Effect of Different Hydrothermal Treatments (Steam and Hot Compressed Water) on Physical Properties and Drying Behavior of Yellow-Poplar (*Liriodendron tulipifera*)

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Abstract

Nonchemical high-pressure steam treatments have been intensively researched and commercialized to produce chemical-free wood products with enhanced properties. However, the utilization of high-pressure steam involves vapor-phase reactions using high-temperature steam generated at the expense of high energy input. In this research, influences of reaction media (steam and hot-compressed water) and temperature (100°C and 140°C) during thermal treatment on physical properties and drying behavior of yellow-poplar (*Liriodendron tulipifera*) heartwood were compared. The length, width, and thickness of the samples were 22.53 mm, 17.18 mm, and 16.72 mm, respectively. After the treatment, the samples were dried under an isothermal temperature condition of 105°C. Data on moisture content and time of drying from drying experiments were fitted with unsteady-state molecular transport equations to calculate overall liquid diffusion coefficients. Dimensions, weight, and true volume of samples were measured for green, thermally treated, and dried samples and the values were used to calculate selected physical characteristics. Additionally, selected mechanical properties were evaluated for samples conditioned to 13 percent moisture content. Results showed that intensified hot-compressed water-treated and control samples had the highest and lowest saturated moisture contents (101% and 44%), respectively, immediately after treatments. Intensified steam-treated and control samples had the highest and lowest total porosity (95% and 82%), respectively. Furthermore, mild hot-compressed water-treated samples showed the greatest compression strength (47.8 MPa) at 13 percent moisture content. Except for steam treatment at 140°C, other treatments significantly decreased the diffusion coefficient. Collectively, samples treated with hot-compressed water at 100°C showed the most improved mechanical properties.

Wood drying accounts for approximately 40 to 70 percent of the total energy consumed in wood processing operations (Zhang and Liu 2006). For this reason, research efforts have been focused on fast-drying methods with high drying quality while maintaining physical properties of dried wood. In addition to improvement of the drying process itself, numerous studies have been devoted to treating wood prior to drying. For example, to improve physical properties and decay resistance of wood, several treatments have been explored, including high-pressure steam (Dashti et al. 2012a, 2012b; Peng et al. 2012; Sayar and Tarmian 2013), steam explosion (Zhang and Cai 2006), ultrasonic treatment (He et al. 2013, 2014), and heat treatment (Rousset et al. 2004). Dashti et al. (2012a) reported that microwave treatment on aleppo oak (*Quercus infectoria*) successfully eliminated the tyloses structure and improved air permeability. However, steam treatment did not alter the tyloses structure. Similarly, Alexiou et al. (1990) documented no change in tyloses structure due to

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presteaming on eucalyptus (*Eucalyptus pilularis*). Wet-wood-related issues have also been researched using steam-explosion treatment of Japanese cedar (*Cryptomeria japonica*; Kanagawa et al. 1992) and subalpine fir (*Abies lasiocarpa*; Zhang and Cai 2006). Additionally, steaming processes and specific drying schedules have been recommended to mitigate issues related to false heart (Trenciansky and Hansmann 2007). Hill (2006) indicated that out of all the modification processes, thermal treatment in presence of steam or liquid water (also referred as hydrothermal treatment) is currently the most commercially advanced method to boost dimensional consistency and improve decay resistance.

Hydrothermal treatments may be performed in presence of steam or liquid water under either pressure or vacuum at various temperatures. This treatment has the potential to affect physical characteristics, mechanical properties, and drying behavior of wood. Influences on wood properties vary based on temperature, pressure, holding time, number of cycles, and medium. Physical characteristics of interest are moisture content (MC), specific gravity (SG), porosity, and shrinkage or swelling. In general, steam treatments have been reported to reduce the MC of hardwoods. Peng et al. (2012) applied steam treatment on poplar (*Populus* sp.) at 100°C and 140°C and observed greater MC reduction after steaming at 140°C than 100°C. Similarly, Cai (2006) reported a reduction in MC after steam-explosion treatment of subalpine fir in the temperature range of 120°C to 160°C. The superheated steam at 140°C acted like a drying medium while steam at 100°C was completely saturated and did not have any drying potential. Specific gravity or density is perhaps the most important physical property of wood, which is affected by mass loss during heat (hydrothermal) treatment. Mass loss during hydrothermal treatment depends on several factors, such as wood species, heating medium, temperature, and treatment time (Esteves et al. 2009). Scheiding et al. (2016) performed hydrothermal treatment at 190°C and 210°C for 3 hours on Scots pine (*Pinus sylvestris*). The mild and strong modification actions decreased and increased the water uptake, respectively. They indicated that cracks along the middle lamella, intercellular cavities, and a degradation of wood cells occur at 190°C, which results in increased permeability and water uptake. Nevertheless, the thermoplastic flow of the torus above 200°C might result in filling of wood cells and could explain decreased water uptake at 210°C. The second reason could be the loss of methoxyl groups of the aromatic ring. Mazela et al. (2003) studied the mass loss in pine species (*Pinus sylvestris*) at 160°C, 190°C, and 220°C during 6-hour and 24-hour treatment periods in the presence and absence of vapor. They observed similar mass loss in different heating media after 6 hours. However, after 24 hours the mass losses in air were greater than in vapor media, especially at temperatures of 190°C and 220°C. In another study, Bourgois and Guyonnet (1988) reported that mass loss of maritime pine (*Pinus pinaster*) treated at 260°C was 18.5 percent for 15-minute treatment durations and 30 percent for 60-minute treatment periods.

Hydrothermal treatments not only affect density or specific gravity, but they also alter ultrastructure and pore characteristic. Wood is a porous material with micropores, mesopores, and macropores present in varying proportions. The porosity of wood largely depends on the density and anatomy of the wood species. Typically, porosity or fractional pore volume of wood ranges from 54 to 80 percent for hardwoods with a green specific gravity of 0.30 to 0.70 (Siau 1995, Plotze and Niemz 2011). Biziks et al. (2013) provided a pioneering study on how anatomical properties (growth rings, cell wall, lumens, and fibers) of birch wood change during hydrothermal treatment. They showed that hydrothermal treatment at 180°C for 1 hour could break up the integrity of wood's morphological structure. Zhang and Cai (2006) reported that 6 to 13 percent of samples treated through steam explosion at 160°C collapsed during posttreatment kiln drying, indicating that the treatment significantly altered the cell wall structure. Hydrothermal treatments have also been reported to influence mechanical properties (modulus of elasticity and strength). Specifically, steam explosion contributed to the reduction in modulus of elasticity and modulus of rupture of wood samples (Cai 2006, Zhang and Cai 2006).

While hydrothermal treatments affect physical, structural, and mechanical properties, they also influence moisture diffusion coefficient during drying and, therefore, drying time (Dashti et al. 2012a, Tarmian et al. 2012). Peng et al. (2012) report that steam treatment prior to vacuum-drying reduces overall drying time of poplar and Manchurian walnut (*Juglans mandshurica*). They compared the influence of steam treatments at 100°C and 140°C on drying rate and time in postvacuum drying. They report that initial drying rates were faster for untreated samples due to high initial moisture content as compared with the steam-treated samples whose initial moisture decreased significantly during steam treatment. In addition, more reduction in MC occurred when steam treatment was performed at 140°C than at 100°C. This occurrence was related to superheated steam at 140°C acting like a drying medium, whereas 100°C steam was effectively saturated and did not have the potential to gain extra moisture vapor. However, below 25 percent MC, drying rates of untreated samples were lower than steam-treated samples because steam treatments enhanced vapor permeability of wood by damaging bordered pits, aspirated pits, and chemical structure of the cell-wall. Murata et al. (2013) indicated that the fiber saturation point of spruce (*Picea* sp.) wood decreased when treated above 150°C. Likewise, steam-explosion treatment has been shown to significantly improve the dryability of subalpine fir lumber because bordered pits among early-wood tracheids were destroyed (Cai 2006, Zhang and Cai 2006). Zhang and Cai (2006) suggested that some fractures transpired in bordered pit pairs between tracheids at 130°C. At 160°C, more fractures happened in pit pairs between earlywood tracheids. However, no damage occurred in latewood because of its small diameter of bordered pits in tracheids walls and thick pit membrane in margo and torus compared with those of earlywood. Margo and torus regions within wood have a large amount of cellulose-based strands (Hoadley 1980), which fracture during steam explosion. Ma et al. (2015) reported a general increased drying rate (above and below fiber saturation point [FSP]) and effective water-diffusion coefficient in poplar samples that were treated five times with a pressure of 1.0 MPa. Ma et al. (2016) claimed that the large capillary system of microexplosion-treated samples was broadened and resulted in a decreased resistance to moisture flow. However, research by Sayar and Tarmian (2013) reported that neither temperature nor treatment time had any effect on water-vapor diffusion

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coefficients for poplar samples because the treated samples showed lower water-vapor diffusion coefficients.

Most of the referenced research on hydrothermal treatments was performed in steam media, so it is anticipated that hydrothermal treatment in liquid medium would likely have a different effect on the physical characteristics and drying behavior of wood. Specifically, liquid water movement is dominated by capillary (convective) flow, whereas vapor water movement is dominated by diffusive flow, which is more random and less uniform. Fluid movement tends to be more uniform in diffuse porous low-density hardwoods (such as yellow-poplar [*Liriodendron tulipifera*]) than in ring porous hardwoods (Siau 1995). Given the uniformity in fluid movement within hardwoods, the objective of this research was to compare the effect of hot-compressed water (HCW) and steam treatments at 100°C and 140°C on the selected physical characteristics, mechanical properties (modulus of elasticity and compressive strength), and drying behavior of yellow-poplar.

Materials and Methods

Procurement and processing of raw material

Yellow-poplar test samples were selected given the prevalence of yellow-poplar harvesting (25.5%) in West Virginia (Grushecky et al. 2012) and historical use by the forest products industry (Wiemann 2010). A freshly cut yellow-poplar log (diameter = 175 ± 15 mm, length = 900 ± 20 mm) was procured from the West Virginia University Research Forest. Three discs with average thickness of 23 ± 1 mm were cut from the log (Fig. 1a). From the discs, 78 small rectangular samples were cut from the heartwood region according to the cutting pattern shown in Figure 1b. The approximate distances between the pith (i.e., center of the disk) and border of juvenile and mature heartwood were 32 and 60 mm, respectively. Heartwood was chosen because of its rich extractives, which were expected to redistribute during the hydrothermal treatment. The redistribution of extractives was expected to make positive significant changes in physical characteristics and drying behavior (Hoadley 1980, 1990). Typical dimensions of the samples were 17.18 mm radial, 16.72 mm tangential, and 22.53 mm longitudinal. Sample cutting was performed using a bandsaw (Rockwell Model 28-350; Pittsburgh, Pennsylvania, USA). Immediately after cutting, the samples were held in deionized water to avoid loss of green moisture, and the water was replaced every 48 hours to prevent microbial spoilage. Six specimens were used as control. Other samples were randomly assigned to a hydrothermal treatment group (18 samples per group). Each experiment was replicated three times (6 samples per replication). Hydrothermal treatments were a combination of following two treatment factors: Factor 1: medium (hot-compressed water and steam) and Factor 2: temperature (100°C and 140°C).

The intention was to prepare only heartwood specimens because of the small diameter of the log; however, some samples (11 out of 78) were taken in proximity to the first 30 growth rings (first 15 yr) and potentially represented some juvenile wood. The juvenile wood generally has different physical properties from those of mature wood (Bowyer et al. 2007). Therefore, specific gravity of those samples was compared to make sure that there was no significant heterogeneity among 78 experimental units. The average green specific gravity of the 11 samples and remaining 67 samples was the same (0.43), while oven-dry specific gravity values were 0.49 and 0.50, respectively. Furthermore, statistical test (Shapiro-Wilk) was performed to check normality of each replication having some of those 11 samples. The Shapiro-Wilk test results did not show any lack of normality among the replications.

Measurements

Each sample was weighed with accuracy of 0.01 g using a weighing balance (Model P603DMDS; Denver Instrument, USA). True volume of the samples (excluding pore volume) was measured using a pycnometer (Model Manual Multipycnometer; Qunatachrome, Florida, USA) using the fluid (nitrogen) displacement method. Length, width, and depth dimensions of cubical samples were measured using a caliper (Model ROHS NORM 2002/95/EC; Digimatic, Mitutoyo, Japan) with accuracy of 0.01 mm at three locations. The identical sets of measurement were done prior to hydrothermal treatments, before drying, and after drying.

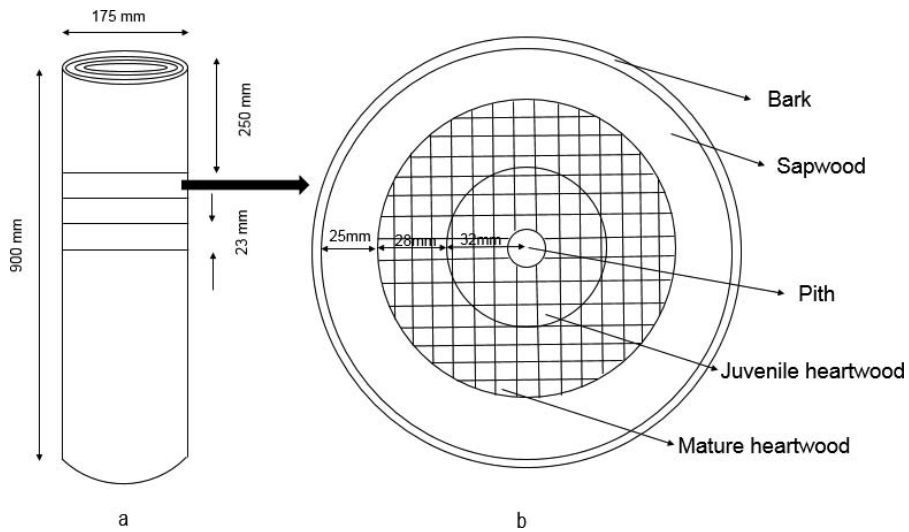

Figure 1.—(a) Cutting pattern of the discs from a log. (b) Cutting pattern of the cubic samples from a disc.

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Hydrothermal Experiments

A combination of two treatment factors (medium and temperature) resulted in the following four types of hydrothermal treatments: (1) Hot-compressed water (HCW) at 100°C (mild HCW); (2) steam at 100°C (mild steam); (3) HCW at 140°C (intensive HCW); and (4) steam at 140°C (intensive steam). However, to determine the temperature for intensive treatment, several trials were performed prior to actual experiments for temperatures ranging from 100°C to 190°C. From the results of trials, 140°C was chosen because all the samples treated in HCW above 140°C (190°C, 170°C, and 150°C) became dark-colored, brittle, and fragile (Fig. 2).

Both the hot compressed water (HCW) and steam media treatments were performed inside a 1-L sealed pressure reactor (Model 4500; Parr Instrument Company, Moline, Illinois, USA). To create HCW medium, the test samples were kept submerged in the hot water during the experiments. For a typical HCW experiment, six specimens were placed inside the pressure reactor. Distilled water was then added to the reactor in an amount (seven times the weight of the specimens) necessary to keep the specimens submerged in water (Singh Seehra et al. 2015). The reactor weight containing wood specimens and water was recorded before and after each experiment. The reactor was sealed and heated to the desired temperature. During treatment, the room's temperature was 22°C. Heating took 20 and 27 minutes for the mild and intensive treatments, respectively. All hydrothermal treatments were performed for a holding time of 60 minutes. To perform treatments in the steam medium, a perforated plate was placed inside the reactor vessel just above water table and all the specimens were placed on the perforated plate without touching the water. During treatment, temperature and pressure were recorded (represented as functions of time in Fig. 3). In the experiments, pressure was not controlled, but rose as the result of rise in temperature in a constant volume. The temperatures of water and steam were equal at the same treatment intensity (100°C for mild and 140°C for intensive), which matched the furnace temperatures. To achieve these temperatures, the furnace was set at, for example, 140°C, and temperatures recorded by the thermocouple (placed inside the thermal well submerged in the water) were monitored along with the furnace temperature. During the holding times both the furnace and water temperatures were the same. The reactor was placed inside the furnace, so it was assumed that the temperature of the headspace containing steam was equivalent to the water temperature.

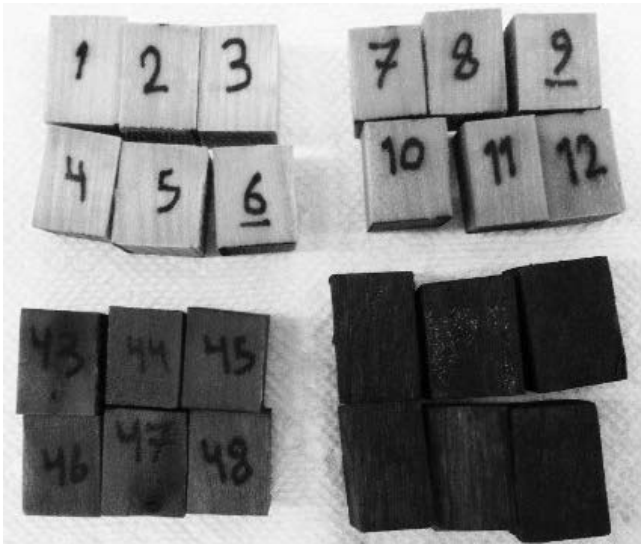

Figure 2.—Yellow-poplar control samples (upper left) and HCW-treated samples at 100°C (upper right), 140°C (lower left), and 140°C (lower right). HCW is hot-compressed water.

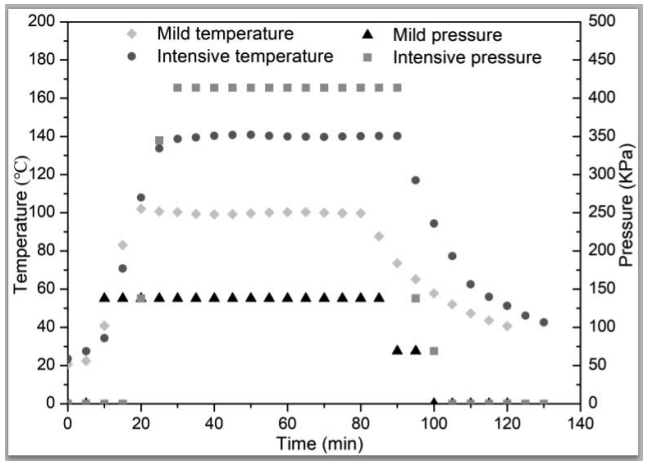

Figure 3.—Temperature and pressure as a function of time over the period of hydrothermal treatment.

After a hydrothermal treatment was complete, the reactor was cooled to room temperature. Cooling required approximately 40 and 50 minutes for the mild and intensive treatments, respectively. Following cooling, the treated samples were collected from the reactor and wiped to remove excess surface water. Weight and dimensions of all the samples were measured and recorded.

Drying and postdrying measurements

The hydrothermally treated samples were dried in a ThermoGravimetric Analyzer (TGA; Model LECO 701; LECO Corporation, St. Joseph, Michigan, USA) at isothermal temperature at 105°C. Drying was done in nitrogen conditions to avoid the effect of humidity. During each drying experiment, data on time, temperature, and weight of the samples were continuously measured and recorded.

After drying, the samples were immediately placed under water for 24 hours. After the 24-hour period, weight and dimensions were measured again to determine water absorption and volumetric swelling of the treated samples. Lastly, compression testing was performed using an Instron Model 825 Digital Electronic Instron/MTS machine (Norwood, Massachusetts, USA and Minneapolis, Minnesota, USA) to determine modulus of elasticity and compression strength of the samples following a modified ASTM D143-14 (ASTM International 2011) procedure (because the samples were smaller than specified in ASTM D143). Prior to the compression test, the samples were placed inside the conditioning chamber at 20°C and relative humidity of 65 percent to reach an estimated target equilibrium moisture content of 12 percent. Samples were weighed before and after being placed in the conditioning chamber to evaluate MC. After conditioning, the average specimen MC for the

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control samples was 12.61 ± 0.68 percent, whereas it was 13.54 ± 0.68 percent and 13.11 ± 0.98 percent for hot compressed water- and steam-treated samples, respectively.

Evaluation of physical properties and drying behavior

Physical characteristics of the samples, including moisture content, specific gravity, dimensional changes, total porosity, and water sorption and mechanical properties (compression strength and compressive modulus of elasticity), were calculated using standard equations (Siau 1995). Furthermore, yield of the treated samples was calculated.

Porosity is fractional void volume of wood expressed in percentage at a given MC (Siau 1995), which can be calculated by the following equation:

$$\mu_{\text{total}} = \frac{V - V_{p0}}{V} \times 100 \tag{1}$$

where μ_{total} = total porosity (%), which is equal to fractional volume occupied by air plus water fraction in green condition V_{p0} = oven-dry true volume of wood cell mass (cm^3).

In addition, drying behavior of the samples, including moisture ratio, drying rate, and diffusion coefficient, were evaluated and compared (Geankoplis 2003).

Using weight data measured by TGA 701 at various times, MC of the samples during drying time was calculated. Then, moisture ratio was calculated, applying Equation 2 (Chen et al. 2012) as given below:

$$MR = \frac{MC - MC_{final}}{MC_{initial} - MC_{final}} \tag{2}$$

where
MR = moisture ratio;
MC = current moisture content (%);
MC_{final} = moisture content at the end of drying (%);
and $MC_{initial}$ = moisture content at the beginning of drying (%).

Then, moisture ratio (MR) as a function of time elapsed was plotted for treated and untreated samples.

Drying rate (R) was calculated using Equation 4 (Geankoplis 2003). Then R was plotted as a function of MR, which was used to calculate overall liquid diffusion coefficient.

$$X_i = \frac{MC}{100} \tag{3}$$

where X_i (kg of water per kg of dry solid) is the moisture fraction at ith time.

$$\mathrm{R} = \frac{dX}{dt} = \frac{1}{100} \times \frac{MC_{t1} - MC_{t2}}{t_2 - t_1} \frac{kgwater}{kgdrysolid.h} \tag{4}$$

where R (kg water per kg dry solid per hour) is the drying rate at the ith time.

Drying behavior comparison was performed by comparing graphs between dX/dt and mean moisture fraction ($[X_1 + X_2]/2$).

Unsteady-state molecular transport equation for mass is written as following for three dimensions:

$$\frac{\partial X}{\partial t} = D_L \left(\frac{\partial^2 X}{\partial x^2} + \frac{\partial^2 X}{\partial y^2} + \frac{\partial^2 X}{\partial z^2} \right) \tag{5}$$

where D_L (m^2/s) is the overall liquid diffusion coefficient in each dimension and x, y, and z are the diffusion lengths (m).

Assuming that initial moisture distribution is uniform at $t = 0$, a simple solution to above equation (Eq. 4) may be written as follows:

$$\frac{X}{X_1} = \frac{8}{\pi^2} \left[e^{-D_L t(\pi/2x_1)^2} + e^{-D_L t(\pi/2y_1)^2} + e^{-D_L t(\pi/2z_1)^2} \right] \tag{6}$$

where X_1 is the initial fractional MC, and x_1, y_1, and z_1 are half of the length, width, and depth (m) of the sample. Solving above equation (Eq. 5) for time of drying will result in Equation 6.

$$t = \frac{4}{\pi^2 D_L} \ln\left(\frac{8X_1}{\pi^2 X} \right) \left[x_1^2 + y_1^2 + z_1^2 \right] \tag{7}$$

Differentiating Equation 7 with respect to time and rearranging resulted in the following expression:

$$\frac{dX}{dt} = -\frac{\pi^2 D_L X}{4\left[x_1^2 + y_1^2 + z_1^2 \right]} \tag{8}$$

Therefore, slope of dX/dt versus X will be $\frac{\pi^2 D_L}{4[x_1^2+y_1^2+z_1^2]}$. The overall liquid diffusion coefficient is calculated from the slope. The values of X range from the FSP to zero. With each temperature increase of 1°C, FSP decreases by 0.1 percent. The FSP at different temperatures can be determined using the following relation:

$$FSP = [0.3 - 0.001(T - 20)] \times 100 \tag{9}$$

where FSP = the fiber saturation point (%), and T = the temperature (°C).

Hence, when the temperature is at 20°C, the value of FSP is equal to 30 percent. In similar fashion, at 105°C, the value of FSP is assumed to be 21.5 percent (Stamm and Loughborough 1935, Siau 1995, He et al. 2012).

Statistical analysis

Two-way analysis of variance (ANOVA) based on two-by-two factorial design was applied to make statistical comparison between different treatments and the effect of temperature (100°C and 140°C), media (HCW and steam), and their interaction. In addition, the least significant difference multiple comparison was used to compare the pair of two treatments. ANOVA was done at 95 percent of confidence level. Data were analyzed using JMP and SAS software (JMP®, Version Pro 12.2 and SAS Institute Inc., Cary, North Carolina, USA).

Results and Discussion

Determination of the maturity of wood samples

In this study, 14 percent (11 out of 78) of the samples came from proximity of the first 30 growth rings. Three of these samples were in HCW treatment at 140°C (two in the first and one in the second replication) and eight of them were in steam treatment at 140°C (two in the second and six in the second replication). On average, green specific gravity of the samples used in HCW treatment at 140°C was in the range of 0.42 and 0.43 and oven-dry specific gravity was in the range of 0.49 and 0.50 for three replications. Likewise, neither green nor oven-dry specific gravity showed considerable changes among replications of the samples treated in steam at 140°C. Uniformity of

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samples used for testing was further confirmed by the results of Shapiro-Wilk test. For instance, occurrence of non-normally distributed data in replications having specimens of questionable maturity was 3 out of 40 (7.5%), which was less than that of all remaining replications, that is, 10 out of 110 (9.1%). Therefore, all the wood samples were considered mature and uniform (i.e., no presence of juvenile wood) for the purpose of hydrothermal and steam treatment.

Physical characteristics

Moisture content.—On average, the green yellow-poplar samples had moisture content of 43.6 percent (Table 1), which was lower than that reported for green yellow-poplar heartwood (83%; Glass and Zelinka 2010). The lower green moisture content in the test samples may be attributed to several factors, such as geographic location, harvest season (Manwiller 1975), tree diameter (Zobel and Van Buitenen 1989), and time elapsed between tree harvest and sample processing due to natural air drying. Samples used in the current research were cut from logs at a logging landing site where previous cut trees were being delimbed and prepared for shipment during the winter season. Therefore, the samples might have significant moisture loss while lying on the landing site. After picking samples from the landing site, they were brought into the lab and then cut into test specimens within a week. Therefore, further moisture loss might have taken place during the sample preparation process, thereby resulting in lower moisture contents than that reported in literature. In the current research, hydrothermal treatment media had a significant effect on MC ($P = 0.001$). The HCW medium reached a MC as high as 101.1 percent, whereas steam medium increased the MC up to 71.9 percent. Additionally, the temperature level did not have a statistically significant effect on MC ($P = 0.137$). The effect of medium was most likely due to liquid medium facilitating better access of water molecules to the smaller pores than steam medium (Siau 1995). Overall, the hydrothermal treatment increased sample MC (Table 1). The increase in MC as a result of hydrothermal treatment may be explained by a change in hydrogen bonding intensity of hydroxyl groups and changes in crystallinity. Heating fibers to 70°C to 80°C decreases intramolecular hydrogen bonding, which results in greater crystallinity. However, heating fibers above 130°C results in significant increase in hydroxyl group intensity, resulting in reduced crystallinity and therefore more water absorption capacity (Dadashian et al. 2005, Singh and Sivanandan 2014). Peng et al. (2012) and Cai (2006) have reported decreases in MC as results of steam treatment and steam explosion. On the contrary, in our research, steam treatment increased sample MC. This differing result was most likely attributable to the different steaming process in our research where the steam was not either injected or released instantaneously. Specifically, the steam was generated in the sealed reactor containing the test samples by heating up the reactor. Also, during postheat treatment, the sample-reactor system was allowed to cool-down to room temperature prior to it being opened, thereby allowing steam to condense back on the test samples.

Shrinkage.—Wood shrinks as its MC drops below fiber saturation point (FSP). In this research, volumetric shrinkage was calculated to represent overall shrinkage for control and hydrothermally treated samples. Table 1 represents average volumetric shrinkage in samples when control and treated samples were oven-dried. The control sample of yellow-poplar showed 12.4 percent of volumetric shrinkage when it was dried from 43.6 to 0 percent MC. The volumetric shrinkage demonstrated by the control sample was equivalent to the 12.7 percent reported by Glass and Zelinka (2010). Additionally, all treatments but HCW at 140°C did not affect volumetric shrinkage. The samples treated in HCW medium at 140°C showed increased volumetric shrinkage of 13.7 percent. Statistical analysis showed that medium had a statistically significant effect on volumetric shrinkage ($P = 0.004$). However, the effect of temperature on shrinkage was minimal ($P = 0.147$). In past literature, only the effect of steam medium and temperature on shrinkage have been reported (Alexiou et al. 1990, Cai 2006). Similar to the results of this research, Alexiou et al. (1990) observed no significant change in volumetric shrinkage of eucalyptus (*E. pilularis*) when it was steamed at 100°C for 3 hours prior to drying. Cai (2006) reported that linear shrinkage in wood increases significantly above 140°C. They found no significant changes in linear shrinkage for the samples treated up to 130°C. The heat treatments did not lead to wood loss; however, wood cell-walls fractured and small pores cleaned-up, leading to increased shrinkage.

Specific gravity.—Typically, green and oven-dry specific gravity for yellow-poplar are reported to be 0.40 and 0.46, respectively (Glass and Zelinka 2010), which are notably lower than the values reported in this research. This may be explained by the fact that all samples used in the current research were from heartwood, which typically has lesser porosity and more density than sapwood (Hoadley 1980, 1990). In our research, samples used to perform various hydrothermal treatments had a green SG of 0.44, which upon oven-drying increased to 0.50 at oven-dry conditions (Table 2). SG values did not change significantly as a result of various hydrothermal treatments. Statistically, neither of the treatment factors (temperature and medium) had significant effect on SG. It is noted that any change in SG

Table 1.—Moisture content (MC; dry basis) and volumetric shrinkage (mean ± standard error) of untreated and hydrothermally treated yellow-poplar.

Treatment (media and temperature)	Dry-basis MC before (%)	Dry-basis MC after (%)	Volumetric shrinkage (%)
Untreated (control)	43.6 ± 0.8 A[a]	43.6 ± 0.8 D	12.4 ± 0.6B
HCW[b] 100°C	46.6 ± 3.0 A	85.5 ± 11.8 B	11.5 ± 0.6B
HCW 140°C	46.0 ± 1.4 A	101.1 ± 6.7 A	13.7 ± 0.8 A
Steam 100°C	50.9 ± 1.1 A	71.9 ± 3.7 C	11.6 ± 0.6 B
Steam 140°C	50.1 ± 4.6 A	69.6 ± 9.9 C	12.4 ± 0.4 B

[a] Letters in the same column show statistically significant differences.
[b] HCW = hot-compressed water.

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Table 2.—Specific gravity and total porosity (mean ± standard error) of untreated and hydrothermally treated yellow-poplar.

Treatment (media and temperature)	Green specific gravity	Oven-dry specific gravity	Total porosity (%)
Untreated (control)	0.44 ± 0.01 A[a]	0.50 ± 0.14 A	82.4 ± 1.8 B
HCW[b] 100°C	0.44 ± 0.01 A	0.49 ± 0.17 A	83.3 ± 4.0 B
HCW 140°C	0.42 ± 0.01 A	0.49 ± 0.13 A	92.2 ± 3.4 A
Steam 100°C	0.44 ± 0.02 A	0.50 ± 0.18 A	92.3 ± 2.1 A
Steam 140°C	0.43 ± 0.02 A	0.49 ± 0.19 A	94.9 ± 2.7 A

[a] Letters in the same column show statistically significant differences.
[b] HCW = hot-compressed water.

is expected from change in volume; for HCW at 140°C, volumetric shrinkage was significantly greater than the other groups, but it did not change oven-dry volume considerably. In the current treatment conditions, both green volume and oven-dried mass remained unchanged. The main effect of media ($P = 0.224$), main effect of temperature ($P = 0.099$), and interaction effect of media and temperature ($P = 0.529$) were statistically insignificant. There was no statistically significant loss of mass following hydrothermal treatment at 140°C. Literature suggests that wood undergoes significant loss of mass when treated at temperatures above 170°C, which significantly affects SG (Yang et al. 2007, Gunduz et al. 2008). All wood polymers are very stable below 155°C (Singh and Sivanandan 2014). Therefore, no change in SG of yellow-poplar is in agreement with literature because no significant loss of mass or volume change took place during our treatment conditions.

Porosity.—The total porosity represents the fraction of green volume that is not occupied by wood cell mass. The control samples indicated that 82.4 percent of the green volume was occupied by either air or water and only 17.4 percent of the green volume was occupied by dry wood mass (Table 2). Theoretically, the total porosity may be calculated using specific gravity and dry-basis moisture content values (Siau 1995). The theoretical porosity for the tested samples was 71.3 percent, which was similar to the measured values. Hydrothermal treatment significantly changed total porosity of yellow-poplar samples. Treatments with HCW medium increased total porosity from 82.4 to 92.2 percent at the temperature of 140°C. Treatment under steam increased total porosity even at the low temperature of 100°C. Both media ($P = 0.022$) and temperature ($P = 0.021$) appeared to have a statistically significant influence on total porosity. However, the interaction effect of temperature and media was not statistically significant ($P = 0.165$). Although specific gravity remained unchanged, the porosity of samples changed during hydrothermal treatments because the hydrothermal treatments were not intense enough to cause chemical decomposition of the cell wall. However, the hydrothermal treatment appeared to be strong enough to open micropores and mesopores and fracture cell walls, leading to enhanced porosity. According to Zauer et al. (2013), heat treatments have varied effect on the porosity of different species. They report that the thermal modification reduced total porosity for dry Norway spruce (*Picea abies*) wood, whereas it increased for sycamore maple (*Acer pseudoplatanu*) wood and it did not change in European ash (*Fraxinus excelsior*) wood.

Water absorption.—The hydrothermal treatments significantly changed water absorption (WA) capability and total volumetric swelling (Table 3). WA after 24 hours soaking was 32.5 percent for untreated yellow-poplar heartwood samples, which was significantly less than the WA values of treated samples. Upon hydrothermal treatments, the WA significantly increased for all treatments except HCW at 100°C. These results, combined with the porosity results, indicated that hydrothermal treatments increased porosity and consequently WA. Among treatment factors, the temperature had a statistically significant influence on WA ($P = 0.013$). However, media had a minimal effect ($P = 0.995$) on WA. Interaction effect of temperature and media was not statistically significant ($P = 0.253$). In contrast, reduction in WA following heat treatment has been reported for black pine (*Pinus nigra*) after treatment at 210°C for 3 hours (Dundar et al. 2012) and for acacia (*Acacia mangium*) wood after treatment up to 230°C (Tuong and Li 2011). The reduction in WA has been attributed to reduction of hydroxyl group in wood (Weiland and Guyonnet 2003, Tjeerdsma and Militz 2005) and owing to formation of cross-linking over heat treatment, which makes the molecules less elastic and decreases the possibility to enlarge the cellulose microfibrils (Tjeerdsma et al. 1998). Literature showed decrease in WA, but temperature and holding time used in current research were not high enough to make aforementioned changes.

Swelling.—All hydrothermal treatments did not have a statistically significant effect on volumetric swelling. Additionally, the main effect of temperature ($P = 0.053$) and media ($P = 0.499$), as well as their interaction effect ($P = 0.144$) on shrinkage, were all statistically insignificant. However, steam treatment at 140°C reduced volumetric swelling from 12.2 percent (after 24 hr for untreated yellow-poplar heartwood samples) to 10.0 percent (Table 3). Theoretically, the maximum volumetric swelling may be calculated by multiplying 30 with oven-dried specific gravity (Siau 1995). The untreated samples had oven-dry specific gravity of 0.50, which would result in 15.0 percent theoretical volumetric swelling. The lower than theoretical measured volumetric swelling in this research was most likely due to a lack of significant time (24 h) for complete saturation of the wood samples. Longer saturation duration would have achieved volumetric swelling close to the theoretical value.

Mechanical properties

Compression strength and modulus of elasticity are important mechanical properties for wood used in structural applications. The untreated samples showed a compression strength of 42.95 MPa (Table 4) and modulus of elasticity of 1.29 GPa. The compression strength of clear yellow-poplar (12% MC) has been reported to be 38.20 MPa (parallel to grain), which is slightly lower than our test samples (Kretschmann 2010).

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Table 3.—Water absorption and volumetric swelling (mean ± standard error) of untreated and hydrothermally treated yellow-poplar.

Treatment (media and temperature)	Water absorption (%)	Volumetric swelling (m^2/s)
Untreated (control)	32.5 ± 0.9 B[a]	12.2 ± 0.5 A
HCW[b] 100°C	38.2 ± 3.0 B	12.1 ± 0.5 A
HCW 140°C	48.8 ± 9.9 A	11.6 ± 0.9 A
Steam 100°C	41.1 ± 3.5 A	12.7 ± 0.8 A
Steam 140°C	45.8 ± 5.8 A	10.0 ± 1.0 A

[a] Letters in the same column show statistically significant differences.
[b] HCW = hot-compressed water.

Table 4.—Modulus of elasticity and compression strength (mean ± standard error) of untreated and hydrothermally treated yellow-poplar.

Treatment (media and temperature)	Modulus of elasticity (GPa)	Compression strength (MPa)
Untreated (control)	1.29 ± 0.10 C[a]	42.95 ± 2.79 ABC
HCW[b] 100°C	1.66 ± 0.13 A	47.75 ± 2.33 A
HCW 140°C	1.36 ± 0.14 BC	43.73 ± 3.78 AB
Steam 100°C	1.17 ± 0.10 C	38.50 ± 3.78 C
Steam 140°C	1.57 ± 0.18 AB	42.27 ± 2.46 BC

[a] Letters in the same column show statistically significant differences.
[b] HCW = hot-compressed water.

Hydrothermal treatment significantly increased the modulus of elasticity of yellow-poplar. For example, the modulus of elasticity increased from 1.29 GPa (control) to as high as 1.66 GPa due to hydrothermal treatment performed in HCW medium at 100°C. Also, steam treatment at 140°C led to increase in modulus of elasticity. Contrary to our results, Dundar et al. (2012) reported that the modulus of elasticity significantly decreased from 5.6 GPa (control) to 4.8 GPa when samples were treated at 180°C. The higher treatment temperature used by Dundar et al. (2012) may account for the different result on modulus of elasticity than that in our work because wood polymers start to degrade when subjected to temperatures above 155°C (Singh and Sivanandan 2014), thereby resulting in more brittle or fragile samples (Fig. 2).

Compression strength of yellow-poplar heartwood was 42.95 MPa, which increased with the hydrothermal treatments, in general, except for steam treatment at 100°C. Statistically, the main effects of medium and temperature were insignificant ($P = 0.933$) and significant ($P = 0.007$), respectively. In addition, interaction effect of temperature and media was significant ($P = 0.031$). The significance of interaction effects is due to there being a considerable difference between HCW and steam at 140°C ($P = 0.001$), whereas these two different media did not have noticeable difference at 140°C. Dundar et al. (2012) reported significant decrease in modulus of rupture resulting from hydrothermal treatment at 180°C for black pine wood, which again was likely due to the higher treatment temperature.

Drying behavior

Moisture ratio (MR) at given time was plotted for the control and hydrothermally treated samples (Fig. 4). The drying curve (MR vs. time) for HCW-treated samples lies above the drying curve for control samples. However, the drying curve for steam-treated samples is close to but below the drying line of control samples. This result indicates that it took longer (i.e., slower rate) to dry HCW-treated samples than control, whereas steam-treated samples dried faster. Additionally, high-temperature (140°C) HCW-treated samples held moisture more tightly than low-temperature (100°C) HCW-treated samples; therefore, they showed high moisture ratios at given time. Holding a high moisture ratio at a given drying time indicates that moisture was likely held with more binding force and/or pores that were blocked, slowing down mass transfer rate. Similar behavior of high moisture ratios and slow drying rates has been reported by Taghiyari et al. (2011) for samples treated in HCW. They reported that treating beech (*Fagus orientalis*) at 180°C in hot water leads to extra extractive settling on perforation plates and cell walls, which slows mass transfer rates during drying. Likewise, Peng et al. (2012) reported low moisture ratio or high drying-rate behavior for steam-treated samples due to steam-treated samples possessing pores and cavities with less blockage. Therefore, the treated

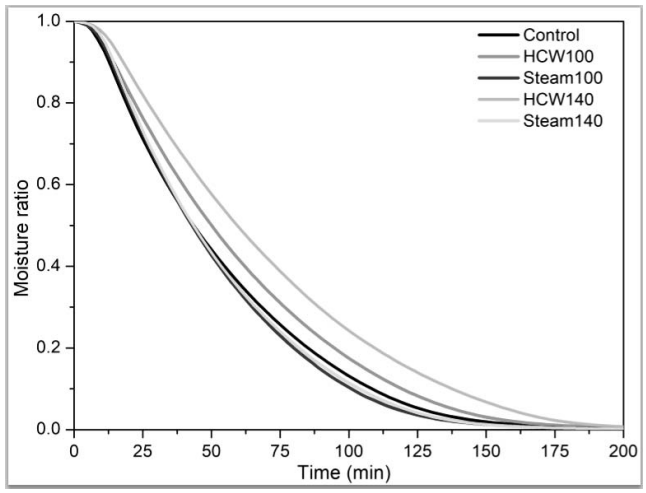

Figure 4.—Moisture ratio as a function of time elapsed during drying at 105°C for untreated and hydrothermally treated samples of yellow-poplar. HCW = hot-compressed water.

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samples dry faster than control samples and bound water diffusion was slower in the samples steam-treated at 100°C or 140°C than in the control samples of poplar wood.

As can be observed in Table 5, untreated samples reached zero MC after 226 minutes, which was faster than treated samples. However, samples treated in HCW at 140°C showed the longest drying time (260 min). Increased drying time for the treated samples in HCW at either temperature can be explained by their elevated MC and slow drying rates. Control samples showed the most uniform drying time (standard deviation = 0.02).

Drying behavior (moisture desorption) of wood in zero humidity and constant temperature is associated with wood properties such as various pore sizes, affinity of wood for water, total porosity, density, and specific gravity. Moisture desorption from wood is fundamentally controlled by two simultaneous mechanisms, which are capillary flow and diffusion flow (Siau 1995, Bergman 2010). The results presented on drying behavior in Figure 5 show the following four phases: (1) increase in drying rate due to sample heating; (2) linear capillary-controlled (convective) falling phase; (3) nonlinear transition falling phase; and (4) linear diffusion-controlled falling period. Over the second phase of drying, free water flows through the cell lumens and pits due to capillary action. In the third phase, moisture loss takes place from lumens (free water) as well as from cell walls (bound water). In the fourth phase, when cell lumens and pits become empty, water vaporizes from cell walls and water vapor flows through the cell lumens and pits by diffusion. Bound water also diffuses through the cell walls. The diffusion coefficient of water vapor is three to four orders of magnitude smaller in lumens than water vapor diffusion coefficient in free water (Siau 1995).

Figure 5 shows that the drying rate curves of all the hydrothermally treated samples were above the drying curves of controlled samples because the treated samples had high initial moisture content (Table 5). Therefore, greater amounts of water were evaporated at given moisture ratio from treated samples than from control samples. In the drying curves, slopes in various phases are important because they are used to calculate overall liquid diffusion coefficients. Overall liquid diffusion coefficient was calculated for the convective controlled drying phase. HCW treatment, especially at elevated temperature, exhibited greater drying rates than controls.

Overall liquid diffusion coefficient of yellow-poplar heartwood was 3.15×10^{-8} m^2/s (Table 5). However, research by Sayar and Tarmian (2013) reported typical diffusion coefficients in the range of 1.34×10^{-9} to 1.90×10^{-9} $m^2 s^{-1}$ for control samples. Hydrothermal treatments tend to reduce overall liquid diffusion from wood. In our research, hydrothermal treatments at low temperature

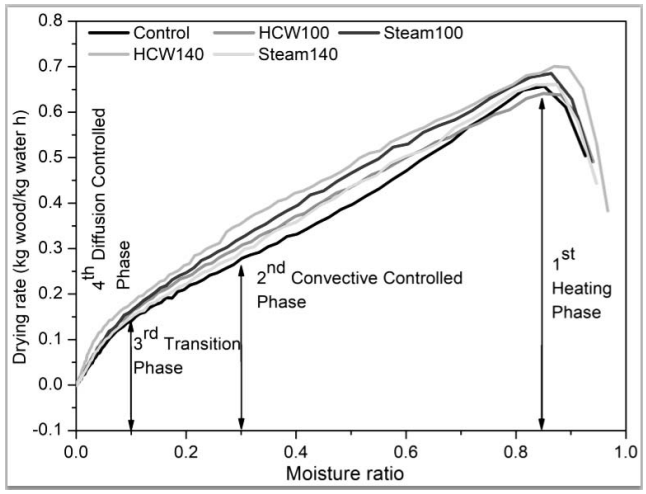

Figure 5.—Drying rate as a function of moisture ratio for untreated and treated samples of yellow-poplar. HCW = hot-compressed water.

(100°C) reduced overall liquid diffusion coefficient from 3.15×10^{-8} to 2.15×10^{-8} m^2/s for HCW and 2.62×10^{-8} m^2/s for steam conditions, which indicated that significantly more pores were blocked with HCW treatment than steam treatment as a result of mobilization of extractives. Treatment at relatively high temperature (140°C) improved overall liquid diffusion coefficient as a result of volatilization of extractives.

Conclusions

In this research, the influence of hydrothermal treatment in steam and hot compressed water at 100°C and 140°C on selected physical properties and drying behavior of yellow-poplar was evaluated. Results showed that the moisture content of the samples greatly increased following hydrothermal treatment, which was influenced by the type of medium. Upon completely drying all the treated samples, greater water absorption was observed for treated samples than the controls. Hydrothermal treatment did not change dimensional stability (shrinkage and swelling) of treated samples, except for HCW at 140°C, which resulted in noticeable increase in volumetric shrinkage. However, neither steam treatment nor HCW treatment appeared to be an improved method for water repellency. Specific gravity was not influenced by the hydrothermal treatment—there was zero loss of mass and a statistically insignificant amount of shrinkage for most of groups following hydrothermal treatment. Concerning mechanical properties, the modulus of elasticity increased following either HCW at

Table 5.—Drying time and diffusion coefficient (mean ± standard error) of untreated and hydrothermally treated yellow-poplar.

Treatment (media and temperature)	Drying time (min)	Diffusion coefficient (m^2/s)
Untreated (control)	225.95 ± 0.02 A[a]	$3.15 \times 10^{-8} \pm 1.19 \times 10^{-9}$ A
HCW[b] 100°C	240.40 ± 4.48 B	$2.15 \times 10^{-8} \pm 3.65 \times 10^{-9}$ C
HCW 140°C	260.14 ± 12.76 C	$1.89 \times 10^{-8} \pm 2.42 \times 10^{-9}$ C
Steam 100°C	231.94 ± 9.07 B	$2.62 \times 10^{-8} \pm 1.63 \times 10^{-9}$ B
Steam 140°C	242.35 ± 13.19 B	$3.04 \times 10^{-8} \pm 3.93 \times 10^{-9}$ A

[a] Letters in the same column show statistically significant differences.
[b] HCW = hot-compressed water.

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100°C or steam treatment at 140°C. However, no treatment resulted in change in compression strength. Regarding drying behavior, the HCW treatments resulted in slow drying that was most likely attributable to blocked pores and elevated initial moisture content resulting from hydrothermal treatment in a liquid medium. Steam treatment at relatively high temperature (140°C) improved overall liquid diffusion coefficient as a result of volatilization of extractives. From a practical standpoint, hydrothermal treatment (in either medium) did not diminish the properties of the final product—no reduction was observed in dimensional stability and compression strength. In addition, increased porosity and water uptake can be helpful in the event wood must be saturated by liquid preservatives. On the whole, prior to any recommendation for industrial wood drying, further research work is needed to reveal any possible effect of hydrothermal treatments (in either medium) on warpage, checks, and splits in lumber.

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