EFFECT OF PRE-STEAMING ON MASS TRANSFER PROPERTIES OF FIR WOOD (*ABIES ALBA* L.); A GYMNOSPERM SPECIES WITH TORUS MARGO PIT MEMBRANE

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In this research, the effect of pre-steaming on mass transfer properties, including air permeability and water vapor diffusivity of fir wood (*Abies alba* L.), a gymnosperm species with torus margo pit membrane, was evaluated. The pre-steaming was performed at temperatures of 120, 140, and 160°C for 1 hour under a pressure of 2-3 bars. Then, the pre-steamed specimens were conventionally dried at a constant temperature of 160°C and a relative humidity of 50% to the final moisture content of 10%. Subsequently, the mass transfer properties of the dried specimens were measured in longitudinal and radial directions. Overall, the pre-steaming was found to be an effective modification method to improve the mass transfer properties of *Abies alba* L. The improvement was more remarkable for the air permeability as well as through the radial direction. The specimens steamed at the temperature of 160°C had higher mass transfer rates than those steamed at the temperatures of 120 and 140°C. Results of chemical analyses, FT-IR spectroscopy, and SEM imaging provide some explanations for the effects of pre-steaming.

Key Words: Air permeability; Fir wood; Pre-steaming; Water vapor diffusivity

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INTRODUCTION

Fir wood (*Abies alba* L.), which is a gymnosperm species with a torus margo pit membrane, is characterized by its low permeability. Drying the gymnosperm species of the Pinaceae family, such as fir wood with a torus margo pit membrane, results in pit aspiration and the pit apertures are closed by a torus. Due to occurrence of this phenomenon, the drying rate in the domain of free water and permeability of the dried wood decrease to an extreme degree, and therefore it can be more difficult to achieve consistent penetration with preservative chemicals.

Some pretreatments applied before drying, such as pre-steaming, can be effective to improve the wood permeability (Alexiou et al. 1990; Kanagawa et al. 1992; Morris et al. 1997; Matsumura et al. 1999). Pre-steaming can cause tremendous changes in the wood quality through its anatomical and chemical modification (Zhang and Cai 2008). For example, chemical reactions can develop between chemical compounds of cell walls and extractive materials due to steaming at high temperatures (Zhang and Cai 2006).

Zhang and Cai (2008) observed some fractures in the pits between ray parenchyma cells and earlywood tracheids of subalpine fir as a result of steaming. The influence of steaming on the ultrastructure of a bordered pit membrane was studied by Nicholas and Thomas (1968). They used loblolly pine and found that components in the pit membrane are hydrolyzed during steam treatment.

Studies by Kanagawa et al. (1992) revealed that steaming at low pressure (2 to 3 atm) can improve the permeability of Japanese cedar wood (*Cryptomeria japonica* L.). In contrast, Cai and Oliveira (2007) reported that there was no significant change in the gas permeability both in longitudinal and transverse directions of subalpine fir (*Abies lasiocarpa* Hook) after a 4 hour steaming pretreatment at green conditions and at FSP. Morris et al. (1997) found that pre-steaming of Western hemlock to a core temperature of 82°C in 4 hours prior to pressure borate treatment can be a very effective means of improving uptake during pressure treatment. Alexiou et al. (1990) found that pre-steaming of regrowth *Eucalyptus pilularis* (blackbutt) can significantly increase the longitudinal permeability, as tyloses appeared unaltered. Jianxiong et al. (1994) investigated the effect of steaming on the permeability of spruce (*Picea jezoensis* var. *komarovii*), fir (*Abies nephrolepis*), and pine (*Pines koraiensis*) woods. They reported that the effect of steaming on the permeability of wood is not similar for different wood species with different properties. They indicated that the increase in permeability of the steamed wood was attributable to the development of cracks in the pit membrane and torus after steaming. Matsumura et al. (1999) found that, in contrast to radiata pine sapwood, the impregnation property of the heartwood was strongly influenced by pre-steaming the green lumber. They also found that samples having increased uptake after pre-steaming also have lower extractive contents. Matsumura et al. (1996) also found that there were significant correlations between the permeability of wood samples and the methanol-soluble extractive content.

In addition to the wood permeability, the water vapor diffusivity of wood also can be modified by steaming. The moisture diffusion coefficient of wood is involved in many industrial operations (drying, bending, peeling, and coating) as well as during the life of wooden structures (flooring, roof structure, and furniture). Choonge et al. (1999) reported that prolonged steaming increased the moisture diffusivities of southern pine above and below the FSP, partially due to changes in the extractive distribution profile. Rousset et al. (2004) stated that the thermal treatment of poplar (*Populus robusta*) at 200°C reduces the value of mass diffusivity, probably due to the chemical modification of the cell wall. However, the permeability value does not change significantly, probably because the thermal treatment has a slight impact on the porous structure of wood. The present research aims to evaluate the effect of pre-steaming at high temperatures on the mass transfer properties of fir wood.

MATERIALS AND METHODS

Sampling and Pre-steaming Method

Fir wood (*Abies alba* L.) flat-sawn boards with green dimensions of 340 × 100 × 50 mm and initial moisture content of 45-50% were selected for the study. Pre-steaming was applied at three temperatures of 120, 140, and 160°C for 1 hour under a pressure of 2-3 bars inside a laboratory steaming device. Four replications were considered for each set of experiment.

Drying Method

The unsteamed and steamed boards were end-coated using oil paint to avoid moisture flow through the end sections. Subsequently, they were conventionally dried inside a laboratory kiln at a constant temperature of 60°C and relative humidity of 50% to the final moisture content of 10%.

Air Permeability Measurement

Eight cylindrical specimens, 18 mm in diameter and 10 mm in thickness, were taken from dried boards in radial and longitudinal directions. Then, the lateral surfaces of the specimens were coated using an epoxy resin to prevent any lateral flow. Figure 1 depicts a schematic of the experimental apparatus used for the air permeability measurement (Taghiyari et al. 2010; Taghiyari and Sarvari 2010). This apparatus applies the falling water displacement method (Siau 1995) to measure the air permeability in specimens. The specific permeability (K) was calculated using Siau's equations (Siau 1995),

$$K = \eta . k_g \tag{1}$$

where K is the specific permeability (m3 m-1), η is the viscosity of air (η =1.81×10-5 Pa s), and k_g is the superficial permeability that can be determined as follows,

$$k_g = \frac{\alpha V_d CL(P_{atm} - \beta \bar{z})}{\beta t A \bar{z}(P_{atm} - \gamma \bar{z})} \tag{2}$$

where k_g is the superficial gas permeability coefficient (m^2/ Pa s), V_d is the volume of apparatus between points 1 and 2 (m^3), P_{atm} is the atmospheric pressure (m Hg), L is the length of wood specimen (m), z is the average height of water over surface of reservoir during period of measurement (m), A is the cross sectional area of wood specimen (m^2), t is the time (s), values of α, β, and γ are given in Siau (1995), and C is the correction factor for gas expansion as a result of change in static head and viscosity of water. The parameter of C can be calculated using equation 3,

$$C = 1 + \frac{\beta V_r z}{V_d (P_{atm} - \beta \bar{z})} \tag{3}$$

where Vr is the total volume of apparatus above point 1 [including volume of hoses] (m^3) and Δz is the change in height of water during time (m).

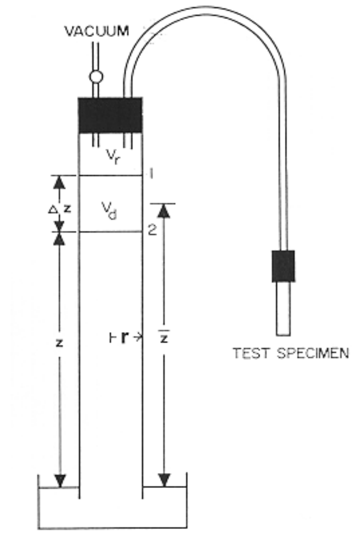

Fig. 1. Schematic view of the air permeability measurement apparatus (Siau 1995)

Water Vapor Diffusivity Measurement

Water vapor diffusivity measurement was carried out on the same specimens as used for the air permeability test. The cup method was used to measure the diffusion coefficient. The method is based on Fick's law of diffusion in steady-state conditions. Before placing the sample in the cup, silicone-based grease was applied on the lateral surfaces of the specimen to avoid any flow in the microporous layer formed between the sample and rubber surface. The saturated salt solution of sodium chloride (NaCl) was used to control the relative humidity inside the cup at about 75%. After preparation, the cups were placed inside a climatic chamber set at 65% RH. Water vapor diffuses from inside the cup with a higher RH_2 (75%) to outside with a lower RH_1 (65%). The cups were weighed every 24 h until a constant weight was reached. Then, dimensionless diffusivity *f* is calculated according to the following formula,

$$f = \frac{Q}{D_v A} \times \frac{L}{\left(RH_2 - RH_1\right) P_{vs}(T)} \times \frac{RT}{M_v} \tag{4}$$

where Q is the measured mass flux (kg.s^{-1}), A is the cross section of sample (m^2), M_v is the molar weight of vapor (kg.mole^{-1}), RH_1 is the relative humidity inside the climatic chamber, RH_2 is the relative humidity inside the cup, R is the constant of perfect gas, L is the sample thickness (m), P_{VS} is the pressure of saturated water vapor in temperature of $T(K)$, and D_V is the binary diffusion coefficient of water vapor in air. For a detailed description of the used method, readers can refer to Tarmian et al. (2012).

Anatomical and Chemical Analyses

To closely analyze the effect of pre-steaming on the structure of cell walls, Scanning Electron Microscope (SEM) studies were conducted. To determine chemical composition, the specimens were ground in Wiley mill to pass a 40-mesh screen. Then, prior to determining the holocellulose and lignin content, wood flour was extracted with acetone overnight in a Soxhlet extractor. Lignin and holocellulose contents of each specimen were analyzed according to TAPPI T 204cm-97 and TAPPI T 249cm-75 standards, respectively. In addition, acetone-soluble and water-soluble extractive contents were also determined based on TAPPI standard methods, TAPPI T 207cm-99 and TAPPI T 222cm-98, respectively. FT-IR testing was also applied for all treatments. For these tests, KBr powder was mixed with the flour of wood samples using a weight ratio of 100:1. From this mixture, small tablets of about 13 mm diameter and 1 mm thickness were prepared using a Beckmann pellet apparatus under high pressure and vacuum. The IR spectra of every sample were analyzed using a Perkin Elmer Spectrum One FTIR spectrometer. Average curves from eight repeated scans were obtained between wave numbers of 4000 and 600 cm^{-1}. This corresponds to the functional group (4000-1300 cm^{-1}) and the finger print (1300-900 cm^{-1}) regions. Precision of the spectrometer was 4 cm^{-1}.

RESULTS AND DISCUSSION

Effect of Pre-steaming on the Diffusion Coefficient

Water vapor diffusivity through the wood specimens slightly increased as a result of pre-steaming (Figs. 2 and 3). Statistical analysis at the 99% confidence level showed significant differences in radial and longitudinal diffusion coefficients between control samples and those steamed at 160 °C. Our results showed that pre-steaming could affect the diffusion coefficient through two ways: 1) effect on the wood porous structure and 2) effect on the wood chemical characteristics. Significant changes were observed in the chemical compositions of pre-steamed specimens when compared to unsteamed ones (Table 1). Both water- and acetone-soluble extractives increased in the specimen surfaces, while the holocellulose and lignin content decreased. Similar to what was reported by Zhang and Cai (2006), our results showed that the extractives could move from the inside to the surface of wood specimens as a result of pre-steaming at a temperature higher than 120°C. The most pronounced changes in the chemical composition were found to be for the specimens pre-steamed at 160°C. Based on FT-IR spectroscopy, the greatest changes in the chemical structure of pre-steamed wood specimens was in the band at 1734 cm^{-1} for steaming at 160°C (Fig. 4). This spectrum represents carbonyl groups, which could correspond to lignin and hemicelluloses structures, but in the literature this band has been predominantly attributed to hemicelluloses structures. Any change in this spectrum indicates destruction of hemicelluloses. In fact, FT-IR results suggest degradation of hemicelluloses at a temperature of 160°C. Based on previous studies, hemicelluloses can enhance the rate of water vapor diffusivity through wood (Siau 1984; Tarmian et al. 2011). This means that any reduction or destruction of hemicelluloses structure of cell walls lower the water vapor diffusivity rate through wood. However, our result here showed that in spite of holocellulose hydrolysis due to steaming, the rate of water vapor diffusivity through the wood specimens slightly increase due to steaming. This unexpected result can be explained by the destructive effect of pre-steaming on the wood porous structure. In this case, the bordered pits on the tracheid walls are damaged due to steaming, and the pit torus is hydrolyzed (Fig. 5). The damage of pit membrane and pit torus due to steaming has also been reported by previous researchers (Nicholas and Thomas 1968; Jianxiong et al. 1994; Zhang and Cai 2008). Although the contribution of pit pairs to water vapor diffusion is usually neglected (Li et al. 2005; Kang et al. 2008), our results revealed that the hydrolysis of pit toruses in *Abies alba* L. due to steaming at a high temperature (160°C) can influence the diffusion characteristics. Indeed, based on the results obtained here, it can be claimed that the effect of pit pairs torus hydrolsis in *Abies alba* L. on its diffusion coefficient is more pronounced compared to that of cell wall holocellulose hydrolysis. Our results are in good agreement with the results of Choonge et al. (1999) who found that prolonged steaming increased the moisture diffusivities of southern pinewood. However, they explained the increase by some changes in the extractive distribution profile. In contrast to the present and Choonge et al.'s studies, Rousset et al. (2004) observed that the thermal treatment of poplar (*Populus robusta*) at 200°C reduces the value of mass diffusivity.

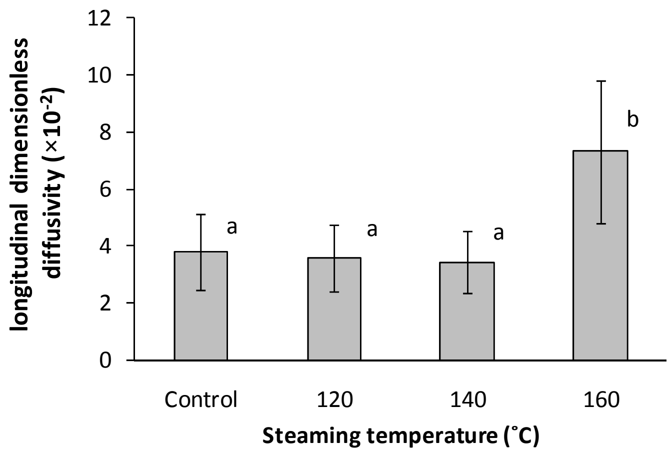

Fig. 2. The effect of steaming on the longitudinal diffusion coefficient of *Abies alba* L.

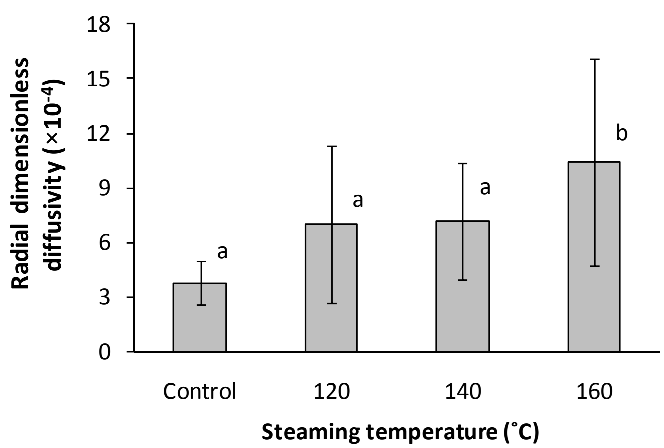

Fig. 3. The effect of steaming on the radial diffusion coefficient of *Abies alba* L.

These contradictory observations can be explained by the fact that the complicated effect of steaming on the wood diffusion coefficient depends on the wood species (i.e., the wood porous structure) and heat treatment condition. In fact, how the wood porous structure is damaged due to the heat treatment determines the ease of fluid migration through the wood. For example, Li et al. (2005) found that the microwave pretreatment was able to damage the structure of pit membrane in Masson pinewood, but could not effectively improve the diffusion coefficient.

Table 1. Chemical Composition of Fir Wood Specimens Steamed at Different Temperatures

Steaming temperature (°C)	Acetone-soluble extractives (%)	Water-soluble extractives (%)	Holocellulose (%)	Lignin (%)
Control	6.58	5.23	65.33	32.88
120	6.92	5.13	62.92	32.29
140	9.30	6.64	61.06	29.22
160	9.78	8.79	59.47	29.91

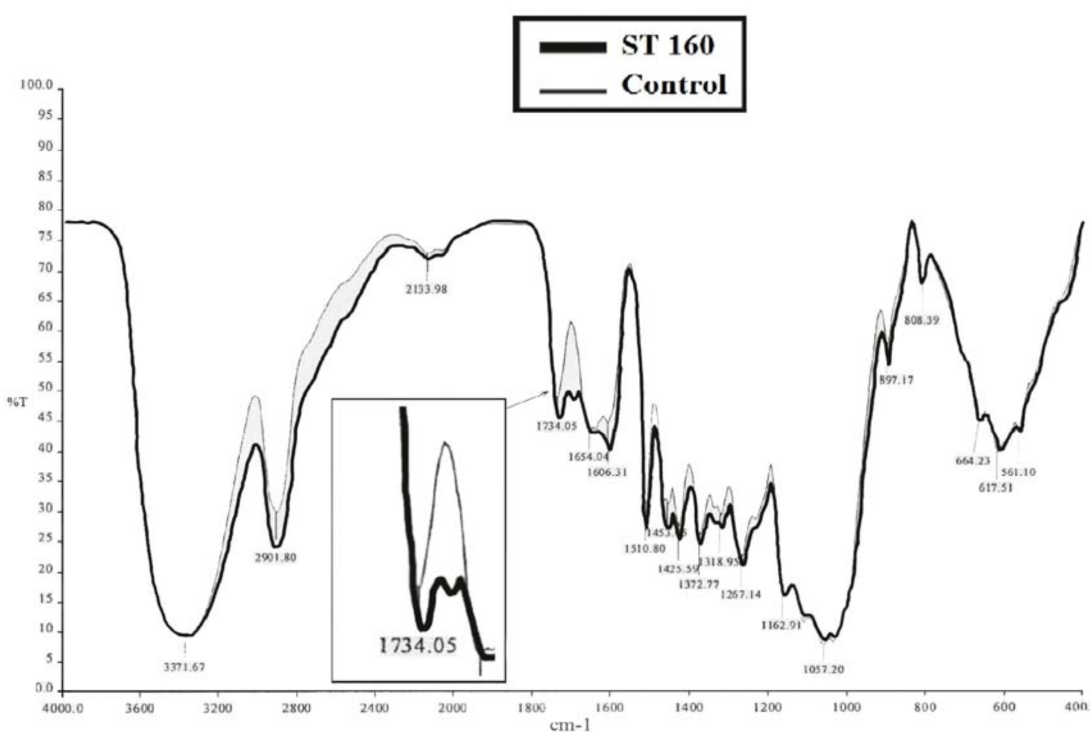

Fig. 4. FT-IR spectroscopy analysis of Fir wood specimen steamed at temperature of 160°C

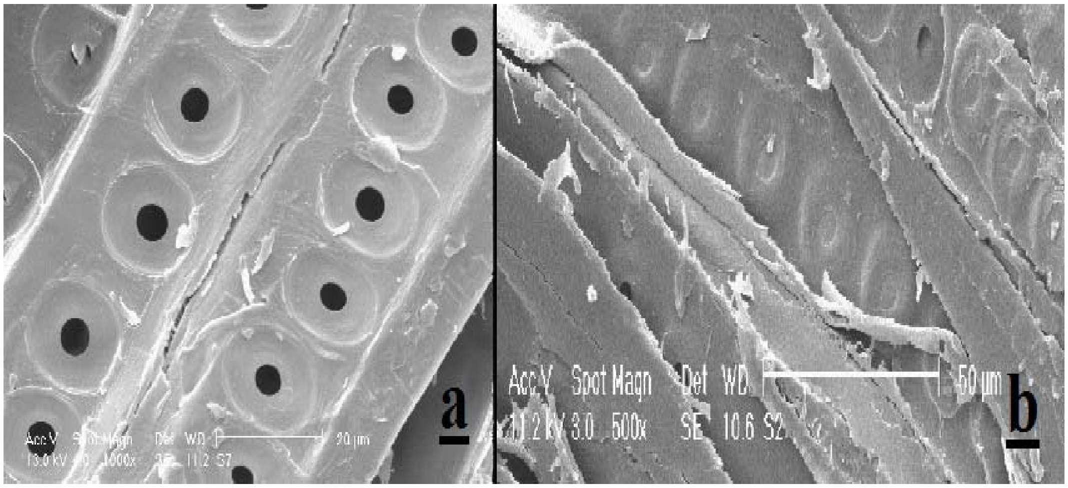

Fig. 5. SEM images of bordered pits of Fir wood specimen steamed at temperature of 160°C (a) compared to control specimen (b)

Effect of Pre-steaming on Air Permeability

Pre-steaming had a relatively strong effect on the radial air permeability, whereas it had no significant effect on the longitudinal permeability, except in the case of pre-steaming at 160°C (Fig. 6). Statistical analysis at the 99% level of confidence showed significant differences in radial permeability between control samples and those steamed at 120, 140, and 160 °C. The statistical analysis also showed a significant difference in longitudinal air permeability between control sample and that steamed at 160 °C. The greatest air permeability was observed for specimens steamed at higher temperature, i.e., 160°C. Overall, the longitudinal and radial permeability of wood specimens increased by 639 and 710% due to presteaming at 160°C, respectively. The increasing effect of pre-steaming on the air permeability can be related to the wood porous structure modification (see SEM images in Fig. 5). SEM studies show some difference in the pit structure between steamed- and unsteamed samples. As previously explained, the torus of bordered pits is completely hydrolyzed due to steaming. Our results are in agreement with some previous studies, reporting the positive effect of steaming on the wood permeability (Alexiou et al. 1990; Kanagawa et al. 1992; Morris et al. 1997; Cai. 2006). In contrast to our result and the mentioned studies, Rousset et al. (2004) and Cai and Oliveira (2007) found that the gas permeability of poplar (*Populus robusta*) and subalpine fir (*Abies lasiocarpa)* Hook do not change significantly due to steaming. Thus, it can be concluded that how steaming or heat treatment can affect the wood permeability depends upon the treatment condition as well as the wood species (i.e., the porous structure of wood). In fact, the steaming treatment can increase the permeability of wood, provided that it impacts the wood porous structure in such a way that facilitates fluid transfer. Lu et al. (1994) also claimed that the effect of steaming on the permeability of wood is not similar for different wood species (*Picea jezoensis* var. *komarovii, Abies nephrolepis*, and *Pinus koraiensis*) with different properties. In addition to the wood porous structure modification, the effect of chemical modification of wood due to steaming on the permeability cannot be neglected. In this case, the role of extractive materials is important. As stated by some researchers (Choonge and Achmadi 1989; Haslett and Kininmonth 1986), before steaming, the extractives are as continuous layers, while after steaming, they turn into discontinuous and fractured layers. Thus, cell walls capability increases with fluid movement.

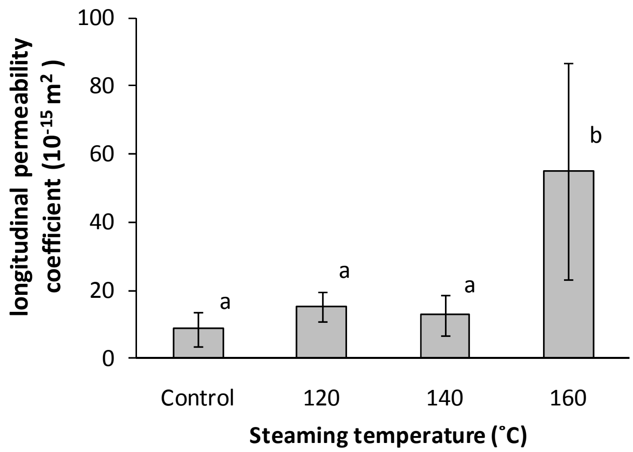

Fig. 6. The effect of steaming on the longitudinal permeability coefficient of *Abies alba* L.

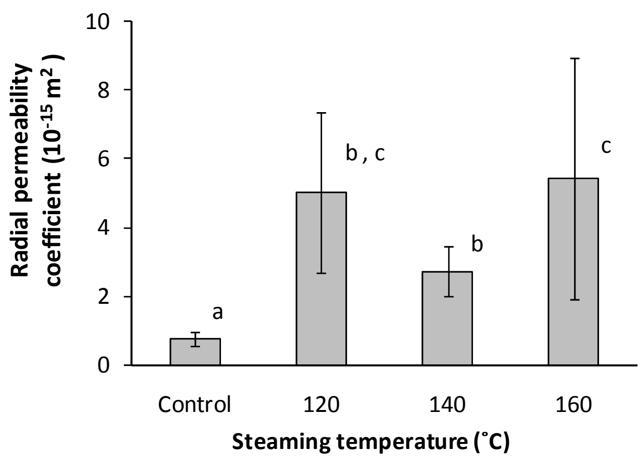

Fig. 7. The effect of steaming on the radial permeability coefficient of *Abies alba* L.

CONCLUSIONS

The effect of pre-steaming at different temperatures (120, 140, and 160°C for 1 hour) on mass transfer properties of fir wood (*Abies alba* L.), a gymnosperm species with torus margo pit membrane, was investigated. On the whole, the pre-steaming was found to be an effective modification method to improve both mass transfer properties of *Abies alba* L. However, a greater improvement was observed for the air permeability. Both mass transfer features improved by increasing steaming temperature from 120 to 160°C, and the greatest mass transfer rates were observed for the wood specimens steamed at 160°C. Compared to longitudinal direction, a more substantial mass transfer improvement due to the steaming was found in a radial direction, and steaming had no significant effect on the longitudinal mass transfer properties, except at the temperature of 160°C. The improvement impact of steaming on the mass transfer through *Abies alba* L. can be explained by some wood porous structure and chemical modifications, such as pit torus and holocellulose hydrolysis. Although the contribution of pit pairs to water vapor diffusion is usually neglected, our results demonstrated that the bordered pit damage in *Abies alba* L. due to steaming can significantly affect its water vapor diffusion coefficient. In fact, our results revealed that the effect of pit pairs torus destruction in *Abies alba* L. on its diffusion coefficient is more pronounced compared to that of cell wall holocellulose hydrolysis. Overall, due to some contradictory observations regarding the effect of wood steaming on its mass transfer properties, it can be concluded that the complicated effect of steaming depends on the wood species, its porous structure and steaming method and condition. The potential use of other heat treatment methods to improve mass transfer characteristics of *Abies alba* L. is recommended for further research.

NOMENCLATURE

k_g = longitudinal specific permeability (m^3 m^{-1})
$V_d = \pi r^2 \Delta z$ [r = radius of measuring tube (m)] (m^3)
C = correction factor for gas expansion as a result of change in static head and viscosity of water.
L = length of wood specimen (m)
P_{atm} = atmospheric pressure (m Hg)
$\bar{z}$ = average height of water over surface of reservoir during period of measurement (m)
t = time (s)
A = cross-sectional area of wood specimen (m^2)
Δz = change in height of water during time t (m)
V_r = total volume of apparatus above point 1 (including volume of hoses) (m^3)
Q = mass flux ($kg.s^{-1}$)
M_v = molar weight of vapor ($kg.mole^{-1}$)
RH_1 = relative humidity inside the climatic chamber
RH_2 = relative humidity inside the cup
R = constant of perfect gas
P_{VS} = pressure of saturated water vapor in temperature of $T(K)$
D_V = binary diffusion coefficient of water vapor in air.

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