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ORIGINAL ARTICLE

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

Soichi Tanaka[1] · **Masako Seki**[2] · **Tsunehisa Miki**[2] · **Kenji Umemura**[1] ·
Kozo Kanayama[1]

Abstract This study has focused on solute diffusing into cell walls in solution-impregnated wood under conditioning, process of evaporating solvent. The purpose of this paper was to clarify the RH- (relative humidity-) schedule that promotes the solute diffusion into shrinking cell walls during conditioning. The wood samples impregnated with a 20 mass% aqueous solution of polyethylene glycol (PEG1540) was conditioned with a temperature of 40 °C to the equilibrium point at the RH where the samples swelled maximally. The samples were subsequently conditioned at 40 °C under the schedules including four ways of RH-decrease steps where the cell walls shrunk. The amount of solute (PEGs) diffused into cell walls during the conditioning logarithmically increased with increasing the number of the RH-decrease steps. This was well explained by the theoretical model that describes the solute diffusion into shrinking cell walls. It is clarified from the model that the RH, or moisture content of the sample, should be decreased as gradually as possible to increase the total amount of diffused solute into shrinking cell walls, and that the amount of diffused solute is smaller for the lower moisture content. The model also suggests that effect of change in RH schedule on change in total amount of diffused solute does not depend on solute diffusivity in the sample under drying in a vacuum over phosphorous pentoxide, and that impregnated wood should be conditioned under natural convection rather than forced convection for promoting the diffusion into shrinking cell walls.

Introduction

Chemical treatment of wood is one of the major methods to change its properties such as biodegradability, combustibility, and dimensional instability (for example, [1]). In the treatments, the chemical substance has been developed and introduced into wood in order to control these properties that disturb the intended usage of wood [2].

For the effective improvement especially of the dimensional stability, the chemical substance is required to exist in wood cell walls rather than in cell cavities (for example, [3]). In the general treatment process, wood is impregnated with a solution of the chemical substance and is subsequently conditioned in the atmosphere to evaporate the solvent from the impregnated wood. Our previous study has shown that the amount of chemical substance, or solute, in cell walls after the impregnation process is smaller than that after the conditioning process [4].

The solute diffusion into cell walls was suggested to be driven by the higher solute concentration in cell cavities than that in cell walls, which is caused by the higher evaporation rate of solvent from cell cavities than that from cell walls [5]. The amount of the solute diffusion is considered to be increased by increasing the following two factors. The first factor is the difference in solute concentration between cell cavities and cell walls. The second factor is the solute diffusivity into cell walls [6], which is considered to increase with increasing moisture content [7] and

✉ Soichi Tanaka
soichi_tanaka@rish.kyoto-u.ac.jp

1 Research Institute for Sustainable Humanosphere, Kyoto University, Uji, Kyoto 611-0011, Japan

2 Structural Materials Research Institute, National Institute of Advanced Industrial Science and Technology, Nagoya, Aichi 463-8560, Japan
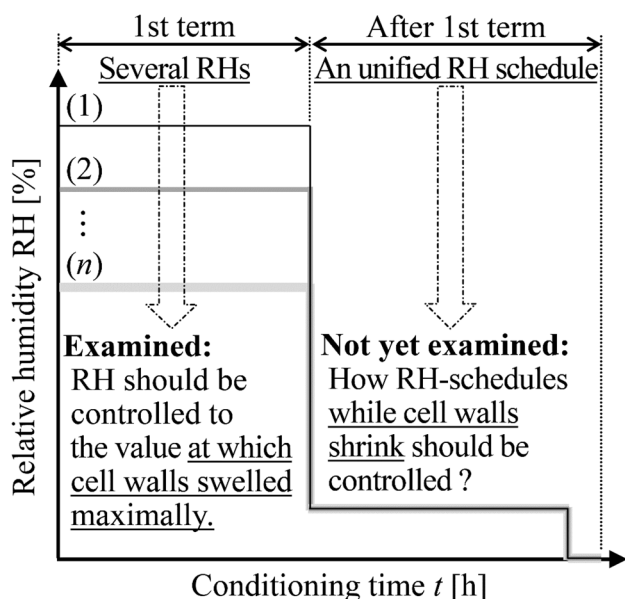
relative swelling [6] of the impregnated wood. It should be noticed that both the concentration difference and the solute diffusivity may be affected by the atmospheric state such as relative humidity (RH) and temperature during the conditioning.

The objective of this study was to determine the atmospheric states that maximize the amount of solute diffused into cell walls during conditioning process. In the previous studies [6–8], we have examined the effect of RH during the conditioning on the solute diffusion into cell walls. In these studies, cross-cut wood samples were used since they are favorable for causing the solute-concentration difference between cell walls and cell cavities. As solute and solvent in these studies, polyethylene glycols (PEGs) and water, respectively, were employed, because the amount of water or PEGs in cell walls and cell cavities can be related to mass or dimension, respectively, of the impregnated wood [9–21]. Most of PEGs are confirmed to diffuse into cell walls during conditioning [4], though the cell walls impregnated with aqueous-PEG solution absorb far smaller amount of PEGs [22]. The RH schedule in our previous study [6–8] is exemplified in Fig. 1. In the first term, the impregnated wood was conditioned at several levels of RHs. The solute (PEGs) was clarified to diffuse maximally into cell walls during the conditioning at the RH causing the cell walls to swell maximally. This is considered to have its origin in the following two relations. The first relation is the increase in the solute diffusivity with increasing the region into which solute can diffuse and replace water that diffuses into cell cavities. The second relation is the increase in this region with swelling of cell walls. If the cell walls swelled maximally in the first term, however, the RH schedule that promotes solute diffusion into shrinking cell walls after the first term (Fig. 1) has not yet been examined.

The purpose of this paper was to clarify the RH schedule that promotes solute diffusion into shrinking cell walls after the first term (Fig. 1). The solute diffusivity was surmised to decrease with both shrinking of cell walls and decreasing of solvent (water) around the solute. In addition, it may take time for the solute to diffuse into cell walls. Therefore, the solute diffusion was expected to be promoted if the water evaporation, or RH-decrease, was as slow as possible so that the solute gained time to diffuse. It was, however, difficult to control experimentally and to analyze theoretically the continuous decrease in RH while monitoring the solute diffusion. The RH, therefore, should be decreased stepwise in the schedule.

In this paper, cross-cut wood samples were impregnated with a solution including PEGs and water as a solute and solvent, respectively. These samples were conditioned at five levels of RH to confirm the RH causing the samples to swell maximally. The other groups of aqueous-PEG solution-impregnated wood samples were conditioned at the confirmed RH to the equilibrium point. These were subsequently conditioned in four schedules including 1, 2, 3, or 5 steps of RH-decrease. The theoretical model that describes solute diffusion into shrinking cell walls when RH decreases stepwise is constructed. The model was used to predict qualitatively the relation between the number of RH-decrease steps and the amount of diffused solute during conditioning. The model was also used to discuss the effect of RH schedule in general, such as continuous decrease in RH, on solute diffusion into shrinking cell walls.

Theory

The model that describes solute diffusion into shrinking cell walls is constructed here to predict qualitatively the relation between the number of RH-decrease steps, n, and the amount of diffused solute during conditioning. The model also can be used to discuss effect of RH schedule in general, such as continuous decrease in RH, on the solute diffusion. The solution-impregnated wood under conditioning swells with decreasing RH down to a level, and subsequently shrinks with decreasing RH to the further lower level, when PEG and water are employed as solute and solvent, respectively [6]. In this paper, the periods of shrinking are called the i-th terms in the schedule including the n times of RH-decrease ($i = 2, \cdots, n + 1$).

Fig. 1 Conditioning schedule of relative humidity (RH) in previous studies, definition of each term, and unclarified issue

Assumption

To simplify the phenomenon, the following assumptions, compared to the experiment in "Results and discussion," are employed.

1. Moisture content of impregnated wood under conditioning, M, affected by stepwise decrease in the RH, approximately shows stepwise decrease in order of $M_1, M_2, \ldots, M_i, \ldots$, and $M_{n+1} (=0)$.
2. Solute diffuses from a point representing cell cavities to another point representing cell walls.
3. The amount of solute diffused during a unit time at the i-the term, dA_i/dt, is approximately equal to the product of the solute diffusivity into cell walls, k_i, and the solute-concentration difference between cell cavities and cell walls, Δx_i, or the approximate equation of $dA_i/dt = k_i \Delta x_i$ consists.
4. The diffusivity, k_i, is approximately in direct proportion to $M_i + \mu$, where M_i represents moisture content at the i-the term and μ is a constant directly proportional to the solute diffusivity in impregnated wood with moisture content of $M_{n+1} (=0)$.
5. The solute-concentration difference just after the beginning of the i-th term, Δx_i, is approximately in direct proportion to the decrease in the moisture content during the same term, $\Delta M_i (=M_{i-1} - M_i)$.
6. The difference, Δx_i, approximately shows exponential decrease after the beginning of the i-th term, or Δx_i is approximately in direct proportion to $\exp(-t/\tau)$, where τ represents relaxation time independent on i.
7. The solute diffusion is almost finished till the end of the i-th term so that Δx_i decreases to be close to zero.

Theoretical equation deduced from assumption

On the basis of the assumptions 1–6, the amount of solute diffused from cell cavities to cell walls during a unit time at the i-the term ($i=2, \cdots, n+1$), dA_i/dt, is formulated as follows:

$$\frac{dA_i}{dt} = C(M_i + \mu)\Delta M_i \exp\left(-\frac{t}{\tau}\right), \tag{1}$$

where C represents normalizing coefficient.

The amount of solute diffused during the i-the term, ΔA_i, is obtained by time integration of the Eq. (1) as follows:

$$\Delta A_i = \int_0^{\Delta t_i} \frac{dA_i}{dt}dt = C(M_i + \mu)\Delta M_i \int_0^{\Delta t_i} \exp\left(-\frac{t}{\tau}\right)dt, \tag{2}$$

where Δt_i represents the duration time of the i-th term, and can be regarded as infinity based on the assumption 7, leading to the following equation.

$$\Delta A_i = \tau C(M_i + \mu)\Delta M_i. \tag{3}$$

If the impregnated wood is placed under the RH schedule causing its moisture decrease as shown in Fig. 2 in the conditioning ($n = 2, 3, \ldots$), the total amount of solute diffused after the first term, A_n, is formulated as follows:

$$A_n = \sum_{i=2}^{n+1} \Delta A_i = \tau C\left(\mu M_1 + \sum_{i=2}^n M_i \Delta M_i\right), \tag{4}$$

where M_{n+1} and $\sum_{i=2}^{n+1} \Delta M_i$ are equal to zero and M_1, respectively, on the basis of the assumption 1.

On the other hand, if the impregnated wood just after the first-term conditioning is placed in vacuum-dried state ($n=1$), ΔM_i ($i = 2, \ldots, n$) is equal to zero in the Eq. (4), on the basis of the assumption 1. Thus, the total amount of the diffusion, A_1, is represented as follows:
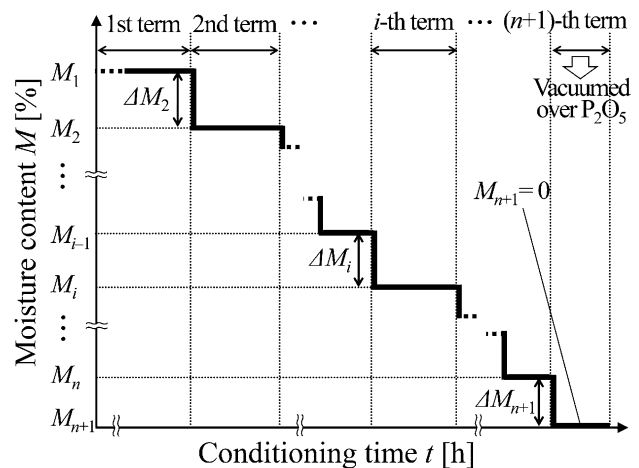
$$A_1 = \tau C\mu M_1. \tag{5}$$

Qualitative prediction using theoretical equation

For qualitative prediction, the decrease in moisture content during each term is assumed to be equal to each other. The decrease in moisture content, ΔM_i, and equilibrium moisture content, M_i, during the i-th term ($i=2, \cdots, n+1$, and $n=1, 2, \cdots$) are thereby represented as follows:

$$\Delta M_i = \frac{M_1}{n}, \tag{6}$$

$$M_i = \left(1 - \frac{i-1}{n}\right)M_1. \tag{7}$$

Fig. 2 Definition of parameters for moisture content employed in theoretical model

The amount of solute diffused during the i-the term, $\Delta A'_i$, and their total amount after the first term are predicted using the Eqs. (3), (4), (6), and (7) as follows:

$$\Delta A'_i = \frac{\tau C M_1^2}{n}\left(\frac{\mu}{M_1} + 1 - \frac{i-1}{n}\right), \tag{8}$$

$$A'_n = \frac{\tau C M_1^2}{2}\left(\frac{2\mu}{M_1} + \frac{n-1}{n}\right). \tag{9}$$

To normalize the Eqs. (3), (4), (8), and (9), the coefficient, C, is determined as follows:

$$C = \frac{2}{\tau M_1^2}. \tag{10}$$

Figure 3 shows the relation between the number of RH-decrease steps, n, and the total amount of diffused solute during conditioning, A'_n, predicted using the Eqs. (9) and (10). With increasing the number of steps, n, the total amount, A'_n, is qualitatively predicted to increase logarithmically.

Materials and methods

Sample preparation and impregnation process

Twenty-seven cross-cut samples, with dimensions of 5 mm×25 mm×25 mm in longitudinal (L), radial (R),
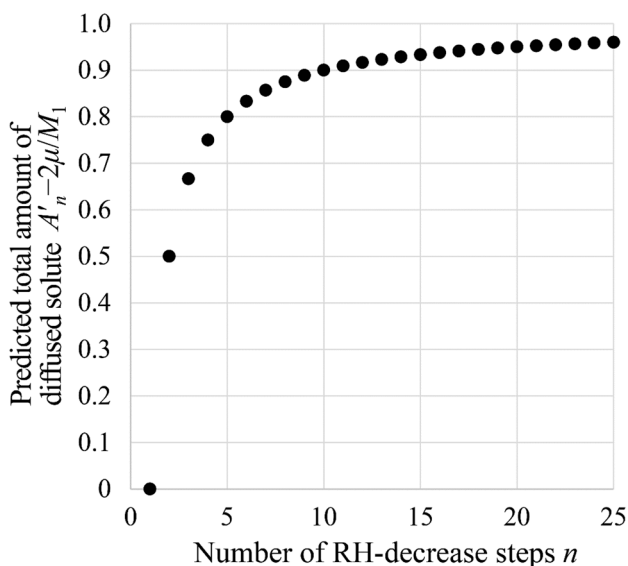
Fig. 3 Qualitative prediction of relation between number of RH-decrease step and total amount of diffused solute into cell walls during conditioning after the first term. $2\mu/M_1$ factor affected by solute diffusivity in impregnated wood dried to zero moisture content

tangential (T) directions, respectively, were subsequently prepared from a block of hinoki (*Chamaecyparis obtusa*). The samples were impregnated with deionized water and left in it for 2 months, and their mass and area in RT cross section (m_A and s_A, respectively) were measured. They were conditioned at 60% RH (20 °C) for over 2 weeks and subsequently dried at 105 °C to relatively constant mass, m_O, their RT cross-sectional area, s_O, and volume, v_O, and their average oven-dry density was 0.379 ± 0.003 g/cm³. The dried samples were impregnated with an aqueous solution of polyethylene glycol (PEG1540, E.P., Wako) with concentration of 20 mass%, and left in the solution for 5 months.

Conditioning process

The closed containers with a diameter of 88 mm and a height of 109 mm inside were prepared in the chamber (Standard industrial oven, PV-211, Espec Co.) with a temperature inside controlled at 40 °C. The temperature in this study was different from that in the previous studies, 35 °C [4, 6–8]. This was because the chamber employed in this study was changed from that in the previous studies, and because the temperature in the chamber in this study was more stable when it was controlled at 40 °C than at 35 °C under a room temperature of 20–25 °C. Atmosphere inside the container was controlled at RH of 11, 32, 53, 66, 75, or 89% with a temperature of 40 °C using saturated aqueous solution of lithium chloride (LiCl), magnesium chloride ($MgCl_2$), sodium bromide (NaBr), potassium iodide (KI), sodium chloride (NaCl), or potassium nitrate (KNO_3), respectively [23], over which the impregnated samples can be hanged over to be conditioned.

Three impregnated samples were conditioned at the RH of 11, 32, 53, 75, or 89% (40 °C) for 2.17×10^3 h, followed by drying in a vacuum over phosphorous pentoxide (P_2O_5). The RT cross-sectional area, $s(t)$, as a function of conditioning time, t, was measured, and the relative swelling, $r(t)$, was calculated using the following equation.

$$r(t) = \frac{s(t) - s_O}{s_A - s_O}. \tag{11}$$

Mass of the samples after dried in a vacuumed state, m_V, was measured to calculate weight percent gain (WPG) of the dried samples using the following equation.

$$W_V = \frac{100(m_V - m_O)}{m_O}[\%]. \tag{12}$$

The temporal variability of $r(t)$ is shown in Fig. 4. The sample was confirmed to show maximum swelling when conditioned at RH of 75%. The values of W_V were 39.9 ± 0.3, 40.1 ± 0.2, 39.4 ± 0.3, 39.3 ± 0.4, and $39.5 \pm 0.6\%$ for RH of 11, 32, 53, 75, and 89%, respectively.
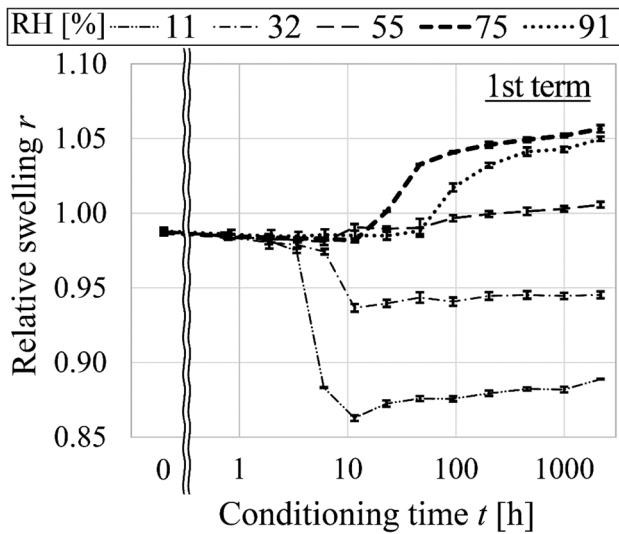
Fig. 4 Temporal variability of relative swelling of impregnated sample during conditioning in the first term. *Error bars* standard deviation
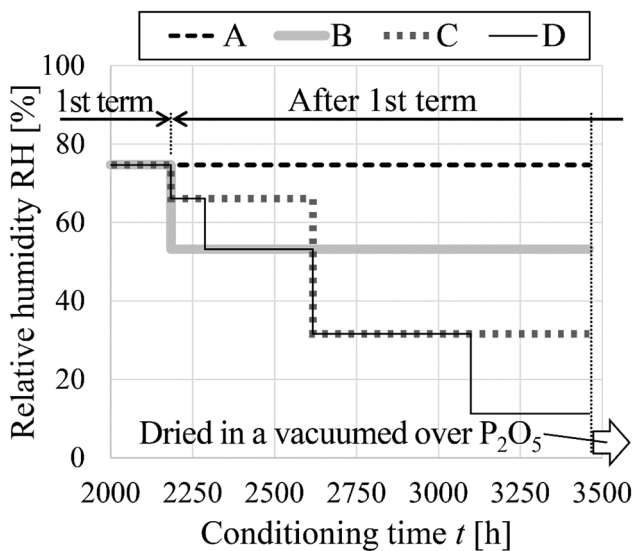
Fig. 5 Conditioning schedule of relative humidity (RH) in this study

This confirms that the total amount of PEGs in each sample after the conditioning is not so different among all RHs.

To clarify the effect of number of steps of RH-decrease when impregnated wood was conditioned during a unified period, the other sets of twelve impregnated samples were conditioned at the confirmed RH of 75% (40 °C) for 2.18×10^3 h, and subsequently conditioned at 40 °C in four schedules including 1, 2, 3, or 5 steps of RH-decrease, called schedule A, B, C, or, D, respectively (Fig. 5). The schedule A contained RH of 75% for 1.28×10^3 h, B RH of 53% for 1.28×10^3 h, C RH of 66% for 4.33×10^2 h and 32% for 8.43×10^2 h, and D RH of 66% for 1.00×10^2 h,

53% for 3.34×10^2 h, 32% for 4.82×10^2 h, and 11% for 3.61×10^2 h, where the final step in all the schedules was drying in a vacuum over P_2O_5. The mass and RT cross-sectional area during the conditioning, $m(t)$ and $s(t)$, respectively, and after the conditioning, mv and s_V, respectively, were measured. The relative swelling, $r(t)$, and the moisture content, $M(t)$, of the impregnated samples under conditioning are calculated using the Eq. (11) and the following equation, respectively.

$$M(t) = \frac{100\{m(t) - m_V\}}{m_V}[\%]. \tag{13}$$

The relative swelling and the WPG after dried in a vacuumed state, r_V and W_V, were calculated using the following equation and Eq. (12), respectively.

$$r_V = \frac{s_V - s_O}{s_A - s_O}. \tag{14}$$

The values of W_V were 39.8 ± 0.2, 39.6 ± 0.4, 40.1 ± 0.1, and $39.9 \pm 0.7\%$ for schedules A, B, C, and D, respectively. This confirms that the total amount of PEGs in each sample after the conditioning is not so different among all schedules.

Results and discussion

### Effect of number of RH-decrease steps on total solute diffusion

Figure 6 shows the relation between the number of RH-decrease steps, n, and the relative swelling of the samples after the conditioning, r_V. The value of r_V, which is proportional to the amount of solute (PEGs) diffused into cell walls during conditioning [7], logarithmically increased with increasing the number of steps, n. This trend showed good agreement with the qualitative prediction (Fig. 3).

Relationship between theory and experiment

To compare the assumption 1 to the experiment, temporal variability of moisture content of the sample during conditioning, $M(t)$, is shown in Fig. 7. The value of $M(t)$ decreased stepwise after the stepwise decrease in the RH (Fig. 5). This indicates that the moisture content approximately shows stepwise decrease, affected by the stepwise decrease in RH. This suggests the assumption 1.

The assumptions 2, 3, and 5 have already been compared to the experiments in our previous studies [4, 6–8].

To compare the assumptions 6 and 7 to the experiment, temporal variability of relative swelling of the sample during conditioning, $r(t)$, is shown in Fig. 8. The value
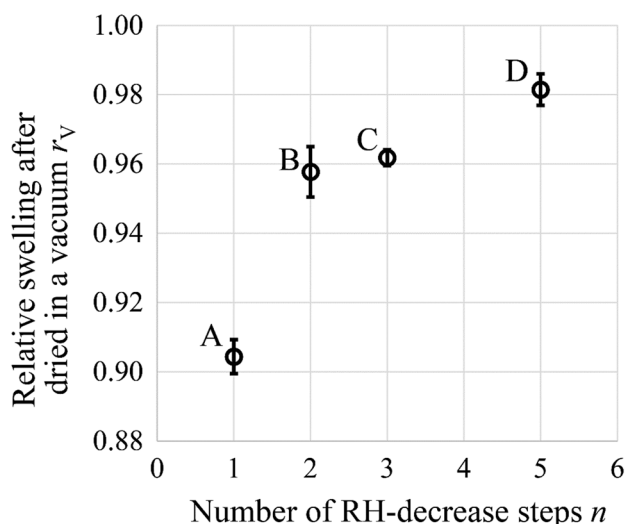
Fig. 6 Relation between number of RH-decrease steps and relative swelling of samples after conditioning at each schedule. *Error bars* standard deviation
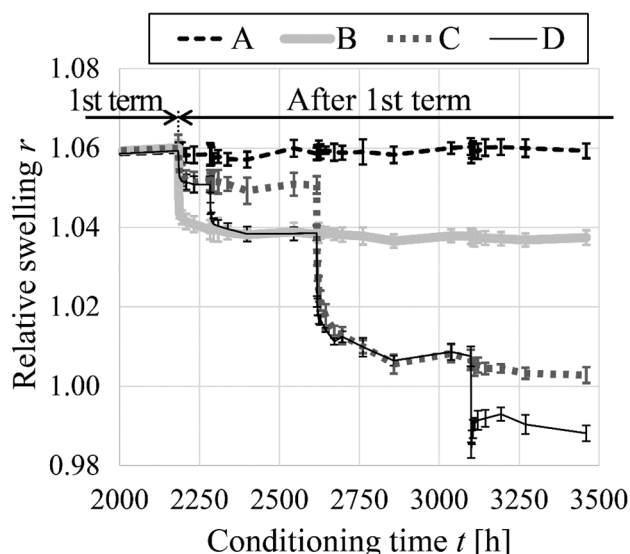
Fig. 8 Temporal variability of relative swelling of impregnated samples during conditioning after the first term. *Error bars* standard deviation
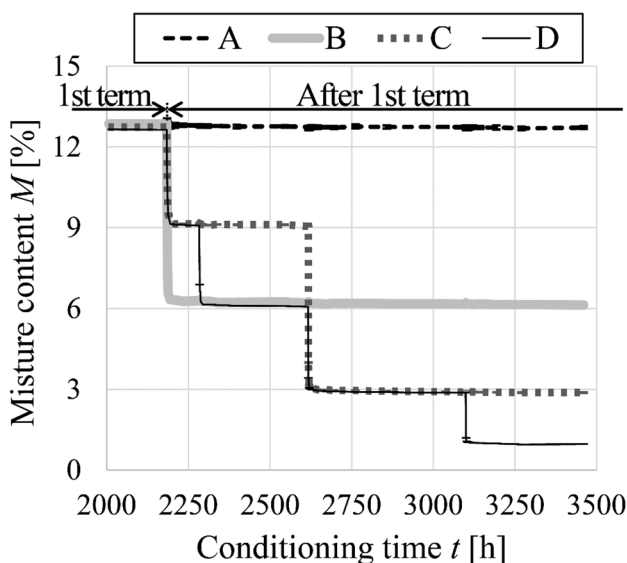
Fig. 7 Temporal variability of moisture content of impregnated samples during conditioning after the first term. *Error bars* standard deviation

of $r(t)$ is known to increase with the total amount of substance in cell walls [9–21], and thus the variation of $r(t)$ implies the migration of substance into and out of cell walls. The value of $r(t)$ decreased after the RH decreased for each term. This value reached to the equilibrium point after the moisture content, $M(t)$, reached to the equilibrium point for each term (Figs. 7, 8). These findings indicate that it takes time for the solute-concentration difference caused by the water evaporation to disappear, and

that the difference disappears to become zero till the end of each term. This suggests the assumptions 6 and 7.

The assumption 4 is related to the mobility of the solute molecules, which increase with increasing the amount of solvent around solute and with increasing the dimension of the cell walls or the samples [6, 7]. In the conditioning after the first term, the samples did not swell but shrunk (Fig. 8), indicating the solute diffusivity decrease with decreasing the moisture content. This suggests the assumption 4. At the beginning of the term of 11% RH conditioning around 3100 h for schedule D (Fig. 8), the samples shrunk rapidly followed by a moderate swelling. In contrast, the samples did not swell during the term of RH over 11%. This was probably because it took longer time for the solute under the 11% RH term than the other terms to diffuse into cell walls after the solvent exudation from cell walls, where the solvent exudation from and solute diffusion into cell walls caused the shrinkage and swelling, respectively, of the samples. This supports the assumption 4.

To compare the theoretical equations to the experiment, the total amount of diffused solute during conditioning for each number of RH-decrease steps, n, was simulated using the Eqs. (4) and (5), and the equilibrium moisture content, M_i, and moisture decrease, ΔM_i, during the i-th term (Table 1) estimated using the moisture content in Fig. 7. Figure 9 shows the relation between the simulated total amount of solute diffused into cell walls, A_n, and the relative swelling, r_V, which is proportional to the amount of diffused solute during conditioning after the first term. The simulated value, A_n, strongly correlated with the experimental value, r_V ($R^2 = 0.988$). This indicates that the

Table 1 Equilibrium moisture content and moisture decrease during each term, employed in simulation

Schedule	Number of RH[a]-decrease n	Equilibrium moisture content (%)					Moisture decrease (%)			
		M_1	M_2	M_3	M_4	M_5	ΔM_2	ΔM_3	ΔM_4	ΔM_5
A	1	12.72	–	–	–		–	–	–	–
B	2	12.86	6.14	–	–		6.72	–	–	–
C	3	12.74	9.05	2.88	–		3.69	6.17	–	–
D	5	12.64	9.08	6.08	2.89	0.96	3.57	3.00	3.19	1.93

[a] Relative humidity
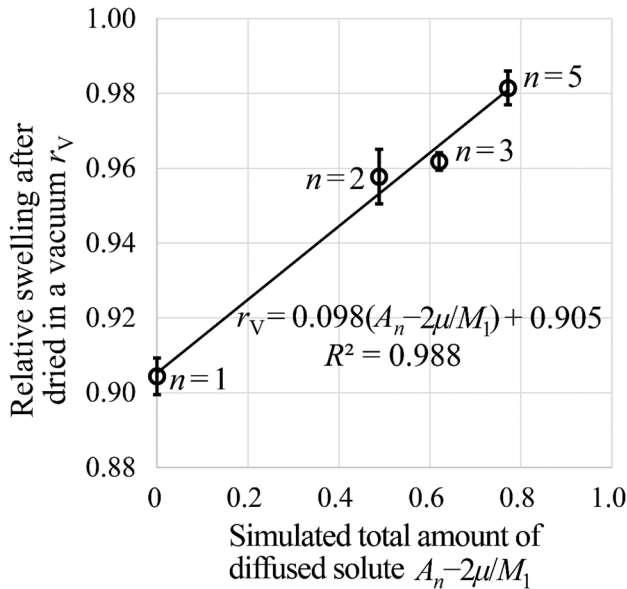
Fig. 9 Relation of relative swelling of samples after conditioning to simulated total amount of solute diffusion during conditioning. $2\,\mu/M_1$ factor affected by solute diffusivity in impregnated wood dried to zero moisture content. *Error bars* standard deviation

theoretical equations can explain the total amount of solute diffused into cell walls after the first term.

Suggestions obtained from theoretical model

The theoretical model represented by Eqs. (4) and (5) can simulate the total amount of diffused solute using the moisture content of the sample. This suggests that the total amount of the diffused solute during conditioning can be simulated by the temporal variability of moisture content of impregnated wood.

In the simulated total amount of diffused solute, A_n ($n = 1, 2,...$), in Eqs. (4) and (5), the term as a function of the number of RH-decrease, n, does not contain the constant μ, which is directly proportional to the solute diffusivity in impregnated wood dried to zero moisture content. This suggests that effect of change in RH schedule on change in total amount of diffused solute does not depend

on solute diffusivity in the sample under drying in a vacuum over P_2O_5.

The relation of the amount of diffused solute, ΔA_i, to equilibrium moisture content, M_i, during the i-th term, is represented as Eq. (15), if the decrease in moisture content during each term is assumed to be equal to each other, or the Eqs. (7), (8), and (10) consist.

$$\Delta A'_i = \frac{2}{nM_1}(M_i + \mu)\cdot \tag{15}$$

This equation suggests that the amount of diffused solute is smaller for the lower moisture content.

To satisfy the assumption 7, the solute-concentration difference is required to disappear due to the solute diffusion till the end of each term. If the RH decreases continuously in the schedule, the quasi-static state in which the solute diffusion constantly finishes is required to be maintained during conditioning to maximize the total amount of the solute diffusion. Although the quasi-static state is hardly realized, this is close to the state in which the time until the moisture content reaches to the equilibrium point is far longer than the relaxation time of the concentration difference, τ, on the basis of the assumption 6. This suggests that moisture content of sample, or RH, should be decreased as gradually as possible to increase the total amount of diffused solute into cell walls.

The convection type as well as the RH is an important factor as the atmospheric state of conditioning, because the evaporation rate of water under forced convection is usually higher than that of natural convection, affecting the solute diffusivity. In the first term, the forced convection is confirmed in our previous study [8] to promote the solute diffusion into swelling cell walls. After the first term, the natural convection is suggested to promote the solute diffusion into cell walls, because the gradual evaporation of water promotes the diffusion as suggested above.

Conclusion

The purpose of this paper was to clarify the RH schedule that promotes solute diffusion into shrinking cell walls after the first term.

Cross-cut wood samples impregnated with a 20 mass% aqueous solution of PEG1540 were conditioned to the equilibrium point at RH of 75% (40 °C) where the samples swelled maximally. The samples were subsequently conditioned at 40 °C under the RHs scheduled in four ways of RH-decrease steps where the cell walls shrunk.

The amount of solute (PEGs) diffused into cell walls during the conditioning, estimated using the relative swelling of the conditioned samples, logarithmically increased with increasing the number of the RH-decrease steps. This was well explained by the theoretical model that describes the solute diffusion into shrinking cell walls. The assumptions in the model were compared to the experiments. The following suggestions were obtained from the model when impregnated wood shrinks during conditioning after the first term.

1. Moisture content of impregnated wood under conditioning, or RH, should be decreased as gradually as possible to increase the total amount of diffused solute into shrinking cell walls.
2. The amount of the diffused solute is smaller for the lower moisture content.
3. Effect of change in RH schedule on change in total amount of diffused solute does not depend on solute diffusivity in the sample under drying in a vacuum over phosphorous pentoxide.
4. For promoting solute diffusion into shrinking cell walls, impregnated wood should be conditioned under natural convection rather than forced convection.

To investigate the applicability of the proposed model is the important challenge to be solved.

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