PYROLYSIS AND HEAT RELEASE OF INORGANIC FLAME-RETARDANTS

Abdul Rashid A.M.

Forest Research Institute Malaysia, Kepong, 52109 Kuala Lumpur, Malaysia

&

R.J. Murphy

Department of Pure and Applied Biology, Imperial College of Science, Technology, and Medicine, London SW7 2BB, United Kingdom

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ABDUL RASHID A.M. & MURPHY, R.J. 1993. Pyrolysis and heat release of inorganic flame-retardants. A study on the fire-propagation and pyrolysis of Malaysian timbers, kempas (*Koompassia malaccensis*), keruing (*Dipterocarpus baudii*) and damar minyak (*Agathis borneensis*) treated with monoammonium phosphate and borax was conducted. A chemical loading required for fire retardancy was achieved in all the three species by pressure impregnation process. The fire-propagation test was conducted based on B.S. 476, Part 6:1968. Both chemicals at a retention level of 40 - 50 $kg\ m^{-3}$ improved the fire-propagation properties of the three timbers. The propagation index values of the treated timbers were comparable to those for Class 1 surface spread of flame rating. The weight loss after fire-propagation test of the timbers treated with monoammonium phosphate was lower than the borax-treated timbers. Presence of resinous material in keruing affected the fire-propagation performance of monoammoium phosphate but not of borax. Monoammonium phosphate and borax behaved differently at various temperature ranges. Monoammonium phosphate increased volatiles liberation at temperature below 300°C.

Key words: Fire-propagation performance - fire-retardants - pyrolysis

ABDUL RASHID A.M. & MURPHY, R.J. 1993. Pirolisis dan pembebasan haba bahan cegah kebakaran yang tak organik. Kajian mengenai rebakan api dan pirolisis ke atas kayu tempatan, kempas (*Koompassia malaccensis*), keruing (*Dipterocarpus baudii*) dan damar minyak (*Agathis borneensis*) yang diawet dengan monoammonium phosphate dan borax telah dijalankan. Satu kandungan kimia yang diperlukan untuk rintangan api telah dapat ditentukan untuk ketiga-tiga spesies kayu tersebut dengan menggunakan kaedah proses serapan tekanan. Ujian rebakan api telah dijalankan mengikut Piawaian B.S. 476, Part 6:1968. Dengan tahap kandungan tertahan 40-50 $kg\ m^{3}$, kedua-dua bahan kimia di atas dapat meningkatkan lagi sifat-sifat rebakan pada api ke atas kempas, keruing dan damar minyak. Nilai indek rebakan pada kayu berawet didapati setanding dengan kadar rebakan permukaan api Kelas 1. Susutan berat selepas ujian rebakan api ke atas kayu diawet dengan monoammonium phosphate adalah lebih rendah daripada kayu di awet dengan borax. Damar dalam kayu keruing telah memberi kesan ke atas performan rebakan api pada kayu yang di awet dengan monoammonium phosphate, tetapi tidak untuk borax. Monoammonium phosphate dan borax menunjukkan sifat berlainan pada tahap suhu yang berbeza. Monoammonium phosphate dan borax menunjukkan sifat berlainan pada tahap suhu yang berbeza. Monoammonium phosphate meninggikan pembebasan bahan-bahan meruap pada suhu di bawah 300°C.

Introduction

Surface flammability test conducted in accordance with various international standards indicates that wood well treated with commercial formulations significantly has lower flame spread values (Juneja 1972, Siau *et al.* 1975, Hirata *et al.* 1981, Ostman 1984). Wood shingles and plywood treated with monoammonium phosphate and borate have also given similar results (Middleton *et al.* 1965, Holmes 1971).

Fire-propagation tests based on British Standard B.S. 476, Part 6: 1968 have shown that timbers sufficiently treated with fire-retardants will have fire-propagation index I of less than 25 (Hall & Dell 1969a, 1969b; Rogowski 1970). Similar results are obtained with chipboards, hardboards and plywood treated with fire-retardant chemicals (Rogowski 1970, Ashton 1977, Abdul Rashid 1984, 1987). Untreated wood and wood based products will have an I value of more than 25 (Hall & Dell 1969a, 1969b, Rogowski 1970, Abdul Rashid 1982b, Abdul Rashid 1988).

Based on temperature rise during fire-propagation test, Rogowski (1970) and Ashton (1977) indicated that softwood and hardboard treated with fire-retardant coatings contribute less heat by about 25%. Using the same method of assessment, monoammonium phosphate and borax reduce the amount of heat released from chipboard by about the same value when they are incorporated at 20% by weight of dry chips (Abdul Rashid 1987).

When fire-retardant treated wood is exposed to heat and fire, it undergoes pyrolysis and char in much the same manner as untreated wood (lmaizumi 1963). However, it does not release sufficient quantities of combustible gases and tars to contribute to flaming along the surface of wood (Holmes & Shaw 1961, Byrnes *et al.* 1966, Fung *et al.* 1972).

Besides reducing the amount of heat released, flame-retardants also increase the charcoal formation. The effectiveness of flame-retardants can be determined by the amount of char formed (Rowell *et al.* 1984, Ellis *et al.* 1987). The charcoal yield for untreated wood at 400°C is about 20%, while treatment with fire-retardant salts results in charcoal yield of about 50% (Brown & Tang 1963, Eickner 1966). From thermogravimetric analysis, fire-retardant treated wood produces more char than untreated timber (Ellis *et al.* 1987).

Smoke evolution can be affected by some fire-retardant chemicals. The smoke density value of fire-retardant treated timber is 25% less than untreated timber (Eickner 1966). Siau *et al.* (1975) stated that inclusion of materials containing aromatic benzene rings in wood can increase smoke evolution while presence of non-aromatic phosgard decrease smoke evolution. Hirata *et al.* (1981) indicated that ammonium sulphamate and boric acid-borate mixture are most effective in reducing smoke from wood. The objectives of this study are to assess the fire-propagation performance of timbers treated with fire-retardant chemicals, borax and monoammonium phosphate, and to evaluate the behaviour of these chemicals during pyrolysis of wood.

Materials and methods

Wood samples

Two Malaysian hardwoods, kempas (*Koompassia malaccensis*) and keruing (*Dipterocarpus baudii*) and one Malaysian softwood, damar minyak or Malayan kauri (*Agathis borneensis*) were used in this study. The species were selected because they are easily amenable to preservative treatment and suitable for panelling and partitioning. The defect-free timbers were ripped into heartwood and sapwood boards. The heartwood boards were used for the tests. They were planed and then cut to the following dimensions depending on the tests to be undertaken:

Fire-propagation test	-	22 x 22 x 1.5 *cm*
Heat test	-	2 x 2 x 3 *cm*

Basic density and moisture content determination

Several discs per board were obtained for density and moisture content determination. Small rectangular blocks measuring 2 x 2 x 1.5 *cm* were obtained from the discs and the basic density and moisture content were determined. The average moisture contents of the timbers were found to be from 20 to 28 % and these were not suitable for fire-retardant treatment. The recommended moisture content of timber is normally less than 18%. Therefore the timbers were allowed to dry in a conditioning chamber prior to fire-retardant treatment.

Conditioning of specimens

The specimens for fire-propagation test and heat test were conditioned at 28°C and 55% relative humidity for one week in a relative humidity chamber in order to get a moisture content of about 12 - 14%. They were then transferred to a perspex cabinet desiccator containing silica gel until impregnation with fire-retardant chemicals was completed.

Fire-retardant chemicals

The chemicals used for the treatment in this study were monoammonium phosphate ($NH_4H_2PO_4$) (MAP) and borax ($Na_2B_40_7.10H_20$). The concentration of the treatment solution was 15% (w/w basis). These chemicals are commonly used as major constituents in fire-retardant formulations (Eickner 1966, Maksimenko & Gorshin 1979).

Impregnation treatment

The impregnation treatment was carried out by vacuum-pressure method. Six replicates per treatment were used for fire-propagation test and five for heat test. Based on preliminary results on the uptake of water by some test specimens, the following treatment cycle was adopted in order to obtain loading of about 50 $kg\ m^3$ in specimens for fire-propagation and heat tests. The loading is a standard treatment recommended in practice for 50 *mm* and thinner board (Anonymous 1963).

Initial vacuum	-	23 *in* Hg for 30 *min*
Pressure	-	80 *p.s.i* for 30 *min*
Final vacuum	-	23 *in* Hg for 30 *min*

The actual chemical loading obtained was 40 $kg\ m^3$ to 80 $kg\ m^3$ depending on the wood.

After treatment the specimens were wrapped in polythene sheet and weighed after 24 *h* to calculate the uptake. Then the chemical loading in each block was calculated:

$$\text{Chemical loading}(kg\ m^3) = \text{Uptake}\ (kg\ m^3) \times \text{Concentration}$$

After weighing the specimens for fire-propagation test and heat test were dried in an oven at 50° *C* for 48 *h* and then transferred into a perspex cabinet desiccator containing silica gel for 24 *h* in order to preclude the effect of water in wood during combustion. Moisture content achieved by this condition was calculated from basic density, sample size and pre-test sample weight giving moisture content of 28.6, 29.5 and 35.6% for kempas, damar minyak and keruing respectively.

Fire-propagation study

The conditioned samples were weighed and subjected to fire-propagation test based on British Standard B.S. 476: Part 6, 1968 using the apparatus shown in Figure 1. Prior to the test proper, the apparatus was calibrated with a non-asbestos board of 220 × 220 × 12 *mm*. The calibration was done in order to ensure that a standard time-temperature curve could be obtained with the apparatus.

After a consistent calibration was achieved, the sample was first set in one of the two furnaces in the testing apparatus. The specimens were heated along the longitudinal direction in order to avoid variation in the burning rate due to grain direction. The specimens were initially heated by butane gas for 2 *min* 45 *sec* and then by electrically heated nichrome heating elements until the total exposure time was 20 *min*. The increase in exhaust temperature given by the burning

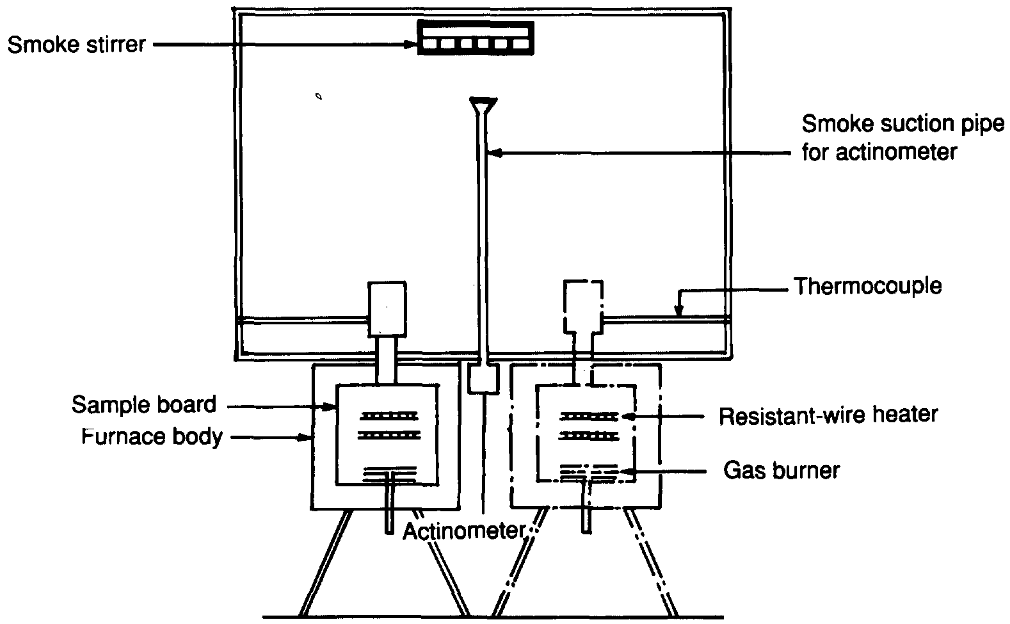

Figure 1. Heating apparatus for conducting the fire-propagation test. (The heaters have been modified to produce temperature - time relationship from JIS A 1321 to B.S. 476: Part 6)

specimen for 20 *min* was recorded using the thermocouples as shown in Figure 1 at the following intervals from the start: at $\frac{1}{2}$ *min* intervals up to 3 *min*, at 1 *min* intervals from 4 to 10 *min*, and at 2 *min* intervals from 12 to 20 *min*.

The following properties were determined from the test:

a) The weight loss after the test. The weight loss was calculated as follows:

$$\text{Weight loss (\%)} = \frac{W_{50} - W_I}{W_I} \times 100$$

where:

W_{50} - weight of sample after conditioning at 50°*C* for 48 *h*

W_I - weight of sample after fire-propagation test.

b) Fire-propagation index. The index provides a comparative measure of the contribution of the material to growth of the fire and thus to fire spread within a compartment. The values may range in descending order-of-merit from 0 to 100.

The index of performance, I, was calculated as follows:

$$I = i_1 + i_2 + i_3$$

where,

$$i_1 = \sum_{1/2}^{3} \frac{Q_m - Q_c}{10t}$$

$$i_2 = \sum_{4}^{10} \frac{Q_m - Q_c}{10t}$$

$$i_3 = \sum_{12}^{20} \frac{Q_m - Q_c}{10t}$$

Q_m = temperature rise recorded for the material
Q_c = temperature rise recorded for non-combustible standard
t = time in seconds

c) The rise in exhaust temperature.

Heat test

The conditioned specimens were weighed and then exposed to temperatures of 250°*C*, 350°*C* and 550°*C* for 30 *min* in a Thermolyne Automatic Furnace. The various temperatures were chosen to correspond to levels of active pyrolysis, pyrolysis with liberation of combustible gases and glowing combustion respectively (Knudson & Williamson 1971).

The following properties were determined from the test:

a) The weight loss at 250°*C*, 350°*C* and 550°*C*. The weight loss was calculated as follows:

$$\text{Weight loss (\%)} = \frac{W_{50} - W_h}{W_h} \times 100$$

where:

W_{50} - weight of sample after conditioning at 50°*C* for 48 *h*.
W_h - weight of sample after heat test at 250°*C*, 350°*C* and 550°*C*.

b) The amount of smoke and moisture liberated at respective temperatures were observed.

Results and discussion

Fire-propagation performance

Weight loss

When heated, wood decomposes and loses weight. At the same time it produces flammable volatiles which accelerate pyrolysis. Therefore weight loss can be used as a measure of the tendency of the wood to burn once ignited (Taylor 1925, Veer & McKnight 1963). The weight losses after the fire-propagation test are shown in Figure 2.

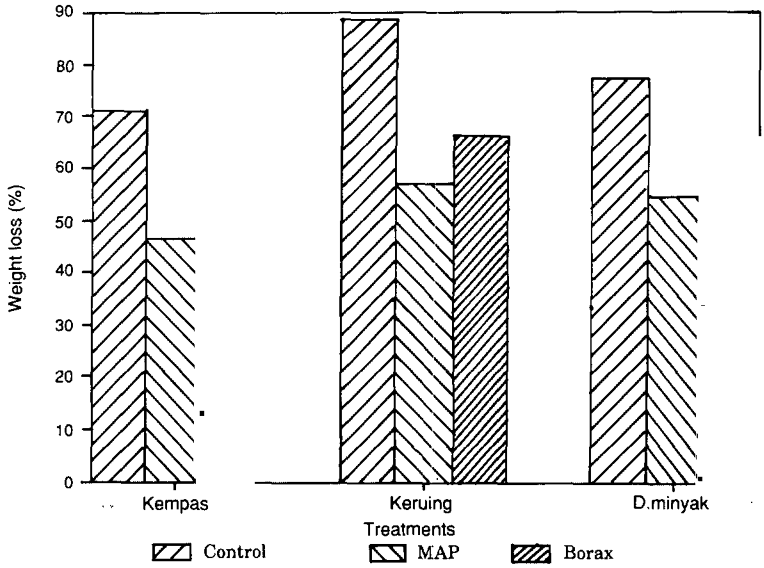

Figure 2. Weight loss after fire propagation test

The percentage loss in weight of untreated hardwoods was influenced by the specific gravity whereby the denser the timber the lower the percentage weight loss (Abdul Rashid 1982a). In the present study untreated keruing with an average basic density of 545 $kg\ m^3$ showed greater weight loss than kempas having basic densities of 759 $kg\ m^3$. Besides density, the permeability of timber might also affect weight loss. Large vessel elements as in kempas facilitate movement of volatile gases and allow 'conduction' of heat into core portion of burning wood for rapid burning. Greater weight loss in the keruing might also be attributed to the presence of naturally occuring resins in the species that could increase rapid burning thus leading to greater thermal degradation of cellulosic materials.

The presence of resinous material in keruing probably increases the amount of levoglucosan during burning of cellulose to yield more flammable products to support flaming. Levoglucosan (1,6-anhydroglucopyranose) or tar is the pyrolysis product of cellulose and it is regarded as highly flammable (Holmes & Shaw 1961, Byrnes *et al.* 1966, Tsuchiya & Sumi 1970).

The weight losses of treated timbers were lower than those of the controls. However, the weight losses of timbers treated with MAP were less than with borax. MAP improved the weight loss by about 30% in keruing and by 20% in kempas and damar minyak. Borax improved the weight loss by about 20, 10 and 5% in keruing, damar minyak and kempas respectively. Hirata *et al.* (1981) found that MAP improved weight loss of meranti (*Shorea* sp.) by about 20% at a chemical loading of 40*kg* m^3. Similarly, sodium borate when combined with boric acid improved weight loss of meranti by not more than 20% at loading of 40*kg* m^3 (Hirata *et al.* 1981).

Lyons (1970) found that fire retardancy in cellulose was improved by incorporating one or more of the following elements: P, N, Sb, Cl, Br or B, and P was most effective than any other elements taken alone. In the present study MAP was found to be more effective than borax.

The effectiveness of MAP and borax is in agreement with the results on weight loss by thermogravimetric analysis (TGA) and differential thermal analysis (DTA) of wood, cellulose, hemicellulose and lignin treated with similar sets of chemicals (Holmes & Shaw 1961, Browne & Tang 1963, Byrne *et al.* 1966).

Fire-propagation index

The fire-propagation index I derived from the fire-propagation test provides a comparative measure of the contribution of material to the heat build-up and also the fire-spread within a compartment (Rogowski 1970). The index may range in descending order-of-merit from 0 - 100 and from this index, material can be assessed into Class 0 rating which is the best rating for lining materials. This class is attained when propagation sub-index i_1 and propagation index, I, are less than or equal to 6 and 12 respectively (Anonymous 1968).

The results of fire-propagation index of untreated and treated timbers are shown in Table 1. The propagation index I of each untreated timbers was 35 to 45 depending on the species. The results correspond to works carried out by Hall and Dell (1969a, 1969b), Rogowski (1970) and Abdul Rashid (1982b). The propagation indices of treated timbers were 10 to 25. Similar results were obtained on chipboards, hardboards and plywood treated with similar chemicals (Rogowski 1970, Abdul Rashid 1984, 1987).

The propagation index of 10 to 25 could satisfy surface spread of flame rating of Class 1 (Malhotra 1979). Therefore treatment with MAP and borax to a loading of 40 to 50 *kg* m^3 improved the surface spread of flame classification to Class 1 rating for kempas, keruing and damar minyak. In fact in this study it appeared that Class 0 was achieved by damar minyak when treated with MAP at chemical loading of 75 *kg* m^3 but not with borax treatment at a loading of 80 *kg* m^3.

Generally, untreated timbers having basic density greater than 400 *kg* m^3 are grouped into Class 2 or 3 ratings in terms of surface spread of flame classification (Hall 1975).

The results can also be evaluated in terms of the reduction of heat release expressed as a percentage of propagation index I of the controls (Table 1). As shown in the table both chemicals showed reduction in the amount of heat release from 40 to 75 % depending on the timbers. The maximum reductions by MAP and borax were found in damar minyak at 74% and 48% respectively. Greater reduction in this species was probably due to relatively greater chemical loading compared to the other two species. MAP performed better than borax in kempas, but in keruing the performance was similar for both chemicals. This relative reduction in performance of MAP in this species could be due to interference with the formation of levoglucosan by the naturally occuring resin in keruing. This suggestion needs to be substantiated in future studies.

Table 1. Fire-propagation indices of untreated and treated timbers tested in accordance to B.S. 476, Part 6:1968

Species	Treatment	Loading *kg m⁻³*	S.D.	Performance sub-indices i_1	i_2	i_3	Propagation index I	Reduction in heat release*%
	Control	-	-	9.6	21.2	4.6	35.5	0
Kempas	MAP	51.0	2.2	1.0	12.2	4.2	17.4	50.9
	Borax	52.0	1.3	2.2	13.9	4.1	20.2	43.1
	Control	-	-	17.2	21.3	3.5	41.9	0
Keruing	MAP	41.0	5.8	4.1	15.7	4.3	24.2	42.3
	Borax	45.0	4.7	3.8	16.6	4.1	24.6	41.4
	Control	-	-	19.2	21.1	3.5	43.8	0
Damar minyak	MAP	75.0	7.4	1.1	6.9	3.5	11.5	73.7
	Borax	81.0	5.3	5.0	13.6	4.2	22.9	47.7

MAP - monoammonium phosphate
S.D. - standard deviation
* - expressed as percentage of propagation index I of controls.

Basically, fire-retardants including MAP and borax lower flammability of the resultant cellulose derivative by reducing the formation of levoglucosan (Holmes & Shaw 1961, Byrnes *et al.* 1966, Tsuchiya & Sumi 1970). In this case it appears that resinous compounds might interfere in the formation of levoglucosan in MAP treated timber to a greater extent than in borax treated timber.

The propagation sub-indices i_1 of treated timbers were less than 6 (Table 1). Therefore the timbers were not easily ignited compared to untreated specimens. Based on the propagation sub-index i_1, timbers treated with MAP were more difficult to ignite than borax treated timbers.

Exhaust temperature-rise

The increases in the exhaust temperature with untreated and treated kempas, keruing and damar minyak are shown in Figures 3, 4 and 5 respectively.

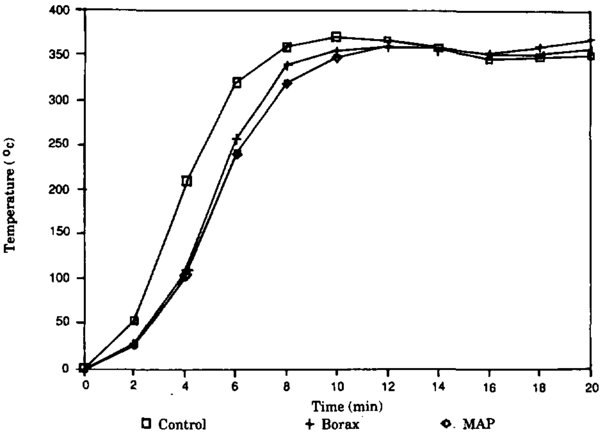

Figure 3. Temperature-time curve of untreated and treated kempas

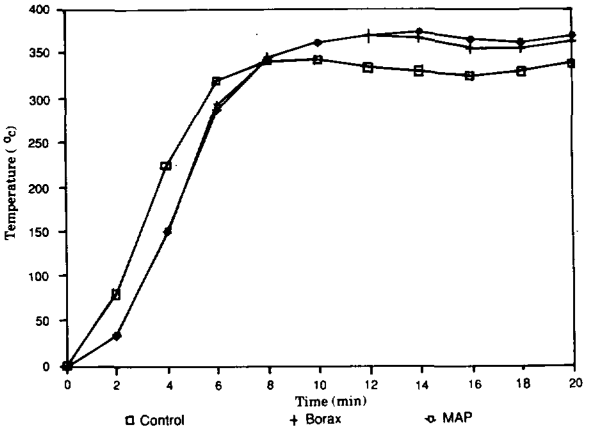

Figure 4. Temperature-time curve of untreated and treated keruing

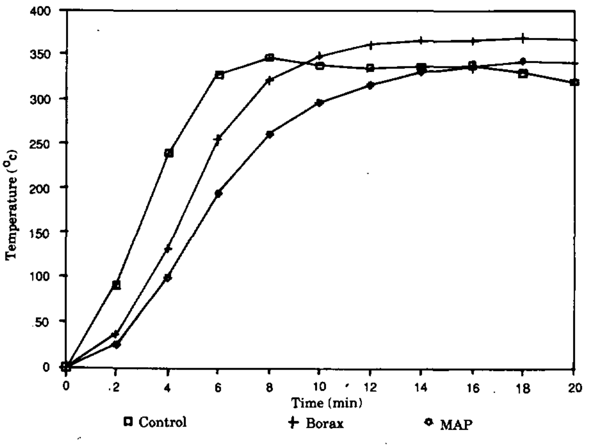

Figure 5. Temperature-time curve of untreated and treated damar minyak

The exhaust temperature-rises of treated specimens were lower than those of the controls for about 8 and 16 *min* depending on the species and chemicals. In kempas the temperature-rises for both chemicals were lower than that of the control for about 15 *min* (Figure 3) while for keruing they were delayed for about 8 *min* (Figure 4). Kempas performed better than keruing due to its relatively greater loading. In damar minyak the temperature-rise for borax and MAP was lower than that of the controls for about 9 and 16 *min* respectively (Figure 5).

The effects of borax and MAP in delaying the temperature-rise have also been noted when chipboard was treated with 20% of these chemicals by weight of dry chips (Abdul Rashid 1987). Similar results were obtained by Hirata *et al.* (1981) in meranti treated with the same chemicals at a loading of 40 $kg\ m^{-3}$.

The delay in the exhaust temperature-rise observed on treated specimens was due to the effects of phosphorus and boron in MAP and borax respectively, in inhibiting flaming and glowing. Phosphorus compounds are known to inhibit both flaming and glowing and boron is effective against flaming (Eickner 1966, Juneja 1972) and thus helps in delaying the temperature-rise during burning.

Heat test

Weight loss

The effects of heat on untreated and treated kempas, keruing and damar minyak at 250°*C*, 350°*C* and 550°*C* are shown in Figures 6, 7 and 8 respectively. The graphs were extrapolated from 0°*C* to 550°*C* in order to get the behavioural pattern in the loss in weight. As shown from the graphs the weight losses increased with each exposure temperature. This is because the burning rate increases with increasing combustion temperature (Mossoudi 1978).

The weight losses of MAP-treated timbers were higher than those of the controls at temperature below 300°*C* in all species. This shows that MAP increases volatiles liberation at temperature below 300°*C*. Knudson and Williamson (1971) found similar results with an intumescent fire-retardant containing MAP, urea and glucose. Zinc chloride and diammonium phosphate behave similarly at temperature below 350°*C* (Brown & Tang 1963). The weight losses of MAP and borax treated timbers were less than those of the controls when the temperature was above 300°*C* showing that both chemicals reduce volatiles liberation at temperature above 300°*C*. In fact borax started to reduce volatiles liberation even at temperature below 300°*C* in kempas and keruing as shown by smaller weight loss compared to the controls from 0°*C* to 300°*C*.

Liberation of smoke and moisture

Liberation of small quantities of water vapour was observed for exposure up to 15 *min* in specimens at 250°*C* and up to 10 *min* at 350°*C*. Much of the water vapour was probably derived from free and bound water.

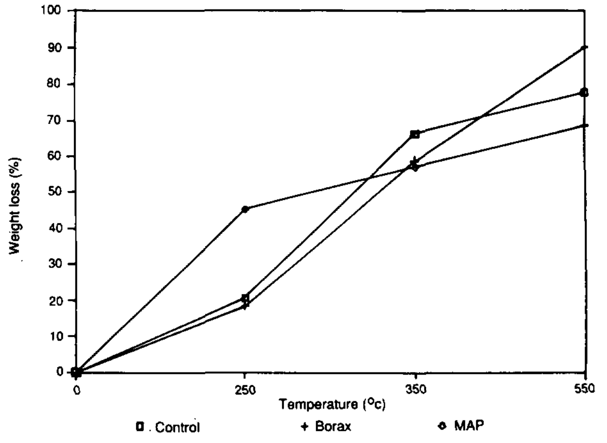

Figure 6. Effect of temperature on weight loss of untreated and treated kempas

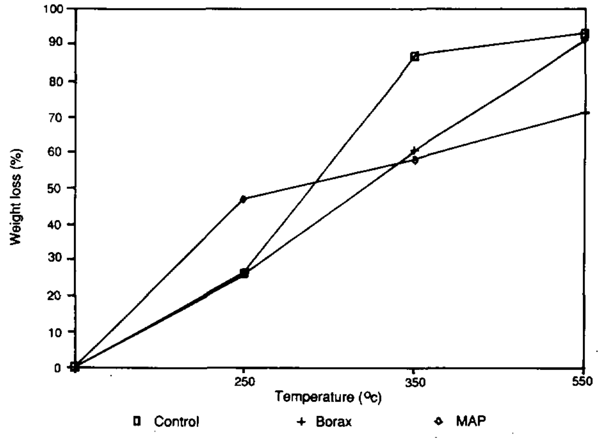

Figure 7. Effect of temperature on weight loss of untreated and treated keruing

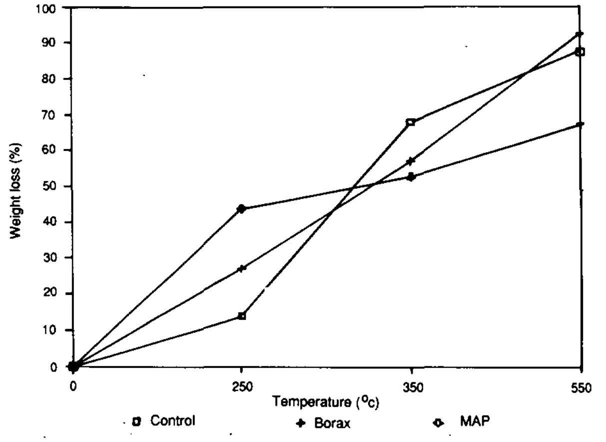

Figure 8. Effect of temperature on weight loss of untreated and treated damar minyak

No smoke liberation was noted at 250°*C*. At 350°*C* untreated specimens liberated more smoke than treated specimens. At 550°*C* no smoke liberation was noted after 10 *min* in untreated specimens and after 5 *min* in treated specimens. The times when no smoke is liberated correspond to the points where reduction in weight loss occurs (Knudson & Williamson 1971).

Some fire-retardants affect the amount of smoke evolution from timbers. This has been demonstrated by Siau *et al.* (1975) and Hirata *et al.* (1981).

Conclusions

The following conclusions can be drawn from the results of this study:

1. Kempas, keruing and damar minyak can be treated with monoammonium phosphate and borax to the required retention for fire retardancy using the concentration specified in the Malaysian Building By-Laws 1984 by pressure treatment process.
2. Monoammonium phosphate can reduce weight loss of untreated timbers by about 20% to 30% whereas borax can do so by less than 20% only.
3. Both monoammonium phosphate and borax treatments at a chemical loading of 40 - 50 $kg\ m^3$ improve the fire-propagation performance of timber. The performance index values are comparable to those in the Class 1 rating for surface spread of flame.
4. Class 0 rating, the best flame spread treatment is demonstrated by damar minyak treated with monoammonium phosphate when the chemical loading is about 75 $kg\ m^3$.
5. Borax treatment fails to improve the flame spread to Class 0 even at a chemical loading of more than 75 $kg\ m^3$.
6. Presence of resinous material as in keruing affects the fire-propagation performance of monoammonium phosphate probably by interferring with the formation of levoglucosan.
7. Monoammonium phosphate can prolong the time at which timber is ignited and this is useful in lowering the surface flammability of the timber.
8. Monoammonium phosphate and borax behave differently at various temperature ranges and this is important in understanding their role in the formulation of fire-retardant chemical.

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