MOISTURE-RELATED PROPERTIES OF *EUCALYPTUS TERETICORNIS* AFTER THERMAL MODIFICATION

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POONIA PK & TRIPATHI S. 2016. Moisture-related properties of *Eucalyptus tereticornis* after thermal modification. Heat treatment of wood is an alternative to chemical treatment. Wood structure changes due to decomposition of hemicelluloses, ramification of lignin and crystallisation of cellulose. These changes improve the hygrophobicity and dimensional stability of wood, increase its resistance to microorganisms and darken its colour. In this study, the effects of heat treatment on physical properties of *Eucalyptus tereticornis* wood were examined. Specimens were subjected to heat treatment at 210 ºC for 15 min, 30 min, 1 hour and 24 hours. Physical properties of heat-treated and control specimens were tested and their densities, weights, water absorption, swelling and shrinkage properties were measured. Results revealed that all parameters measured decreased with increasing treatment time.

Keyword: Heat treatment, shrinkage, swelling, weight loss, wood

INTRODUCTION

Wood is widely used in different applications. However, due to the hygroscopic nature of wood, it has some undesirable properties such as poor resistance against fungal and insect attacks as well as swelling and shrinkage caused by water adsorption and desorption. The service life of wood products mostly depends on their natural durability and a lot of research work has been carried out to enhance the durability of wood. Chemical treatment is the most widely used method for prolonging the service life of wood, but due to very toxic behaviours of these chemicals, namely, pentachlorophenol, copper chrome arsenate and creosote many countries ban their use in wood preservation (Drysdale 2002). Eco-toxicity concerns may restrict the use of chemicals, forcing the industry to opt for non-biocidal methods to improve durability and stability of wood.

Thermal treatment can be an alternative method for protecting wood against decay and insects without using preservatives (Homan & Jorissen 2004). Various thermal treatment methods have been commercialised in Europe (Vernois 2001, Militz 2002). Developed in France, retification involves slow heating of pre-dried wood (12% moisture content) from 210 to 240 °C in a nitrogen atmosphere with less than 2% oxygen content (Vernois 2001). Using this technique, durability increases with increase in temperature but it also induces more strength loss such as bending strength losses up to 40% (Homan & Jorissen 2004). Durability is dependent on species, temperature, time and accuracy of the process. Density, swelling and surface roughness decreased with increasing temperature and times of treatment (Korkut et al. 2008). In a study involving *Eucalyptus grandis* and *Eucalyptus* sp., when temperature rose from 200 to 220 °C, equilibrium moisture contents and wood densities were affected and volumetric contractions decreased (Araújo et al. 2012).

Maximum reductions in volumetric swelling for *E. grandis* wood is about 50% (Batista et al. 2011). Maximum reduction in swelling was obtained when thermal modification was at 230 °C and not 200 °C. *Eucalyptus globulus* and *Pinus pinaster* heat treated at 170 and 210 °C produced significant changes in chemical composition and structure of wood as well as in

lignin and polysaccharides contents (Esteves et al. 2013). Wood of *E. grandis* improved physical and calorimetric properties after heat treatment at 140, 170, 200 and 230 °C for 3 hours (Zanuncia et al. 2014).

Thermal treatment of wood is carried out at higher temperatures (150–240 °C) than those of conventional drying process. This treatment not only removes water from wood but also causes significant modification in chemical composition. Degradation of wood components takes place through dehydration, hydrolysis, oxidation, decarboxylation and transglycosylation, and the wood becomes less hygroscopic (Kocaefe et al. 2008). Water re-absorption decreases and dimensional stability increases during heat treatment (Pavlo & Niemz 2003). Hemicelluloses are the most reactive component of the cell wall. They degrade at lower temperatures (160–220 °C) than the other components due to their relatively low molecular weight (Fengel & Wegener 1989). Ramification of lignin occurs (Nuopponen et al. 2004) and the relative amount of crystalline cellulose increases due to degradation of amorphous components at this temperature range. This affects the hardness of wood. However, certain mechanical properties of wood decrease during heat treatment. Decomposition of long-chain polymers decreases elasticity and makes the wood more brittle. Moisture content of wood and some water-soluble products of heat treatment, such as organic acids, promote degradation of long-chain polymers by hydrolysis (Fengel & Wegener 1989).

Eucalyptus tereticornis is one of the most important fast-growing plantation species in India and was the first eucalypt exported from Australia. This species is now cultivated throughout the tropics and on large scale in India and Brazil. *Eucalyptus tereticornis* wood has densities of 660–1060 kg m^{-3} and rates of shrinkage from green to oven dry are high, i.e. 4.2–10.6% radially and 7.4–13.5% tangentially. The wood has strong tendency to warp during drying (Brink 2008). Improvement of the properties of *E. tereticornis* in an eco-friendly way is one of the biggest challenges for wood technologists. The objective of this study was to investigate the effects of heat treatment duration on physical properties of *E. tereticornis*.

MATERIALS AND METHODS

Preparation of wood specimens

Fifteen-year-old *E. tereticornis* trees were harvested from the Forest Research Institute, Dehradun, India (30° 19' N, 78° 04' E). Planks (62.5 mm thick) were cut along the full log width and lengths and air seasoned. The seasoned planks were cut into different dimensions according to IS (1986), i.e. for determination of density (2 cm × 2 cm × 6 cm), moisture content (2 cm × 2 cm × 2.5 cm), volumetric swelling and shrinkage (2 cm × 2 cm × 6 cm), radial and tangential swelling and shrinkage (2 cm × 2 cm × 5 cm) and water absorption (2 cm × 2 cm × 6 cm). A total of 180 these cut specimens were randomly collected for treatment.

Heat treatment

Specimens for mass loss were oven dried to constant weight at 105 °C (initial dry weight), while other test specimens were conditioned at 21 °C and 65% relative humidity. Six specimens for each test were used as control, while another set of six was heat treated at 210 °C in nitrogen atmosphere with less than 2% oxygen content for 15 min, 30 min, 1 hour or 24 hours. The specimens were treated in a tightly closed heating unit. Vacuum (75 kPa) was applied for 15 min followed by nitrogen gas flushing into the heating chamber. Temperature of heating chamber was raised to 210 °C and held at that temperature for the desired time. The unit was allowed to cool to 25 °C.

Physical properties

Treated samples were characterised and compared with untreated control specimens based on colour. Presence of any treatment defects such as checks and cracks was noted.

After treatment, the specimens were cooled to room temperature and weighed to determine mass loss on oven dry basis. Initial moisture contents of conditioned samples were determined using parallel sets of samples that were oven dried at 105 °C and weighed (Esteves et al. 2008). Density was measured

before and after treatment on air dry basis (Korkut et al. 2008).

Water absorption and dimensional stability (measured by tangential, radial and volumetric swelling) were determined following IS (1986) with some modifications. Dimensions and weights were measured prior to and after submersion in water using vernier callipers and digital balance respectively. Samples were submerged horizontally in water (20 ± 2 °C) and removed after 72 hours, suspended vertically in air to drain for 10 min and the excess surface water was wiped off with dry cloth. The amount of water absorbed and thickness swelling were calculated as the percentage of difference in weight and dimension before and after submersion.

Radial, tangential and volumetric shrinkage were determined as per IS (1986). Specimens were weighed and their dimensions measured to the nearest 0.002 cm. The specimens were allowed to air season and periodically weighed and measured until moisture content of 12% was reached. The specimens were oven dried at 103 ± 2 °C and weighed and measured again. Radial, tangential and volumetric shrinkage values were calculated from differences in dimensions before and after oven drying and expressed as percentage of the initial dimensions.

Statistics

The effects of thermal treatment on physical properties were determined using analysis of variance (ANOVA) in SPSS15.0 version. Homogeneity test was carried out to analyse the significance and mean separation of the treatments (Scheffé 1959).

RESULTS AND DISCUSSION

The colours of heat-treated specimens changed from pale reddish to dark brown or chocolate (Figure 1). Colour became much darker when treated at 210 °C for 24 hours. Other schedules also produce colour changes but they were difficult to assess visually. Cellulose and hemicelluloses in untreated wood do not absorb light in the visible region and do not contribute to changes in the colour of *E. tereticornis* wood (Kocaefe et al. 2008). The dark colour of wood after heating for 24 hours occurred due to formation of byproducts during degradation of hemicelluloses (Kocaefe et al. 2008). Oxidation of polysaccharides and lignin produce phenolic compounds that can also induce colour change (Fengel & Wegener 1989). Decrease in hemicellulose content reduces lightness and increases colour intensity (Bekhta & Niemz 2003). The darker colour in treated *E. tereticornis* can be an aesthetic advantage for certain applications.

There was positive correlation ($r^2 = 0.79$, $p < 0.01$) between weight loss and treatment time (Table 1), meaning weight loss gradually increased with treatment time (Table 2). Decrease in weight during heat treatment is due to losses in extractive substances and degradation of hemicelluloses (Korkut et al. 2008). Density of wood decreased with increasing treatment time

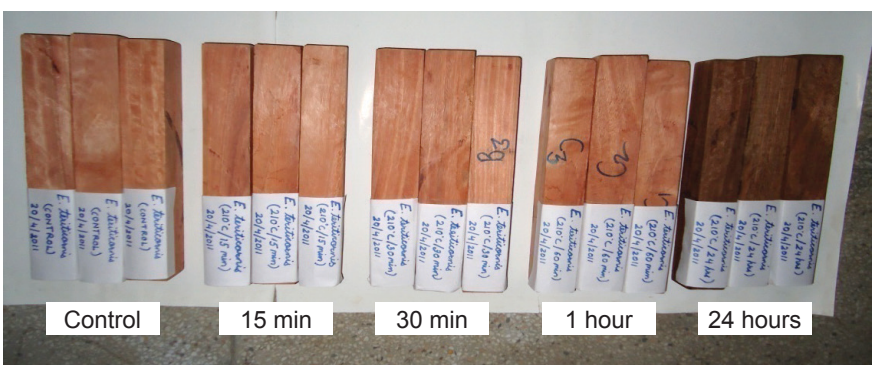

Figure 1 Colour changes of *Eucalyptus tereticornis* samples heated at 210 °C for different times

Table 1 Correlation between physical properties of thermally-modified *Eucalyptus tereticornis* wood at 210 °C for different durations

	Time	Weight loss	Loss of density	Water absorption	Tangential swelling	Radial swelling	Volumetric swelling	Volumetric shrinkage	Tangential shrinkage	Radial shrinkage
Time	1.00	0.79**	0.98**	-0.98**	-0.85**	-0.79**	-0.88**	-0.82**	-0.84**	-0.83**
Weight loss		1.00	0.83**	-0.75**	-0.67**	-0.75**	-0.69**	-0.81**	-0.61**	-0.74**
Loss of density			1.00	-0.95**	-0.85**	-0.82**	-0.86**	-0.84**	-0.84**	-0.84**
Water absorption				1.00	0.85**	0.79**	0.88**	0.78**	0.81**	0.81**
Tangential swelling					1.00	0.74**	0.67**	0.61**	0.83**	0.72**
Radial swelling						1.00	0.66**	0.74**	0.70**	0.78**
Volumetric swelling							1.00	0.77**	0.70**	0.78**
Volumetric shrinkage								1.00	0.64**	0.74**
Tangential shrinkage									1.00	0.80**
Radial shrinkage										1.00

**Correlation is significant at the 0.01 level (2-tailed)

Table 2 Effects of duration of heat treatment at 210 °C on physical properties of *Eucalyptus tereticornis* wood

Physical property	Mean value at different treatment times				
	Control	15 min	30 min	1 hour	24hours
Weight loss (%)	0	0.30	0.38	0.53	2.91
Density(g cm^{-3})	0.84	0.82	0.80	0.77	0.74
Water absorption (%)	39.19	31.82	27.79	23.91	19.68
Tangential swelling (%)	7.74	6.65	6.13	5.85	5.08
Radial swelling (%)	4.27	3.47	3.40	3.28	2.21
Volumetric swelling (%)	6.32	6.03	5.34	4.95	4.39
Volumetric shrinkage (%)	9.18	8.83	8.64	8.02	6.64
Tangential shrinkage (%)	8.12	7.72	6.53	5.77	5.42
Radial shrinkage (%)	5.31	5.18	4.26	4.06	3.06

Critical difference$_{(0.05)}$of loss in density = 0.37, water absorption = 0.54, volumetric swelling = 0.23, volumetric shrinkage = 0.30, radial swelling = 0.24, radial shrinkage = 0.31, tangential swelling = 0.31, tangential shrinkage = 0.40 and weight loss = 0.15, values are highly significant at $p < 0.05$

($r^2 = 0.984$, $p < 0.01$). Samples treated at 210 °C for 24 hours experienced the highest density loss, i.e. 11.9% compared with the rest of the treatment times (Table 2). Densities of treated samples were significantly lower compared with control.

Water absorption of control was significantly higher than treated samples (Table 2). Water absorption of thermally-treated samples ranged from 19.68 (210 °C, 24 hours) to 31.82% (210 °C, 15 min) after 72 hours being dipped in water. Relative to control which gained 39.19% weight,

water absorption in thermally-treated samples was reduced by 7.37–19.51%. Heat treatment produced substantial reduction in hygroscopicity of treated materials after 24 hours. Negative correlation (-0.982, $p < 0.01$) was observed between water absorption and treatment time, i.e. water absorption capacity of treated samples was reduced (Table 1). Reduction in water absorption after thermal treatment was also reported by Kocaefe et al. (2007). Modified lignin decreases water absorption, consequently reducing the shrinkage and swelling of wood (Kollmann & Schneider 1963).

Wood swelling occurs as water is absorbed by the cell wall. Heat treatment resulted in decreased water absorption which ultimately reduced swelling. Swelling of treated samples in the tangential direction ranged between 5.08–6.65% while that of control, 7.74%. At 210 °C, the highest tangential swelling of treated samples was achieved by wood treated for 15 min and the lowest, 24 hours (Table 2). In general, tangential swelling decreased as treatment duration increased. Radial swelling in control specimens was 4.27% while that of treated samples ranged from 3.47% (15 min) to 2.21% (24 hours). Radial thickness swelling of all treated samples exhibited similar trends to tangential thickness swelling except that the values were lower due to different cell arrangement (Maruzzo et al. 2003). Values for volumetric swelling were between 4.39 and 6.03% compared with control (6.32%). For treated samples, highest volumetric swelling was observed for samples treated for 15 min and the lowest, 24 hours. Reduction in volumetric swelling in treated samples were significantly lower ($p < 0.05$) compared with control samples. Decreased swelling increases dimensional stability, which is required for several applications of wood.

Wood shrinks as moisture declines below the fibre saturation point. Volumetric shrinkage values of treated samples were lower (8.83–6.64%) than control (9.18%). Tangential shrinkage values of treated samples were between 5.42 and 7.72%, while control was 8.12%. Radial shrinkage values of treated samples were in the range of 5.18–3.06%, lower than control (5.31%). Highest shrinkage for treated samples was achieved after 15 min treatment while the lowest, 24 hours (Table 2). Reduction in mean shrinkage of treated samples was significantly ($p < 0.05$) lower compared with control. Fibre saturation point decreases because there is less water in the cell wall after heat treatment (Repellin & Guyonnet 2005).

Generally, this study showed that *E. tereticornis* had decreased water absorption as well as reduced swelling and shrinkage with increasing heat treatment time. Treatment time was positively correlated with density and weight loss but was negatively correlated with water absorption as well as volumetric, radial and tangential swelling and shrinkage.

CONCLUSIONS

Heat modification of *E. tereticornis* produced increased mass loss, decreased water absorption as well as reduced swelling and shrinkage with increasing treatment time. Heat treatment modified the colour of samples to produce darker and more even appearance. The results suggested that thermal modification was useful to improve some properties of this species.

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