Improving impregnation properties of fir wood to acid copper chromate (ACC) with microwave pre-treatment

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Effects of microwave pre-treatment on impregnation properties in fir wood (*Abies alba* L.) with acid copper chromate (ACC) were studied here. Flat-sawn specimen boards were prepared with moisture content (MC) of 40 ± 5% and were exposed to microwave radiation with 2450 MHz frequency for 10, 12, 14, and 16 minutes at four different radiation treatments. Microwave-treated specimens, along with the control specimens, were conditioned to the final MC of 12% and then impregnated with 5%-ACC solution, using an empty-cell process. The impregnation properties were then measured, including retention, maximum and minimum depths of penetration, impregnated area in the cross-section, and ACC-leaching. Image J software was used to determine depths of penetration. Results showed that microwave pre-treatment significantly improved all the impregnation properties, with the exception of leaching. Clear direct relation was found between the duration of microwave radiation with the properties. It can be concluded that microwave pre-treatment can be used to significantly improve impregnation properties in fir wood.

Keywords: ***Abies Alba*, Fir Wood, Impregnation, Microwave Radiation, Pre-treatment**

Introduction

Species with low wood permeability, such as fir (*Abies alba* L.), cause many problems during the wood impregnation process with preservatives. Fir is a gymnosperm species with *torus margo* pit membrane; the low permeability is mainly due to the pit aspiration during drying. Different modification methods, such as steaming, mechanical incising, drilling techniques and bio-incising were tested to improve the impregnation properties of such refractory wood species (Schwarze et al. 2006, Lehringer et al. 2009, Dashti et al. 2012a). Microwave (MW) radiation is an innovative method to increase the wood permeability and thus to improve the preservative penetration in the wood of various species (Vinden et al. 2003, Brodie 2009, Torgovnikov & Vinden 2009, Dashti et al. 2012b). Due to the high efficiency in converting electricity into microwave radiation, energy saving, in-depth heating of materials and reduced costs, microwave pretreatment is spreading in many industries (Torgovnikov & Vinden 2010). The increasing effect of microwave radiation on the wood permeability was reported to be due to changes in the wood porous structure (Lu et al. 1994, Zhao et al. 1998, Yu et al. 2002). Torgovnikov & Vinden (2009) mentioned that when the microwave energy was applied to wood, the steam pressure generated within the wood cells provokes the rupture of the pit membranes on cell walls and the weak ray cells, allowing an easier fluid transfer. Liu et al. (2005) reported that the permeability of larch wood can be improved without noticeable reduction in the strength and stiffness if the conditions of microwave pretreatment were optimized. Treu & Gjolsjo (2008) also reported that the microwave processing of Norway spruce (*Picea abies* L.) caused a significant increase in the uptake of a 2% copper-based preservative after wood modification by microwave energy of more than 50 kWh/m^3 at a frequency of 2.45 GHz. Hong-Hai et al. (2005) also found that the water uptake of Larch (*Larix olgensis*) wood irradiated by microwave was 2.5 to 3.3 times greater than that of non-radiated wood. Dashti et al. (2012b) found that the microwave radiation of fir wood (*Abies alba* L.) at frequency of 2.45 GHz for 7 and 10 minutes can increase the wood radial permeability. They also reported that the torus of some bordered pits was hydrolyzed due to the microwave radiation; however, they did not study the relationship of MW pre-treatment on the amount of preservative uptake, depth of penetration, or other impregnation properties. This species is vastly grown in different parts of the world and therefore provide a continuous raw material for commercial applications; huge amount of fir lumbers are also imported to Iran on a regular basis to satisfy the growing needs for commercial woods. Because of the importance of fir species in timber industry and its various applications, the present research was carried out to evaluate the effects of microwave radiation conditions on the impregnation properties of fir wood (*Abies alba* L.) with ACC (acid copper chromate) preservative.

Materials and methods

Sampling and microwave pre-treatment

Ten flat-sawn boards of fir wood (*Abies alba* L.) with green dimensions of 400 × 15 × 5 cm and average moisture content (MC) of 40 ± 5% were used for the study. From each board, five specimens were prepared; one as the control specimen without MW-radiation, and the other four specimens to be MW-radiated under four different MW-treatments. The dimensions of specimens were 250 × 100 × 50 mm. Density of the specimens at moisture content of 8% was 0.32 g cm^{-3}. A domestic Samsung microwave oven (CE3780AT) with frequency of 2450 MHz was used for microwave radiation; the output power was 900 W. Four MW-treatments were used (Tab. 1). The microwave radiation was stopped for 60 to 90 sec (rest time) after every 60 to 150 sec of microwave exposure to prevent cracking due to the rapid wood moisture removal. Specimens were exposed to MW one at a time. The total MW exposure times were 300, 450, 480, and 600 sec for MW1, MW2, MW3, and MW4, respectively. Before and after the MW-radiation, specimens were weighted using a digital scale with 0.01 g precision, in order to measure the reduced weight. Due to hygromechanical behavior of wood (Figueroa et al. 2012), after microwave treatment the sam-

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Tab. 1 - Microwave radiation treatments applied to fir wood specimen boards (*Abies alba* L.).

Micro-Wave Treatment	MW power (kW)	Time of MW power application (s)	Number of MW power exposures	Total time of MW exposure (s)	Energy applied in every exposure (kJ)	Time of cooling between MW exposures (s)	Total cooling time (min)
MW1	0.90	60	5	300	54	60	5
MW2	0.90	90	5	450	81	60	5
MW3	0.90	120	4	480	108	90	9
MW4	0.90	150	4	600	135	90	9

ples were conditioned to 12% moisture content at relative humidity of 65% and temperature of 20 °C.

Impregnation method

Before impregnation, the samples were end coated using two layers of paraffin to prevent the preservative flow through the longitudinal direction. Samples were then impregnated with ACC, using Rueping method. The used preservative solution was a mixture of Copper Sulfate, Sodium Dichromate and Chromic Acid with concentration of 50%, 48.3% and 1.7%, respectively. The diagram of impregnation process is shown in Fig. 1.

Measuring impregnation properties

After the impregnation, the preservative retention was calculated as follows (eqn. 1):

$$R=\frac{M_i-M_n}{V_w}\cdot\frac{C_p}{100}$$

where R (retention) is the amount of wood preservative retained in the wood specimen (kg of preservative per m^3 of wood), M_i is the mass of wood specimen after impregnation (kg), M_n is the mass of wood specimen before impregnation (kg), V_w is the volume of the specimen (m^3) and C_p is the concentration of the preservative in the impregnation solution (kg kg^{-1}).

The depth of preservative penetration was measured using Chrome Azurol S (color index No.43825, also known as mordant blue 29) reagent according to AWPA-A3-84 standard (AWPA 1986). To prepare this reagent, 0.5 g Chrome Azurol S and 5 g sodium acetate were dissolved in 80 ml distilled water and the solution was diluted to a volume of 300 mL. Then, this reagent was sprayed on the cross section of impregnated specimens. As a result, ACC-impregnated areas appeared in blue color and un-impregnated surfaces changed to red color. Depth of penetration was measured at four different sides in each specimen with a digital caliper with 0.1 mm precision; finally, the maximum and minimum depths of penetration were determined. The impregnation area (%) on the wood cross section was measured by the aid of the software Image J. The leaching tests were conducted based on EN-84 standard. Five specimens with dimensions of 50 × 25

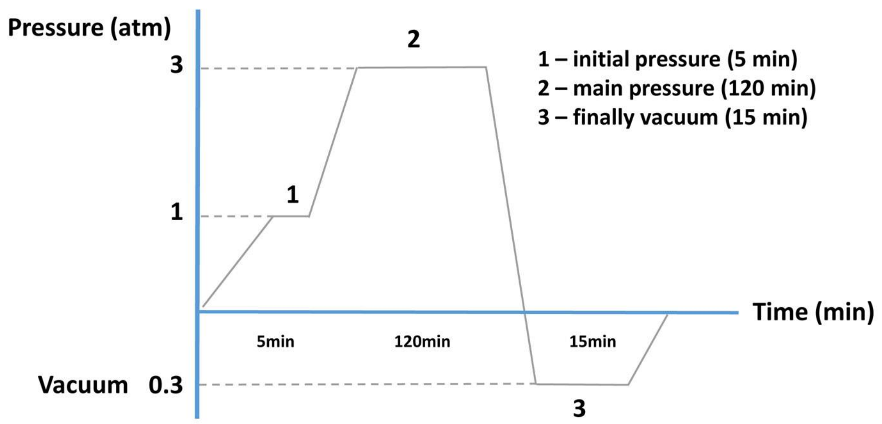

Fig. 1 - Diagram of the impregnation method used (Rueping).

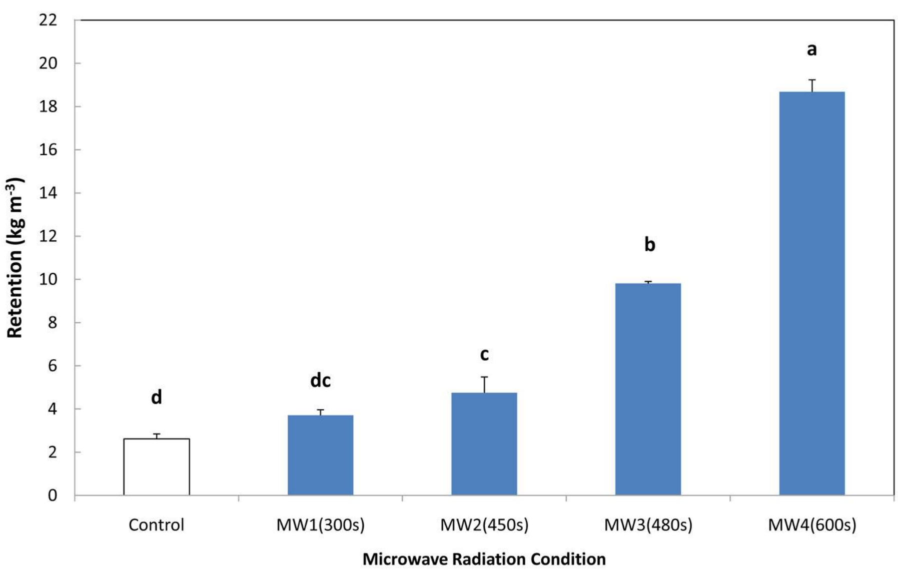

Fig. 2 - Acid Copper Chromate (ACC) retention in the microwave radiated and un-radiated wood specimens of fir wood ± SD. Different letters indicate significant differences among the means according to Duncan's test (N=5).

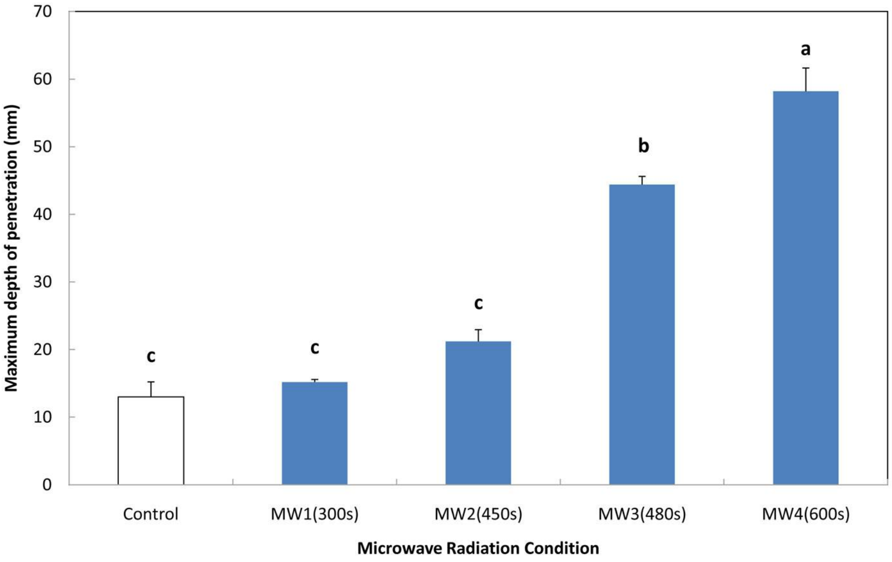

Fig. 3 - Maximum depth of ACC penetration (mm) through the microwave radiated and un-radiated wood specimens of fir wood ± SD. Different letters indicate significant differences among the means according to Duncan's test (N=5).

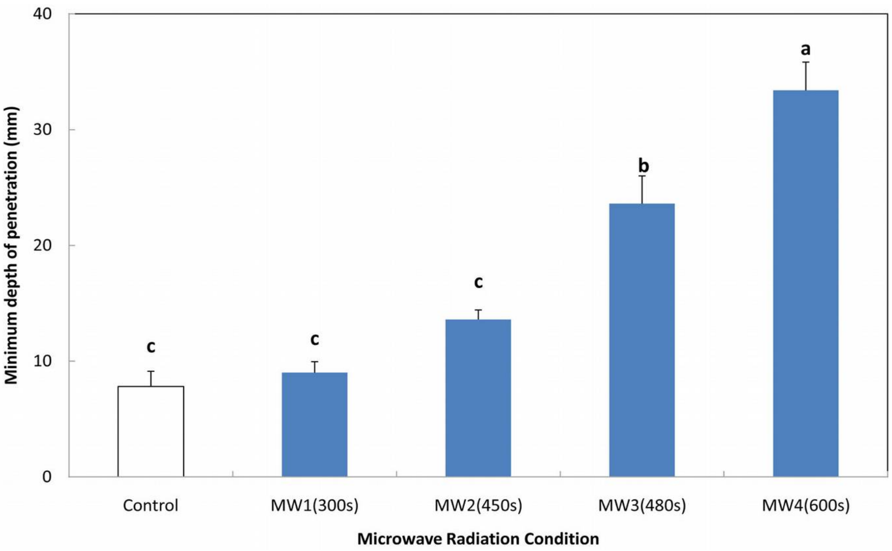

Fig. 4 - Minimum depth of ACC penetration (mm) through the microwave radiated and un-radiated wood specimens of fir wood ± SD. Different letters indicate significant differences among the means according to Duncan's test (N=5).

× 15 mm were prepared from each treatment and kept in conditioning chamber for 3 weeks (relative humidity of 65 ± 5% at 20 ± 2 °C). Specimens were then placed in a desiccator located in separate test vessels, fitted with a vacuum pump, capable of maintaining a pressure of 4 kPa. Specimens were ballasted with weights to prevent from floating and then flooded with water. Specimens were immersed in water for 14 days. At the end of the first and second day of immersion, water was changed; water was further changed seven times in the remaining 12 days at intervals of two days. For each MW-treatment, five replications were used.

ANOVA was applied to the collected data ($\alpha = 0.05$) after their normal distribution was verified. The significance of the differences among the means of MW-treatments was tested using the Duncan's multiple range test by SPSS (v. 2011) software. To summarize the differences among MW-treatments based on more than one property simultaneously, a hierarchical cluster analysis was carried out using the Ward's method on a squared Euclidean distance matrix.

Results

All the microwave treatments increased the preservative retention in the wood specimens (Fig. 2). The preservative retention was improved by increasing the microwave radiation period from 10 to 16 minutes. However, the difference between non-impregnated wood specimens and those MW-radiated for 10 min was not statistically significant. The preservative retention for the specimens MW-radiated for 16 min was outstanding and ranged from 16.55 to 19.56 (kg m^{-3}). The retention of 16 min MW-radiated specimens was improved by almost 7 times (612%) compared to that for the un-radiated ones. The preservative retention for non-impregnated wood specimens ranged from 1.74 to 2.98 (kg m^{-3}).

The preservative penetration through the wood specimens was improved due to microwave exposure and the improvement was remarkable for the samples that were MW-radiated for 14 and 16 min (Fig. 3, Fig. 4). The average maximum depth of preservative penetration at 16 min of radiation was 58.2 mm compared to 13 mm for the un-radiated specimens. The minimum depth of penetration was also improved by almost 4 times (328%) as a result of microwave radiation for 16 min.

Preservative penetrated along the ray cells easily reached the central parts of the MW-radiated specimens (Fig. 5). In the control specimens though, no penetration along the ray cells was observed. The significant effect of MW-radiation on penetration of preservative to the central parts of specimens was quite obvious through the measurement of the impregnated area on the cross-sections of specimens (Fig. 6); the impregnated area was the lowest in the control specimens in comparison to other treatments.

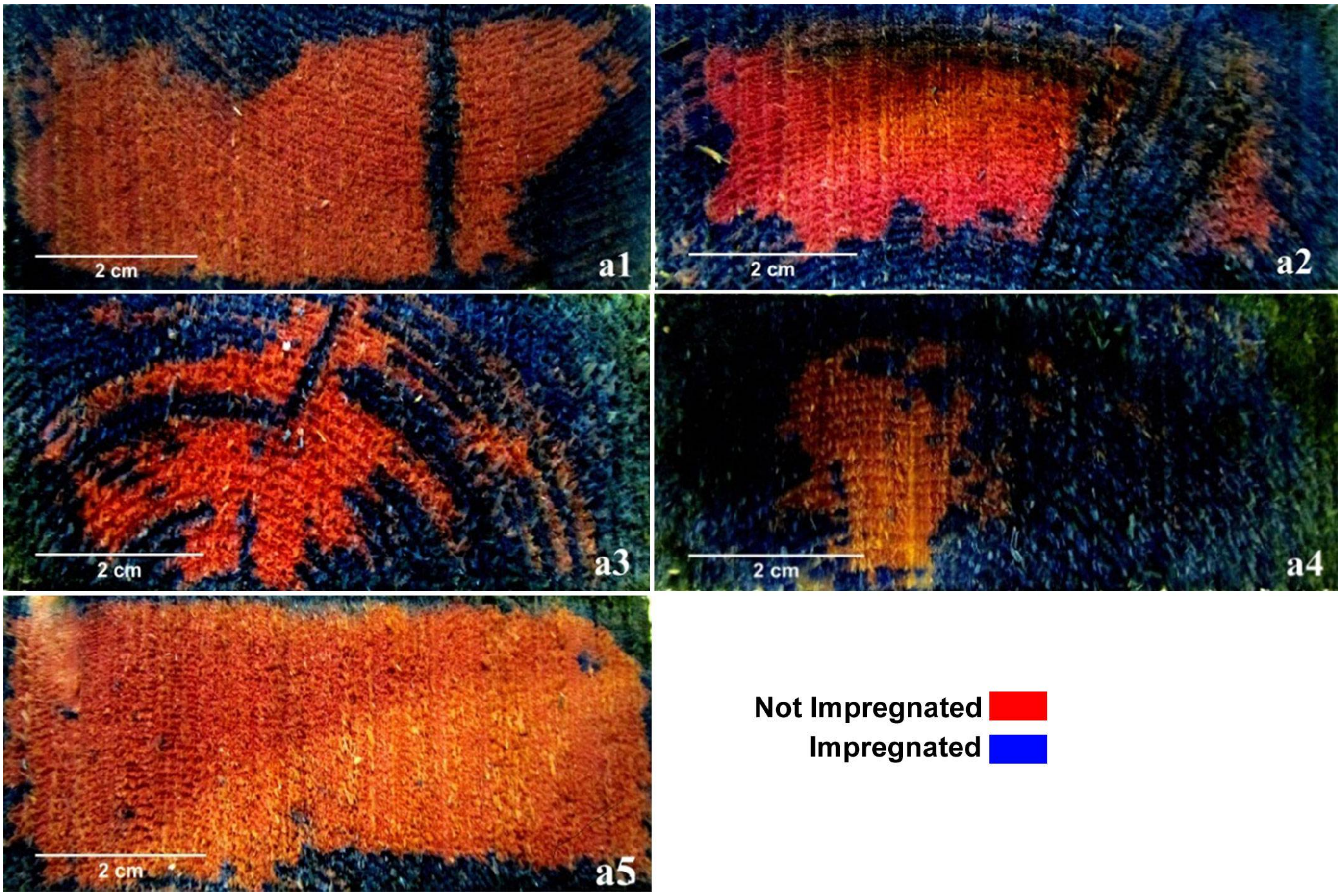

Fig. 5 - Cross-section of impregnated samples, (a1): MW1; (a2): MW2; (a3): MW3; (a4): MW4; (a5): control.

There was no significant difference in the leaching of ACC preservative between MW-radiated and un-radiated specimens (Fig. 7). However, the amount of leaching tended to decrease as the MW-energy increased (refer to Tab. 1). The lowest leaching were observed in MW3 and MW4, although not significantly different from other MW-radiated or the control specimens. The increase in MW also increased the amount of the reduced weight (Fig. 8). In fact, the reduced weight increased as the MW-energy increased. The highest reduced weights were observed in MW3 and MW4 treatment.

Cluster analysis, based on all the impregnation properties measured in the present study (retention, maximum and minimum depths of penetration, impregnated area in the cross-section, and ACC leaching) showed that microwave pre-treatments for 14 and 16 minutes were significantly grouped (Fig. 9), while pre-treatments of 10 and 12 minutes were closely clustered to the control specimens.

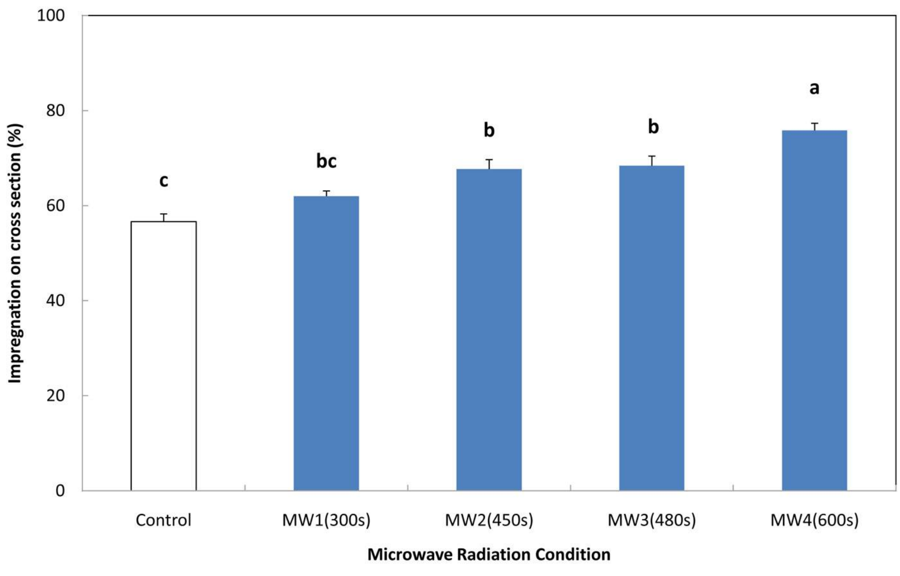

Fig. 6 - Impregnated area (%) measured at the cross-sectional view of the specimens cut from the middle ± SD. Different letters indicate significant differences among the means according to Duncan's test (N=5).

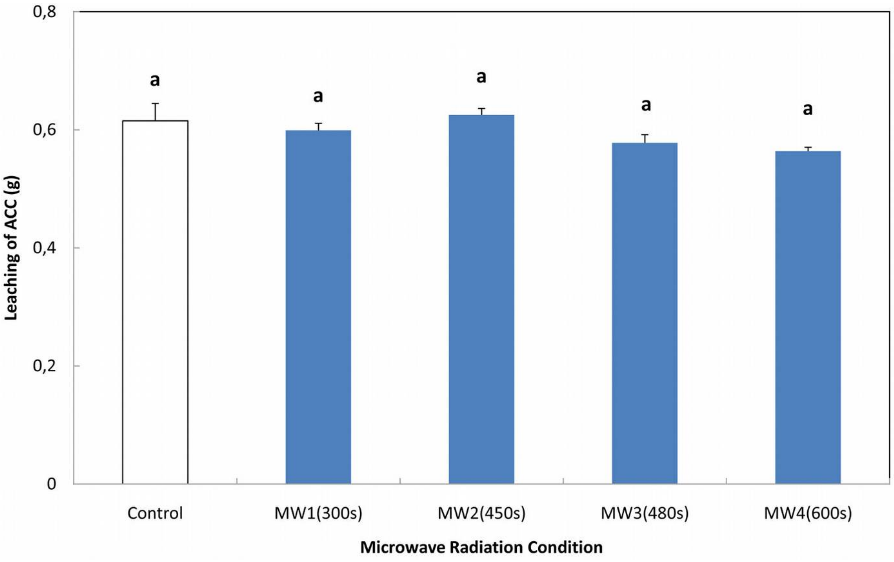

Fig. 7 - ACC leaching from the microwave radiated and un-radiated wood specimens of fir wood ± SD. Different letters indicate significant differences among the means according to Duncan's test (N=5).

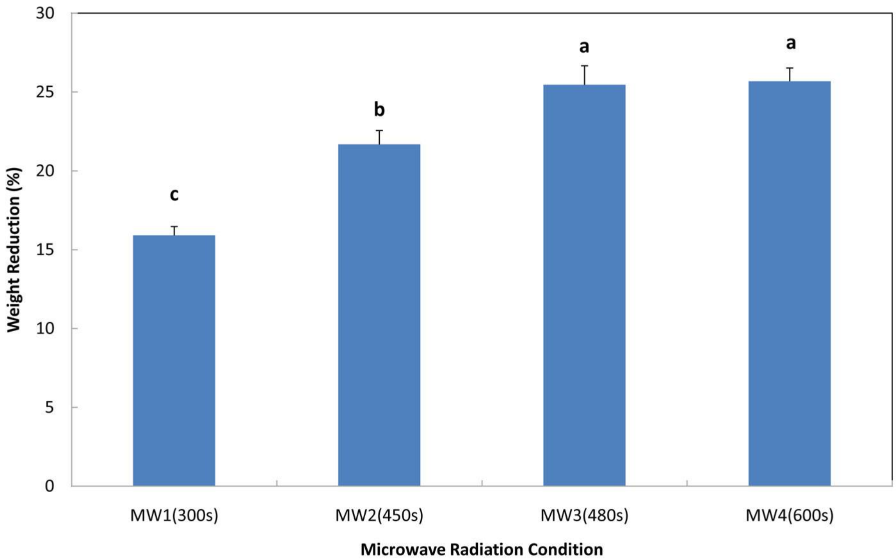

Fig. 8 - Reduction in the weight of specimens after the microwave radiation in comparison to their initial weight (before MW-radiation) ± SD. Different letters indicate significant differences among the means according to Duncan's test (N=5).

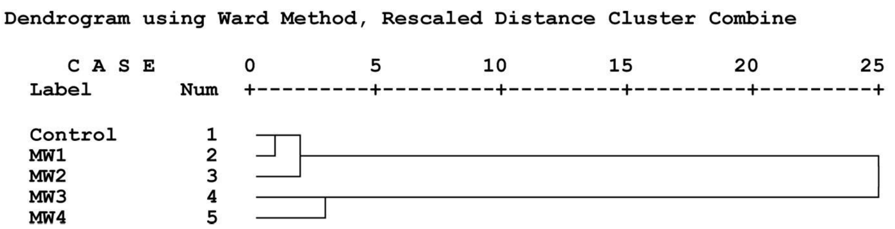

Fig. 9 - Cluster analysis of different treatments based on all the impregnation properties ± SD (retention, maximum and minimum depths of penetration, impregnated area in the cross-section, and ACC leaching).

Discussion

The retention of 16 min MW-treated specimens was improved by almost 7 times compared to that of the un-radiated ones. This improving effect caused by MW-treatment was also reported by Treu & Gjolsjo (2008) for Norway spruce (*Picea abies*). Hong-Hai et al. (2005) also found that the water uptake of Larch (*Larix olgensis*) wood radiated by microwave was 2.5 to 3.3 times greater than that of the un-radiated wood. The effect of MW on the preservative retention can be attributed to ray cell rupture. The effect of MW on preservative penetration can also be attributed to a radial wood permeability improvement as reported by Dashti et al. (2012b). The ruptures in the ray parenchyma caused by the microwave radiation facilitated the penetration of preservative. In fact, penetration of any kinds of fluid into a porous media is directly related to its structure (Taghiyari 2013); therefore, any small alterations in the porous structure by pressure (Taghiyari 2012), heat (Ghorbani et al. 2012), or cold (Taghiyari et al. 2012) would significantly change the permeation properties. Moreover, the maximum weight reduction by microwave treatment occurred in MW4; consequently, it can be inferred that the maximum shrinkage and widening of pits was also happened in this treatment. Ultimately, this widening of pits resulted in a significant improvement in penetration of ACC. The radial permeability of softwood is significantly controlled by ray parenchyma rather than by bordered pits (Tarmian & Perre 2009). Previous studies have shown that the permeability of some wood species in the radial direction can be increased by

170-1200 times due to microwave radiation (Torgovnikov & Vinden 2000). Results of Image J software studies also showed that the impregnation amount on the cross section increased due to microwave radiation; the greatest improvement was observed for specimens radiated for 16 min.

Results of the present study showed that the effect of microwave pre-treatments on leaching of ACC as a water-based preservative was not statistically significant. However, the increasing effects of microwave post-treatment on leaching resistance of copper-based preservatives were previously reported (Zhang & Kamdem 2000, Cao & Kamdem 2004, Yu et al. 2010). Therefore, further complementary studies should be done to understand these contradictions. In the meantime, a fixation method is recommended to be applied to prevent the preservative from leaching.

Cluster analysis, based on all the impregnation properties measured in the present study showed that microwave pre-treatments for 14 and 16 minutes were significantly grouped. So, from an economical point of view, microwave pre-treatment of 14 minutes may be recommended due to the shorter time and thus lower costs. Furthermore, pre-treatments of 10 and 12 minutes were closely clustered to the control specimens. It can then be concluded that these two exposure time may not be recommended. As the difference between MW2 and MW3 was only 2 minutes, it may be concluded that the critical point was between 12 and 14 min of microwave exposure. Further studies on optimal time-intervals as well as on mechanical properties are recommended.

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