IMPREGNATION OF RUBBERWOOD AND OTHER MALAYSIAN TIMBERS WITH COPPER NAPHTHENATE AND TRIMETHYL BORATE

S Muhammed[1, 2], OC Musgrave[1,*] & JA Petty[3]

[1]*Chemistry Department, The University of Aberdeen, Old Aberdeen AB24 3UE, UK*
[2]*Universiti Teknologi MARA, 26400 Jengka, Pahang Darul Makmur, Malaysia*
[3]*Forestry Department, The University of Aberdeen, Old Aberdeen AB24 3UE, UK*

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MUHAMMED S, MUSGRAVE OC & PETTY JA. 2009. Impregnation of rubberwood and other Malaysian timbers with copper naphthenate and trimethyl borate. The objective of this study was to determine the distribution of copper naphthenate and trimethyl borate within four Malaysian woods, namely, rubberwood, kempas, keruing and dark red meranti. The impregnation of end-sealed blocks of rubberwood and kempas by copper naphthenate in white spirit was much more rapid, and the fractions of the void volumes filled were greater than was observed with keruing and dark red meranti. After oven-drying all four species treated showed higher concentrations of copper in the peripheral regions of the blocks compared with the centre. Kempas contained an extractive which formed a brown copper complex. Gaseous trimethyl borate $(MeO)_3B$ and its methanol azeotrope $(MeO)_3B,MeOH$ were absorbed by end-sealed rubberwood blocks having moisture contents below the fibre saturation point with the formation, initially, of boric acid and methanol. After oven-drying the boric acid equivalent (BAE) of the peripheral region of the blocks was greater than that of the central portion and the gaseous azeotrope had higher values than the pure ester. The use of the liquid azeotrope resulted in higher BAE values. After being oven-dried the weight increase of treated blocks suggested that the final impregnation product was not boric acid itself but a mixture of esters formed from boric acid and the polyhydroxy components of the wood. Only part of the water present in the wood appeared to react with trimethyl borate, the rest presumably being sequestered in the microvoids of the cell walls.

Keywords: Dark red meranti, kempas, keruing, boric acid equivalent, trimethyl borate-methanol azeotrope

MUHAMMED S, MUSGRAVE OC & PETTY JA. 2009. Impregnasi kayu getah dan beberapa kayu lain dari Malaysia dengan kuprum naftenat dan trimetil borat. Tujuan kajian ini adalah untuk menentukan taburan pengawet kuprum naftenat dan trimetil borat dalam empat spesies kayu dari Malaysia iaitu kayu getah, kempas, keruing dan *Shorea* sp. Blok kayu getah dan kempas yang dipateri hujung-hujungnya menunjukkan kadar impregnasi oleh kuprum naftenat dalam spirit putih yang lebih cepat berbanding keruing dan *Shorea* sp. Pecahan isi padu lompang yang terisi juga lebih tinggi bagi kedua-duanya. Selepas dikering dalam ketuhar, keempat-empat spesies menunjukkan kepekatan kuprum yang lebih tinggi di bahagian pinggir blok berbanding dengan bahagian tengah. Kempas mempunyai hasil ekstraktif yang membentuk kompleks kuprum berwarna perang. Trimetil borat bergas $(MeO)_3B$ dan azeotrop metanol $(MeO)_3B,MeOH$ diserap oleh blok kayu getah yang dipateri hujungnya dan mempunyai kandungan lembapan di bawah tahap tepu gentian dan pada awalnya membentuk asid borik serta metanol. Selepas dikering dalam ketuhar, kandungan asid borik setara (BAE) bahagian pinggir lebih tinggi daripada bahagian tengah dan sebahagian besar daripadanya dihasilkan oleh azeotrop bergas berbanding ester tulen. Penggunaan azeotrop cecair menghasilkan nilai BAE yang lebih tinggi. Selepas dikering dalam ketuhar, peningkatan berat blok yang dirawat mencadangkan bahawa hasil impregnasi akhir bukanlah asid borik tetapi sebaliknya merupakan campuran ester yang terbentuk daripada asid borik dan komponen polihidroksi kayu. Nampaknya tidak semua air dalam kayu bertindak balas dengan trimetil borat; sebahagiannya diandaikan telah tersimpan dalam lompang mikro di dinding sel.

INTRODUCTION

The potential uses of rubberwood (Hong & Sim 1999) are limited by its vulnerability to fungal and insect attack and, in tropical regions particularly, to termites. Copper-containing preservatives such as copper-chrome-arsenate (CCA) have been used successfully in protecting the wood but such treatment requires aqueous conditions which cause problems of swelling and distortion (Jusoh & Kamdem 2001, Remadevi & Raja 2004). In addition compositions containing chromium(VI) and arsenic are becoming less attractive because of their toxicity to higher

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*Author for correspondence. E-mail: o.c.musgrave@abdn.ac.uk animals and man. The use of less toxic organo-soluble preservatives such as copper naphthenate overcomes these difficulties and, despite being more expensive, provides a suitable alternative to these preservatives (Freeman & McIntyre 2008).

The attractive colour and texture of rubberwood make it very popular in furniture manufacturing (Lew & Sim 1982). This is the reason why coloured preservatives such as copper compounds are considered inappropriate for use with rubberwood and instead colourless materials of lower toxicity such as boron compounds have been employed as alternative preservatives (Tan 1991).

Accurate knowledge of the distribution of preservatives within treated wood is important but, unfortunately, very few quantitative data are available for rubberwood. In a study that utilized aqueous solutions of borax and boric acid, it was reported that, after being dried, the innermost regions of treated wood had much lower boric acid equivalent (BAE) concentrations than had the outermost layers (Salamah & Dahlan 2008). A similar observation was reported with rattan (Zaidon *et al.* 1998).

The aim of the present study was to examine the distribution of preservatives in rubberwood using non-aqueous conditions. We investigated impregnation with copper naphthenate dissolved in white spirit and measured the concentrations of copper throughout the wood. For comparative purposes three other important Malaysian timbers, namely, kempas, keruing and dark red meranti were treated similarly.

Trimethyl borate, either in the liquid or in the gaseous state, has the advantage of not requiring a solvent for its application. It has been used in the impregnation of woods such as *Pinus radiata* and *Eucalyptus regnans* (Ahmed *et al.* 2004, Evans *et al.* 1997a, b, c, Przewloka *et al.* 2007) and wood-based products including strand- and fibre-boards (Murphy & Turner 1989, Turner & Murphy 1995, 1998, Joseph 2000, Barnes & Murphy 2006). We investigated the impregnation of rubberwood by pure ester and its methanol azeotrope and determined the resulting BAE concentrations throughout the wood.

MATERIALS AND METHODS

Preparation of wood samples

Square cross-section blocks (typically 35 × 35 × 150 mm or 50 × 50 × 150 mm) with the grain parallel to the length were cut from air-dried samples of rubberwood (*Hevea brasiliensis*), kempas (*Koompassia malacensis*), keruing (*Dipterocarpus* sp.) and dark red meranti (*Shorea* sp.) supplied by the Forest Research Institute Malaysia (FRIM), Kepong, Malaysia. Each block was sanded until smooth and the end-faces were sealed with plastic padding (AB Hisingeplast, Goteborg, Sweden) so that liquid penetration could take place only through the radial and tangential surfaces of the block and not via the more highly permeable transverse surfaces. The blocks were oven-dried to constant weight at 105 °C and stored in closed containers out of contact with laboratory air. Some of the rubberwood blocks prepared as above were then allowed to equilibrate either with laboratory air at 20 °C (LA samples) or with air saturated with water vapour at 20 °C in a closed container (SA samples) after which they were reweighed and the moisture contents (%) calculated. All measurements reported in Tables 1 and 2 were made in duplicate or triplicate.

Preparation and analysis of copper naphthenate solution

A commercial solution of copper naphthenate (~8% w/w copper) was diluted with white spirit to give a solution containing 1% w/w copper. One millilitre of this solution was further diluted with light petroleum to 100 ml. Aqueous HCl (1 M, 20 ml) was added to the diluted solution (10 ml) and the mixture was shaken by hand for 1.5 min. The organic layer was separated and the treatment with acid was repeated three times. The resulting four aqueous layers were combined, diluted to 100 ml with 1 M aqueous HCl and centrifuged (3000 rpm, 5 min) to remove any traces of white spirit. A portion (10 ml) of the solution was diluted with water to 100 ml. The copper concentration of the solution was determined by a Perkin-Elmer model 290B atomic absorption spectrophotometer at 324.8 nm using the method by Thompson and Reynolds (1978). Copper concentrations were determined from the linear calibration curve for the absorbance of standard solutions of copper chloride.

Treatment of wood samples with copper naphthenate solution

A block of rubberwood, oven-dried at 105 °C and end-sealed as described above, was placed in a glass vessel which was evacuated to 0.1 mm Hg for 10 min. A solution of copper naphthenate (1% w/w copper) in white spirit, prepared as above, was then immediately introduced into the vessel until it was completely filled. The weight of solution absorbed by the block was measured periodically (Petty 1975). After 400 min the rate of absorption was very low and the block was removed from the vessel, wiped dry and immediately weighed prior to air-drying overnight at 20 °C followed by 50 °C for three days and finally oven-drying at 105 °C to constant weight. Blocks were kept in a closed container prior to the measurement of their copper content. Similar experiments were made using kempas, keruing and dark red meranti.

Measurement of fractional volumetric absorption F

The fraction of the theoretical void space in a wood which is filled with non-swelling liquid is given by equation (Siau 1984):

$$F = \frac{W}{d_L V(1 - d/1.46)}$$

where

W = weight of liquid of density d_L
V = volume of the wood sample
d = dry density of the wood sample
1.46 g cm^{-3} = density assumed for dry cell-wall material

In the present work the densities of the copper naphthenate solution, rubberwood, kempas, keruing and dark red meranti were 0.898, 0.672, 0.834, 0,801 and 0.518 g cm^{-3} respectively.

Distribution of copper in impregnated wood samples

The middle section of part of the rubberwood block was cut into five numbered strips as shown in Figure 1 and these were ground separately to pass a 40-mesh screen. The resulting wood meal (0.5 g) was heated with 5 M aqueous HCl (10 ml) at 55 °C in a polyethylene flask with intermittent swirling for 30 min. After being cooled the mixture was diluted with water to 50 ml and centrifuged at 3000 rpm for 5 min to remove all insoluble material. Part of the clear solution (10 ml) was diluted to 100 ml and used in the determination of copper content by atomic absorbance spectrophotometry as previously described.

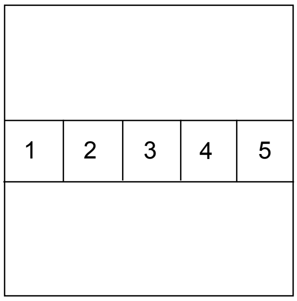

Figure 1 Wood block portions for copper analysis

Formation of a copper complex from a kempas extractive

Air-dried kempas wood (1165 g) was ground to 40 mesh and Soxhlet-extracted for 16 hours with light petroleum (boiling point 60–80 °C), chloroform and finally 95% ethanol. Evaporation of the ethanolic solution under reduced pressure produced a crude extractive (23.94 g, 2.05%) and a portion of this (0.5 g) was boiled with methanol (100 ml) and then filtered. The addition of 15 ml of 5% w/v methanolic solution of copper acetate gave an immediate brown precipitate. The mixture was then centrifuged (3000 rpm, 5 min) after which the supernatant liquid was decanted off. More methanol was added to the precipitate, the mixture was shaken and the liquid again decanted. This procedure was repeated until the liquid was completely colourless ensuring the removal of unreacted copper acetate. Finally the brown copper complex was dried at 0.1 mm Hg and 100 °C before being analysed for copper by the procedure previously described.

Preparation of boron compounds

Commercial trimethyl borate [$(MeO)_3B$, boiling point 65 °C] was purified by redistillation under anhydrous conditions. The trimethyl borate-methanol azeotrope, boiling point 55 °C, was prepared from boric oxide and methanol (Schlesinger *et al.* 1953). The azeotrope, which contains 75.5% w/w trimethyl borate and 24.5% w/w methanol corresponding approximately to the formula $(MeO)_3B$,MeOH, was easier to prepare than was trimethyl borate itself.

Treatment of rubberwood with trimethyl borate and with methanol azeotrope in gas phase

The end-sealed wood block which had been conditioned to the appropriate moisture content was placed in a glass vessel and heated to 65 °C. The vessel was evacuated to 0.1 mm Hg pressure and after 10 min gaseous trimethyl borate was introduced until the internal pressure reached 760 mm Hg. After 20 min, the vessel was again evacuated to remove excess trimethyl borate and air was allowed in. The block was air-dried overnight at 20 °C and then oven-dried to constant weight at 105 °C. The difference in weight of the oven-dried block before and after the methyl borate treatment gave the amount of boron-containing products retained in the wood.

The procedure was repeated using methanol azeotrope with an impregnation temperature of 55 °C.

Treatment of rubberwood with liquid trimethyl borate-methanol azeotrope

The moisture-conditioned rubberwood block was placed in a glass vessel which was evacuated to 0.1 mm Hg at 20 °C. After 10 min, liquid azeotrope was introduced until the vessel was full and the wood block was completely immersed. Dry air was admitted after 2 hours and excess of azeotrope was siphoned out. The block was removed and air-dried overnight at 20 °C before being oven-dried to constant weight at 105 °C. It was then stored in a desiccator prior to determination of its BAE content.

Determination of boric acid equivalent in treated wood

Sealed end-sections were sawn off and the resulting block was cut longitudinally into 25 numbered strips. These were grouped together into six types according to their positions in the original block (see Figure 2) and ground separately to 40 mesh. Each wood meal sample (1 g) was heated at 65 °C for 30 min in a polyethylene vessel containing aqueous 5 M NaOH (10 ml) and water (20 ml) and occasionally swirled. The mixture was cooled, diluted to 50 ml with water, shaken repeatedly and allowed to settle. After being centrifuged at 3000 rpm for 5 min, a portion (0.5 ml) of the clear solution was mixed with 3 ml of a solution of curcumin (0.12 g) in acetic acid (100 ml), followed after 5 min by a mixture of concentrated sulphuric acid and acetic acid (1:1 v/v; 3 ml). The mixture was kept at room temperature for 20 min before being diluted to 100 ml with aqueous acetone (1:1 v/v). The intense red solution of the curcumin-borate complex was diluted appropriately to give a suitable BAE concentration (20–60 µg/100 ml). Absorbance of the solution was measured at 555 nm using a Perkin-Elmer model 402 UV/VIS spectrophotometer. The BAE concentration was determined by comparison with standard boric acid/curcumin solutions which exhibit a linear relationship between absorbance and boric acid concentration. The total theoretical weight of boric acid present in a block was calculated using the weight of the block after the borate treatment and the %BAE content of the 25 component strips (grouped together in portions 1 – 6).

1	2	3	2	1
2	4	5	4	2
3	5	6	5	3
2	4	5	4	2
1	2	3	2	1

Figure 2 Rubberwood block portions for boric acid equivalent (BAE) analysis

RESULTS AND DISCUSSION

Treatment of the woods with copper naphthenate solution

Table 1 outlines the data for the *in vacuo* application of the solution of copper naphthenate in white spirit to the four types of woods. Kempas (330 g dm^{-3}) and rubberwood (321 g dm^{-3}) absorbed much more solution than did keruing (53 g dm^{-3}) and dark red meranti (42 g dm^{-3}). The fractions of the void volumes filled followed the same pattern, as expected, i.e. kempas (86%) and rubberwood (66%) had much larger values than had keruing (13%) and dark red meranti (7%).

The solution uptakes of the four wood species are summarized in Figure 3 where the fractional volumetric absorption F is plotted against (time/min)$^{1/2}$ (Petty 1975). Kempas and rubberwood took up the solution rapidly during the first

36 min and then more slowly up to 400 min. On the other hand, keruing and dark red meranti underwent slow initial impregnation followed by even slower absorption.

Although rubberwood and kempas absorbed much more copper naphthenate solution than did keruing and dark red meranti the distribution of copper in all dried wood blocks followed the same general pattern—the greatest concentrations being found in the outermost portions of the blocks (Figure 4). The concentrations in the central portions were low and only rubberwood had > 0.2% w/w copper, the toxic limit quoted for wood-destroying insects (Bulman 1961). A similar concentration protects southern pine effectively against termite attack (Grace *et al.* 1993).

After being treated with copper naphthenate solution rubberwood developed the expected pale green colour associated with the presence of copper. In contrast kempas became dark brown and an ethanol-soluble extractive obtained from the untreated wood formed an insoluble brown copper complex which contained 16.5% copper. The formation of such a complex would be expected to affect the penetration of kempas by a copper-containing preservative.

Treatment of rubberwood with trimethyl borate

Trimethyl borate is hydrolyzed very rapidly by both liquid water and water vapour (Steinberg & Hunter 1957) with the formation of methanol and

Table 1 Treatment of Malaysian timbers with copper naphthenate (1% w/w copper) in white spirit

Timbers	Weight of oven-dried block before treatment (g)	Volume of oven-dried block before treatment (cm^3)	Weight of block after treatment (g)	Solution uptake (kg m^{-3})	Copper retention (kg m^{-3})	Void volume filled (%)
Rubberwood	114.86 (0.70)	170.83 (1.53)	169.81 (0.75)	321.66 (6.71)	3.22 (0.07)	66.38 (1.07)
Kempas	240.05 (2.36)	287.88 (6.50)	335.03 (2.39)	329.93 (11.21)	3.30 (0.11)	85.82 (4.23)
Keruing	228.70 (1.76)	285.69 (1.21)	243.88 (1.63)	53.15 (1.94)	0.53 (0.02)	13.10 (0.38)
Dark red meranti	156.74 (3.54)	302.58 (0.22)	169.30 (2.83)	41.52 (2.34)	0.42 (0.02)	7.16 (0.32)

Standard deviations are in parentheses.

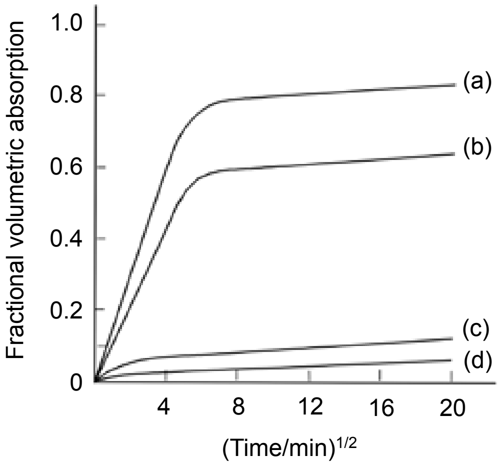

Figure 3 Mean absorption variation of solution of copper naphthenate in white spirit by (a) kempas, (b) rubberwood, (c) keruing, and (d) dark red meranti

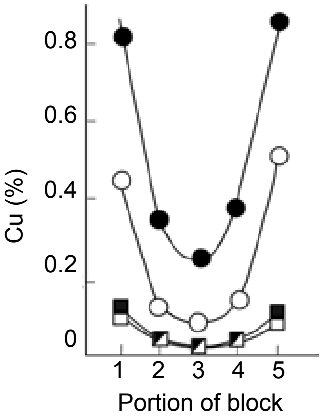

Figure 4 Distribution of copper in woods after treatment with a solution of copper naphthenate (1% w/w Cu) in white spirit; ● rubberwood ○ kempas ■ keruing □ dark red meranti

the relatively involatile boric acid. Rubberwood samples with moisture content of 9.76% (SA samples) when treated with gaseous trimethyl borate showed greater increases in oven-dried weights and higher values of BAE concentrations in their outermost regions (portions 1–3) compared with LA samples which had lower moisture content of 5.17% (Table 2). In both cases BAE contents of the inner regions (portions 4–6) were very low. The treatment of similar blocks with gaseous trimethyl borate-methanol azeotrope resulted in significant increases in BAE contents of the corresponding regions, particularly those of the inner group (portions 4–6). Blocks with higher moisture contents again showed higher BAE values in the outer regions. It appeared that the azeotrope, despite having a trimethyl borate content of only 76.4%, diffused more readily than pure ester. It is suggested that the molecule of methanol in the azeotrope effectively reduces the rate of reaction of trimethyl borate with water via a mass action effect, thus, permitting more efficient penetration into the wood.

For comparison the interaction of rubberwood having a high (16.3%) moisture content with liquid trimethyl borate-methanol azeotrope was examined. Substantial increases in BAE contents of the outermost parts of the blocks (portions 1–4) were observed (Table 2) but the innermost region (portions 5 and 6) showed relatively low values. Although the gaseous azeotrope might be expected to penetrate the wood more easily than the liquid, the much higher concentration of the latter ensured that a greater weight of trimethyl borate entered the wood.

The nature of BAE in treated wood

The total theoretical weight of boric acid present in the blocks was in each case much greater than the measured increase in weight of the blocks. This suggested that the boric acid formed had reacted with polyhydroxy components of the wood such as lignin, cellulose and hemicelluloses. Simple diols can react with boric acid to give cyclic esters (Steinberg 1964) types A, B, C and D as shown in Figure 5 with the production of two, three, five or six molecules of water respectively. The formation of type A ester would be accompanied by the theoretical gain of a molecule of boric acid (molecular weight 61.84) and the loss of two molecules of water (molecular weight 18.015). The percentage increase in weight on the esterification of the diol

Table 2 Analytical data for the treatment of rubberwood blocks with gaseous trimethyl borate, gaseous trimethyl borate-methanol azeotrope and liquid azeotrope

	With gaseous trimethyl borate		With gaseous trimethyl borate-methanol azeotrope		With liquid trimethyl borate-methanol azeotrope
	SA samples*	LA samples*	SA samples	LA samples	SA samples
Moisture content at equilibrium (%)	9.76 (2.21)	5.17 (0.02)	13.03 (0.38)	4.09 (0.23)	16.13 (0.60)
Weight increase after treatment (g)	0.355 (0.04)	0.269 (0.05)	0.944 (0.12)	0.984 (0.47)	3.428 (0.45)
Boron uptake (%BAE) of block portions**					
1	4.51 (0.52)	1.91 (0.08)	5.02 (0.68)	2.64 (0.08)	12.80 (1.27)
2	3.50 (1.20)	1.73 (0.06)	4.12 (0.32)	2.51 (0.03)	11.52 (1.23)
3	3.33 (1.21)	1.65 (0.10)	3.98 (0.41)	2.41 (0.01)	11.08 (0.79)
4	0.18 (0.02)	0.55 (0.49)	1.39 (0.15)	1.27 (0.09)	2.79 (0.66)
5	0.08 (0.06)	0.40 (0.29)	1.02 (0.16)	1.15 (0.19)	1.74 (0.27)
6	0.03 (0.01)	0.16 (0.20)	0.28 (0.09)	0.99 (0.14)	0.93 (0.28)

*SA samples were equilibrated using air saturated with water vapour at 20 °C; LA samples were equilibrated with laboratory air at 20 °C; **portions of the blocks were numbered as shown in Figure 2; standard deviations are in parentheses.

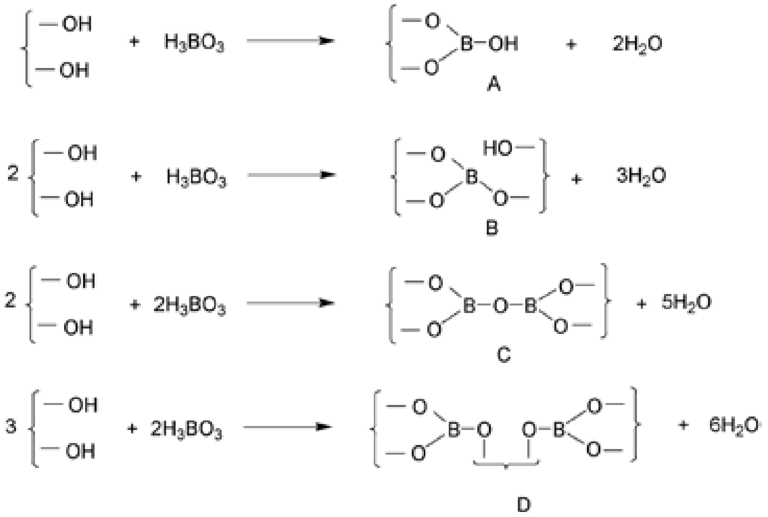

Figure 5 Types of borate ester formed by reactions between boric acid and diols

is therefore: [(61.84 – 2 × 18.015)/61.84] × 100 = 41.74% of the weight of boric acid used. Similar calculations for the formation of esters of types B, C and D would lead to increases in weights of 12.60, 27.17 and 12.60% respectively.

The total boric acid content calculated for the 25 SA sample strips treated with gaseous trimethyl borate was 2.307 g but the increase in weight of the block was only 0.355 g, i.e. 15.4% (Table 2), suggesting that an ester of type B or D was the main product. The related LA sample had a calculated total boric acid content of 1.249 g but an increase in weight of only 0.269 g (21.5%), corresponding to the formation of a mixture of type B and C or D and C esters. The interaction of the azeotrope and SA samples gave a calculated total boric acid content of 2.941 g and a net increase in weight of 0.944 g (32.1%), corresponding to the presence of a type C ester. The related LA samples with a total BAE content of 1.939 g showed a weight gain of 0.984 g (50.8%), suggesting that a mixture of a type A ester with some boric acid was present. Finally, with the liquid azeotrope, the calculated total boric acid content was 7.805 g and the net increase in weight was 3.428 g (43.9%), a value close to that (41.8%) required for the formation of type A ester. The above figures, although only approximates, indicated that most of the BAE was present in the form of borate esters in the oven-dried wood. It is possible that direct transesterification could occur between the wood components and trimethyl borate with the elimination of methanol; the overall result would, of course, be the same. Such borate esters are hydrolyzed by water (Steinberg 1964) and leaching of boric acid from such treated wood would be expected under wet conditions. Thus, impregnated timbers would be unsuitable for outdoor use.

The theoretical weights of boric acid formed in the blocks did not account for all the moisture present in the wood samples. For example in the treatment of SA samples with trimethyl borate-methanol azeotrope (Table 2) the theoretical weight of boric acid in the whole block was 7.805 g; its formation from the ester required 6.821 g water. Despite the presence of a considerable excess of trimethyl borate much (10.672 g) of the original weight of water (17.493 g) present in the wood failed to react. It would seem likely that the unreacted water was present in microvoids of cell walls of the wood. While small water molecules can penetrate microvoids easily (Siau 1984) the much more bulky trimethyl borate molecules are apparently unable to do so.

CONCLUSIONS

Rubberwood absorbed 1% w/w copper naphthenate solution rapidly, as did kempas. In both cases, a large fraction of the available voids were filled by the solution. In contrast, both keruing and dark red meranti absorbed the solution slowly and inefficiently. Of the four woods, only rubberwood had a copper concentration in the central portion which was above the toxic limit (0.2% w/w) for wood-destroying insects. The central portion concentrations measured for kempas, keruing and dark red meranti were significantly lower (Figure 4).

Rubberwood containing water below the fibre saturation point absorbed gaseous trimethyl borate or its gaseous methanol azeotrope. After being oven-dried it contained boric esters formed from the polyhydroxy components of the wood. The use of the liquid azeotrope gave improved BAE content (Table 2), with concentrations even at the centre of the blocks being in the range known to be toxic to termites.

The various impregnations of rubberwood were effected with a gaseous and a liquid borate, and with a solution of a copper salt in a hydrocarbon. Despite the widely differing conditions the relative concentrations of the preservative species in the various portions of the dried blocks showed strong similarities, all resulting in U-shaped distributions as exemplified in Figure 4.

What are the practical implications of these studies? To prevent termite attack on rubberwood, copper naphthenate solution should contain at least 1% w/w copper, preferably more. However, kempas, keruing and dark red meranti would not be protected under these conditions. The protection of rubberwood could also be achieved by the use of liquid trimethyl borate-methanol azeotrope.

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