Response Surface Modeling of Hydrothermal Treatment Conditions on Color Changes, Strength, and Durability Properties of Rubberwood

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The effect of hydrothermal treatment at various temperatures (100 to 160 °C) and treatment times (30 to 720 minutes) on color changes (ΔE^*), equilibrium moisture content (EMC), tensile strength (TS), shear strength (SS), brown-rot fungal decay mass loss (FML), and termite attack score (TAS) of rubberwood was examined. Response surface methodology (RSM) with a two-factor, four level (4^2) full factorial was employed. The mathematical models describing those properties as functions of treatment temperature and logarithm of treatment time were obtained. Hydrothermal treatment adversely and positively influenced mechanical properties (TS and SS) and durability (FML and TAS), respectively, of rubberwood. Strong correlations between ΔE^*, TS, SS, and FML of hydrothermally treated rubberwood, proposed to be a consequence of degradation of hemicelluloses, were observed. Finally, ΔE^* proved to be a good indicator of TS, SS, and FML but not that of EMC and TAS of hydrothermally treated rubberwood.

Keywords: Hydrothermal treatment; Rubberwood; Color changes; Strength properties; Fungal resistance; Termite resistance

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INTRODUCTION

Rubberwood (*Hevea brasiliensis*) lumber obtained from 25- to 30-year-old plantation-grown rubber trees has long been used as raw material for furniture productions (Balsiger *et al.* 2000). Because rubberwood has low natural durability (Scheffer and Morrell 1998), the lumber is generally impregnated with chemical preservatives such as boron to protect it against insect borers for indoor uses (Gnanaharan and Dhamodaran 1993). To extend the uses of this lumber to outdoor applications, treatment of chemical preservatives such as copper-chrome arsenic (CCA) are needed (Dhamodaran and Gnanaharan 2001). However, because of their toxicity and consumer health concerns (Hemond and Solo-Gabriele 2004), more environmentally friendly treatments of wood that improve wood durability, such as thermal treatment, should be developed. Thermal treatment processes such as ThermoWood, oil heat treatment, Retification, Bois Perdure, and PLATO have been successfully developed for commercial uses in various European countries (Rapp 2001; Esteves and Pereira 2009). Reports suggest that thermal treatment of wood improves dimensional stability and durability, but has a negative effect on mechanical properties (Tjeerdsma *et al.* 2000; Bekhta and Niemz 2003; Boonstra *et al.* 2007a; Ayrilmis *et al.* 2011).

Chemical changes during hydrothermal treatment have been extensively examined (Tjeerdsma *et al.* 1998; Tjeerdsma and Militz 2005; Boonstra and Tjeerdsma 2006). Thermal treatment in the presence of moisture at high temperature has been reported to largely depolymerize hemicelluloses, the most reactive wood component, into oligomers and monomers. This involves cleavage of acetyl groups of hemicelluloses, which leads to the formation of carbonic acids, largely acetic acid. These acids further catalyze carbohydrates cleavage to form formaldehyde, furfural, and other aldehydes and cause some lignin cleavage and some aldehydes production from lignin units (Tjeerdsma *et al.* 1998; Tjeerdsma and Militz 2005; Boonstra and Tjeerdsma 2006). Condensation reactions of lignin fragments with high reactivity and degradation products of hemicelluloses can result in an increased cross-linking of the lignin network. The effect of hydrothermal treatment on cellulose degradation is rather limited (Boonstra and Tjeerdsma 2006). However, the relative amount of crystalline cellulose has been reported to increase, which might be a result of crystallization of amorphous cellulose to a certain degree (Tjeerdsma *et al.* 1998).

In Southeast Asian countries, rubberwood sawmills usually carry out lumber impregnation within a pressure vessel before the lumber has been subjected to drying in a kiln (Gnanaharan and Dhamodaran 1993). Therefore, it would be cost effective to modify this pressure vessel to perform hydrothermal treatment in which the lumber is heated up to a high temperature in the presence of liquid water under pressure. This work was intended to investigate the effect of hydrothermal processing parameters *i.e.* temperature and time on color changes, equilibrium moisture content, and mechanical and durability properties of rubberwood lumber. An attempt was also made to model properties of the treated lumber as functions of processing parameters by means of response surface methodology (RSM).

EXPERIMENTAL

Materials

Rubberwood logs, approximately 25 years old, were taken from a local saw mill in the Chawang district, Nakhon Si Thammarat, Thailand and were sawn to dimensions of 80 mm (width in tangential direction) × 30 mm (thickness in radial direction) × 1000 mm (length in longitudinal direction). Ten pieces of lumber were randomly allocated for each experimental treatment. The average moisture content of lumber prior to hydrothermal treatment process was 49±2%.

Hydrothermal Treatment

Hydrothermal treatment of rubberwood lumber was carried out by boiling the lumber in hot liquid water above atmospheric pressure within the pressure vessel. After loading the lumber into the pressure vessel, impregnation of water into the lumber was first performed by reducing pressure within the pressure vessel to -0.85 bar for 15 minutes, then filling the pressure vessel with water and increasing the pressure of water to 10 bar for 45 minutes until the lumber was saturated with water. The water inside the pressure vessel was then heated to the required temperature of 100 to 160 °C, by passing saturated steam through the heating coil fitted inside the pressure vessel. During the boiling process, pressure inside the vessel was maintained sufficiently to keep water in its liquid state. After performing the hydrothermal treatment for the specified period between 30 to 720 minutes, temperature and pressure of the water within the pressure vessel were reduced with the cooling rate of about 3 °C/min. After reaching atmospheric pressure, hot water was drained from the pressure vessel. The hydrothermally treated lumber was then dried to the required moisture content of about 8% in the laboratory drying kiln using a single step drying at dry-bulb and wet-bulb temperatures of 90 °C and 60 °C, respectively. Prior to further testing, the kiln dried lumber was cut to size and conditioned in a walk-in conditioning room (KBO, Sweden) at 20 °C and 65%RH for at least 4 weeks to attain equilibrium moisture content (EMC).

Measurements of Color and Strength Properties

Color measurement was performed on the core section of hydrothermally treated rubberwood specimens using a tristimulus color analyzer (ColourFlex colorimeter, Hunter Associates Laboratory, Inc., USA). A total of ten replicates per treatment were carried out. Hunter color coordinates were used to determine the degree of lightness (L^*), redness-greenness (+ or- a^*), and yellowness-blueness (+ or- b^*). Total color difference (ΔE^*) between the treated specimens and the controls (without hydrothermal treatment) was calculated using the equation:

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \qquad (1)$$

Tests for tensile strength (TS) and shear strength (SS) parallel to grain of hydrothermally-treated rubberwood specimens were performed in accordance with the ISO-3345 (1975) and ISO-3347 (1976) methods, respectively, using a 10 kN universal testing machine (Lloyd, UK). For each treatment, a total of ten replicates were carried out. Information was also gathered to determine equilibrium moisture content (EMC) at 20 °C and 65%RH of specimens for each hydrothermal treatment.

Fungal Decay Test

Five hydrothermally-treated rubberwood specimens (20 mm × 20 mm × 20 mm) per treatment were subjected to fungal decay against the brown-rot decay fungus (*Gloeophyllum* sp.), identified from rubberwood, in accordance with the ASTM D1413-05. (2005) with some modifications. Soil block culture was prepared in screw-capped bottles. Rubberwood feeder strips (3 mm × 28 mm × 35 mm) were placed on the surface of the soil. After autoclaving at 121°C for 30 minutes, the feeder strips were inoculated with the fungal inoculum section from freshly grown culture. The bottles were incubated at 25 °C and 80%RH in an environmental chamber (Binder, Germany) for 2 weeks until mycelium of the decay fungus completely colonized the feeder strips. The specimens were then placed on the feeder strips in contact with fungal mycelium. The bottles containing the hydrothermally-treated specimens were then incubated at 25 °C and

80%RH for 12 weeks. After incubation, surface mycelium was removed. The specimens were reconditioned at 20 °C and 65%RH in a conditioning room (KBO, Sweden) to a constant weight. Average percentage fungal decay mass loss (FML) was determined from the conditioned weight before and after exposure to the decay fungus.

Termite Test

Five hydrothermally-treated rubberwood specimens (20 mm × 20 mm × 20 mm) per treatment were subjected to a termite bioassay according to a no-choice test procedure. Each specimen was placed in a glass bottle with 50 g sand, 8.5 ml DI water, and 1 g subterranean termites (*Coptotermes gestroi*). The bottles were then stored at 28 °C and 75%RH for 4 weeks. A visual rating of termite attack score (TAS) (0, failure; 4, heavy; 7, moderate; 9, light; and 10, sound) according to the ASTM D3345-74. (1999) was recorded for each specimen at the end of the test.

Experimental Design

The response surface methodology (RSM) combined with a two-factor, four level (4^2) full factorial design was employed to describe ΔE^*, EMC, TS, SS, FML, and TAS of hydrothermally treated rubberwood (dependent variables) as functions of two independent variables: treatment temperature (x_1) and logarithm of treatment time (x_2) as shown in Table 1. The behavior of the system was described using the following second-order polynomial,

$$Y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \beta_{ij} x_i x_j \tag{2}$$

where Y is the predicted response, β_o is the interception coefficient, β_i are the linear terms, β_{ii} are the quadratic terms, and β_{ij} are the interaction terms. The models of the six responses were expressed in terms of the two independent variables. The quality of fit was checked with the coefficient of determination R^2, and its statistical significance was determined by the F-test. The statistical analysis was performed using Statistica software (StatSoft, USA).

Table 1. Independent Variables and Level in a Two-factor, Four-level (4^2) Full Factorial Design

Factor		Level			
Temperature(°C)	x_1	100	120	140	160
Time (minutes)		30	120	360	720
ln(Time, minutes)	x_2	3.40	4.79	5.89	6.58

RESULTS AND DISCUSSION

The experimental data of color changes (ΔE^*), equilibrium moisture content (EMC), tensile strength (TS), shear strength (SS), fungal decay mass loss (FML), and termite attack score (TAS) are summarized in Table 2. ANOVA analysis was first employed to examine correlations among six dependent parameters regardless of treatment temperature and treatment time. Linear correlations were found between ΔE^* and TS, SS, and FML with R^2 of 0.89 (Fig. 1a), 0.81, and 0.90 (Fig. 1b), respectively. This is in agreement with the previous work of Bekhta and Niemz 2003, González-Peña and Hale 2009, and Todorović *et al.* 2012, who reported that physical and mechanical properties of thermally treated wood strongly correlate with color changes. In addition, the results presented within this work indicate that color changes could also be used as an indicator of fungal resistance property of the hydrothermally-treated wood (Fig. 1b). Correlations between ΔE^* and EMC and between ΔE^* and TAS, on the other hand, were very weak, with an R^2 of 0.40 and 0.11, respectively. As a result, color changes may not be a suitable indicator for those values.

Table 2. Experimental Data with Different Combinations of Treatment Temperature (x_1) and Logarithm of Treatment Time (x_2)

Run	Temp (°C) (x_1)	ln(Time,min) (x_2)	ΔE^* (n=10)	EMC (%) (n=10)	TS (MPa) (n=10)	SS (MPa) (n=10)	FML (%) (n=5)	TAS (n=5)
1	100	3.40	4.0	11.2	108.7	19.7	19.6	3.8
2	100	4.79	6.4	10.1	103.8	20.9	17.5	1.6
3	100	5.89	5.0	9.5	96.3	22.3	21.0	4.0
4	100	6.58	1.1	9.3	99.5	21.6	20.4	5.2
5	120	3.40	1.1	11.4	118.4	20.1	22.6	4.0
6	120	4.79	3.8	10.5	93.3	20.8	20.3	4.0
7	120	5.89	3.6	9.3	96.9	21.1	19.9	6.3
8	120	6.58	4.3	9.1	94.2	19.9	18.6	4.0
9	140	3.40	2.6	11.0	102.7	19.8	23.5	5.8
10	140	4.79	7.7	9.7	94.4	19.6	21.9	5.1
11	140	5.89	9.3	8.2	76.7	20.1	17.2	5.4
12	140	6.58	19.7	7.8	57.1	19.5	13.9	4.4
13	160	3.40	9.8	10.2	98.0	19.1	19.4	5.2
14	160	4.79	16.8	8.4	82.2	20.1	17.5	6.1
15	160	5.89	31.1	7.7	39.6	15.4	10.1	7.3
16	160	6.58	37.8	7.8	37.1	14.1	9.9	8.1
Control	-	-	-	11.9	109.1	19.3	19.9	3.2

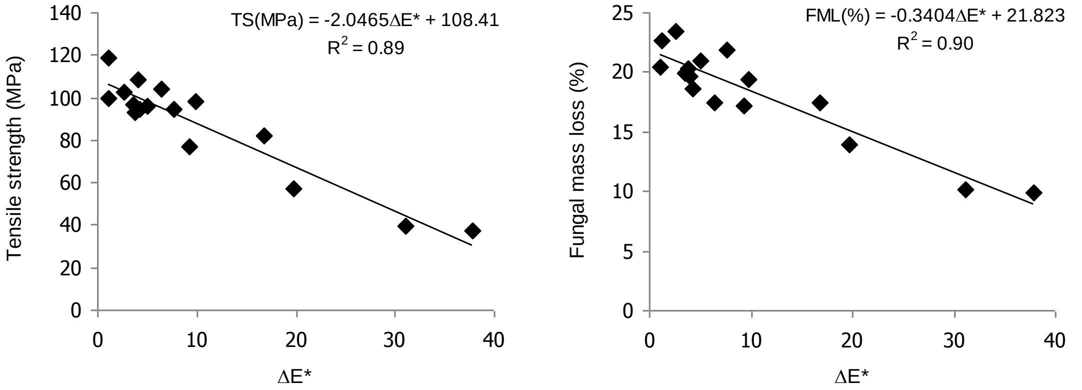

Fig. 1. Correlations between color changes (ΔE^*) and (a) tensile strength (TS) and (b) brown-rot fungal decay mass loss (FML) of hydrothermally treated rubberwood

Multiple regression coefficients using a second-order polynomial model for Δ*E**, EMC, TS, SS, FML, and TAS are summarized in Table 3. Corresponding *P*-values suggest that temperature (x_1), its cross product (x_1x_2), and its quadratic (x_1^2) are significant terms (*P*-values < 0.05) in the models describing Δ*E**, TS, and FML. Logarithm of treatment time (x_2) is also a significant term in the models describing Δ*E**, SS, and FML.

Table 3. Regression Coefficients of the Second Order Polynomial Equations

Factor	ΔE*	EMC	TS	SS	FML	TAS
β_o	222.2238[a]	9.7595[b]	-151.1538	-25.4762	-67.9114[a]	9.1650
β_1	-2.96120[a]	0.1078	3.6750[b]	0.4406[b]	1.1753[a]	-0.0600
β_2	-21.46190[b]	-1.3728	30.1433	8.8763[b]	8.6085[b]	-1.9068
β_{11}	-0.0094[a]	-0.0004	-0.0107[b]	-0.0012	-0.0035[a]	0.0004
β_{12}	0.1660[a]	-0.0041	-0.3001[a]	-0.0369[a]	-0.0674[a]	0.0029
β_{22}	0.3399	0.1108	-0.2541	-0.4372	-0.1618	0.1887

[a] *P*-Value <0.01 and [b] *P*-Value <0.05

The statistical significance of the six models checked with the *F*-test and the analysis of variance (ANOVA) is presented in Table 4. The model *F*-ratios for Δ*E**, EMC, TS, SS, FML, and TAS (Table 4) reveal that all six model terms were significant with *P*-values of less than 0.05. The goodness of fit represented by R^2 was found to be greater than 0.90 for the models describing Δ*E**, EMC, TS, and FML and greater than 0.80 for the model of SS. The parameter effects of linear terms, quadratic terms, and cross-product terms on the models of Δ*E**, SS, and FML and linear terms and cross-product terms on the model of TS were significant with *P*-values of less than 0.05. However, only the effect of the linear terms was predominant in the models describing EMC and TAS. This fact might be a reason why the values of EMC and TAS weakly correlate, while the values of TS, SS, and FML strongly correlate with the value of Δ*E** (Fig. 1).

Table 4. Analysis of Variance Shows the Effect of Processing Variables as a Linear Term, Quadratic Term, and Interactions on the Responses Considered

Source	Degree of freedom	Sum of squares	Mean square	F-ratio	Source	Degree of freedom	Sum of squares	Mean square	F-ratio
(a) ΔE^*					(b) EMC				
Model	5	1693.89	338.78	49.61[a]	Model	5	21.62	4.32	37.98[a]
Linear	2	1148.91	574.46	84.12[a]	Linear	2	20.71	10.36	90.94[a]
Quadratic	2	226.42	113.21	16.58[a]	Quadratic	2	0.72	0.36	3.17
Cross product	1	318.56	318.56	46.65[a]	Cross product	1	0.19	0.19	1.68
Residual	10	68.29	6.83	-	Residual	10	1.14	0.11	-
R^2	0.96				R^2	0.95			
(c) TS					(d) SS				
Model	5	7795.57	1559.11	28.09[a]	Model	5	56.32	11.26	12.69[a]
Linear	2	6459.57	3229.79	58.20[a]	Linear	2	33.25	16.63	18.73[a]
Quadratic	2	294.48	147.24	2.65	Quadratic	2	7.30	3.65	4.11[b]
Cross product	1	1041.52	1041.52	18.77[a]	Cross product	1	15.77	15.77	17.76[b]
Residual	10	554.99	55.50	-	Residual	10	8.88	0.89	-
R^2	0.93				R^2	0.86			
(e) FML					(f) TAS				
Model	5	217.59	43.52	19.98[a]	Model	5	22.51	4.50	3.34[b]
Linear	2	132.84	66.42	30.49[a]	Linear	2	21.42	10.71	7.92[a]
Quadratic	2	32.13	16.07	7.38[b]	Quadratic	2	1.00	0.50	0.37
Cross product	1	52.62	52.62	24.16[a]	Cross product	1	0.10	0.10	0.07
Residual	10	21.78	2.18	-	Residual	10	13.53	1.35	-
R^2	0.91				R^2	0.62			

[a] *P*-value <0.01 and [b] *P*-value <0.05

Response surface plots of ΔE^*, EMC, TS, SS, FML, and TAS of hydrothermally-treated rubberwood specimens as functions of temperature and logarithm of treatment time are shown in Fig. 2a-f, respectively. In general, hydrothermal treatment with higher temperature and longer treatment time appeared to increase the value of ΔE^* (Fig. 2a), reduce the values of EMC (Fig. 2b), adversely affect strength properties (reduction in values of TS and SS shown in Fig. 2c and Fig. 2d, respectively), improve fungal resistance (reduction in value of FML shown in Fig. 2e), and improve termite resistance (increase in the value of TAS shown in Fig. 2f). These effects are in general agreement with those according to thermal treatments reported elsewhere (Tjeerdsma *et al.* 2000; Boonstra *et al.* 2007a; Esteves and Pereira 2009). It should be noted that response surface plots of ΔE^*, TS, SS, and FML, in which the linear, the quadratic, and the cross-product terms are predominant, are similar in shape. It also appears from those plots that mild hydrothermal treatment at around 120 °C and 60 minutes slightly improved TS (Fig. 2c) and SS (Fig. 2d), while this treatment reduced fungal resistance with a slight increase in the value of FML (Fig. 2e). Mild thermal treatment has been reported to increase strength properties (Kubojima *et al.* 2000). For the surface plots of EMC (Fig. 2b) and TAS (Fig. 2f), the effect of predominant linear terms on the curves can be clearly seen.

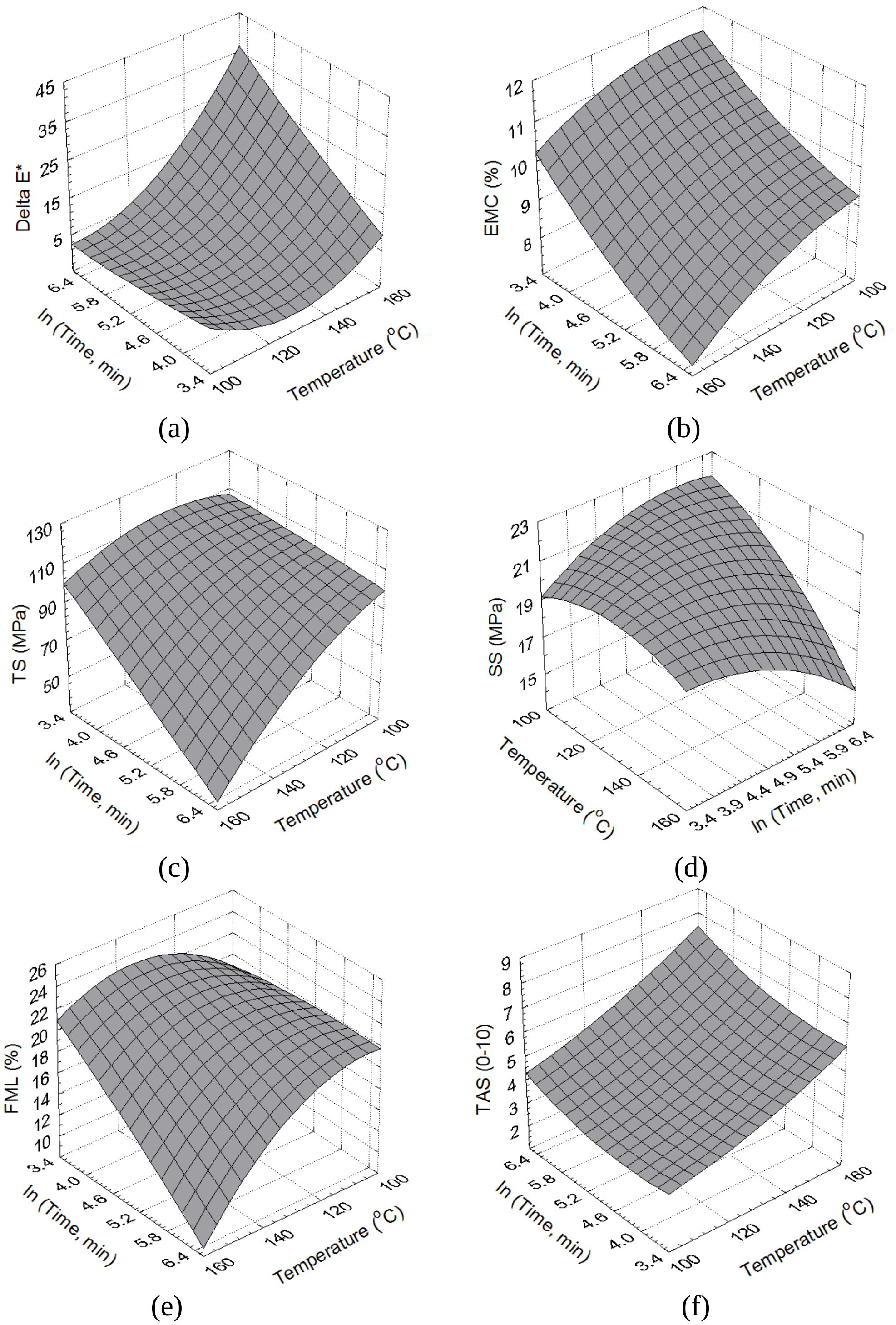

Fig. 2. Response surface plots of (a) color changes (Δ*E**), (b) equilibrium moisture content (EMC), (c) tensile strength (TS), (d) shear strength (SS), (e) fungal decay mass loss (FML), and (d) termite attack score (TAS) of rubberwood hydrothermally treated at temperatures between 100 °C to 160 °C for 30 to 720 minutes

Strong correlations between color changes (Δ*E**), strength properties (TS and SS), and brown-rot fungal resistance property (FML) might be explained as a direct consequence of hemicelluloses degradation during hydrothermal treatment. Color change of treated wood has been attributed to leaching and/or caramelizing of the cleaved products from hemicelluloses (Sehlstedt-Persson 2003; Sundqvist 2004; Boonstra and Tjeerdsma 2006). Degradation of hemicelluloses has been proposed to cause the decrease of the tensile strength and shear strength as a result of cleavage of the chemical bonds within hemicelluloses, between hemicelluloses and celluloses, and between hemicelluloses and lignin. These reduce load-sharing capacities of lignin-hemicelluloses matrix and cellulose microfibrils and/or fibrils (Boonstra *et al.* 2007a). Finally, glucose in hemicelluloses is reacted with enzyme glucose oxidase, produced by brown-rot decay fungi, to generate hydrogen peroxide which then oxidizes cellulose and modifies lignin (Ritschkoff 1996; Deacon 1997). Reduction in the amount of hemicelluloses as a result of hydrothermal treatment reduces the amount of hydrogen peroxide produced, which should therefore lead to a reduction of fungal mass loss observed (Boonstra *et al.* 2007b). On the other hand, both EMC and termite resistance (TAS) weakly correlated with the properties mentioned above and should not be solely a consequence of hemicelluloses degradation. The reduction of water adsorption has been suggested to be mainly the effect of increased cross-linking of the lignin network during hydrothermal treatment (Boonstra and Tjeerdsma 2006). Lignin has also been suggested to play a role as a barrier in protecting the polymer carbohydrates from termite attack (Geib *et al.* 2008). Increased cross-linking of the lignin network should therefore increase resistance to termites. Future research studies are needed, however, to clarify these issues.

CONCLUSIONS

1. The mathematical models describing color changes ($R^2 = 0.96$), equilibrium moisture content ($R^2 = 0.95$), tensile strength ($R^2 = 0.93$), shear strength ($R^2 = 0.86$), fungal resistance ($R^2 = 0.91$), and termite resistance ($R^2 = 0.62$) properties as functions of treatment temperature (100 to 160°C) and logarithm of treatment time (30 to 720 minutes) were obtained for hydrothermally-treated rubberwood.
2. Property changes as a result of hydrothermal treatment were more pronounced at higher temperature and at longer treatment time. Hydrothermal treatment improves durability against brown-rot decay fungi and termites but deteriorates tensile and shear strengths of rubberwood.
3. Strong correlations between color changes, tensile strength, shear strength, and brown-rot decay resistance properties of hydrothermally-treated rubberwood ($R^2>0.80$) were obtained. Degradation of hemicelluloses and its consequence during hydrothermal treatment was proposed to be responsible for the correlations observed.
4. Color change was a good indicator of tensile strength, shear strength, and brown-rot decay resistance of hydrothermally-treated rubberwood but it was not a good indicator of equilibrium moisture content and termite resistance properties.

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