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The Behaviour of Boron Compounds in Treated Rattan when Dehydrated at High Temperatures

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ABSTRAK

Sifat kompaun boron dan kompaun boron di dalam batang rotan manau (Calamus manan Miq.) yang telah dirawat dan diikuti dengan pengeringan pada suhu tinggi diselidiki. Apabila boraks dan asid borik dicampurkan terjadi natrium 1-5 borat. Selepas pengeringan pada suhu 100±3, 50±1, atau < 0°C, peratus berat hilang dari campuran menurut pengukuran dan perkiraan (seperti dalam kurungan) adalah masing-masing 40.34 (40.10), 39.31 (36.39) dan 29.43% (30.11%). Nilai penurunan yang diukur melalui percubaan digunakan untuk menganggarkan bahan larut yang terkandung di dalam sampel rotan yang telah dirawat menggunakan campuran asid borik dan boraks dan dikeringkan. Penurunan berat campuran yang berlebihan di dalam rotan selepas dikeringkan pada suhu tinggi berkemungkinan diakibatkan oleh 1. peruapan komponen rotan yang berat molekulnya rendah 2. peruapan asid borik di dalam wap air semasa pengeringan dan 3. Kehilangan cecair pengawet secara mekanikal di awal peringkat pengeringan. Untuk menjamin penahanan persamaan asid borik (BAE) yang optimum, rawatan rotan atau bahan berselulosa lain yang melibatkan asid borik dalam larutan berair, pengeringan menggunakan suhu tinggi perlu dihindari

ABSTRACT

The behaviour of boron compounds per se and boron compounds in treated stem of rotan manau (Calamus manan Miq.) dried at elevated temperatures wasstudied. When borax and boric acid are mixed, sodium 1:5 borate is formed. After drying at 100±30, 50±10, or <0°C, the measured and the calculated (in parentheses) percentage weight losses from the mixtures were 40.34 (40.10), 39.31 (36.39) and 29.43% (30.11%), respectively. The experimentally measured values were used to estimate the amount of solute retained in the rattan samples which had been treated with such a boric acid-borax mixture and then dried. The further weight losses of the mixture in treated rattan dried at higher temperatures are presumably attributable to: 1. the volatilization of low molecular weight rattan components; 2. the volatilization of boric acid in water vapour during drying; and 3. the mechanical loss of permeating liquid during the early stage of drying. Treatments of rattan or other cellulosic material which involve boric acid in aqueous solution should avoid high temperature drying in order to ensure optimum retention of boric acid equivalent (BAE).

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INTRODUCTION

Rattans can be treated with boron compounds to enhance their durability against pests. Zaidon (1995) reported that by using vacuum-pressure, thermal and diffusion processes boron compounds can be successfully impregnated into the rattan stems. A standard retention requirement of boric acid equivalent (BAE, % w/w) and a complete penetration of BAE were achieved in the rattans when treated by these processes. According to Zaidon (1995), a significant reduction of BAE in the treated rattan was noted when the materials were dried at elevated temperatures following treatments. This decrement ranged from 10.93% for *rotan jelayang* (*Calamus peregrinus* Furtado) to 23.37% for *rotan manau* (*Calamus manan* Miq.) after they were dried at ca.100°C overnight. It is believed that the reduction of BAE in the treated rattans is probably through the loss of boric acid in the steam during drying.

Kininmonth (1963) also reported that during kiln drying at high temperature, boron compounds can be lost from the treated wood as a consequence of steam volatilization. Parkes (1967) stated that boron compounds, for instance orthoboric acid are appreciably volatile in steam. To understand the behaviour of the boron compounds in treated rattan dried at high temperatures, it was thought advisable to investigate the behaviour of the boron compound itself when subjected to dehydration at similar temperatures.

This paper reports the behaviour of boron compounds in treated rattan stems dried at high temperatures.

MATERIALS AND METHODS

Preparation of Materials

Three different boron solutions were prepared for this study: 5% (w/w) aqueous solutions of orthoboric acid (H_3BO_3, molecular weight (MW) 247.32 g), and of sodium tetraborate decahydrate ($Na_2B_4O_7.10H_2O$, MW 381.36 g), and a solution of boric acid (2.5 g) and borax (3.85 g) in water (100 ml), i.e. 6.35% (w/v) solute. The mixture of boric acid and borax was equivalent to 5% (w/v) boric acid equivalent (BAE) since

$$\begin{aligned} 4H_3BO_3 &= Na_2B_4O_7.10H_2O \\ \text{i.e. } (247.32\text{ g}) &= (381.36\text{ g}) \\ \text{or } (2.5\text{ g}) &= (3.85\text{ g}) \text{ molar equivalent.} \end{aligned}$$

The concentration of the mixture was converted from w/v to w/w by determining its density. The weight of 10 ml of the solution was 10.22 g, giving a density of 1.022 g/ml. The concentration of solute in the treatment solution on a w/w basis was (6.35/102.2) 6.21% solute, i.e. 2.44 g boric acid and 3.77 g borax in 100 g solution, corresponding to (5.0/102.2) 4.89% BAE.

Approximately 2 - 4 g of each of the solutions was placed in a preweighed flask (25 ml). A small funnel was used to cover the mouth of the flask to prevent any loss of solution by splashing at high temperature. Each of the solutions was evaporated for one day at 100±3°C, or for three days at 50±1°C in a conventional drying oven, or for one day at < 0°C in a freeze-dryer at 0.1 mm Hg. The calculated weights of solute from the aqueous solution before drying were compared with the measured weights of solute after drying. The concentration of the BAE in the solute after the drying processes was measured by spectrophotometric measurement of the rosocyanin complex formed between boric acid and curcumin (after Williams 1968).

Preparation of Samples

The material used in this study was mature stem of Calamus manan Miq. (*rotan manau*). Small samples of approximately 3 cm diam. x 3 cm long were cut from the internode section of the rattan stem. The samples were then oven-dried at 100±3°C to constant weight. The oven-dried samples were then treated with an aqueous solution of boric acid (2.44 g) and borax (3.77 g) in 100 g solution by a vacuum-pressure process. The rattan samples were placed in a glass vessel which was evacuated to 0.1 mm Hg for 10 min. The boron solution was introduced into the vessel and the samples fully immersed. The pressure was allowed to rise to atmospheric and the samples were kept in the liquid until no more solution was absorbed; this took approximately 6 h. The solution was then drained out of the vessel and the vessel with the treated samples was again evacuated for 10 min to remove surface liquid. The samples were then removed, wiped free of solution, and weighed.

The weights of the samples before and after treatment were used to calculate the weights of solute and of BAE contained in them. For ease of comparison, the amounts of solute and the

BAE retention were calculated as percentages of the oven-dried weight of the rattan. The initial BAE retention and the weight of retained solute were calculated using the following equations:

$$\text{BAE (\%)} = [(\text{Wat-Wo}) \times \text{conc. (w/w) of BAE} \times 100] / \text{Wo} \quad [1]$$

$$\text{Solute (\%)} = [(\text{Wat-Wo}) \times \text{conc. (w/w) of solute} \times 100] / \text{Wo} \quad [2]$$

where Wat is the weight of the treated sample after final vacuum treatment and Wo is the od-weight of the sample.

The treated samples were dried for one day at 100±3°C, or for three days at 50±1°C, or for one day at < 0°C in a freeze-dryer. After being dried the samples were again weighed (Wff), and the weight of retained solute was calculated:

$$\text{Retained solute (\%, w/w)} = [(\text{Wff - Wo}) \times 100] / \text{Wo}] \quad [3]$$

All the dried treated samples were analysed for the retention of BAE (% w/w) using UV-spectrophotometer (after Williams 1968).

Analysis of Boric Acid Equivalent (BAE)

The dried material was then ground separately into sawdust (40 mesh) using a Wiley mill. The boron-containing compounds were leached from the sawdust using aqueous sodium hydroxide solution. The weighed sawdust (0.1 g) was transferred into a boron-free soda lime boiling tube, after which 5 M sodium hydroxide solution (10 ml) and water (20 ml) were added. The boiling tube and its contents were heated in a water-bath at 65°C for 30 min with occasional swirling to ensure thorough mixing of the contents. The boiling tube was then removed from the water bath, water (15 ml) added and the tube swirled to mix the contents. It was then cooled to room temperature. The mixture from the boiling tube was transferred to a 50-ml graduated flask and diluted to 50 ml with water. The flask was shaken several times at 5-min intervals before being allowed to settle for 10 min.

A 0.5 ml aliquot of the leachate solution was transferred by pipette to a clean soda lime boiling tube. Three ml of curcumin solution, made by dissolving curcumin (0.12 g) in glacial acetic acid (100 ml), was added using a burette, followed by gentle swirling of the tube to mix the contents. After being allowed to stand for 5 min, a mixture of concentrated sulphuric acid and acetic acid (1:1, v/v; 3 ml) was added from a burette, the mixture shaken and allowed to stand in a water-bath at 30°C for 30 min. With the aid of a filter funnel, the mixture was then slowly poured with swirling into a 100-ml graduated flask containing acetone-water solution (1:1 v/v; 70 ml). Any of the mixture remaining in the boiling tube was washed into the graduated flask with acetone-water solution, the mixture diluted to 100 ml with the solution, and thoroughly shaken.

Optical density (absorbance) of the rosocyanin complex was measured against a reagent blank (prepared in a similar way with the omission of the sawdust) in 1-cm cells at 555 nm on a CECIL 6000 series UV spectropho-tometer (Cambridge, England). The calibration curve was produced by plotting the boric acid mass of standard solutions optical density (*Fig. 1*). The fitted calibration curve is represented by the equation below:

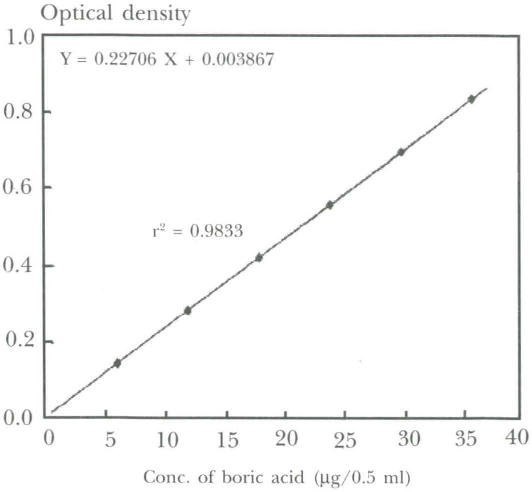

Fig. 1. Calibrationcurve for boric acid obtained form spectrophotometric measurements of the rosocyanin complex at 555 nm

$$Y = 0.003867 + 0.227056\ X \quad (r^2 = 0.983) \quad [4]$$

where Y is optical density measured for X µg of BAE in 0.5 ml of solution.

Having obtained the amount of BAE (µg/0.5 ml solution) from the calibration graph, the concentration of boric acid in the original sawdust from each layer can be calculated using the equation below (Suhaimi 1989):

$$\text{BAE}~(\%) = \frac{\text{conc. of } H_3BO_3~(\mu g/0.5~\text{ml}) \times 100}{\text{oven-dry weight of rattan sawdust}} \times 100 \qquad [5]$$

where 100 is the dilution factor.

RESULTS AND DISCUSSION

The Nature of Boron Behaviour

The weights of the boron compounds before and after the different types of drying processes are presented in Table 1, whilst the weights and concentrations of the boric acid-borax solution expressed as BAE before and after the drying processes are shown in Table 2.

From Table 1, the boric acid solution lost 50.0% (SE, 0.002%) of the solute when dried at 100±3°C, 4.74% (SE, 0.0001%) at 50±10C, and almost none when freeze-dried at < 0°C. Orthoboric acid itself when heated at ca. 100°C forms metaboric acid $(HOBO)_3$, usually referred to as "HBO_2," (Parkes 1967).

$$\underset{(61.83\text{ g})}{H_3BO_3} \xrightarrow{100^{\circ}C} \underset{(43.83\text{ g})}{HBO_2} + \underset{(18.016}{H_2O} \qquad [6]$$

The loss of one mole of water is equivalent to 29.11% of the original weight of boric acid. The additional loss of 20.89% at 100±3°C may be attributed to the steam volatilization of some of the orthoboric acid. Solid boric acid heated under the same condition lost 31.91% of its

TABLE 1
Weights of boron compounds before and after evaporation of aqueous solutions at different temperatures

	No. of samples	Weight of solution before drying (g)	Calculated weight of solute before drying (g)	Weight of solute after drying (g) and (% loss in weight)					
				At 100°C		At 50°C		At < 0°C	
				Boric acid solution (5% w/w)					
Mean	5	3.60	0.180	0.090	(50.00)	-	-	-	-
SE[1]		0.001	0.002	0.002					
Mean	5	3.80	0.190	-	-	0.181	(4.74)	-	-
SE		0.002	0.0001			0.0001			
Mean	5	3.54	0.177	-	-	-	-	0.176	(0.56)
SE		0.001	0.0001					0.0001	
				Borax solution (5% w/w					
Mean	5	3.56	0.175	0.112	(36.00)	-	-	-	-
SE		0.001	0.001	0.001					
Mean	5	3.62	0.181	-	-	0.116	(35.91)	-	-
SE		0.002	0.0001			0.0001			
Mean	5	3.16	0.158	-	-	-	-	0.151	(7.59)
SE		0.011	0.0001					0.0001	
				Boric acid-borax solution (6.21 % w/w)[2]					
Mean	5	2.38	0.119	0.071	(40.34)	-	-	-	-
SE		0.002	0.001	0.002					
Mean	5	3.10	0.155	-	-	0.093	(39.31)	-	-
SE		0.003	0.0001			0.001			
Mean	5	3.44	0.214	-	-	-	-	0.151	(29.43)
SE		0.012	0.0001					0.0001	

- [1] SE, standard error
- [2] The solution contained boric acid (2.44 g) and borax (3.77 g) in 100 gm solution

TABLE 2

Concentrations (% w/w) and weights (g) of boric acid equivalent (BAE) in an aqueous solution of borax-boric acid before and after evaporation at different temperatures

No. of samples	Initial concentration	Weight of solution before drying	Calculated weight of BAE before drying[1]	After drying at 100°C, measured by chemical analysis		After drying at 50°C measured by chemical analysis		
	(% w/w)	g	g	Conc. of BAE (% w/w)	Weight of BAE g	Conc. of BAE (% w/w)	Weight of BAE g	% loss in weight
3	4.89	5.07 0.021	0.248 (0.001)	4.04 (0.295)	0.205 (0.001)	-	-	17.30 (0.035)
3	4.85	5.11 0.018	0.250 (0.001)	-	-	4.40 (0.210)	0.225 (0.003)	10.00 (0.041)

- [1] The solution contained boric acid (2.44 g) and borax (3.77 g) in 100 g solution
- Values in parentheses are standard errors

original weight, i.e. only slightly more than the theoretical amount. In this case, of course, a much smaller quantity of water is involved.

After drying at either 100±3°C or 50±1°C the borax ($Na_2B_4O_7.10H_2O$) solutions had lost 36% of the original weight of borax. Borax itself contains 10 molecules of water of crystallization and when heated at 88°C loses 8 of these; this is equivalent to a weight-loss of 37.79% (Menzel *et al.* 1935):

$$\underset{(381.38\ g)}{Na_2B_4O_7.10H_2O} \xrightarrow{88^{0}C} \underset{(237.25\ g)}{Na_2B_4O_7.2H_2O} + \underset{(144.13)}{8H_2O} \quad [7]$$

The loss of a similar amount of water after drying at 50±10C is clearly a consequence of the much longer drying time employed. When the solution was evaporated in a freeze-dryer at < 0°C, the weight loss was only 7.59% which corresponds approximately to the loss of 1.5 molecules of water (theoretically 7.09%).

The Reaction of Borax with Boric Acid

When solid borax and boric acid are mixed a reaction occurs and water is liberated with the formation of sodium decaborate decahydrate ($Na_2O.5B_2O_3.10H_2O$) (Kemp 1956). Due to the ratio of $Na_2O:B_2O_3$ the latter is conveniently referred to as sodium 1:5 borate.

$$6H_3BO_3 + Na_2B_4O_7.10H_2O \longrightarrow Na_2O.5B_2O_3.10H_2O + 9H_2O \quad [8]$$

Sodium 1:5-borate crystals rapidly lose six molecules of water at ambient temperature and then a further two, more slowly, at 80°C. One molecule of water remains at 200° and is lost only at higher temperatures (Atterberg 1906).

$$Na_2O.5B_2O_3.10H_2O \xrightarrow{20^{0}C} Na_2O.5B_2O_3.4H_2O + 6H_2O \quad [9]$$

$$\xrightarrow{80^{0}C} Na_2O.5B_2O_3.2H_2O + 2H_2O \quad [10]$$

$$\xrightarrow{200^{0}C} Na_2O.5B_2O_3.H_2O + H_2O \quad [11]$$

From Equations 8, 9 and 10, it is apparent that the number of molecules of water lost after heating a suitable mixture of boric acid and borax at 50±1°C will be 15 and at 100±3°C will be 17.

Weight Loss of Boric Acid-Borax Mixture after Drying at 100±3°C

The amount of water lost from a mixture of 3.77 g borax and 2.44 g boric acid can be calculated as follows. From Equation 8,

$$\begin{array}{lcl} 4H_3BO_3 & = & Na_2B_4O_7.10H_2O \\ 370.98\ g) & = & (381.38\ g) \end{array}$$

Therefore 2.44 g of H_3BO_3 = (2.44/370.98) 3 381.38 = 2.51 g of borax. This weight was used in the formation of sodium 1:5-borate. The original weight of borax was 3.77 g, and it follows that 1.26 g of borax remained after the formation of the sodium 1:5-borate.

From Equations 8, 9 and 10, six moles of boric acid (370.98 g) react with 1 mole of borax (381.38 g) with the loss of 17 moles of water (306.27 g). Hence 2.44 g of boric acid corresponds to the loss of [(306.27/370.98) x 2.44 g]= 2.01 g of water. When a borax solution is evaporated at 100±30C the loss of weight is 37.79%, corresponding to the loss of 8 molecules of water. As the excess of borax over that used for reaction with boric acid was 1.26 g the loss of water from this was [(144.13/381.38) x 1.26 g] = 0.48 g. Thus the total weight of water lost from the mixture after drying at 100±30C was 2.49 g. As the total weight of solute used was 6.21 g, the calculated loss of weight is 40.10%. This is in excellent agreement with the value (40.34%) found by experiment (Table 1).

Weight Loss of Boric Acid-Borax Mixture after Drying at 50±1°C

If 15 molecules of water are lost from the boric acid-borax mixture (Equations 8 and 9) after drying to constant weight at 50±1°C, the weight loss from 2.44 g of boric acid is 1.78 g. When dried for three days at 50±1°C, borax loses 8 molecules of water (Table 1). Hence the loss of water from the excess of borax over that used in reacting with the boric acid, namely 1.26 g, is 0.48 g. So the calculated total loss of water from the mixture is 2.26 g i.e. 36.39%. This value is comparable with the value (39.31%) found experimentally (Table 1).

Weight Loss of Boric Acid-Borax Mixture after Freeze-drying at < 0°C

Nine molecules of water are liberated during the formation of sodium 1:5-borate and another six molecules are lost during freeze-drying (Equations 8 and 9). Consequently the weight of water corresponding to 2.44 g boric acid is again 1.78 g. Under those conditions borax lost about 1.5 molecules of water (observed, 7.59%,

TABLE 3
Mean retention of solute in rotan manau treated with a mixture of borax and borax and boric acid dried at different temperatures

Drying conditions	No. of samples	Od weight of samples	Weight of solution absorbed	Calculted weight of solute in sample[1]		Weight of retained solute after drying				Weight loss of solute
						Estimated[2]		Measured[3]		
			g	g	%w/w[3]	g	%w/w	g	%w/w	g
At 100°C										
Mean	5	6.87	6.95	0.432	6.29	0.258	3.75	0.180	2.62	0.078
SE		0.514	0.497	0.031	0.139	0.018	0.113	0.012	0.096	0.008
At 50°C										
Mean	5	5.24	5.30	0.329	6.29	0.200	3.81	0.183	3.27	0.017
SE		0.418	0.387	0.024	0.097	0.015	0.062	0.019	0.170	0.006
At < 0°C	5	3.55	3.59	0.223	4.84	0.157	4.53	0.150	4.34	0.007
Mean		0.475	0.312	0.019	0.078	0.014	0.063	0.010	0.253	0.005

- [1] The treatment solution contained boric acid (2.44 g) and borax (3.77 g) in (100 g) solution, (6.21% solute w/w)
- [2] Estimation values based on the loss of solute after drying, 40.34% at 100°C, 39.31% at 50°C and 29.43% at <0°C (Table 1).
- [3] Measured retentions as percentages of the od-weights of rattan
- SE, standard error

calculated, 7.09%) during the drying process. The calculated loss of water from the excess of borax was [(27.02/381.38) x 1.26 g] i.e. 0.089 g. Hence the total loss of weight on freeze-drying the boric acid-borax mixture should be 1.87 g i.e. 30.11%. This is in excellent agreement with the value (29.43%) found experimentally (Table 1).

Loss of BAE Concentration from the Mixture of Boric Acid-Borax after Drying at Higher Temperatures

From Table 2, it can be seen that the concentrations of BAE (w/w) in the solution of boric acid-borax mixture after drying at ca. 100°C and 50°C were reduced respectively by 17.3% to 4.04% and by 10.0% to 4.40% from its original concentration, 4.89%. The reduction of the BAE concentration appears to be mainly due to the loss of some of the boric acid in water vapour during the drying. The volatilization of boric acid in the steam produced during the evaporation of an aqueous solution at high temperature (*ca.* 100°C) has been described earlier; some 20.89% was lost in this way.

The results of weight loss of solute obtained by experiment and by calculation are in good agreement and show that when borax and boric acid are mixed, sodium 1:5 - borate ($Na_2O.5B_2O_3.10H_2O$ i.e. $Na_2B_{10}O_{16}.10H_2O$) is formed. After drying at 100±3, at 50±1 or < 0°C, the measured and the calculated (in parentheses) percentage weight losses from the mixtures were 40.34 (40.10), 39.31 (36.39) and 29.43% (30.11%), respectively. These measured values will be used to estimate the amount of solute retained in the rattan samples treated with such a boric acid-borax mixture and then dried. The percentage loss of H_3BO_3 (% w/w) from the boric acid-borax mixture in the steam volatilization will be used to estimate the amount of BAE in the same samples, i.e. 17.3% after drying at 100°C and 10.0% at 50°C. Preliminary experiment showed that there was no loss of BAE in treated rattan after drying at < 0°C in a freeze-dryer.

The Behaviour of Boron Compounds in Rattan when Dehydrated at Different Temperatures

The mean calculated and the measured values for the retention of the solute after the different drying processes are summarized in Table 3, while the mean retention, expressed as the BAE values, is presented in Table 4. The values under the headings '% w/w' in the Tables are the percentage retentions of the oven-dried weights of the rattan samples. The mean measured weight of the solute (0.180 g) for the treated samples dried at *ca.* 100°C was significantly lower than the mean estimated weight (0.258 g) (Table 3). The difference (30.23%) was too big to be attributed to experimental error. The same was also observed for the BAE values. The mean measured retention BAE value (0.248 g) was 11.74% lower than the mean estimated value (0.281 g) (Table 4). For samples which had been dried at *ca.* 50°C (Table 3), the mean measured solute retention (0.183 g) was 8.50% lower than the mean estimated retention (0.200 g). Under these drying conditions, the measured weight of the BAE (0.210 g) in the treated samples was lower by 7.59% than the estimated value (0.227 g) (Table 4).

Results in Table 3 show that the mean measured weight of the solute (0.150 g) in the samples which had been freeze-dried was slightly lower than the calculated weight (0.157 g). The BAE values (Table 4) in the material before and after freeze-drying were similar.

Several factors could be responsible for the weight loss of the solute and the decrease in the BAE in the treated rattan samples which had been dried at the higher temperatures. These include:

1. the loss of steam-volatile organic compounds,
2. the loss of steam-volatile boric acid, and
3. the loss of some of the permeating liquid during the early stage of drying.

Loss of lower molecular weight components of the rattan may occur by steam volatilization. Many organic compounds which are not themselves appreciably volatile at 100°C are readily volatile in steam (Ault 1968). It seems likely that the drying of the rattan samples which had initially been saturated with water would be accompanied by the loss of some lower molecular weight components. To test this idea six small pieces of rattan which had been dried previously to constant weight at 100°C were saturated with water and then again dried at 100±3°C. All the samples showed a decrease in weight (an average of 0.307% of the original od weight). As the average oven-dried weight of the samples used is 6.87 g (Table 3), the loss of steam volatile components would be 0.021 g. This, however, is

TABLE 4

Mean retention of boric acid equivalent (BAE) in rotan manu treated with a mixture of borax and boric acid and dried at different temperatures

Drying temperatures	No. of samples	Od-weight of sample	Before drying			Weight of retained BAE after drying				Weight loss of BAE
			Weight of solution absorbed	Calculated weight of BAE in sample1		Estimated[2]		Measured by chemical analysis3		
	g	g	g	g	% w/w[3]	g	% w/w	g	% w/w	g
At 100°C										
Mean	4	6.87	6.95	0.340	4.94	0.281	4.09	0.248	3.61	0.043
SE		0.514	0.497	0.024	0.152	0.021	0.170	0.012	0.152	0.025
At 50°C										
Mean	4	5.24	5.30	0.253	4.84	0.227	3.41	0.210	4.01	0.017
SE		0.418	0.387	0.019	0.078	0.019	0.117	0.022	0.117	0.010
At < 0°C										
Mean	4	3.55	3.59	0.176	4.99	-	-	0.175	4.95	-
SE		0.312	0.312	0.023	0.077			0.016	0.078	

- [1] The treatment solution is equivalent to 4.89% (w/w) BAE
- [2] Estimation values based on the loss of H_3BO_3, 17.30 at 100°C, 10.0% 50°C (Table 1)
- [3] Measured retentions as percentages of the od-weights of rattan
- SE, standard error

still not enough to account for the average observed weight losses (0.258 - 0.180 g) i.e. 0.078 g (Table 3) in the treated samples.

The volatilization of boric acid in the steam produced during the evaporation of an aqueous solution at 100±30C has been described earlier; some 20.89% was lost in this way. From Table 3 it can be seen that an average 0.078 g of boric acid was lost when rattan samples containing an average 0.432 g of boric acid and borax were dried at ca. 100°C. The weights of boric acid and borax in the original solution (100 g) were 2.44 and 3.77 g, respectively, and it follows that the average weight of boric acid in the rattan samples initially was [(0.432 x 2.44)/6.21] = 0.169 g while the weight of borax was [(0.432 x 3.77)/ 6.21] = 0.262 g.

Since 0.078 g of the boric acid would be lost in the drying process, that remaining is 0.091 g. As shown earlier (Equation 8) this weight would react with 0.094 g of borax with the formation of sodium 1:5 borate. As the original weight of borax present was 0.262 g, it follows that 0.169 g remained after the formation of the sodium 1:5 borate.

From Equations 8, 9, and 10, the loss of water corresponding to 0.091 g boric acid is 0.075 g. The loss of water from partial dehydration of the excess of borax present, namely 0.169 g, is 0.064 g. So the calculated loss of weight resulting from dehydration and volatilization of boric acid is 0.075 + 0.064 + 0.078 = 0.217 g. To this must be added the weight of the steam volatile components, 0.021 g. The total loss of weight of the treated rattan samples is therefore 0.238 g and the average weight of solute remaining will be (0.432 - 0.238), i.e. 0.194 g. This is somewhat higher than the measured value (0.180 g) (Table 3) and an explanation of this discrepancy is needed.

The further loss in weight (0.194 -0.180) i.e. 0.014 g could be a result of loss of the permeating liquid from the sample during the early stage of drying. The imbalance of temperature in the samples with the oven condition might have caused the air in the samples to expand, which would force the liquid out of the samples through the porous end surfaces. The loss of permeating liquid in this way could have been minimized if the samples had been end-coated before drying. This precaution was not taken in this particular experiment.

Similar calculations to those detailed above show that loss of solute also occurred from the samples which were dried at ca. 50°C for a longer period of time (Table 3). The estimated weight of solute after drying was 0.20 g, which was slightly higher than the measured value (0.183 g). The calculation was made assuming that under the milder drying conditions there had been no loss of steam-volatile components from the rattan. The further loss of weight of solute under these conditions would be due mainly to the loss of some boric acid in the water vapour during the drying process together with a little loss of the permeating liquid.

The decrease in the BAE of treated rattan (Table 4) which had been dried at *ca.* 100°C and or 50°C appears to be due mainly to the loss of some boric acid in the water vapour. The percentage loss of BAE from the mixture of boric acid-borax after drying at *ca.* 100°C (17.3%) found in this study (Table 2) seems to be comparable with the loss of BAE calculated for rattans which were treated by the hot and cold-bath process (Zaidon 1995), i.e. between 11-23%, but a higher loss of BAE was found in treated *rotan manau* in the present study; i.e. 0.033 g or 11.74% higher (Table 4). The significant difference in the loss of the BAE between these two treated rattans is probably due to the process of drying the samples. The hot and cold-bath treated samples were initially dried at a milder temperature (50°C) for approximately 3 days before being dried at a higher temperature (100°C). In contrast, the vacuum-pressure treated samples in the present study were dried at 100°C following treatment; the loss of permeating liquid from the porous end surfaces on being heated is greater, thus resulting in further loss of boric acid.

CONCLUSION

When borax and boric are mixed, sodium 1:5-borate is formed. After drying at 100±3, 50±1, or at < 0°C, the measured percentage weight losses from the mixtures were 40.34, 39.31 and 29.43%, respectively. The further weight losses of the mixture in treated rattan which had been dried at higher temperatures are presumably attributable to: (1) the volatilization of low molecular weight rattan components, (2) the volatilization of boric acid in water vapour during drying and (3) the mechanical loss of permeating liquid during the early stage of drying.

The decrease in the BAE of the treated rattan which had been dried at ca. 100°C, or at 50°C for a longer time appears to be due mainly to the loss of boric acid in the water vapour. The measured BAE (% w/w) values in treated rattan in the previous experiments would have been *ca.* 17% higher if a lower temperature had been used in the drying process.

Freeze-drying of rattan which has been treated with a mixture of boric acid-borax or with boric acid itself is essential if all the boric acid is to be retained. On a commercial scale, treatments of rattan or wood which involve boric acid in an aqueous solution should avoid high temperature drying to ensure optimum retention of BAE.

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