

Solute diffusion into cell walls in solution-impregnated wood under conditioning process I: effect of relative humidity on solute diffusivity

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Abstract This study focused on solute diffusing into cell walls in solution-impregnated wood under conditioning. The purpose of this paper was to clarify the effect of relative humidity (RH) of the conditioning on solute diffusivity in the impregnated wood. Water evaporation, swelling, and shrinkage of wood samples impregnated with an aqueous solution of polyethylene glycol (PEG) polymer were examined under conditioning at an RH of 11, 32, 55, or 80 % followed by drying under vacuum. Dried samples were observed using a micro-focus X-ray computed tomography instrument. The total amount of PEG polymer diffusing into cell walls during conditioning increased with RH. Theoretical interpretation indicated that this was caused by an increase in polymer (solute) diffusivity as the amount of water (solvent) in samples increased. Temporal variability of the evaporation rate of water and of the swelling rate of the sample also were examined. Solute diffusivity, which was similar at each RH at the beginning of conditioning, decreased during conditioning; this decrease was greater at lower RH values due to a higher evaporation rate.

Keywords Impregnated wood · Conditioning · Relative humidity · Solute diffusivity · Solvent evaporation

Introduction

Treatment of wood with a non-volatile substance has been used to overcome the disadvantages of wood, such as a lack of homogeneity, its tendency to deform in water and moist air, and its ability to be degraded by sunlight, fungi, and insects. Wood properties are well known to be improved, to some extent, by treatment with an appropriate substance (for example, [1]). The method of treatment, however, has not been well studied. Several studies have shown that the amount of a substance in wood cell walls mainly determines the wood properties such as dimensional stability (for example, [2]). Thus, filling up cell walls with a substance is necessary for the efficient treatment.

Treatment processes include impregnation and conditioning (Fig. 1a). For impregnation, the wood is soaked in a solution of a non-volatile substance (solute), and then placed under a vacuum (or subjected to pressure) while in contact with the treatment solution. For conditioning, the impregnated wood is placed under an atmosphere. For both processes, migration of solute and solvent occurs at a cellular level (Fig. 1b):

- During impregnation, the solution uniformly permeates the cell walls and cell cavities.
- During conditioning, a greater amount of solvent evaporates from the cell cavities than from the cell walls, which increases the concentration of solute in the cavities than that in the walls. This concentration difference causes diffusion of the solute from the cavities to the walls [3].

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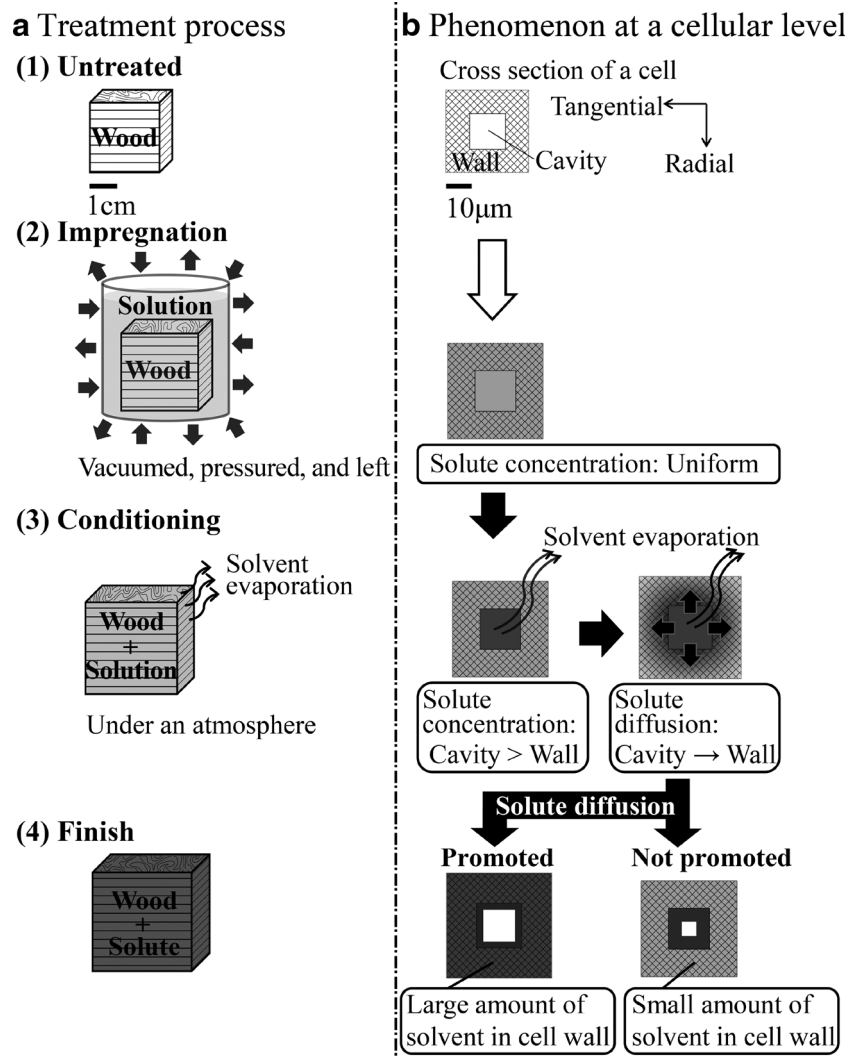
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Fig. 1 **a** Process and **b** phenomenon during the treatment of wood by a non-volatile substance (solute)



The solute permeability during impregnation has been studied by many researchers using polyethylene glycol (PEG) polymer as a solute [3–14]. Jeremic et al. [5] examined the effect of nature of solvent on amount of the polymers in cell walls and estimated the diffusion rate of the polymers into cell walls [7]. Tarkow et al. [14] examined the effect of the molecular size on amount of the polymers penetrated into wood. Stamm [12, 13] investigated a variety of factors that affect amount of the polymers in cell walls and cell cavities during impregnation. Stamm [3] also proposed the mechanism of solute diffusing into cell walls during conditioning. This mechanism has, however, never been verified. Solute amount in the cell walls will increase if the solute diffusion is promoted during the conditioning, while the amount will not increase if the diffusion is not promoted (Fig. 1b). Therefore, there exists the possibility of maximizing the solute amount in the cell walls in short time by optimizing the conditioning.

The objective of this study is to determine the conditions that maximize amount of solute diffusing into the cell walls of solution-impregnated wood during the conditioning process in short time. Amount of solute diffusing into cell walls is determined by:

- Concentration difference of the solute between the cell walls and cell cavities.
- Solute diffusivity.

These two factors are affected mainly by solution conditions (such as concentration and type of solute and solvent), material conditions (such as wood species and dimension), and atmospheric conditions (such as temperature and vapor pressure of the solvent). Control of the atmospheric conditions is necessary since the other conditions often depend on the demanded properties of the treated wood. Solvent vapor pressure is especially important, because it is easily controlled for determining its effect on solution-impregnated wood.

Relative humidity (RH) is often used as an indicator of solvent vapor pressure, because water is used often as a solvent due to its high affinity toward wood. In our previous studies [15, 16], the effect of RH on the concentration difference was determined, but the effect on solute diffusivity could not be evaluated by investigating the swelling and shrinkage behavior of wood impregnated with an aqueous solution of PEG during conditioning at high RH values (close to the saturated vapor pressure). Solute diffusivity was expected to experience greater change within the lower RH range where the cell walls contain less bound water, on the basis of the fact that diffusion of the polymers into cell walls is much more rapid if moisture is present in wood [13].

The purpose of this paper was to clarify the effect of RH on the solute diffusivity. Water evaporation and the swelling-shrinkage behavior of wood impregnated with an aqueous solution of PEG polymers were examined during conditioning at several RHs including the lower RHs. Conditioned wood was dried under vacuum and its mass and dimension were measured. A cross section of a dried sample was observed using a micro-focus X-ray computed tomography (CT) system. The effect of RH on the polymer diffusivity was interpreted based on theoretical factors. Temporal variability in the water evaporation rate and swelling rate were examined in terms of solute diffusivity.

Theory

Effect of RH on solute diffusivity could not be directly observed. Therefore, the relation between RH and amount of solute diffusing into cell walls, which could be estimated using dimension of sample, was qualitatively predicted by the model of substance migration (Fig. 2) considering the solute diffusivity. The process of the prediction is shown as follows.

Solvent evaporation

Solvent evaporated from the samples can be separated into those from the cell cavities and from the cell walls. The amount of solvent evaporated from the cell cavities and cell walls, $-\Delta n_{C0}$ and $-\Delta n_{W0}$, respectively, can be formulated as [17]:

$$-\Delta n_{C0} = K(P_S - P_A - P_C)\Delta t, \tag{1}$$

$$-\Delta n_{W0} = K(P_S - P_A - P_W)\Delta t, \tag{2}$$

where K represents surface evaporation coefficient (>0), P_S is saturated vapor pressure of the solvent, P_A is vapor pressure of the solvent in the atmosphere, P_C and P_W are evaporation resistance pressure on the boundary between the atmosphere and the wood elements (cell cavity and cell wall, respectively).

The relation $P_S - P_A = P_W = P_C$ exists immediately before the conditioning. In the unsteady state during conditioning, the inequality of $P_S - P_A > P_W > P_C > 0$ exists, because vapor pressure decreases to a level corresponding to RH, and water retentivity of the cell wall is higher than that of the cell cavity due to higher energy to remove bound water from the cell wall [18]. From this inequality, and Eqs. (1) and (2), the following expression can be deduced:

$$-\Delta n_{C0} > -\Delta n_{W0}. \tag{3}$$

This inequality indicates that more solvent evaporates from the cell cavities than from the cell walls.

Causal relations among solute diffusion, solute concentration difference, and solvent evaporation

The amount of solute diffusing into cell walls during infinitesimal time Δt can be expressed as:

$$\Delta n_{W1} = K_1(x_{C1} - x_{W1})\Delta t, \tag{4}$$

where K_1 is solute diffusivity (>0), and x_{C1} and x_{W1} represent molar fractions of solute in the cell cavity and cell wall, respectively.

The solute concentration difference between the cell walls and cell cavities changes as solvent evaporates from the wood. Immediately before conditioning, the difference is absent producing the following equation:

$$x_{C1} - x_{W1} = \frac{n_{C1}}{n_{C0} + n_{C1}} - \frac{n_{W1}}{n_{W0} + n_{W1} + n_W} = 0, \tag{5}$$

where n_{C0} and n_{W0} represent moles of solvent in cell cavity and cell wall, respectively, n_{C1} and n_{W1} are moles of solute in the cell cavity and cell wall, respectively, n_W is number in moles of sorption sites of solution in the cell wall. The concentration difference changes with time (Δt) after the start of conditioning [Eq. (6)]; the inequality relation of Eq. (6) was deduced from Eq. (3).

$$x_{C1} - x_{W1} = \frac{n_{C1}}{n_{C0} + n_{C1} + \frac{\Delta n_{C0}}{n_{W1}}} - \frac{n_{W1}}{n_{W0} + n_{W1} + n_W + \Delta n_{W0}} > 0. \tag{6}$$

The inequality indicates that a solute concentration in the cell cavities greater than that in the cell walls is caused by solvent evaporation. The inequality $\Delta n_{W1} > 0$ is deduced from Eqs. (4) and (6), which indicates that the solute diffuses from the cell cavities to the cell walls due to the concentration difference.

Relation between RH and total amount of solute diffusing during conditioning

Let us consider the case where the solution-impregnated samples are conditioned at several RH values.

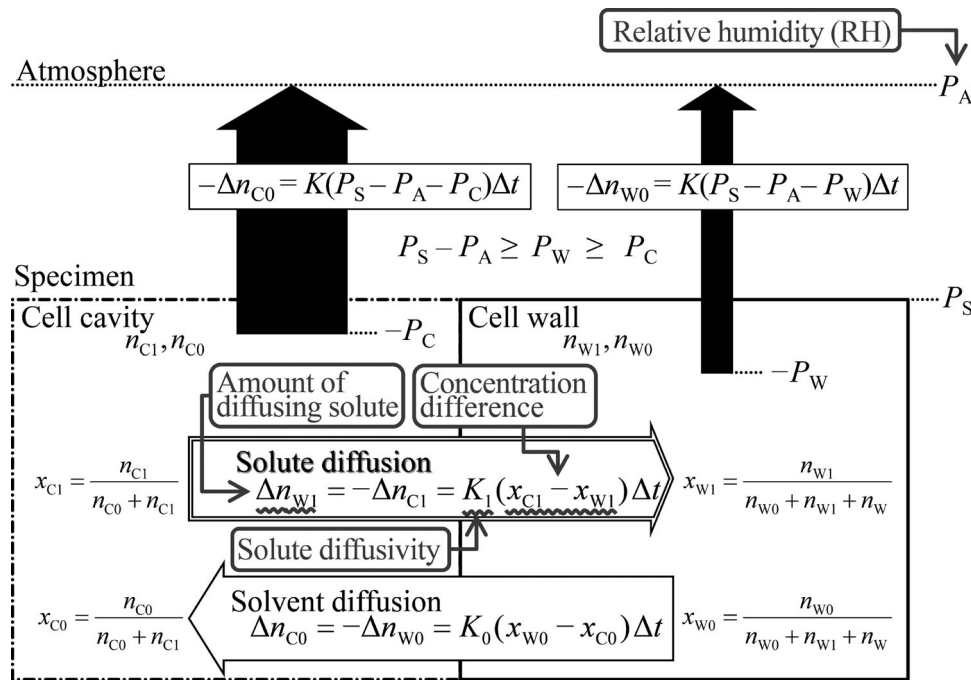


Fig. 2 Model of substance migrating in and around a sample during an infinitesimal time, Δt . P_S , saturated vapor pressure of solvent; P_A , vapor pressure of solvent in the atmosphere; P_C and P_W , evaporation resistance pressure on the boundary between the atmosphere and wood elements (cell cavity and cell wall, respectively); K , surface evaporation coefficient; n_{C0} and n_{W0} , moles of solvent in cell cavity

and cell wall, respectively; n_{C1} and n_{W1} , moles of solute in the cell cavity and cell wall, respectively; n_W , number in moles of sorption sites of solution in the cell wall; x_{C0} and x_{W0} , molar fractions of solvent in cell cavity and cell wall, respectively; x_{C1} , and x_{W1} , molar fractions of solute in cell cavity and cell wall, respectively; K_0 and K_1 , solvent and solute diffusivity, respectively

The solute diffusivity, K_1 , was assumed in this paper to increase with the amount of solvent in the sample. If it was also assumed that the amount of solvent (water) in the sample increases with RH during conditioning (Fig. 3b), the value of K_1 also increases with RH (gray dotted line in Fig. 3a).

The solute concentration difference, $x_{C1} - x_{W1}$, increases as RH or P_A (Fig. 2) decreases (gray broken line in Fig. 3a), because Δn_{C0} and Δn_{W0} , which have negative values, decrease with P_A [Eqs. (1) and (2)], and because the value of $x_{C1} - x_{W1}$ increases as Δn_{C0} and Δn_{W0} decreases [Eq. (6)]. This tendency was also supported by our previous study [16].

The concave-downward curve showing the relation of the amount of solute diffusing during Δt to RH (gray solid line in Fig. 3a) can be deduced by Eq. (4), and the relations of K_1 and $x_{C1} - x_{W1}$ to RH (see “Appendix” section). The total amount of solute diffusing during conditioning was the time integration of the amount of solute diffusing during Δt . Thus, the relation of the total amount to RH (black line in Fig. 3a) was approximately the same shape as the relation during Δt .

Materials and methods

Twelve cross-cut-wood samples, with dimensions of 5 mm \times 25 mm \times 25 mm in longitudinal (L), radial (R), and tangential (T) directions, respectively, were subsequently prepared from a block of hinoki (*Chamaecyparis obtusa*). The samples were dried at 105 °C to relatively constant mass, followed by measurement of their width in the T direction.

The oven-dried samples were soaked in a 30 mass % aqueous solution of polyethylene glycol with an average molecular weight of 1500 (PEG1540, E.P., Wako), placed under vacuum at 0.01 MPa in 1 h, and then subjected to pressure at 0.8 MPa in 20 h in an impregnation plant (YA-10, Yasujima). The samples then were placed in the solution at ambient pressure for 1 week.

The impregnated samples were blotted and their T width was measured. The samples were divided equally into four groups and conditioned for 900 h in a desiccator (MD-1, Sanplatec) at an RH of 11, 32, 55, or 80 % and temperature of 35 °C using supersaturated solutions of lithium chloride (LiCl), magnesium chloride (MgCl₂), sodium bromide

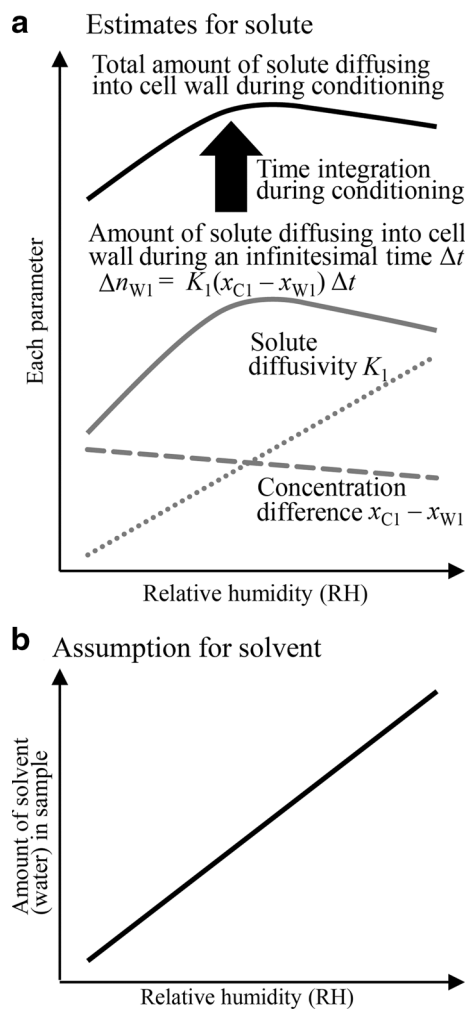


Fig. 3 **a** Qualitative estimates for solute and **b** theoretical assumptions for the solvent

(NaBr), or potassium bromide (KBr), respectively [19]. The mass and T width of the samples were monitored over 1–900 h of conditioning.

All of the conditioned samples were removed from the desiccator and dried to relatively constant mass and T width in a vacuum chamber (0.01 MPa) with phosphorus pentoxide (P_2O_5).

The moisture content and relative swelling of the impregnated samples during conditioning as a function of conditioning time t , $M(t)$ and $r(t)$, and the relative swelling after conditioned and subsequently dried in a vacuum, r_V , were defined as follows:

$$M(t) = \frac{100\{m(t) - m_V\}}{m_V} [\%], \tag{7}$$

$$r(t) = \frac{w(t) - w_0}{w(0) - w_0}, \tag{8}$$

$$r_V = \frac{w_V - w_0}{w(0) - w_0}, \tag{9}$$

where w_0 represents T width of the oven-dry sample before impregnation, $w(0)$ is T width of the impregnated sample just before conditioning ($t = 0$), $m(t)$ and $w(t)$ are mass and T width, respectively, of the impregnated sample during conditioning as a function of t , m_V and w_V are mass and T width, respectively, after conditioned and subsequently dried in a vacuum.

The evaporation rate and swelling rate of the impregnated samples during conditioning, $-\Delta M/\Delta t$ and $\Delta r/\Delta t$, as a function of t were defined as follows:

$$-\frac{\Delta M}{\Delta t} = -\frac{M(t_{i+1}) - M(t_i)}{t_{i+1} - t_i}, \tag{10}$$

$$\frac{\Delta r}{\Delta t} = \frac{r(t_{i+1}) - r(t_i)}{t_{i+1} - t_i}, \tag{11}$$

where t_i ($i = 0, 1, 2, \dots$) is the conditioning time when the mass and width were measured ($t_0 = 0 < t_1 < t_2 < \dots$).

A block with a dimension of 5 mm (L) \times 8 mm (R) \times 8 mm (T) was cut from the center of the sample that was conditioned at each RH and subsequently dried. The center of the block was scanned using a micro-focus X-ray CT system (SMX-160CT-SV3S, Shimadzu) [20, 21] to obtain a two-dimensional tomogram with a field of view size of 1.68 mm in both the R and T directions and with a pixel size and slice thickness of 3 and 18 μ m, respectively.

Results and discussion

Water evaporation and swelling-shrinkage behaviors of impregnated wood under conditioning

Figure 4a shows the temporal variability in moisture content of the aqueous PEG-solution-impregnated sample during conditioning. The moisture content or amount of water in the sample increased with RH during conditioning, which satisfied the assumption (Fig. 3b) in the theory.

Figure 4b shows the temporal variability in relative swelling of an impregnated sample during conditioning. The samples shrunk over time at RH values of 11 and 32 %, while they swelled over time at RH values of 55 and 80 %. The relative swelling at the conditioning time of 900 h (equilibrium point) increased with RH. This swelling and shrinkage behavior was considered to be caused by the PEG polymers diffusing into the cell walls and by the water exuding from the cell walls, respectively [16]. To evaluate the effect of RH on polymer diffusing into the cell walls, the effect of water on the dimensions of the samples had to be eliminated.

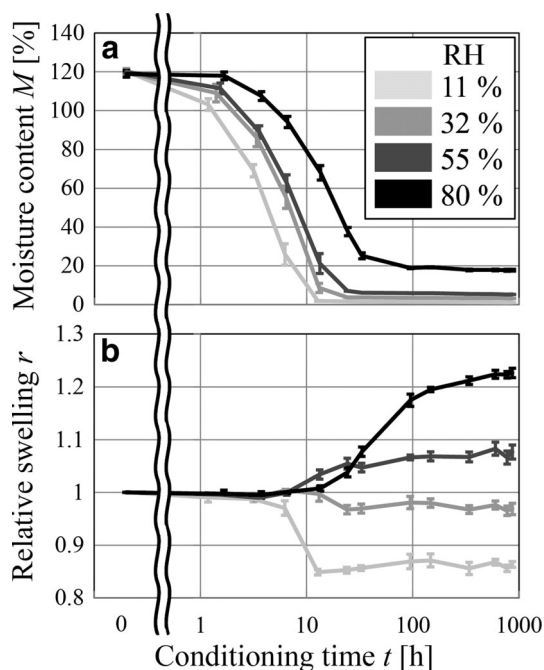


Fig. 4 Temporal variability in **a** moisture content, M , and **b** relative swelling, r , of impregnated samples during conditioning at each relative humidity (RH). Bars standard deviations

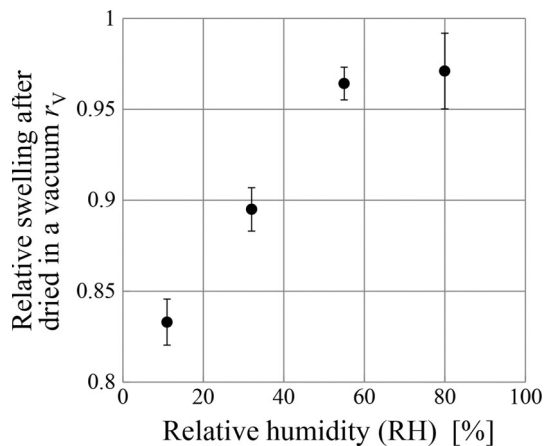


Fig. 5 Relation between RH and relative swelling of samples conditioned and subsequently dried under vacuum, r_v . Bars standard deviations

Effect of RH on polymer diffusivity

Figure 5 shows the relation between RH and relative swelling of samples that were conditioned and subsequently dried in a vacuum to eliminate the effect of water on the samples. During drying in vacuum, the amount of solute diffusing into cell walls was considered to be small. This is because the polymer with molecular weight higher than 400 lose its diffusivity if the sample is dried up [10] and because it generally takes shorter time to evaporate

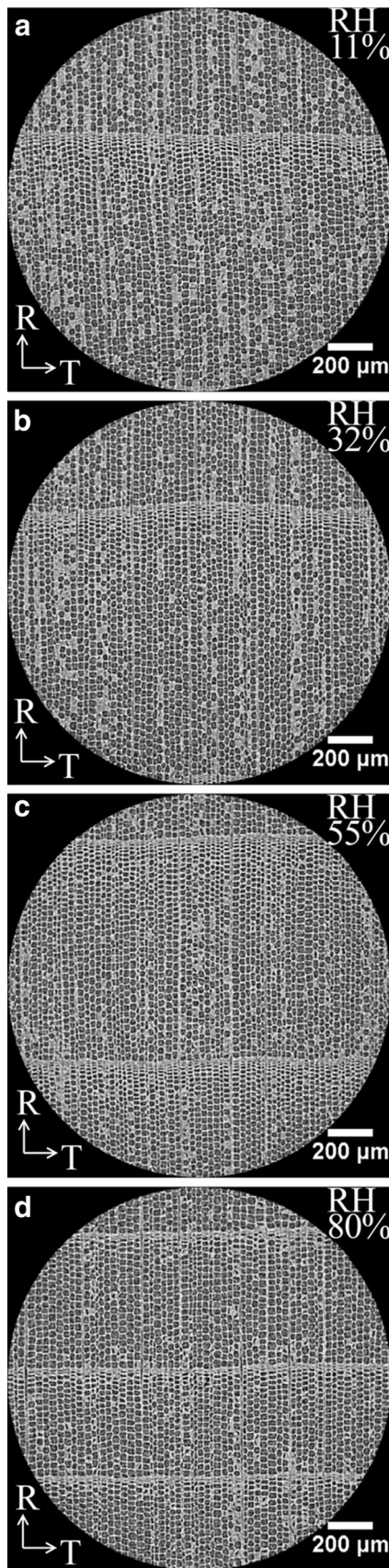
water in drying in a vacuum than in the conditioning at $RH = 11\%$ at which the solute diffusion will be indicated in the latter description to be small. Thus, the relative swelling in Fig. 5 is considered to show the amount of polymer migration into the cell walls. The amount of polymer diffusing into the cell walls during conditioning increased with RH, producing a concave-downward curve (Fig. 5), which agrees with the theoretical trend (black solid line in Fig. 3a). This agreement indicates that the theory was valid. On the basis of the theory, the tendency of the increase and decrease in the amount of the polymer diffusing into the cell walls with the RH is caused by the solute diffusivity and concentration difference, respectively (Fig. 3a). Thus, it was indicated in the measured RH range (Fig. 5) that the solute diffusivity affected the amount of the diffusing polymers much more than the concentration difference did. It was also indicated in the theory that the solute diffusivity was affected by the RH.

To confirm the polymer distribution in the samples that were conditioned and dried, the CT images of the RT cross section of the samples at each conditioning RH value were observed (Fig. 6a–d). The cell cavities completely filled with the polymers in each image were counted by the visual observations. The number of the filled cavities showed a strong negative correlation with the relative swelling for the same samples as shown in Fig. 7. This indicates that the relative swelling increased with the polymer amount in the cell walls, because the non-volatile polymers did not evaporate from the samples during conditioning and drying processes. Thus, these findings support the increase in polymer diffusing into cell walls during conditioning with RH.

Temporal variability of polymer diffusivity

Figure 8a shows the temporal variability in evaporation rate, $-\Delta M/\Delta t$, of water from impregnated samples during conditioning. The lower the RH, the greater the maximum value of evaporation rate, and the earlier the time until the evaporation rate reached zero.

Figure 8b shows the temporal variability in swelling rate, $\Delta r/\Delta t$, during conditioning. The phase in which the sample swells, when the polymers diffuse into the cell walls, existed except at an RH of 11%. The phase in which the specimen shrunk, when the water exudates from the cell walls, existed except at an RH of 80%. Maximum swelling rate (Fig. 8b) appeared after the maximum evaporation rate (Fig. 8a) and occurred earlier at lower RH values. This indicates a shorter time between the initiation of conditioning and polymer diffusion at lower RH values because of the larger concentration difference (gray broken line in Fig. 3a) caused by the higher evaporation rate (Fig. 8a). The slowest swelling rate (Fig. 8b) occurred



◀**Fig. 6** Tomogram of a RT cross section of the sample conditioned at each relative humidity (RH) and subsequently dried under vacuum

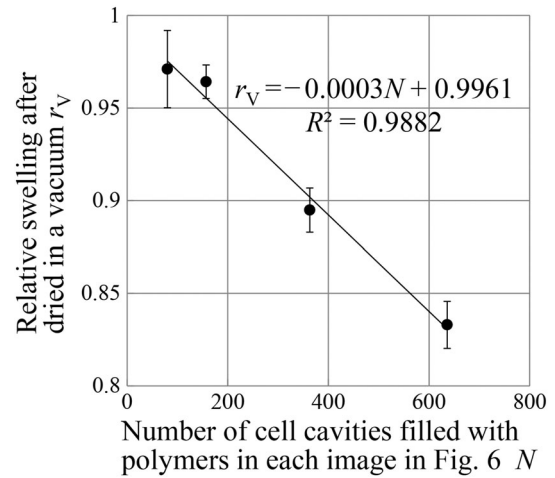


Fig. 7 Relation of relative swelling of samples conditioned and subsequently dried under vacuum, r_V , to number of cell cavities filled with polymers in each image in Fig. 6, N . Bars standard deviations

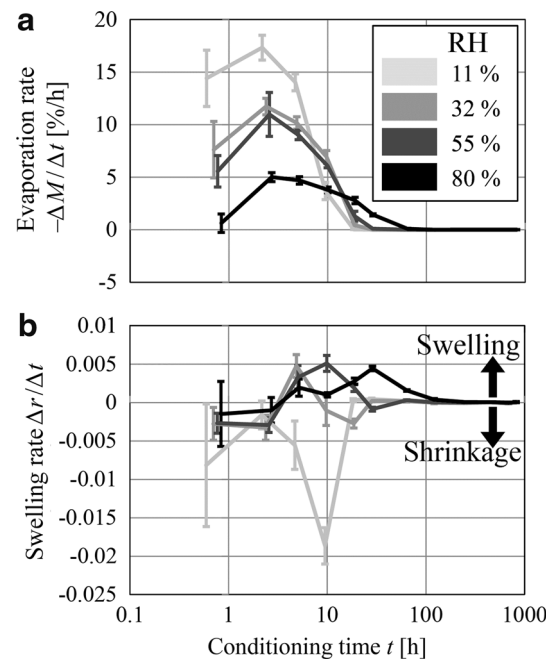


Fig. 8 Temporal variability in **a** evaporation rate of water ($-\Delta M/\Delta t$) and **b** swelling rate of samples ($\Delta r/\Delta t$) during conditioning at each relative humidity (RH). Bars standard deviations

earlier at lower RH values. This indicates that the polymer diffusivity, which was consistent at each RH value at the start of conditioning, decreased during conditioning; the decrease was greater at lower RH due to a higher evaporation rate.

Conclusions

Water evaporation and swelling and shrinkage of aqueous PEG-solution-impregnated wood samples were examined during conditioning at an RH value of 11, 32, 55, or 80 %, followed by drying under vacuum, and compared against theoretical prediction. The dried samples were observed using a micro-focus X-ray CT system. Results showed:

1. The total amount of PEG polymers diffusing into the cell walls during conditioning increased with RH. Theoretical interpretation indicated that this trend was caused by an increase in the polymer (solute) diffusivity with the amount of water (solvent) in the samples.
2. Temporal variability in the evaporation rate of water and of the swelling rate of the samples indicated that solute diffusivity was consistent at each RH value at the start of conditioning but decreased during conditioning; the decrease was greater at lower RH due to a higher evaporation rate.

Thus, conditioning of wood impregnated with a solution is efficient for the treatment of wood with solute if solvent vapor pressure is appropriately controlled. To develop a successful conditioning schedule, temporal variability in RH remains to be optimized, and the effect of factors such as temperature and solution concentration on the solute diffusing into cell walls should be investigated.

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Appendix

The relation of the amount of solute diffusing during Δt , Δn_{W1} , to RH, H , was shown to be a concave-downward curve as follows.

According to the assumption in theory, K_1 and $x_{C1} - x_{W1}$ were formulated as follows:

$$K_1 = aH, \quad (12)$$

$$x_{C1} - x_{W1} = -bH + c, \quad (13)$$

where a and b represent constant of proportionality and c is intercept ($a > 0$, $b > 0$, $c > 0$). The reason for the absence of the intercept in Eq. (12) is that the polymer completely loses its diffusivity at $H = 0$, which is supported by the fact that the PEG polymer with molecular weight higher than 400 do not swell dried wood [10]. The relation of Δn_{W1} to H can be deduced by Eqs. (4), (12), and (13) as follows:

$$\Delta n_{W1} = (-abH^2 + acH)\Delta t. \quad (14)$$

The slope of the curve, $\Delta n_{W1}(H)$, can be deduced by taking the derivative of Δn_{W1} with respect to H as follows:

$$\frac{d}{dH}(\Delta n_{W1}) = (-2abH + ac)\Delta t. \quad (15)$$

In the RH range of $H < c/2b$, $d(\Delta n_{W1})/dH > 0$ consists, and thereby Δn_{W1} increases with H . In the range of $H > c/2b$, $d(\Delta n_{W1})/dH < 0$ consists, and thereby Δn_{W1} decreases with the increase in H . Thus, the relation of Δn_{W1} to H has concave-downward shape.

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