Wood Sci Technol (2008) 42:39–45
DOI 10.1007/s00226-007-0139-4

ORIGINAL

Determination of optimum borate concentration for alleviating strength loss during heat treatment of wood

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Received: 23 December 2006

Abstract Based on the already established mitigating potentials of borate salt on the negative effects of heat treatment on the strength properties of wood (Awoyemi and Westermark 2005), the optimum concentration of the alkali buffer solution required to minimize strength loss was determined. Wood samples were impregnated with 0.1, 0.3 and 0.5 M sodium borate solution and exposed to heat treatment for 4 h at 200°C. The mitigating effect of borate salt on the degree of strength loss during heat treatment increases significantly with increasing concentration from 0.1 to 0.3 M. Increasing the concentration of sodium borate from 0.3 to 0.5 M did not produce significant differences in the degree of strength loss during heat treatment. The increase in the buffering effect observed with increasing concentration of the sodium borate preservative is more pronounced on the modulus of rupture than on the modulus of elasticity. It is evidenced therefore that the buffering effect of borate salt on the modulus of elasticity of wood exposed to heat treatment did not start significantly until the 0.3 M concentration is reached and further increase in concentration beyond this point did not produce any appreciable improvement in strength properties.

Introduction

Heat-treatment is a wood modification method which has been used to some extent in improving timber quality. Among the benefits associated with this technique are reduction in the emission of volatile organic compounds (Manninen

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et al. 2002), stress reduction (Dwianto et al. 1998; Kamdem et al. 2002; Nogi et al. 2003; Tejada et al. 1997; Tejada et al. 1998), improved dimensional stability (Kamdem et al. 2000; Rapp and Sailer 2001; Tejada et al. 1997), enhancement of colour uniformity (Sailer et al. 2000) and improved durability (Hanger et al. 2002; Sailer et al. 2000).

During heat treatment, there are considerable changes in the chemical composition of wood. These changes result mostly in degradation of amorphous carbohydrates (Kamdem et al. 2002; Udaka and Furuno 2003) and consequently increase in cellulose crystallinity (Bhuiyan et al. 2000; Tejada et al. 1997; Udaka and Furuno 2003) and in apparent lignin content (Kamdem et al. 2002), decrease in extractive content (Kamdem et al. 2002) and increase in wood acidity (Hodgin and Lee 2002; Kamdem et al. 2002). The increase in wood acidity is due to the formation of acetic acid liberated from the hemicelluloses, which further catalyses carbohydrates cleavage causing a reduction of degree of polymerisation of the carbohydrates (Tjeerdsma et al. 1998).

The various chemical changes invariably lead to the alteration of the various physical properties of wood such as reduction in the equilibrium moisture content (Obataya et al. 2000; Obataya and Tomita 2002), reduction in electrical resistance (Geissen and Du 1995), increase in brittleness (Hodgin and Lee 2002; Kubojima et al. 2000) but also reduction in strength properties (Kamdem et al. 2002).

The various changes associated with heat treatment have been linked with the changes in the chemical composition of the material during the process. For instance, increase in acidity is associated with colour changes (Maruyama et al. 2001). Pretreatment of wood with borate salt has been found to reduce thermal induced degradation in wood (Awoyemi and Westermark 2005; Winandy 1997) and this is clearly due to the counterbalancing of the liberation of acetic acid from the hemicelluloses. It is therefore essential to investigate the effect of different borate concentrations on the degree of strength loss during heat treatment in order to derive the maximum benefit from its application.

Materials and methods

Wood material

Wood samples ($10 \times 10 \times 100$ mm^3) were obtained from two boards of birch (*Betula pubescens*) wood grown in Northern Sweden. The samples were matched into 8 groups labeled A to H and allocated into different treatment units (Table 1).

Borate impregnation

In order to facilitate impregnation, the samples were soaked in boiled water for about 20 min. Samples not intended for borate impregnation were also soaked in boiled water in the same medium in order to minimise the possibility of bias so as to ensure that the greater proportion of the differences that could occur in the properties will be due to the effects of heat treatment and variations in borate

Table 1 Experimental units

Sample group	Treatment	
	Borate concentration (M)	Heat treatment (200°C for 4 h)
A	Unbuffered	Yes
B	0.1	Yes
C	0.3	Yes
D	0.5	Yes
E	Unbuffered	No
F	0.1	No
G	0.3	No
H	0.5	No

concentrations. The samples were then impregnated with different concentrations of di-sodium tetraborate ($Na_2B_4O_7.10H_2O$; pH = 9.4) as shown in Table 1.

Conventional drying

As a pre-requisite for heat treatment, all the samples were dried by conventional method using kiln at a temperature of 60°C and 60% relative humidity, which corresponds to 9% equilibrium moisture content.

Heat treatment

Heat treatment was carried out by heating in an autoclave (about 33 mm internal diameter and 125 mm long). About 5 ml of water was put into the autoclave to drive out air and generate a steam atmosphere. An iron mesh was placed on top of the water upon which the samples were placed inside the autoclave. The autoclave was then placed inside a laboratory oven at 200°C for 4 h.

Determination of strength properties

After heat treatment, both the heat-treated and untreated samples were kept in a temperature and humidity control chamber at a temperature of 33°C and 52% relative humidity in order to equilibrate to 9% moisture content. Static bending test was carried out with Hounsfield Tenseometer. The modulus of elasticity and modulus of rupture were determined as follows:

$$\mathrm{MOE} = \frac{WL^3}{4\Delta bd^3}$$

$$\mathrm{MOR} = \frac{3WL}{2bd^2}$$

where MOE = modulus of elasticity in MPa, MOR = modulus of rupture in MPa, W = breaking load, L = span, Δ = deflection at the proportional limit, b = width in mm and d = thickness in mm.

Results and discussion

Modulus of elasticity

A close look at the results in Table 2 shows that prior to heat treatment there was a slight decrease in the modulus of elasticity in wood pre-treated with 0.1 M sodium borate. Increasing borate concentration to 0.3 M resulted in a sharp increase in the property. A further increase in the concentration of sodium borate led to higher value of modulus of elasticity. The increase in MOE value resulting from the increase in borate concentration from 0.3 to 0.5 M is however lower than that resulting from the increase from 0.1 to 0.3 M. Similarly, after heat treatment the modulus of elasticity of the unbuffered wood was higher than wood pre-treated with 0.1 M sodium borate. Increasing the concentration of the buffer solution to 0.3 M reversed the loss of strength producing wood stronger than both unbuffered wood and wood pre-treated with 0.1 M borate. However, contrary to expectation, a further increase in the concentration of sodium borate to 0.5 M led to lower MOE value compared to that observed for wood pre-treated with 0.3 M solution.

In a chi-square analysis of the interactions between the effects of borate concentration and heat treatment, the statistical chi-square (18.62) is greater than the critical chi-square at 95% confidence level (7.81). Therefore, there is a very strong interaction between the effects of the concentration of sodium borate used and heat treatment on the modulus of elasticity (Fig. 1).

Table 2 Strength properties of heat-treated and untreated wood pre-treated with different cconcentrations of sodium borate

Heat treatment	Sodium borate concentration (M)	Modulus of elasticity (MPa)		Modulus of rupture (MPa)		Number of samples
		Mean	Standard deviation of mean	Mean	Standard deviation of mean	
Yes	Unbuffered	6,678	1,583	76	32	10
	0.1	6,246	1,053	70	23	10
	0.3	7,930	2,606	95	19	10
	0.5	7,865	1,041	99	15	10
No	Unbuffered	7,207	1,177	98	16	10
	0.1	6,837	617	98	10	10
	0.3	8,091	1,117	112	9	10
	0.5	8,801	1,215	110	7	10

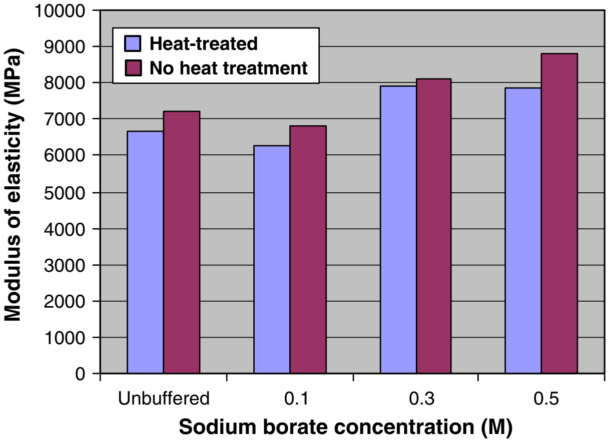

Fig. 1 Modulus of elasticity of heat-treated and untreated wood pretreated with different concentrations of sodium borate

Modulus of rupture

In untreated wood (wood not subjected to heat treatment), pretreatment with 0.1 M sodium borate did not change the modulus of rupture. Increasing the concentration of the alkali medium to 0.3 M resulted in appreciable increase in the property. However, further increase in the borate salt produced a negative result manifesting in slight reduction of the MOR. After heat treatment, the unbuffered wood was slightly higher in MOR than wood pretreated with 0.1 M sodium borate. Increase in borate concentration to 0.3 M resulted in large increase in MOR over both unbuffered wood and wood pretreated with 0.1 M borate. However, the improvement in MOR became minimal with further increase in the concentration of the preservative to 0.5 M resulting in lower MOR value than in wood pretreated with 0.3 M. Nevertheless, the modulus of rupture of wood pretreated with 0.5 M solution is conspicuously higher than that of the unbuffered and 0.1 M pretreatment.

In a chi-square analysis of the interaction between the effects of borate concentration and heat treatment, the statistical chi-square (1.26) is less than the critical chi-square at 95% confidence level (7.81). Therefore, there are no significant interactions between the effects of heat treatment and the concentration of sodium borate used on the modulus of rupture (Fig. 2).

The increase in strength properties due to impregnation with 0.3 and 0.5 M borate in untreated wood (no heat treatment) is due to the fact that *B. pubescens* is naturally acidic and addition of alkaline salt will reduce the acidity and thus increase the strength even before heat treatment. Generally the mitigating effect of borate salt on the degree of strength loss during heat treatment of wood increases significantly with increasing concentration from 0.1 to 0.3 M. Increasing the concentration of sodium borate from 0.3 M to 0.5 M did not produce significant improvement in strength properties of heat treated wood. The increase in the buffering effect observed with increasing concentration of the sodium borate preservative is more

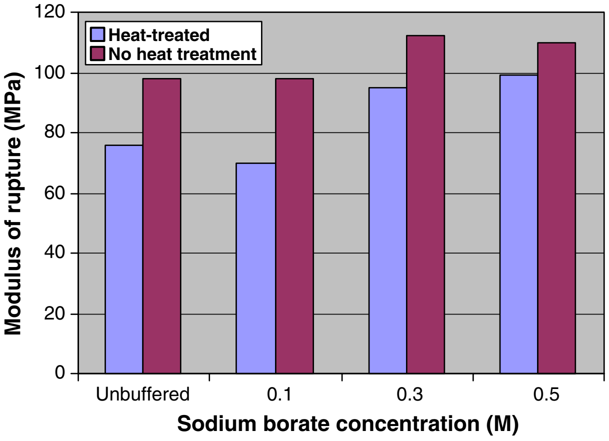

Fig. 2 Modulus of rupture of heat-treated and untreated wood pretreated with different concentrations of sodium borate

pronounced on the modulus of rupture than on the modulus of elasticity. This might be connected with the fact that heat treatment affects MOR more than MOE (Kubojima et al. 2000; Santos 2000) and generally the ameliorating effect of borate is more pronounced on the MOR than on the MOE. It is evidenced therefore, that the buffering effect of borate salt on the strength properties of wood exposed to heat treatment did not start significantly until the 0.3 M concentration was reached and further increase in borate concentration beyond this point did not produce any appreciable improvement in strength properties.

Since the release of acetic acid from the hemicelluloses is the principal process involved in the modification of wood resulting in improvement of the quality of wood, in particular improved dimensional stability, the use of buffer in decreasing the release of acetic acid despite its positive effect of reducing strength loss will invariably affect negatively other qualities particularly the dimensional stability of wood.

Conclusions

The buffering effect of borate salt in minimizing strength loss during heat treatment of wood increases with increasing concentration of the preservative up to a level of 0.3 M. Conclusively, in order to ensure maximum prevention of strength loss at a reasonable cost, impregnation with 0.3 M of sodium borate preservative is recommended especially when carrying out heat treatment in the vicinity of the temperature used in this study.

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