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BORON ANALYSIS IN TREATED WOOD AND SOLUTION

Salamah Selamat, Zaitun Said & Habibah Mohamad

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

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SALAMAH SELAMAT, ZAITUN SAID & HABIBAH MOHAMAD. 1989. Boron analysis in treated wood and solution. Analysis of boron in solution and treated wood samples using Atomic Absorption Spectrophotometer and Inductively Coupled Plasma are found to be the most rapid, simple and accurate methods . These methods are useful when low concentrations of boron are to be detected. Colorimetric method gives good results, but is not suitable when a large number of samples are involved. Titration method is lengthy and time consuming and is not suitable for boron determination at low concentration.

Key words: Boron analysis - titration method - Atomic Absorption Spectrophotometer - Inductively Coupled Plasma

Introduction

Boron compounds in the form of disodium octaborate tetrahydrate, $Na_2B_8O_{13}.4H_2O$, disodium tetraborate decahydrate,$Na_2B_4O_7.10H_2O$ and boric acid, H_2BO_3, in combination with other chemicals are very effective against insect and fungi attack (Carr 1959). Boron compounds can either be used for temporary or permanent treatment of wood. Temporary treatment includes spraying, brushing or dipping processes. Permanent treatment can be achieved through dip diffusion or vacuum pressure (Carr 1959). Analysis on the distribution and penetration of boron compounds in treated wood needs to be carried out in order to comply with the Malaysian Standard Specification (Anonymous 1986a). The current interest in these compounds for preserving timber has resulted in the need for a rapid, simple and yet accurate method for determining boric acid equivalent in treated wood.

At present, such assessments are made by spraying chemical reagents on the treated wood sample (Anonymous 1979, Anonymous 1980, Wilson 1958), but its accuracy is doubtful. The titration method, although more accurate, is not practicable when large batches of specimens are involved. In addition, it cannot be used to determine the preservative distribution over a small area and at low concentrations.

The objectives of this study are to establish a simple method for separating boron compound from the matrix and to come up with a reliable procedure for determining boric acid equivalent. This study will cover three different methods of boron determination, that is, colorimetry, Atomic Absorption

Spectrophotometry (AAS) and Inductively Coupled Plasma (ICP) and to compare them against the titration method.

Materials and methods

Three sets of samples having a range of boric acid equivalent of 1 to 60 ppm, 0.01 to 4.0% weight/volume in solution and 1.0 to 4.0% weight/volume in treated sawdust (rubberwood) were used in this study. The chemicals used were also freshly prepared.

Titration method

The titration method is based on the Australian Standard (AS 1605) method (Anonymous 1974) and British Wood Preserving Association (BWPA) method (Anonymous 1977). This method is currently being used for determining the amount of boron in solution or treated wood. However, it is tedious and lengthy. The method involves digestion of boron from treated sawdust by the process of dry ashing in the presence of barium hydroxide, followed by extraction with hydrochloric acid. The boric acid content is then determined by titrating the solution against standard solution of sodium hydroxide. The only difference between the AS 1605 method and the BWPA method is the process of freeing CO_2 from the solution. The amount of boron as percentage boric acid equivalent (% BAE) is calculated by using the following equations:

AS1605 method,

$$\text{\% Boric acid equivalent in preservative solution (weight/volume)} = \frac{\text{Titre} \times 247}{\text{Volume of preservative solution}}$$

$$\text{\% Boric acid equivalent in treated wood (weight/weight)} = \frac{\text{Titre} \times 250}{\text{Oven dry weight of treated sawdust}}$$

BWPA 105 method,

$$\text{\% Boric acid equivalent in treated solution (weight/volume)} = \frac{61.84 \times T2 \times N2 \times 100}{V1 \times V2}$$

$$\text{\% Boric acid equivalent in treated wood (weight/weight)} = \frac{T \times N2 \times 6.184}{W}$$

where **N2** is the normality of standard sodium hydroxide, **T2 and Titre** are the amount of standard NaOH to reach the phenolphthalein end point, **V1** is the volume of preservative solution taken for analysis, **V2** is the volume of the diluted solution actually titrated; **T** is T1 - T2, where **T1** is the amount of standard NaOH to reach the phenolphthalein end point for the treated wood and **T2** is for the untreated wood, and **W** is the dry weight of wood sawdust.

Colorimetry method

This colorimetry method is based on the Japanese Standard on analytical method of treated plywood (Anonymous 1986b). Boron from solution and treated sawdust is digested using hydrogen peroxide, phosphoric acid and sulphuric acid. In the presence of boron, Carmine in concentrated sulfuric acid will change from a bright red to a stable purple or blue.

Colour development would depend on temperature (Anonymous 1968). It develops after approximately 1 *h* at 30°*C*. The absorbance is read at 600 *nm* against a blank solution using Bousch and Lomb 1001 Spectrophotometer. A standard curve is prepared by plotting the absorbance of sample against that of standard samples. The boric acid equivalent is then calculated based on the following formula:

$$\text{\% Boric acid equivalent in treated solution (weight/volume)} = \frac{(\text{ppm B}) \times (61.84/10.81) \times \text{f} \times 100}{\text{Volume of wood preservative} \times 10}$$

$$\text{\% Boric acid equivalent in treated wood (weight/weight)} = \frac{(\text{ppm B}) \times (61.84/10.81) \times \text{f} \times 100}{\text{Oven dry weight} \times 10}$$

where **ppm B** is the concentration of boron from colorimetry analysis and **f** is the dilution factor.

Atomic Absorption Spectrophotometer and Inductively Coupled Plasma methods

The digestion procedure for both methods is also based on the Japanese Standard for analytical method of treated plywood (Anonymous 1986b). The digested solutions are analysed by either the AAS or ICP. The % B.A.E in solution and treated wood is calculated based on the same formula as in the colorimetry method.

Results and discussion

From the series of determinations on different concentration levels of boron, the average amounts of boron as % boric acid equivalent (% B.A.E) detected in the treated solution are given in Table 1. The amounts of boron as % B.A.E in the range 0.01 to 0.1% detected by those methods, against the real values of % B.A.E, indicate that there are differences in sensitivity of detection between the titration method and AAS, ICP and colorimetry at the lower and the higher concentrations of boron. Further experiments using % B.A.E in the higher range of 0.1 to 1.0% and 1.0 to 4% showed that differences between the five methods are not significant.

Table 1. The amount of boric acid percentage detected from known concentration of boric solution by using different methods of analysis

% H_3BO_3 in solution	Average of boric acid percentage detected by using									
	AS 1605		BWPA		AAS		ICP		COLOR	
	×	SD	×	SD	×	SD	×	SD	×	SD
0.0100	0.0019	1.88E-05	0.0025	1.70E-05	0.0110	1.21E-05	0.0108	5.26E-05	0.0099	4.12E-05
0.0250	0.0240	3.57E-05	0.0242	1.71E-05	0.0252	1.80E-05	0.0249	1.30E-05	0.0251	1.58E-05
0.0500	0.0480	4.12E-05	0.0470	3.14E-05	0.0500	1.07E-05	0.0504	6.17E-05	0.0501	1.32E-05
0.0750	0.0740	3.00E-05	0.0730	1.80E-05	0.0740	6.00E-05	0.0745	7.45E-02	0.0003	7.46E-02
0.1000	0.1050	4.00E-05	0.1060	3.10E-05	0.1008	3.00E-05	0.1009	5.00E-05	0.1002	4.00E-05
0.2500	0.2510	3.00E-05	0.2478	2.10E-05	0.2508	1.00E-05	0.2510	1.00E-05	0.2500	5.00E-05
0.5000	0.5050	2.15E-05	0.5030	3.44E-04	0.5010	1.21E-05	0.5010	1.81E-05	0.5003	1.11E-05
0.7500	0.7600	1.01E-04	0.7560	1.11E-04	0.7517	1.42E-05	0.7510	1.17E-05	0.7509	1.21E-04
1.0000	1.0130	3.14E-03	1.0100	1.60E-03	1.0020	2.13E-04	1.0011	1.14E-04	1.0010	1.31E-03
2.0000	2.0098	3.12E-03	2.0018	2.75E-03	2.0015	1.17E-03	2.0011	1.83E-03	2.0010	1.17E-03
4.0000	4.0500	2.79E-03	4.0190	3.13E-03	4.0024	2.17E-03	4.0013	3.14E-03	4.0010	1.13E-02

(AS 1605 = Australian Standard, BWPA = British Wood Preserving Association, AAS = Atomic Absorption Spectrophotometry, ICP = Inductively Coupled Plasma, COLOR = colorimetry, × = an average of four replicates, SD = standard deviation - abbreviatons follow for all tables)

Regression analysis showed that the result obtained from titration was quite reliable ($R^2 > 95\%$) for boron detection at higher concentration ($\geq 0.1\%$). Further analysis on the recovery of boron compound from sawdust treated with 1, 2, 3, 4, 5 and 10% of boric acid also showed that all the five methods gave high detection of boron (Table 2). However, at lower concentration (≤ 60 ppm), AAS, ICP and colorimetry produced better results with $R^2 \geq 95\%$ (Table 3). On the other hand, both titration methods were unable to detect boron at this range of concentration.

Table 2. Percentage of boric acid in treated sawdust determined by using atomic absorption spectrophotometer, inductive couple plasma, colorimetry and titration (AS 1605 and BWPA) methods

Sample number	Amount of average boric acid percentage by using										
	H_3BO_3 added (%)	AS 1605		BWPA		AAS		ICP		COLOR	
		×	SD	×	SD	×	SD	×	SD	×	SD
1	1.0000	0.9675	3.72E-03	0.9677	3.18E-03	0.9800	2.87E-03	0.9850	1.40E-03	0.9878	1.27E-03
2	2.0000	1.9773	4.50E-03	1.9800	3.91E-03	1.9872	3.13E-03	1.9875	1.13E-03	1.9887	1.33E-03
3	3.0000	2.9840	5.13E-03	2.9875	4.16E-03	2.9900	4.11E-03	2.9881	2.11E-03	2.9824	2.98E-03
4	4.0000	3.9535	4.71E-03	3.9836	3.87E-03	3.9830	4.78E-03	3.9867	1.78E-03	3.9889	1.17E-03
5	5.0000	4.9000	5.22E-03	4.9310	6.31E-03	4.9807	5.11E-03	4.9867	3.21E-03	4.9880	2.67E-03
10	10.0000	9.9180	7.57E-03	9.9470	6.72E-03	9.9797	5.41E-03	9.9800	3.17E-03	9.9870	3.27E-03

Table 3. Determination of boron at the lower range of concentration 0 - 60 ppm

Boron (ppm)	Average of boron concentration in ppm detected by using AAS		ICP		COLOR	
	×	SD	×	SD	×	SD
0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
2.2000	2.1800	0.0281	2.2150	0.0148	2.2050	0.0053
4.4000	4.3576	0.0312	4.4080	0.0171	4.4027	0.0018
8.6000	8.4960	0.0711	8.5433	0.0712	8.5518	0.0033
13.000	12.6817	0.0611	12.7435	0.0352	12.7630	0.0018
15.200	14.9500	0.0521	15.0021	0.0453	15.0010	0.0021
30.400	30.3438	0.0461	30.0173	0.0432	30.3855	0.0017
60.800	60.5426	0.0551	60.6207	0.0567	60.7353	0.0215

From these results, it was found that AAS, ICP and colorimetry methods are suitable for boron determination especially for detection of very low concentration of boron. However, the use of colorimetry method is limited by the colour reaction which tends to change after 24 *h*, thus affecting the absorption reading.

Conclusion

Results show there was a good linear correlation between the amounts of detected boron and the actual boron concentration prepared. Generally, AAS and ICP are the most simple, rapid and accurate methods for determining boron in both solution and treated wood. The advantages of these two methods are that they can be used for a wide range of boron concentrations and a large number of samples can be handled with ease.

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