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EFFECTS OF STEAMING AND MICROWAVE PRETREATMENTS ON MASS TRANSFER CHARACTERISTICS OF ALEPPE OAK (*QUERCUS INFECTORIA*)

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In this research, effects of steaming and microwave pretreatments on the mass transfer properties of never-dried *Q. infectoria* were investigated. Specimens at green moisture content were exposed to microwaves of frequency 2450 MHz for 10 minutes. The pre-steaming was performed at a temperature of 160°C for 1 hour under a pressure of 2 to 3 bars. Air permeability values were measured to be 4.8 and 4.9 ($\times 10^{-16}$ m^3 m^{-1}) in the sapwood and heartwood, respectively. Results showed a significant general increase in the air permeability and diffusion coefficients in the pretreated sapwood specimens. The presence of tyloses in the heartwood prevented the penetration of steam to the inner parts of the specimens, resulting in the diffusion coefficient remaining constant. The pressure gradient caused by the microwave heating resulted in the distortion of the tyloses structure in the heartwood, thus resulting in a significant increase in the air permeability. It may be concluded that the presence of tyloses has a significant effect on the final impact of either of the pretreatments.

Keywords: Drying; Microwave; Steaming; Diffusion; Permeability; Aleppe oak

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INTRODUCTION

Aleppe oak (*Q. infectoria*) is one of the most important species of forests in Iran. The Zagros region forest, which is situated in the western part of Iran, accounts for 5 million hectares, with oak species as a predominant species, accounting for a massive 3.5 million hectares. In addition, the oak wood has a wide range of utilization, for instance, in veneer industry, barrels, as well as wooden parquet flooring. Given the importance of this species, numerous studies have been carried out on it (Sandoval-Torres *et al.* 2010; Todaro *et al.* 2010; Beakler *et al.* 2007).

Wood drying is considered to be a costly process. Therefore, reduction in wood drying time for the purpose of saving in energy consumption, and consequently the drying costs, is a great challenge in this industry. It is also worth noting that reduction in wood drying time should not be accompanied with a decrease in the quality of dried boards. Reduction of drying time is affected by various factors, such as permeability (Cai 2006) and diffusion coefficients (Brodie 2009). Many studies have been carried out by

means of different methods concerning an increase of these coefficients. In this regard, the pre-steaming method has drawn lots of attention to improve the mass transfer coefficients and consequently increase the rate of the drying process. The study of Kanagawa *et al.* (1992) revealed that steaming at low pressure (2 to 3 atm) could improve the permeability of Japanese cedar wood (*Cryptomeria japonica* L.). Alexiou *et al.* (1990) found that pre-steaming of regrowth *Eucalyptus pilularis* (blackbutt) can significantly increase the longitudinal permeability, as tyloses appeared unaltered. In another study, the effect of applying steam explosion on Mongolian oak and red oak was also evaluated (Ping *et al.* 2007). Their results revealed that permeability of treated specimens noticeably increased relative to the untreated ones. Choong *et al.* (1999) reported that prolonged steaming increased the moisture diffusivities of southern pine above and below the fiber saturation point (FSP), partially due to changes in the extractive distribution profile. Results of a study conducted by Harris *et al.* (1989) regarding the effect of steaming pre-treatment of red oak (*Quercus spp.*) showed that as a result of this treatment the moisture gradient decreased, while drying rate climbed. Ping and Zhang (2009) investigated the implications of pre-steaming on the two hardwood species of *Nothofagus* spp. and *Toona sinensis*. They mentioned that the transverse permeability of treated specimens as opposed to control ones increased significantly.

Another method, which has been considered for the sake of increasing the drying rate and improving mass transfer coefficients, is microwave treatment. Vinden and Torgovnikov (2000) showed that controlled use of powerful microwave energy for drying newly cut timbers of hardwoods can directly affect both permeability and density factors of wood due to producing early rupture in wooden ray cells. Results of studies by Liu (2005) indicated that wood drying by microwave led to improved permeability of birch wood, without any depression in modulus of elasticity (MOE) and modulus of rupture (MOR). Fei *et al.* (2003) and Zhao *et al.* (2003) reported that microwave pretreatment resulted in improved water vapor diffusion coefficient and reduced drying time of eucalyptus.

Nowadays, applying microwave and steaming treatments are of potential interest, and they are considered as common methods to improve the permeability and diffusion coefficients, as well as speed up the drying process. On the other hand, there is a dearth of research regarding microwave and steaming application as a pretreatment for increasing of mass transfer coefficients in oak wood. Hence, this research is aimed at evaluating the influence of these treatments to find a new possibility of enhancing the permeability and diffusion coefficients of Aleppe oak (*Q. infectoria*) wood.

EXPERIMENTAL

Sampling

Boards of Aleppe oak (*Q. infectoria*) were cut to bastard-grain with the thickness of 6 cm by length and width of 35 and 12 cm, respectively. Moisture content of the boards was 35 to 40%. Moreover, the specimens were separately provided from the sapwood and heartwood regions. Three replications were considered for each set of the experiments.

Steaming Pretreatment

The steaming was done by means of a laboratory steaming vessel equipped with heat and pressure indicators. There was a tap for controlling the input steam and another one for controlling the pressure of the tank. The pre-steaming was performed at a temperature of 160°C for 1 hour under a pressure of 2 to 3 bars. After steaming, steam was also relieved gradually. Once the specimens were steamed, they were kept at environment temperature without air circulation for about 6 hours to prevent occurrence of crack or rupture and to create thermal and moisture equilibrium.

Microwave Pretreatment

Microwave pretreatment was performed for a total time period of 10 minutes. After placing the specimens inside the microwave oven (frequency of 2450 MHz), every minute microwave performance was stopped for a minute in order to prevent carbonization and occurrence of cracks as a result of quick moisture evaporation.

Drying Procedure and Internal Cracks

After treating, cross sections of control specimens and pretreated ones were covered by oil-base paint so that moisture transfer took place only through their thickness and width. Subsequently, all specimens were dried with a convectional laboratory kiln at a constant temperature of 60°C and relative humidity (RH) of 50% up to the final MC of 10%.

Five samples with 2 cm thickness were cut from each of the dried board, and frequency of internal cracks was then evaluated.

Air Permeability Measurement

Twenty cylindrical specimens, 18 mm in diameter and 10 mm in length, were taken from dried boards in the radial direction. The radial direction, in fact, was oriented through the thickness of the samples. The lateral surfaces of the specimens were coated with epoxy resin to prevent lateral flow. Air permeability was measured with a USPTO-patented apparatus No. 8,079,249 B2, approved by The Iranian Research Organization for Scientific and Technology under certificate No. 47022 (Taghiyari 2011; Taghiyari and Efhami 2011; Taghiyari *et al.* 2012; Taghiyari 2012). Figure 1 depicts a schematic of the experimental apparatus used for the air permeability measurement (Taghiyari *et al.* 2010; Taghiyari and Sarvari Samadi 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 (m^3 m^{-1}), η is the viscosity of air (η =1.81×10^{-5} Pa s), and k_g is the superficial permeability, which can be determined as follows,

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

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,

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

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).
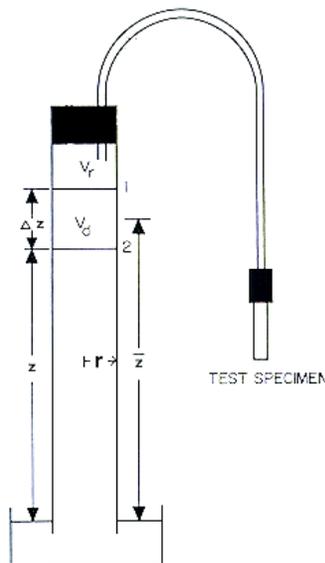
Fig. 1. Schematic view of the air permeability measurement apparatus (Taghiyari & Sarvari Samadi 2010) (USPTO No. US 8,079,249, B2; Pub. No. 2010/0281951 A1)

Water Vapor Diffusivity Measurement

Water vapor diffusivity measurement was carried out on the same specimens 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, the dimensionless diffusivity f was calculated according to the following formula,

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

(4)

where Q is the measured mass flux (kg.s^{-1}), A is the cross section of the specimen (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 specimen 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.

Chemical Analyses

To determine chemical composition, the specimens were ground in Wiley mill to pass a 40-mesh screen. Then, prior to determination of the holocellulose and lignin contents, 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, water-soluble and acetone-soluble extractive contents were also determined based on TAPPI standard methods, TAPPI T 207cm-99, and TAPPI T 222cm-98, respectively. It is also worth noting that chemical analyses were performed in the superficial layers of the specimens, with the thickness of 5 mm, to determine the effects of applied treatments on the movement of extractives and also changes in other chemical compositions.

RESULTS AND DISCUSSION

Air Permeability

Results of the present study revealed that microwave treatment had no statistically significant effect on radial permeability in both sapwood and heartwood treated specimens. However, a pronounced difference was observed between the mean values of the control and microwave-treated heartwood specimens (Fig. 3). In fact, the high statistical variance in the air permeability, which is the natural property of solid woods (Taghiyari *et al.* 2010; Taghiyari and Sarvari Samadi 2010), did not allow the difference between the two treatments of control and microwave-treated specimens to be revealed. The increase in the air permeability of the heartwood specimens can be rooted in the distortion of tyloses in the heartwood by microwave pretreatment (Fig. 4b). The microwaves increased the temperature of the inner parts of the specimens, causing the water content to be abruptly evaporated in this part; the resulting pressure gradient between the inner part and the surrounding environment gave rise to rapid high flow, causing part of the tyloses structure to become distorted and torn out. This process ended up in the easier fluid flow and the consequent increase in the air permeability. Similar increasing results in fluid flow caused by microwave pretreatment were also reported in other research projects; some studies (Lu *et al.* 1994; Zhao *et al.* 2003; Yu *et al.* 2002) have also indicated that wood pretreatment by microwave heating destroys the pore structure in the cell walls, which in turn leads to an improved fluid flow within the treated woods. Furthermore, as a result of microwave pretreatment and, consequently, the internal

pressures induced by quick moisture evaporation, microscopic checks developed in the cell walls, which lead to facilitation of the fluid flow within wood structure (Kanagawa *et al.* 1992; Li *et al.* 2010).

The obtained results also showed that steaming pretreatment increased the air permeability coefficient in both the sapwood and heartwood specimens of *Q. infectoria* (Figs. 2 and 3). Permeability of steamed-specimens of sapwood was 22.9% more than that of control ones; the mean permeability coefficient was increased from 4.8 to 5.9 m^2 (10^{-16}). The number of superficial and internal cracks increased noticeably in the steam-pretreated specimens in comparison to that of the control ones (Tables 1 and 2). These cracks have facilitated fluid flow through the steam-treated specimens, resulting in the increase in air permeability.
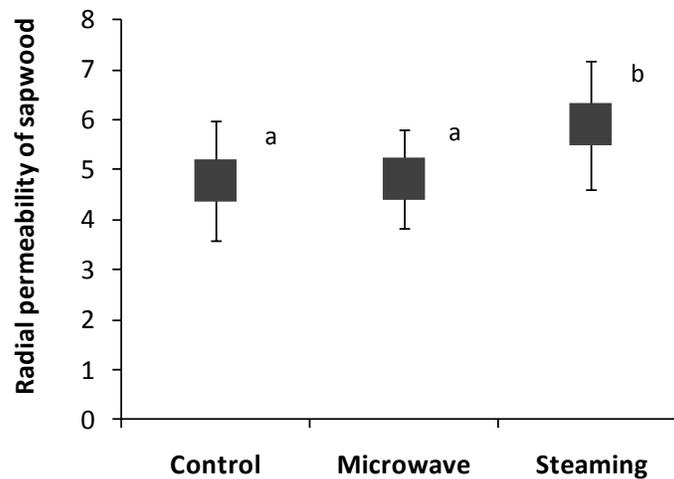
Fig. 2. Radial permeability of the sapwood for different treatments of control, microwave-pretreated, and steam-pretreated ($\times 10^{-16}$ m^3 m^{-1})
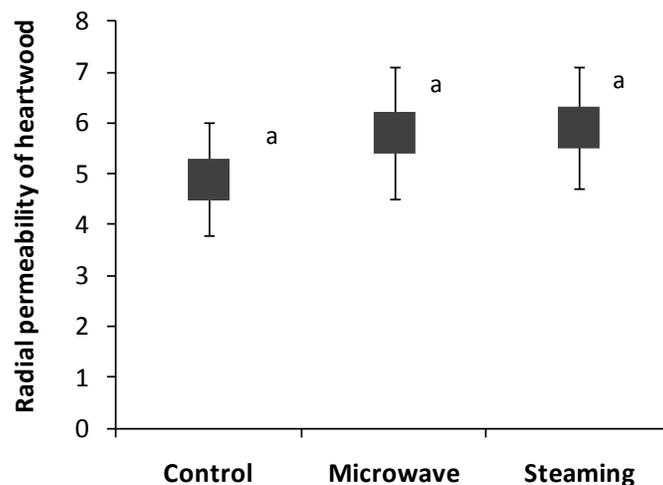
Fig. 3. Radial permeability of the heartwood for different treatments of control, microwave-pretreated, and steam-pretreated ($\times 10^{-16}$ m^3 m^{-1})

Table 1. Intensity of Internal Cracks in the Control and Pre-treated Specimens

Crack length (mm)	Control specimen	Microwave treated specimen	Steamed specimen
1-10	2	2	13
11-20	1	0	3
21-30	1	0	2

Table 2. Intensity of Superficial Cracks in the Control and Pre-treated Specimens

Crack length (mm)	Control specimen	Microwave treated specimen	Steamed specimen
1-30	3	2	9
31-60	0	0	3
61-90	0	0	1
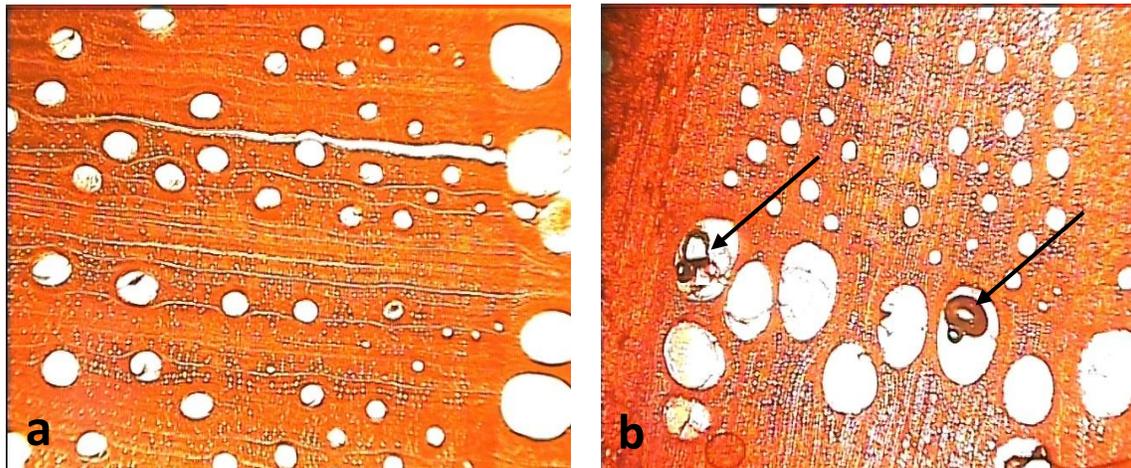
Fig. 4. Light microscope feature of sapwood (a) and heartwood (b) of *Q. infectoria* (arrows show the tyloses blocking the way for fluid flow)

Diffusion Coefficient

The cups for measurement of the diffusion coefficient reached a nearly constant weight change after 15 days for both sapwood and heartwood specimens. The mean weight depressions of the cups containing sapwood and heartwood were 0.0218 and 0.0185 g, respectively. The mean diffusion coefficient of the heartwood specimens was 2.35 ± 0.32 (10^{-7}), and that of the sapwood was 3.6 ± 0.39 (10^{-7}) (Figs. 5 to 6). A prior study suggests that water vapor diffusion depends on the cell wall thickness and the amount of substance it contains, that is, the density (Perre 2007; Tarmian *et al.* 2012).

Based on the previous studies, hemicelluloses can enhance the rate of water vapor diffusivity through wood (Siau 1984; Tarmian *et al.* 2012). This means that any reduction or destruction in hemicelluloses structure of the cell walls lowers the water vapor diffusivity rate through wood. In this connection, many studies have also reported the great impact of the period of steaming on fluid flow (Schmidt 1982a, b; Kubinsky and Ifju 1974; Oltean *et al.* 2007). The same results were observed in the steam-treated sapwood specimens in the present study. However, as to the steam-treated heartwood specimens, the presence of tyloses, as well as the low steaming time, lessened the flow of steam, and consequently the amount of steam penetrated to the inner parts of the specimens decreased, resulting in the low change in the diffusion coefficient of heartwood (Fig. 6).

Chemical analyses revealed that the amount of holocellulose and lignin in the pre-steamed sapwood specimens decreased (Table 3). The temperature of 160°C and the pressure of 2 to 3 bars resulted in chemical degradation, particularly in the holocellulose content, decreasing it by about 4% compared to the corresponding control specimens. This change in the chemical composition of the steam-treated specimens may be considered the root of the change in the diffusion coefficient. Similar conclusions have been made by Zhang and Cai (2008).

The high pressure gradient between the inner part and outer layer of the specimens caused by the microwave heating and abrupt evaporation of water tore out part of the tyloses structure, resulting in the significant increase in the diffusion coefficient of the heartwood (Fig. 6). A similar increase in diffusion coefficient has been also reported in *Eucalyptus* (Fei *et al.* 2003). However, the effect of microwave pretreatment on the diffusion coefficient of sapwood was significantly lower (Fig. 7) due to the lack of tyloses in the sapwood (Fig. 4a). It can therefore be concluded that the structure of woody cells and components can significantly affect the impact of microwave pretreatment on the diffusion coefficient of the specimens.
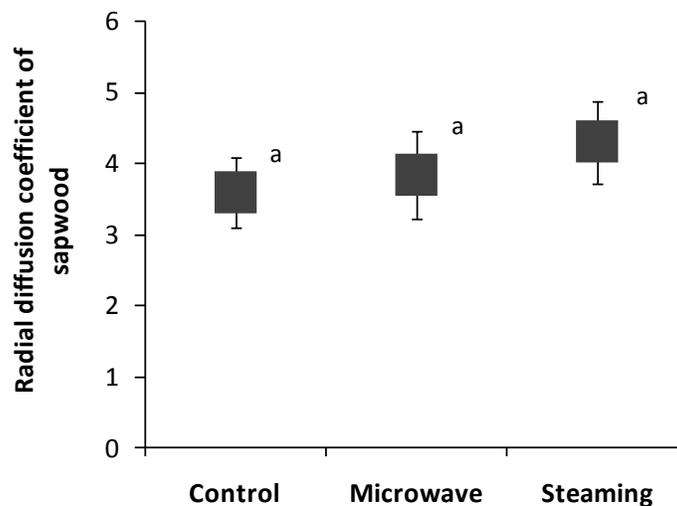
Fig. 5. Radial diffusion coefficient in the sapwood treated specimens toward the control ones ($\times 10^{-7}$)
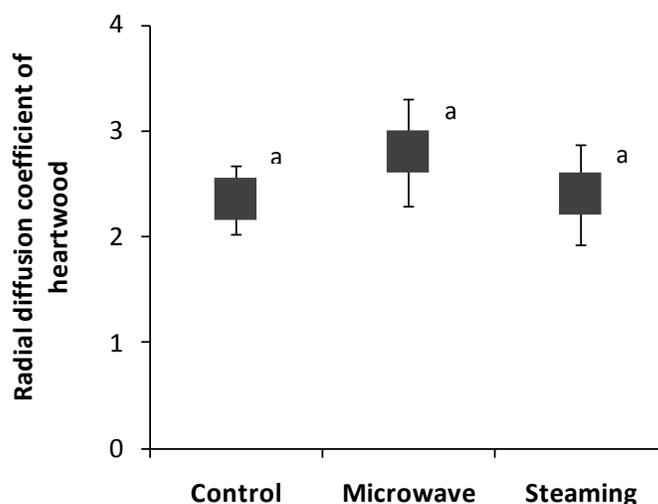
Fig. 6. Radial diffusion coefficient in the heartwood treated specimens toward the control ones ($\times$ 10^{-7})

Table 3. Chemical Composition of the Control and Treated Sapwood Specimens

Treatment	Acetone-soluble extractives (%)	Water-soluble extractives (%)	Holocellulose (%)	Lignin (%)
Control	10.30	4.84	67.20	28.00
Microwave	11.70	4.42	66.80	28.00
Steaming	11.14	3.85	62.40	27.00

CONCLUSIONS

1. The pretreatment drying system significantly affects the final results in the air permeability and diffusion coefficients of oak wood specimens.

2. Distortion of the tyloses structure in the heartwood of oak by the microwave drying system would result in a significant increase in the air permeability and diffusion coefficients.

3. Steam pretreatment significantly increased air permeability and diffusion coefficients by the cracks it makes in the internal and external parts of the specimens, as well the change in the chemical composition of oak wood.

4. Tyloses in the heartwood of oak react differently in different pretreatments. In microwave pretreatment, tyloses are torn out and therefore facilitate fluid flow resulting in the increase in the air permeability of oak. However, in the steaming pretreatment, they prevent steam to enter the inner parts of the specimens resulting in keeping the diffusion coefficient constant.

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