard.

Suggestions for future work

- deeper research on the ammonium phosphate MDF made from both rubberwood fibres and EFB with different variables and conditions to achieve a satisfactory strong MDF.
- Study on different process conditions such as hot pressing temperatures, pressing time, press pressure and the profile of the thickness change during pressing in order to achieve better uniform density which leads to high IB strength and eventually an overall good strength properties of the final product.
- More thermal analysis and fire evaluation on the fire retardant MDFs.
- More analysis on the anatomical features on the lignin, cellulose and hemicellulose after thermal degradation occur.
- More study on the MDF made from EFB, and probably mixing with rubberwood fibres because of its compatibility to achieve better strength properties.

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