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Formulation of maximum weight percent gain of wood impregnated with resin considering cell wall swelling and resin concentration distribution: in the impregnation of six softwood species with phenol formaldehyde resin

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Abstract

Weight percent gain (WPG) is used as an index of the quantity of impregnated resin into wood. It is well-known that the maximum value of WPG is roughly calculated using the volume of cell lumen included in wood. In this study, we proposed the theoretical formula that calculates the maximum value of WPG (*W*-value), with higher accuracy by considering the cell wall swelling and resin concentration distribution as well as the cell-lumen volume. In this paper, the WPG of wood for six softwood species was measured after the injection with phenol–formaldehyde resin solution and after the subsequent conditioning and drying. The measured WPG values after each process were successfully explained by the *W*-value calculated using the theoretical formula. It was suggested from the calculation that the WPG was greatly influenced by the cell wall swelling and by the difference in solution concentration between the cell lumen and the transient pore in the cell wall. The influences were also suggested to be larger for wood with higher density.

Keywords Impregnation, Weight percent gain, Cell wall swelling, Concentration distribution of solution, Resin treatment, Physical-property control

Introduction

Impregnation of wood with chemicals is widely used to stabilize the dimension of wood [1], to improve the durability of wood [2, 3], and to fix the shape after deformation processing of wood [4]. In the process of the impregnation, a solution of the chemicals is injected into wood, followed by conditioning and drying to evaporate the solvent or water [5] for adding various performances. During the injection phase, irregular treatment occurs due to obstructions in liquid permeation [6] caused by aspirated pits in the tracheid and by tyloses in the vessel. Irregular treatment can cause the problem of reducing the performance such as decay resistance [2] and fire retardance [3]. Therefore, for enhancing the product quality, many studies have aimed to improve the liquid permeability and thereby to address the treatment irregularities [3, 6]. For the quality control, it is necessary to have an indicator to evaluate the treatment irregularities.



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The weight percent gain (WPG) due to the treatment has been widely used as such an indicator. As the measured WPG approaches the theoretical maximum WPG, referred to as "*W*-value" in this paper, the treatment becomes increasingly uniform. However, few studies have focused on calculating *W*-value after the injection phase. The *W*-value is determined by the mass of solution required to fill all the voids included in wood. Furthermore, the mass of the solution should be determined by "the volume of all voids in wood" and "the solution concentration, or solution density, in wood".

All voids in wood are mainly composed of cell lumen and transient pore in the cell wall. If the volumetric change in the cell lumen with swelling and shrinkage of wood is considered negligible [7], the volume of the cell lumen can be determined by the density and the volume of oven-dry wood [8]. The transient pore, on the other hand, is generated when the cell wall is swollen by a swelling agent represented by water. The maximum specific volume of the transient pore can be considered as that of the cell wall swelling, referred to as "cell wall swelling coefficient" in this paper. The cell wall swelling coefficient is equal to the proportionality constant in the directly proportional relationship between the oven-dry density and the external swelling rate of wood, and it has been estimated to $0.28 \text{ cm}^3/\text{g}$ when the swelling agent is water [7]. It is well-known that the cell wall swelling varies with the type of swelling agent such as water, organic solvent [9], and resin causing the bulking effect on wood [10] and with their mixing ratio [11], or solution concentration. Thus, the cell wall swelling coefficient should be affected by these factors. Consequently, the volume of all voids in wood can be explained by the cell-lumen volume and the cell wall swelling coefficient.

The solution concentration should not be considered uniform throughout wood. In the cell lumen, the concentration can be equal to the injected solution concentration, since the solution can be free from the cell wall as water in the cell lumen is regarded as free water [8]. While in the transient pore in the cell wall, the solution concentration can be different from that in the cell lumen. This is supported by the study [12] on the positive or negative adsorption of polymers on the cell wall when wood flour is immersed in polymer solutions.

Previous studies have established the formula for calculating the *W*-value after the injection phase considering only the cell-lumen volume [13, 14]. However, no formula has been established that takes account of the cell wall swelling coefficient and of the solution-concentration distribution as well as of the lumen volume.

Even if the measured WPG became closer to the *W*-value after the injection phase, it is nevertheless of practical issue to calculate the *W*-value for predicting

the WPG after the phase of the conditioning and drying following the injection phase. In recent years, new deformation processing techniques, such as wood flow forming [15], have been developed to promote the use of wood as an industrial material. In such a technique, the resin impregnation has been applied as a pre-treatment or post-treatment. It was reported [4] that the WPG after conditioning and drying of the resin-solution injected wood greatly affected the density and mechanical property of the produced material. It is therefore necessary to predict the *W*-value after conditioning and drying for controlling the quality of the material.

The purpose of this study was to formulate the *W*-values after the solution injection into wood and after the subsequent conditioning and drying. The cell wall swelling and the difference in solution concentration between the cell lumen and the transient pore were incorporated into the formula as well as the volume of cell lumen. In this paper, the WPG after injection of phenol–formal-dehyde (PF) resin solution into wood and after the subsequent conditioning and drying were measured and compared with the *W*-value obtained from the theoretical formula for investigating the validity of the formula.

Theory

The model for the formulation of the *W*-value in the resin impregnation is shown in Fig. 1. The main assumptions of the model are shown as the following:

- 1. Wood is composed of cell lumen and cell wall, and the cell wall includes pure wood substance (pure cellwall substance) [8] and transient pore.
- Injection allows the solution to fill up all the cell lumen, and it also causes the maximum swelling of the cell wall where all the transient pore is filled up with the solution.
- 3. The specific volume of cell wall swelling is proportional to the mass of oven-dried wood, or pure wood substance, m_0 , and the proportionality coefficient is defined as the cell wall swelling coefficient, *k*.
- 4. When the cell wall swells, the volume and density of pure wood substance are constant, and the volume of the transient pore changes in the range from 0 to km_0 . This means that the cell wall has the volume of $v_{\rm ws}$ in oven-dry state and it has the volume of $v_{\rm ws} + km_0$ after the cell wall swelling due to the injection.
- 5. The volume of the cell lumen, ν_{cl} , remains constant even when the cell wall swells.
- 6. The solution concentration and density are equal to u and ρ_s , respectively, in the cell lumen and to u' and ρ_s , 'respectively, in the transient pore.



Fig. 1 Model for *W*-value formulation in resin impregnation when considering cell wall swelling and concentration distribution of resin solution. m_o, m_i , and m_i ; mass of wood without treatment in oven-dry state, after solution injection, and after conditioning and drying, respectively. ρ_o , ρ_{wsr}, ρ_s , and ρ'_{sr} ; densities of oven-dry wood, pure wood substance (pure cell-wall substance), solution in cell lumen, and solution in transient pore, respectively. k_i cell wall swelling coefficient. u and u'_i ; concentrations of solution in cell lumen and in transient pore, respectively. v_o, v_{wsr} , and v_{cl} ; volumes of wood, pure wood substance, and cell lumen, respectively

Being based on this model, the mass of wood without treatment in oven-dry state, after solution injection, and after conditioning and drying, m_o , m_i , and m_f , respectively, are expressed as the following:

$$m_{\rm o} = \rho_{\rm o} \nu_{\rm o} = \rho_{\rm ws} \nu_{\rm ws},\tag{1}$$

$$m_{\rm i} = m_{\rm o} + \rho_{\rm s} \nu_{\rm cl} + \rho_{\rm s}' k m_{\rm o},\tag{2}$$

$$m_{\rm f} = m_{\rm o} + u\rho_{\rm s}v_{\rm cl} + u'\rho'_{\rm s}km_{\rm o}, \qquad (3)$$

where $\rho_{\rm o}$ and $\rho_{\rm ws}$ are the densities of oven-dry wood and pure wood substance, respectively, and $\nu_{\rm o}$ is the volume of wood, and the following equation holds.

$$\nu_{\rm o} = \nu_{\rm ws} + \nu_{\rm cl},\tag{4}$$

Substituting the Eqs. (1)-(4) for the WPG after solution injection, $W_i = (m_i - m_o) / m_o$, and the WPG after conditioning and drying, $W_f = (m_f - m_o) / m_o$, the *W*-values of W_i and W_f respectively, are obtained as the following:

$$\frac{W_{\rm i}}{\rho_{\rm s}} + \frac{1}{\rho_{\rm ws}} = \frac{1}{\rho_{\rm o}} + \frac{\rho_{\rm s}'}{\rho_{\rm s}}k,\tag{5}$$

$$\frac{W_{\rm f}}{u\rho_{\rm s}} + \frac{1}{\rho_{\rm ws}} = \frac{1}{\rho_{\rm o}} + \frac{u'\rho'_{\rm s}}{u\rho_{\rm s}}k,\tag{6}$$

where ρ_s is the density of the impregnated solution and can be estimated using the following equation, which is quoted from the reference [16].

$$\rho_{\rm s} = \frac{\rho_{\rm u} \rho_{\rm w}}{\left(1 - u\right) \rho_{\rm r} + u \rho_{\rm w}},\tag{7}$$

where $\rho_{\rm r}$ and $\rho_{\rm w}$ are the densities of resin and solvent (water), respectively.



Fig. 2 Relationship between solution concentration in cell lumen, *u*, and cell wall swelling coefficient, *k*. Each line is a function given in Eqs. (8)-(10). EW, earlywood; LW, latewood



Fig. 3 Relation **a** of $k\rho'_{s}$ to *k* for calculating ρ'_{s} and **b** Relation of $u'k\rho'_{s}$ to $k\rho'_{s}$ for calculating *u'* in case of u = 0.2. EW, earlywood; LW, latewood

Materials and methods Measurement of WPG in impregnation

Twelve pieces of specimens with a dimension of 3 mm (R, radial)×10 mm (T, tangential)×40 mm (L, longitudinal) were cut from hinoki (*Chamaecyparis obtusa*), sugi (*Cryptomeria japonica*), or akamatsu (*Pinus densiflora*). For Radiata pine (*Pinus radiata*), twelve specimens with the same dimension were cut from the earlywood

or latewood. All the specimens were dried in the drying chamber at 105 °C for at least 24 h. The mass, m_o , and volume, v_o , of the specimens in oven-dry condition were measured to evaluate the oven-dry density, ρ_o .

The specimens were then injected with an aqueous solution of PF resin (PX-341, Aica Kogyo Co., Ltd.) with a solid concentration of u=0, 0.1, 0.2, or 0.3 at room temperature. In the injection, the specimens were placed under a vacuumed pressure of 1.0 kPa for 1 h and then submerged in the solution with the vacuumed pressure maintained, followed by being placed in the solution under an atmospheric pressure for 1–2 days. The mass, m_i , and volume, v_i , of the specimens were measured after being taken out from the solution. The specimens were conditioned at 20 °C under 59%RH for 5–7 days, and subsequently dried at 40 °C under a vacuumed pressure of 1.0 kPa in the sealed container with P_2O_5 . The mass m_f of the sample after conditioning and drying was measured.

The WPGs of each specimen after the injection and after the subsequent conditioning and drying were determined to be $W_i = (m_i - m_o) / m_o$ and $W_f = (m_f - m_o) / m_o$, respectively.

Setting of coefficients in calculation

In all the calculation in this paper, the densities of pure wood substance, PF resin, and solvent (water) were constant to be, $\rho_{\rm ws} = 1.5$ g/cm³ [8], $\rho_{\rm r} = 1.4$ g/cm³, $\rho_{\rm w} = 1.0$ g/ cm³, respectively. The density of resin solution, $\rho_{\rm s}$, was estimated using Eq. (7) for each concentration, *u*.

Calculation of cell wall swelling coefficient

For each concentration, *u*, and for each wood species, the cell wall swelling coefficient, *k*, was calculated as β_i / ρ_o , where β_i is volumetric swelling coefficient of wood, calculated as $(v_i - v_o)/v_o$.

The relationship between u and k is shown in Fig. 2. The measured values for u=0 were distributed in the range of k=0.18-0.36 cm³/g. For u=0.1-0.3, the distributed range was shifted to about k=0.22-0.44 cm³/g. The average value for all species of k increase with increasing u in the range of u=0-0.1 and was nearly constant in the range of u=0.1-0.3. For the calculation of W-value as shown below, it is necessary to specify the range over which k is distributed for each u. Thus, the representative function for this tendency, $k=k_{mid}(u)$, shown by the dashed line in Fig. 2, was given as the following:

$$k_{\rm mid}(u) \,/\, {\rm cm} \cdot {\rm g}^{-3} = \begin{cases} 0.60 \,\, u + 0.27 \quad (0 \le u \le 0.1) \\ 0.33 \qquad (0.1 < u \le 0.3) \end{cases} \,. \tag{8}$$

The maximum and minimum function, $k = k_{max}(u)$, and $k = k_{min}(u)$, shown by the chain-dotted and dotted lines in



Fig. 4 Calculated relations to solution concentration in cell lumen, u, of **a** solution density in transient pore ρ'_s and of **b** solution concentration in transient pore, u'. Solid lines are the expressions given in Eqs. (11)-(12)

Fig. 2, were also given by considering the measured values as follows:

$$k_{\min}(u) / \operatorname{cm} \cdot \operatorname{g}^{-3} = \begin{cases} 0.40 \ u + 0.18 & (0 \le u \le 0.1) \\ 0.22 & (0.1 \le u \le 0.3) \end{cases}, \quad (9)$$

$$k_{\max}(u) / \operatorname{cm} \cdot \operatorname{g}^{-3} = \begin{cases} 0.80 \ u + 0.36 & (0 \le u \le 0.1) \\ 0.44 & (0.1 \le u \le 0.3) \end{cases}$$
(10)

Calculation of density and concentration of solution in transient pore

In the solution impregnated wood, the concentration of the solution has been suggested to differ between in cell lumen



Fig. 5 Relationship **a**–**d** between inversed oven-dry density, $1/\rho_{or}$ and normalized *W*-value, $W_t/\rho_s + 1/\rho_{ws}$, after injection of solution with u = 0-0.3. **e**–**g** Relationship between inverse of oven-dry density, $1/\rho_{or}$, and normalized *W*-value, $W_t/(u\rho_s) + 1/\rho_{ws}$, after conditioning and drying of specimen injected with solution of u = 0.1-0.3. EW, earlywood; LW, latewood

and in transient pore [11]. This also suggests that the density of the solution can also differ between them. The density and concentration in transient pore in cell wall were calculated here. For each concentration, u, and for each wood species, the values of $k\rho'_s$ and $u'k\rho'_s$ were calculated using Eqs. (5) and (6), by substituting the measured values for W_i and W_p respectively. Figure 3a, b shows the relation of $k\rho'_s$ to k and the relation of $u'k\rho'_s$ to $k\rho'_{s'}$, respectively, for u=0.2. The slope of the regression line for the relation in Fig. 3a or Fig. 3b can be calculated as the density or concentration of the solution in transient pore, ρ'_s or u'. The calculated values of ρ'_s and u' means the representative density and concentration of the solution in transient pore for all species. The same calculations for u=0, 0.1, and 0.3, were also performed.

The calculated relation of ρ'_{s} to u and the calculated relation of u' to u are shown in Fig. 4a, b, respectively. These relations are the representative of all species, since these were calculated using Fig. 3a, b. Both the solution density ρ'_{s} and solution concentration u' in the transient pore increased with increasing solution concentration in the cell lumen, u. The solution concentration in the transient pore was especially indicated to be considerably larger than in the cell lumen. Equations for ρ'_{s} and u' as a function of u, calculated by regression of a linear equations on the plots in Fig. 4, are expressed as the following:

$$\rho'_{\rm s} / \operatorname{g-cm^{-3}} = \begin{cases} 0.635 \, u + 1.03 & (0 \le u \le 0.2) \\ 1.16 & (0.3 < u \le 0.3) \end{cases}, \quad (11)$$

$$u' = \begin{cases} 4.32 \ u & (0 \le u \le 0.1) \\ 1.71 \ u + 0.261 & (0.1 \le u \le 0.3) \end{cases}$$
(12)

Calculation of W-value

For comparing *W*-value with the measured WPG, the normalized *W*-values, $W_i/\rho_s + 1/\rho_{ws}$ and $W_f/(u\rho_s) + 1/\rho_{ws}$, as a function of $1/\rho_o$ were calculated using Eqs. (5) and (6), respectively, with the substitution of the Eqs. (8)-(12) for *k*, *u*', and ρ'_s .

For discussing the factors affecting the WPG, the W-values, W_i and W_i , were also calculated.

Results and discussion

Comparison of W-values with measured WPG

Figure 5a–d shows the relationship between the inversed oven-dry density, $1/\rho_{o}$, and the normalized *W*-values, $W_i/\rho_s + 1/\rho_{ws}$, after injection of the solution with u = 0-0.3. The normalized *W*-values obtained in calculation, shown by the lines, were directly proportional to the inversed density. The measured values, shown by plots, were distributed around the lines. This indicates that the maximum WPG increased with increasing the volume of cell lumen in wood being inversely proportional to the



Fig. 6 Calculated relationship between solution concentration, u, and W-value after conditioning and drying, W_f without (k=0) and with (others) considering cell wall swelling or resin concentration distribution

density. The normalized *W*-value obtained in calculation increased with increasing the value of *k* from k_{\min} to k_{\max} . The measured values were located between the lines for the minimum and maximum function, k_{\min} and k_{\max} . For the specimens with the *k*-values closer to k_{\min} and k_{\max} in Fig. 2, the measured values showed the tendency to

be closer to the line for k_{\min} and k_{\max} , respectively. These findings indicate that the measured values were well-explained by the *W*-values and that the maximum WPG was increased by the cell wall swelling.

Figure 5e–g shows the relationship between the inversed oven-dry density, $1/\rho_{o}$, and the normalized *W*-value, $W_{\rm f}/(u\rho_{\rm s}) + 1/\rho_{\rm ws}$, after the conditioning and drying of the specimen injected with solution of u = 0.1-0.3. The calculated *W*-value and the measured values for $W_{\rm f}/(u\rho_{\rm s}) + 1/\rho_{\rm ws}$ showed the same tendency as those for $W_{\rm f}/(u\rho_{\rm s}) + 1/\rho_{\rm ws}$ (Fig. 5a–d). The increase in $W_{\rm f}/(u\rho_{\rm s}) + 1/\rho_{\rm ws}$ with the increasing *k* from $k_{\rm min}$ to $k_{\rm max}$ was more evident in comparison with $W_{\rm i}/\rho_{\rm s} + 1/\rho_{\rm ws}$. This indicates that the influence of cell wall swelling on the maximum WPG was more evident after the conditioning and drying than after the injection.

Essential factors affecting maximum WPG

As suggested above, the measured WPG was wellexplained by the maximum WPG calculated using the proposed formula of *W*-value. It is well-known that the maximum WPG is mainly affected by the volume of cell lumen in wood [13, 14]. The WPG may also be affected by other two factors, or the cell wall swelling and concentration difference between cell lumen and transient pore. For demonstrating the extent of the influence of these two factors on the maximum WPG, the *W*-values after the conditioning and drying were calculated in various cases.

The calculated relationships between solution concentration, u, and W-value, W_{f} , are shown for wood with oven-dry density of $\rho_0 = 0.3$ and 0.7 g/cm³ in Fig. 6a, b, respectively. The W-value without considering cell wall swelling, shown by gray solid line, was smaller than the value with consideration of the swelling, shown by other lines. The W-value was especially higher with larger cell wall swelling in order of k_{max} , k_{mid} , and k_{min} , shown by chain-dotted, dashed, and dotted lines, respectively. The W-value was also higher when considering the solutionconcentration difference between cell lumen and transient pore, u and u'_{i} respectively, shown by black lines in Fig. 6a, b. These trends became more pronounced for higher wood density. It was suggested from these findings that the WPG was greatly influenced by the cell wall swelling and also by the difference in solution concentration between the cell lumen and the transient pore. It was also suggested that the influences were larger for wood with higher density because of the larger volume of the transient pore in cell wall which was included in wood.

Conclusion

For resin impregnation of wood, the theoretical formula that calculates the maximum value of WPG, *W*-value, was proposed when considering the cell wall swelling and resin concentration distribution as well as the cell-lumen volume. The WPG of wood impregnated with phenol-formaldehyde resin solutions was measured for several softwood species after injection and after the subsequent conditioning and drying. The validity of the proposed formula was investigated by being compared with the measured WPG in normalized form.

The measured WPG values in normalized form were successfully explained by the *W*-value calculated as the normalized form using the theoretical formula. The *W*-value was increased with increasing the cell wall swelling coefficient. It was also increased when considering the difference in solution concentration between the cell lumen and the transient pore. These findings suggest that the WPG was greatly influenced by the cell wall swelling and by the difference in solution concentration between the cell lumen and the transient pore in the cell wall. The influences were also suggested to be larger for wood with higher density.

Appendix

See Fig. 7 here.



Fig. 7 Relationship between concentration, u, and density, ρ_{sr} of the injected solutions of PF resin. Plot, measured value; line, calculated value using Eq. (7)

The density of the resin solution is represented in Eq. (7), which is quoted from the reference [16]. The purpose here was to confirm the accuracy of this equation by comparison with the experimental results. The volume and mass of the aqueous solution of PF resin (PX-341, Aica Kogyo Co., Ltd.) were measured for estimating the density of the solution for concentrations of u = 0, 0.1, 0.2, 0.3, 0.4, and 0.5. Figure 7 shows the relationship between the concentration and the density of the solution. The plotted values, which were obtained through measurement, were well-explained by the calculated line using Eq. (7). This indicates that Eq. (7) is valid in the concentration range of u = 0-0.5. However, it is necessary to verify the applicability of Eq. (7) each time a solution containing a different chemical or solvent is used.

Abbreviations

- WPG Weight percent gain
- RH Relative humidity
- EW Earlywood
- LW Latewood

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Author contributions

ST conceptualization; ST and MS methodology; ST calculation; ST investigation; ST writing-original draft; MS, MA and TM supervision; ST and TM project administration; ST and TM funding acquisition. All authors reviewed the manuscript.

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Availability of data and materials

All data and code supporting the findings of this study are available from the corresponding author upon reasonable request.

Declarations

Ethics approval and consent to participate Not applicable.

Consent for publication

Not applicable.

Competing interests

Not applicable.

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