

# Effects of Acid Copper Chromate Preservative and Hydrothermal Treatment on the Dimensional Stability, Hardness, and Decay Resistance of Poplar Wood

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The effects of acid copper chromate (ACC) and hydrothermal treatments were determined relative to the hardness, decay resistance, and dimensional stability of poplar wood. Test specimens, prepared from poplar wood (*Populus nigra* L.), were first heat-treated under saturated steam in a digester and then impregnated with ACC solution and by a long-term (21 days) dipping technique to reach complete saturation. Impregnated specimens were exposed to white-rot fungus (*Trametes versicolor*) for 14 weeks, using the Kolle flask method. The weight loss and Brinell hardness were determined after impregnation, thermal treatment, and exposure to *T. versicolor*. The combination of thermal treatment and ACC preservative on the poplar wood showed improved hardness and decay resistance properties of wood, depending on the treatment time and temperature. The highest weight loss (37.78%) was observed for control specimens, and the lowest (3.03%) occurred in 1% ACC-treated specimens. The highest Brinell hardness on a tangential surface was observed in 1% ACC-treated specimens (6.45 kN), and the lowest was noted in the specimens heat-treated at 130 °C and 180 min (0.52 kN).

*Keywords:* Heat treatment; ACC preservative; Decay resistance; Hardness; Water absorption; Volumetric swelling

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## INTRODUCTION

The genus *Populus* (Salicaceae), having a wide geographical distribution in Iran, fast growth, ease of propagation and good economical revenue, in addition to its value for wood products, provides a range of ecological services, including carbon sequestration, bioremediation, nutrient cycling, and bio-filtration (Taylor 2002; Brenner *et al.* 2004, Alimohamadi *et al.* 2012). In Iran, *P. nigra* L. has been cultivated in plantation forests and used as construction materials. Poplar plantations, which have been planted in Iran for many years (Nori *et al.* 2008), are regarded as an appropriate alternative to unplanted, naturally occurring forest trees.

Recently, various chemical techniques have been used to modify wood structure and lignocellulosic materials to enhance their properties. Thermal treatments use no chemicals, resulting in an environment-friendly process. Hydrothermal treatment is a thermal correction that uses water for heat transfer. Chemical changes in wood cell walls caused by the application of heat affect the mechanical and physical properties of wood (Rowell and LeVan-Green 2005; Charani *et al.* 2007). Drying at high temperatures decreases the equilibrium moisture and the consequent swelling of wood (Tiemann 1920).

The degradation of hemicelluloses and the amorphous region of cellulose has been reported by the heating of wood at high temperature, which results in an increase in the degree of crystallinity of this polymer. Subsequently, a cross-linkage between the lignin and the polymers occurs because of the thermal degradation of the wood, which is responsible for the decrease in the hygroscopicity of wood and the improvement of the dimensional stability (Jämsä and Viitaniemi 2001; Waskett and Selmes 2001; Bekhta and Niemz 2003; Wikberg and Maunu 2004; Metsä-Kortelainen *et al.* 2006; Calonego *et al.* 2010). In addition, a decrease in mechanical properties, as well as weight loss, was found with the heating process (Haygreen and Bowyer 1996; Homan *et al.* 2000; Waskett and Selmes 2001).

Thermal treatments have been used by many researchers to improve wood composite materials (Kazemi Najafi *et al.* 2007; Kaboorani *et al.* 2008; Arwinfar *et al.* 2016) and to lower the water absorption of wood by the crystallization of cellulose and extraction of hemicelluloses from wood (Wallenberger and Weston 2004; Yildiz and Gumuskaya 2007). Additionally, enhancement of the dimensional stability, reduction of the swelling, an increase in the decay resistance, and alteration of the chemical composition of wood has been found in thermally modified wood (Tjeerdsma *et al.* 2000; Militz and Tjeerdsma 2001; Yildiz 2004a; Temiz *et al.* 2006; Charani *et al.* 2007; Koubaa *et al.* 2011). On the other hand, reduction in strength (MOR, MOE) has been found to occur in thermally modified wood (Bengtsson *et al.* 2002; Yildiz *et al.* 2002; Poncsak *et al.* 2006).

Anti-swelling efficiency (ASE) (the difference between the swelling of the treated and untreated wood) in the radial and tangential directions of modified Plato-treated beech can be decreased significantly, with values of 10%, and 13%, respectively, in comparison with the control treatment (Militz and Tjeerdsma 2001). Another study revealed that ASE was observed to be 31% and 44% at 190 °C and 200 °C, respectively, as the maximum treatment in the radial direction. Also, ASE was not additionally improved at higher temperature (Charani *et al.* 2007). In addition, Ding *et al.* (2012) reported that ASE values suggested that incorporating methyl methacrylate effectively improved the dimensional stability of poplar wood at the early soaking stage, but was less effective in the long term and increased the density of all poplar woods.

The dimensional stability of the treated wood is improved after treatment, which could be related to filling the void spaces in the wood (Schaudy and Proksch 1982; Ellis 1994; Zhang *et al.* 2006) or because of a reduction in number of free hydroxyl groups with chemical reactions (Ellis 1994; Deka and Saikia 2000; Zhang *et al.* 2006). For low-grade wood, the dimensional stability, mechanical strength, and decay resistance can be improved by wood modification (Yildiz *et al.* 2005; Gao and Li 2007; Koubaa *et al.* 2011).

Tests with stakes and posts that are exposed to decay and termite attack in wood have indicated that acid copper chromate (ACC) provides an acceptable average service life. But the wood used in ground contact may suffer occasional early failure from attack by copper-tolerant fungi (Lebow *et al.* 2003). The ACC was found to control the beech wood decay by fungus *Trametes versicolor* (Feraydoni and Hosseinihashemi 2012). The ACC has better decay resistance against *Coriolus versicolor* (*T. versicolor*) and *Gloeophyllum trabeum* in comparison with methanol and acetone extracts of heartwood of *Maackia amurensis* (Su *et al.* 2007) where the fungal resistance was further increased by using 2% ACC. The impregnation of *Acer insigne* wood with ACC increased decay resistance and natural durability, and subsequently converted the non-durable sample to durable (Tazakor Rezaei 2010) sample.

In the present study, the effects of hydrothermal treatment of modified poplar wood treated by acid copper chromate (ACC) at various concentrations (0%, 0.5%, and 1%) were investigated to study some physical properties, as well as the effect of the treatments on the mass loss and hardness of incubated wood exposed to degradation by *Trametes versicolor*.

## EXPERIMENTAL

### Preparation of Wood Samples

Wood boards (3 × 10 × 50 cm) were prepared from poplar wood (*Populus nigra* L.) logs and cut into samples with dimensions of 2 × 2 × 2 cm and 1.5 × 2.5 × 5 cm to meet ASTM D143-94 (2007) and BS 838 (1961) requirements, respectively. Samples were dried in an oven for 24 h at 103 ± 2 °C to determine the dry weight before treatment. Samples (four replicates) for each treatment were placed in a stainless steel chamber (digester) filled with water (liquid/dry wood=10 g/g) and then heated at 130 °C and 160 °C for 120 or 180 min. After the hydrothermal treatment, they were cured in an oven for 24 h based on their initial treatment temperatures (130 °C and 160 °C). The impregnation with ACC solution (0%, 0.5%, and 1% w/w in distilled water) was achieved by a long-term (21 days) dipping technique to reach complete saturation. All treatments used in the present study are summarized in Table 1. Instead of complete saturation by application of pressure method (such as the Bethel method), dipping was used in the pressing work.

**Table 1.** Thermal Modification and ACC Treatment of Poplar Wood

Temperature (°C)	Time (min)	ACC Concentration (%)	Replicates
130	120	0, 0.5, and 1	4 for each concentration
130	180	0, 0.5, and 1	4 for each concentration
160	120	0, 0.5, and 1	4 for each concentration
160	180	0, 0.5, and 1	4 for each concentration
-	-	0.5 and 1	4 for each concentration
-	-	Control	4

### Dimensional Stability Measurements

Wood samples of 2 × 2 × 2 cm dimensions were used for water absorption, volumetric swelling, and anti-swelling efficiency (ASE) tests. The samples were dipped in water for 2, 24, or 432 h. Wet weights and dimensions were measured to determine the water absorption and swelling after each soaking. Next, samples were oven-dried and their dry weights and dimensions were again determined. ASE and swelling were calculated based on the following equation,

$$ASE (\%) = (S_2 - S_1) / S_1 \times 100 \quad (1)$$

where ASE (%) is the anti-swelling–efficiency,  $S_2$  (%) is the volumetric swelling of untreated wood after each soaking time, and  $S_1$  (%) is the volumetric swelling of treated wood after each soaking time.

### Density

Samples with nominal dimensions of 20 × 20 × 20 mm (longitudinal × radial × tangential) were used to determine oven-dried density ( $D_o$ ). The samples were oven-dried at 103 ± 2 °C until achieving a constant weight. Wood sample dimensions were determined and the weight was recorded.  $D_o$  was calculated according to the following equation,

$$D_o = \frac{M_o}{V_o} \quad (2)$$

where  $M_o$  and  $V_o$  are the mass and volume of the oven-dried sample, respectively.

## Decay Test

### *Fungus culture*

Decay tests were conducted in accordance with BS 838 (1961) as applied by the Kolle flasks method for 14 weeks of exposure to *Trametes versicolor*. The fungus was grown and maintained on malt extract agar (MEA). The medium was autoclaved and sterilized for 30 min at 105 kPa and 125 °C and cooled to room temperature before inoculation. After cooling the medium, the purified Turkey Tail fungus was transferred to Kolle flasks containing MEA under a sterile hood using sterile pincers. The flasks were kept at 23 °C for one week until the culture medium was fully covered by the fungus.

### *Trametes versicolor poplar wood colonization*

Four wood samples of *P. nigra* wood (1.5 × 2.5 × 5 cm) from each treatment were mounted on 3-mm platforms and placed in the Kolle flasks. The flasks containing *T. versicolor* and wood specimens were incubated for 14 weeks at 23 °C and 75% relative humidity until the specimens were heavily colonized by the test fungus.

### *Weight loss*

At the end of the exposure period, the test blocks were removed from the Kolle flasks, and their surfaces carefully brushed. The blocks were then dried to constant weight at 103 ± 2 °C for 24 h. The wood blocks were weighed to the nearest 0.01 g to determine the decayed weight ( $W_2$ ). Weight loss (WL) was calculated as a percentage of the initial sample weight ( $W_1$ ) using the following equation:

$$WL (\%) = [(W_1 - W_2)/W_1] \times 100 \quad (3)$$

## Hardness Test

Control samples of wood (Undecayed), as well as the impregnated samples with ACC, and thermal treatments were tested for their Brinell hardness after exposure to white-rot fungus. The wood samples were prepared in dimensions of 1.5 × 2.5 × 5 cm. Hardness was measured according to the Janka scale, using a ball 11.3 mm in diameter. Specimens were kept at a temperature of 20 ± 3 °C and moisture content of 8 ± 2% at the time of testing. For all mechanical tests, the number of replications was four.

## Reduction of Wood Mass

The relative mass reduction (MR) after thermal modification was calculated using the following equation:

$$MR = [(m_1 - m_2)/m_2] \times 100 (\%) \quad (4)$$

where  $m_1$  is the oven-dry mass before heat treatment and  $m_2$  is the oven-dry mass after the process.

## Statistical Analysis

Statistical analysis was performed using (SPSS 17, USA). The 15 formulation designs, which are shown in Table 1, were all analyzed for variance using a complete randomized block design. Testing of mechanical, physical, and durability properties was performed using four replicates of each formulation. Property means were compared using Duncan's new multiple range test at a 95% confidence level.

## RESULTS AND DISCUSSION

### Weight Loss

Statistically, results in Table 2 showed that ACC concentration and time\*ACC concentration had a significant effect on the weight loss (WL) of *P. nigra* wood as affected by *T. versicolor*. Table 3 shows that the lowest WL values of poplar wood observed by the treatment of 1% ACC without hydrothermal treatment (3.03%). The next-lowest results, respectively, were for the heated wood at 160 °C for 120 min with 0.5% ACC, and unheated wood with 0.5% ACC with WL values of 4.66%, and 7.21%. On the other hand, the highest WL values was observed by non-treated wood (37.98%), 0% ACC-heated wood with 160 °C for 120 min (35.28%) and 0% ACC-heated wood with 130 °C for 120 min (32.91%). It could be observed that with un-impregnated wood heated with increasing time, the WL values was higher than those found with impregnated wood at the same heating temperature and time.

**Table 2.** Univariate Analysis of Weight Loss

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	5278.630	11	479.875	33.876	0.000
Intercept	15069.797	1	15069.797	1063.826	0.000
Temperature	25.201	1	25.201	1.779	0.191
Time	0.340	1	0.340	0.024	0.878
ACC concentration	4973.630	2	2486.815	175.552	0.000
Temperature * Time	48.481	1	48.481	3.422	0.073
Temperature * ACC concentration	26.666	2	13.333	0.941	0.400
Time * ACC concentration	178.814	2	89.407	6.312	0.004
Temperature * Time * ACC concentration	25.498	2	12.749	0.900	0.416
Error	509.964	36	14.166		
Total	20858.391	48			
Corrected Total	5788.594	47			

Statistically, the analysis in Table 4 shows that the ACC concentration and the interaction between temperature and time had significant effects on the hardness values of the incubated wood samples with *T. versicolor*.

**Table 3.** Weight Loss of Poplar Wood by *T. versicolor* as Affected by Heating Temperature, Time, and ACC Concentration

Treatment	Weight Loss (%)		
	0% ACC	0.5% ACC	1% ACC
130 °C- 120 min	32.91 ± 3.94 ef	8.12 ± 5.93 abcd	11.54 ± 4.61 cd
130 °C- 180 min	32.08 ± 6.04 ef	13.14 ± 3.04 cd	12.88 ± 1.62 cd
160 °C- 120 min	35.28 ± 4.09 f	4.66 ± 1.94 ab	14.31 ± 3.61 d
160 °C- 180 min	27.67 ± 2.25 e	9.69 ± 3.09 bcd	10.36 ± 1.25 bcd
Control	37.78 ± 5.32 f	7.21 ± 4.64 abc	3.03 ± 1.18 a

**Table 4.** Univariate Analysis of Hardness

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	16.314	11	1.483	3.194	0.004
Intercept	72.591	1	72.591	156.353	0.000
Temperature	1.415	1	1.415	3.048	0.089
Time	1.310	1	1.310	2.823	0.102
ACC concentration	3.736	2	1.868	4.024	0.026
Temperature * Time	4.437	1	4.437	9.557	0.004
Temperature * ACC concentration	2.508	2	1.254	2.701	0.081
Time * ACC concentration	1.575	2	0.788	1.697	0.198
Temperature * Time * ACC concentration	1.332	2	0.666	1.434	0.252
Error	16.714	36	0.464		
Total	105.620	48			
Corrected Total	33.028	47			

Table 5 shows that the highest hardness values were observed by the wood treated with 1% and 0.5% ACC without heating, with values of 6.447 and 4.184 kN.

**Table 5.** Hardness Values as Affected by Treatment

Treatment	Mean* (kN)	Std. Deviation	Std. Error	95% Confidence Interval for Mean (kN)		Minimum (kN)	Maximum (kN)
				Lower Bound	Upper Bound		
130 °C- 120 min	0.851a	0.145	0.072	0.620	1.081	0.654	0.974
130 °C- 120 min- 0.5% ACC	1.615a	0.835	0.417	0.286	2.945	0.913	2.805
130 °C- 120 min- 1% ACC	1.123a	0.514	0.257	0.305	1.941	0.767	1.883
130 °C- 180 min	0.521a	0.115	0.057	0.338	0.704	0.381	0.644
130 °C- 180 min- 0.5% ACC	1.349a	0.448	0.224	0.636	2.063	0.928	1.893
130 °C- 180 min- 1% ACC	0.886a	0.182	0.091	0.595	1.177	0.712	1.144
160 °C- 120 min	0.776a	0.122	0.061	0.581	0.971	0.652	0.893
160 °C- 120 min- 0.5% ACC	1.053a	0.393	0.196	0.427	1.679	0.617	1.568
160 °C- 120 min- 1% ACC	0.966a	0.270	0.135	0.536	1.396	0.777	1.354
160 °C- 180 min	1.203a	0.423	0.211	0.529	1.876	0.611	1.592
160 °C- 180 min- 0.5% ACC	1.521a	0.695	0.347	0.414	2.628	0.811	2.478
160 °C- 180 min- 1% ACC	2.887b	1.853	0.926	-0.060	5.836	0.719	5.189
0.5% ACC	4.184c	2.107	1.053	0.831	7.537	1.319	6.099
1% ACC	6.447d	1.095	0.547	4.705	8.190	5.144	7.699
Control	1.100a	0.146	0.073	0.867	1.333	0.938	1.288

\* Means with the same letter are not significantly difference according to Duncan's new multiple range test at a 95% confidence level

The heating temperature caused a significant increase in hardness values, and these results were similar to those of Homan *et al.* (2000) and Waskett and Selmes (2001), which demonstrated that the strength properties of wood were reduced by approximately 30% as affected by heat treatments.

A significant decrease in Janka hardness of up to 20.7% was found for thermally modified *Eucalyptus grandis* wood (Calonego *et al.* 2012). On the other hand, it was reported that, especially above 200 °C, a slight hardness increase in heat-treated birch wood with increasing treatment temperature was observed (Poncsak *et al.* 2006). Additionally, minimizing the decrease in mechanical properties of the compressed wood resulted from heat plasticization

of the thermoplastic matrix (lignin and hemicellulose) which is reset in its compressed status and not by degradation of the hemicelluloses (Inoue *et al.* 1993; Yildiz *et al.* 2004a).

### Oven-Dry Density ( $D_o$ )

Statistical results in Table 6 show that there were no significant effects on the oven-dry densities ( $D_o$ ) of wood samples for any treatment used. However, the highest  $D_o$  (Table 7) was found for wood samples treated with 0.5% ACC and heated at 160 °C for 120 and 180 min, with values of 0.420 and 0.425 g/cm<sup>3</sup>, respectively. On the other hand, the lowest  $D_o$  was observed with treatment of 0% ACC-heated at 160 °C for 120 min, with a value of 0.343 g/cm<sup>3</sup>.

**Table 6.** Univariate and One-Way Analysis of Density

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	0.018	11	0.002	1.172	0.340
Intercept	7.254	1	7.254	5130.583	0.000
Temperature	0.003	1	0.003	2.128	0.153
Time	0.000	1	0.000	0.094	0.761
ACC concentration	0.007	2	0.003	2.444	0.101
Temperature * Time	0.004	1	0.004	2.853	0.100
Temperature * ACC concentration	0.001	2	0.000	0.231	0.795
Time * ACC concentration	0.003	2	0.002	1.204	0.312
Temperature * Time * ACC concentration	7.917E-5	2	3.958E-5	0.028	0.972
Error	0.051	36	0.001		
Total	7.323	48			
Corrected Total	0.069	47			
Source	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	0.036	36	0.001	0.720	0.870
Within Groups	0.155	111	0.001		
Total	0.191	147			

The reduction or loss in  $D_o$  values can be related to thermal degradation. The hydrothermal treatment of beech wood at 150 to 170 °C led to a small decrease in the specific gravity of treatment samples (Charani *et al.* 2007), but the highest loss of  $D_o$  values were obtained at 170 °C for 7 h with eight cycles of soaking/oven drying. When the temperature is elevated above 200 °C, the loss in specific gravity values can be achieved rapidly (Kotilainen 2000; Charani *et al.* 2007).

**Table 7.** Values of Oven-Dry Density as Affected by Treatment

Treatment	Before Treatment (g/cm <sup>3</sup> )	After Heat-Treatment (g/cm <sup>3</sup> )	After Heat and ACC Preservative Treatment (g/cm <sup>3</sup> )
130 °C- 120 min	0.383 ± 0.04 ab	0.380 ± 0.03 ab	-
130 °C- 120 min- 0.5% ACC	0.390 ± 0.04 ab	0.370 ± 0.04 ab	0.393 ± 0.04 ab
130 °C- 120 min- 1% ACC	0.390 ± 0.03 ab	0.383 ± 0.04 ab	0.395 ± 0.03 ab
130 °C- 180 min	0.390 ± 0.05 ab	0.390 ± 0.03 ab	-
130 °C- 180 min- 0.5% ACC	0.380 ± 0.04 ab	0.375 ± 0.05 ab	0.385 ± 0.05 ab
130 °C- 180 min- 1% ACC	0.405 ± 0.02 ab	0.385 ± 0.04 ab	0.400 ± 0.05 ab
160 °C- 120 min	0.378 ± 0.03 ab	0.343 ± 0.05 a	-
160 °C- 120 min- 0.5% ACC	0.420 ± 0.03 b	0.398 ± 0.03 ab	0.385 ± 0.02 ab
160 °C- 120 min- 1% ACC	0.383 ± 0.03 ab	0.373 ± 0.09 ab	0.400 ± 0.03 ab
160 °C- 180 min	0.373 ± 0.02 ab	0.375 ± 0.03 ab	-
160 °C- 180 min- 0.5% ACC	0.403 ± 0.02 ab	0.418 ± 0.04 b	0.425 ± 0.03 b
160 °C- 180 min- 1% ACC	0.408 ± 0.05 ab	0.395 ± 0.04 ab	0.413 ± 0.04 b
0.5% ACC	0.395 ± 0.02 ab	-	0.395 ± 0.02 ab
1% ACC	0.385 ± 0.03 ab	-	0.383 ± 0.03 ab
Control	0.393 ± 0.03 ab	-	-

It should be mentioned that the specific gravity is not the most suitable property for evaluating the quality of thermally modified wood, according to Calonego *et al.* (2012).

### Reduction of Wood Mass

According to the ANOVA analysis (Table 8), temperature has a significant effect on the mass reduction (MR) of wood. Generally, an increase in temperature led to an increase in MR. Table 9 shows the MR of the treated poplar wood with various treatments. The highest MR was observed in wood treated with 0% and 1% ACC and heated at 160 °C for 180 min, with values of 16.20%, and 16.17%, respectively. The lowest MR was observed in wood samples treated with 0% and 0.5% ACC and heated at 130 °C for 180 min, with values of 3.72%, and 3.99%, respectively.

**Table 8.** Univariate Analysis of Reduction of Wood Mass

Source	Type III Sum Of Squares	df	Mean Square	F	Sig.
Corrected Model	1437.760	11	130.705	6.707	0.000
Intercept	4322.505	1	4322.505	221.803	0.000
Temperature	1314.823	1	1314.823	67.468	0.000
Time	11.682	1	11.682	0.599	0.444
ACC concentration	21.901	2	10.950	0.562	0.575
Temperature * Time	30.592	1	30.592	1.570	0.218
Temperature * ACC concentration	16.257	2	8.129	0.417	0.662
Time * ACC concentration	29.665	2	14.832	0.761	0.475
Temperature * Time * ACC concentration	12.840	2	6.420	0.329	0.721
Error	701.570	36	19.488		
Total	6461.835	48			
Corrected Total	2139.330	47			



Heating wood at high temperatures causes thermal degradation of the main polymers and formation of furfural monomers, resulting in weight loss (Haygreen and Bowyer 1996; Homan *et al.* 2000; Waskett and Selmes 2001), which causes a reduction in wood strength properties (Homan *et al.* 2000; Waskett and Selmes 2001). For example, *Eucalyptus camaldulensis* wood thermally treated at 150 and 180 °C for 10 h showed a decrease in the compressive strength parallel to the grain (Unsal and Ayrilmis 2005).

**Table 9.** Reduction of Poplar Wood Mass as Affected by Treatment

Treatment	Mass Reduction of Poplar Wood (%)		
	0% ACC	0.5% ACC	1% ACC
130 °C- 120 min	4.74 ± 1.54 a	4.96 ± 1.43 a	3.99 ± 0.77 a
130 °C- 180 min	3.72 ± 0.25 a	3.99 ± 0.58 a	4.14 ± 0.42 a
160 °C- 120 min	15.94 ± 5.21 b	14.42 ± 8.01 b	9.95 ± 3.34 ab
160 °C- 180 min	16.20 ± 11.19 b	15.68 ± 0.60 b	16.17 ± 0.44 b

## Dimensional Stability

### Water absorption

Table 10 presents the water absorption (WA) in the treated wood samples after 2, 24, and 432 h, respectively. The lowest WA after 2, 24, and 432 h was found by the wood treatment of 1% ACC and heat at 130 °C for 120 min, 1% ACC and heat at 130 °C for 120 min, and 1% ACC and heat at 130 °C for 180 min, with values of 42.6%, 44.3%, and 115.6%, respectively. The highest water absorption was found in the control treatment, with values of 119.9%, 120.4%, and 203.3% after 2, 24, and 432 h, respectively. Previous results have shown that the WA of wood is reduced after thermal treatment by crystallization of cellulose and also by the extraction of hemicelluloses from wood (Wallenberger and Weston 2004; Charani *et al.* 2007; Yildiz and Gumuskaya 2007). Also, at 150 °C, a low percentage of WA occurred, related to the effect of treatment on the chemical structure of wood (Charani *et al.* 2007).

**Table 10.** Change in Water Absorption of Poplar Wood as Affected by Treatment after 2 h, 24 h, and 432 h

Treatment	Water Absorption (%)		
	Immersion Time (2 h)	Immersion Time (24 h)	Immersion Time (432 h)
130 °C- 120 min	102.1 ± 30.00 fg	102.8 ± 29.88 d	148.2 ± 26.23 cd
130 °C- 120 min- 0.5% ACC	46.1 ± 6.95 ab	47.8 ± 6.89 a	125.9 ± 5.72 ab
130 °C- 120 min- 1% ACC	42.6 ± 2.54 a	44.3 ± 2.65 a	126.8 ± 12.46 ab
130 °C- 180 min	90.4 ± 9.72 ef	74.8 ± 36.44 bc	143.0 ± 9.42 bcd
130 °C- 180 min- 0.5% ACC	46.0 ± 0.73 ab	47.9 ± 1.00 a	130.6 ± 4.09 abc
130 °C- 180 min- 1% ACC	43.7 ± 5.45 ab	45.4 ± 5.36 a	115.6 ± 8.10 a
160 °C- 120 min	104.1 ± 8.68 fgh	105.0 ± 8.62 d	140.1 ± 1.91 bcd
160 °C- 120 min- 0.5% ACC	52.5 ± 2.97 abc	53.4 ± 2.92 ab	131.0 ± 10.21 abc
160 °C- 120 min- 1% ACC	61.3 ± 8.44 bcd	63.0 ± 8.60 abc	132.0 ± 11.77 abc
160 °C- 180 min	117.2 ± 20.13 gh	116.7 ± 20.04 d	158.2 ± 21.53 d
160 °C- 180 min- 0.5% ACC	64.5 ± 4.97 cd	66.7 ± 5.38 abc	132.6 ± 7.51 abc
160 °C- 180 min- 1% ACC	74.9 ± 10.32 de	81.1 ± 13.38 c	137.8 ± 9.01 bc
0.5% ACC	52.2 ± 3.03 abc	54.4 ± 2.33 ab	139.6 ± 8.81 bcd
1% ACC	51.9 ± 4.98 abc	53.7 ± 5.01 ab	140.7 ± 8.32 bcd
Control	119.9 ± 7.91 h	120.4 ± 6.64 d	203.3 ± 8.26 e

### Anti-swelling efficiency

Table 11 presents the anti-swelling efficiency (ASE, %) of the treated wood samples after 2, 24, and 432 h, respectively. The highest ASE was found in the treatment of 0% ACC and heat at 160 °C for 180 min, with values of 75.0%, 62.1%, and 54.6% after 2, 24, and 432 h, respectively. The lowest ASE after 2, 24, and 432 h was found with the wood treatment of 0.5% ACC without heating, with values of 20.6%, 20%, and 15.9%, respectively. Another study reported that volumetric swelling was decreased by 53.3% in thermally modified *E. grandis* wood (Calonego *et al.* 2012). In the absence of ACC preservative the cell cavity is empty, and therefore the main function of water sorption was due to imbibition of the cell lumen, but in case of use of ACC preservative water sorption was mainly due to the cell wall. It seems that water vapor from water in the cell cavity decreased ASE of samples.

Three soaking times were used, during which the ASE of poplar wood samples (%) was reduced; these results are in agreement with those of Yildiz (2004a) for beech wood. Charani *et al.* (2007) found that the best ASE value for beech wood was achieved at 170 °C with 1 h of treating time and three steps of soaking measurement by hydrothermal treatment. Also, an increase in the ASE of wood was reported with increasing exposure temperature (130 and 150 °C) and time (2, 6, and 10 h) (Yildiz *et al.* 2004b).

**Table 11.** Change in Anti-swelling Efficiency of Poplar Wood as Affected by Treatment after 2 h, 24 h, and 432 h

Treatment	Anti-swelling Efficiency (%)		
	Immersion Time (2 h)	Immersion Time (24 h)	Immersion Time (432 h)
130 °C- 120 min	35.7 ± 12.92 bcd	35.2 ± 12.55 bcdef	29.0 ± 9.34 abcd
130 °C- 120 min- 0.5% ACC	34.1 ± 13.06 bcd	29.1 ± 2.15 abcd	20.9 ± 4.64 ab
130 °C- 120 min- 1% ACC	27.3 ± 7.15 ab	25.9 ± 7.23 abc	21.3 ± 7.99 ab
130 °C- 180 min	46.1 ± 4.71 d	43.0 ± 5.27 ef	37.8 ± 7.06 cd
130 °C- 180 min- 0.5% ACC	38.7 ± 12.80 bcd	32.6 ± 5.87 bcde	32.0 ± 14.70 bcd
130 °C- 180 min- 1% ACC	27.4 ± 9.72 ab	26.0 ± 8.56 abc	21.1 ± 7.49 ab
160 °C- 120 min	46.0 ± 5.53 d	45.5 ± 5.31 f	40.7 ± 5.55 d
160 °C- 120 min- 0.5% ACC	36.3 ± 5.42 bcd	35.7 ± 5.15 cdef	28.0 ± 4.23 abcd
160 °C- 120 min- 1% ACC	30.8 ± 3.72 abc	29.2 ± 3.51 abcd	24.6 ± 2.08 abc
160 °C- 180 min	75.0 ± 4.55 e	62.1 ± 11.26 g	54.6 ± 13.91 e
160 °C- 180 min- 0.5% ACC	41.8 ± 4.41 cd	40.2 ± 3.97 def	26.7 ± 11.45 abc
160 °C- 180 min- 1% ACC	31.0 ± 4.02 abc	29.2 ± 4.06 abcd	27.6 ± 4.74 abcd
0.5% ACC	20.6 ± 9.46 a	20.0 ± 9.14 a	15.9 ± 7.81 a
1% ACC	25.4 ± 4.92 ab	24.0 ± 4.80 ab	24.0 ± 4.02 ab

### Volumetric swelling

Table 12 present the volumetric swelling (VS, %) in the treated wood samples after 2, 24, and 432 h, respectively. The same trend was found as in VS. The lowest VS was found in treatment of 0% ACC and heat at 160 °C for 180 min, with values of 4.6%, 7.2%, and 9.3% after 2, 24, and 432 h, respectively. The highest VS with respect to the control after 2, 24, and 432 h was found by wood treatment at 0.5% ACC without heating, with values of 14.6%, 15.2%, and 17.1%, respectively.

**Table 12.** Change in Volumetric Swelling of Poplar Wood as Affected by Treatment after 2 h, 24 h, and 432 h

Treatment	Volumetric Swelling (%)		
	Immersion Time (2 h)	Immersion Time (24 h)	Immersion Time (432 h)
130 °C- 120 min	11.8 ± 2.37 bcd	12.3 ± 2.38 bcd	14.5 ± 1.91 bcde
130 °C- 120 min- 0.5% ACC	12.1 ± 2.40 bcde	13.4 ± 0.41 def	16.1 ± 0.94 de
130 °C- 120 min- 1% ACC	13.4 ± 1.31 def	14.1 ± 1.37 def	16.0 ± 1.63 de
130 °C- 180 min	9.9 ± 0.87 b	10.8 ± 1.00 bc	12.7 ± 1.44 bc
130 °C- 180 min- 0.5% ACC	11.3 ± 2.35 bcd	12.8 ± 1.12 cde	13.9 ± 3.00 bcd
130 °C- 180 min- 1% ACC	13.3 ± 1.79 def	14.0 ± 1.63 def	16.1 ± 1.53 de
160 °C- 120 min	9.9 ± 1.01 b	10.3 ± 1.01 b	12.1 ± 1.13 b
160 °C- 120 min- 0.5% ACC	11.7 ± 1.00 bcde	12.2 ± 0.98 bcd	14.7 ± 0.86 bcde
160 °C- 120 min- 1% ACC	12.7 ± 0.68 cdef	13.4 ± 0.67 def	15.4 ± 0.42 de
160 °C- 180 min	4.6 ± 0.84 a	7.2 ± 2.14 a	9.3 ± 2.84 a
160 °C- 180 min- 0.5% ACC	10.7 ± 0.81 bc	11.3 ± 0.75 bc	14.9 ± 2.33 cde
160 °C- 180 min- 1% ACC	12.7 ± 0.74 cdef	13.4 ± 0.77 def	14.8 ± 0.96 cde
0.5% ACC	14.6 ± 1.74 f	15.2 ± 1.73 f	17.1 ± 1.59 e
1% ACC	13.7 ± 0.90 ef	14.4 ± 0.91 ef	15.5 ± 0.82 de
Control	18.4 ± 0.72 g	19.0 ± 0.74 g	20.4 ± 0.38 f

With hydrothermally treated poplar wood, the thickness swelling was reduced (Yildiz 2004a), which could be related to the chemical modification in the fiber cell walls during the hydrothermal treatment (Rowell and LeVan-Green 2005). Hemicelluloses that were degraded due to thermal elevation could affect the dimensional stability of wood (Garrote *et al.* 1999; Tjeerdsma and Militz 2005). Thermal treatment of *Picea abies* at 180 to 220 °C caused a reduction of 5% in density at 0% moisture content (Arnold 2010). *Pinus sylvestris* wood thermally modified at 165 to 180 °C presented reductions in tangential and radial swellings of up to 33% and 44%, respectively (Militz and Tjeerdsma 2001). Also, the reduction of the thickness swelling could be related to the increase of the crystalline regions in the cellulose microfibrils (Wallenberger and Weston 2004; Yildiz and Gümüşkaya 2007). The swelling loss may have occurred as a result of esterification of the cellulose microfibrils (Tjeerdsma and Militz 2005; Boonstra and Tjeerdsma 2006).

Overall, the degradation of wood components and particularly of hemicelluloses weakens the wood (Yildiz *et al.* 2006; Korkut *et al.* 2008). Further progress of thermo-modified materials will depend on developments that permit their utilization in use Classes 3 and 4 (EN 335-2 2007). Subsequently, before thermal treatment, the impregnation with borax, which means the combination of boron impregnation and thermo-modification, could be an interesting method to improve the properties of thermally modified wood. In particular, properties such as resistance to fungi and insects and resistance to wood fire could be improved by this method (Salman *et al.* 2014).

## CONCLUSIONS

In this study, the effects of hydrothermal treatment of modified poplar wood treated with acid copper chromate (ACC) at various concentrations was investigated to study some physical properties, as well as effect of the treatments on mass loss and hardness of wood incubated with white-rot fungus (*Trametes versicolor*).

1. The highest values of hardness of wood degraded by *T. versicolor* were observed in wood treated with 1% and 0.5% ACC without heating temperature, with values of 6.447 and 4.184 kN, respectively.
2. The respective treatments with 1% ACC without hydrothermal treatment, treatment by heating at 160 °C for 120 min with 0.5% ACC, and unheated wood with 0.5% ACC showed the lowest WL values of poplar wood, at 3.03%, 4.66%, and 7.21%.
3. None of the treatments used showed significant effects on the oven-dry density ( $D_o$ ) of the wood samples.
4. The lowest mass reduction (MR) was observed in wood samples treated with 0% and 0.5% ACC and heated at 130 °C for 180 min, with values of 3.72% and 3.99%, respectively.
5. The lowest water absorption (WA) after 2, 24, and 432 h was found for wood treatment of 1% ACC and heat at 130 °C for 120 min, 1% ACC and heat at 130 °C for 120 min, and 1% ACC and heat at 130 °C for 180 min, with values of 42.6%, 44.3%, and 115.6%, respectively.
6. The highest anti-swelling efficiency (ASE) was found with treatment of 0% ACC and heat at 160 °C for 180 min, with values of 75.0%, 62.1%, and 54.6% after 2, 24, and 432 h, respectively.
7. The lowest volumetric swelling (VS) was found with treatment of 0% ACC and heat at 160 °C for 180 min, with values of 4.6%, 7.2%, and 9.3% after 2, 24, and 432 h, respectively.

## ACKNOWLEDGMENTS

The authors are grateful for the support of the Department of Wood Science and Paper Technology, Karaj Branch, Islamic Azad University, Karaj, Iran.

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Article submitted: January 24, 2016; Peer review completed: March 30, 2016; Revised version received and accepted: April 7, 2016; Published: April 14, 2016.

DOI: 10.15376/biores.11.2.4850-4864