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Impregnation of radiata pine wood by vacuum treatment II: effect of pre-steaming on wood structure and resin content

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Abstract Radiata pine sapwood and heartwood were dried using high-temperature, conventional-temperature, and air drying schedules with and without pre-steaming. They were then impregnated by vacuum treatment with double-distilled water, toluidine blue, and fluorescein dye. For sapwood, there were only minor differences in uptake between drying methods and when pre-steaming was used. Using microscopy, the primary flow pathways in sapwood were found to be the resin canal network and ray parenchyma cells, which provided conduction without large resistance. In heartwood, uptake was strongly influenced by pre-steaming the green lumber. After pre-steaming heart-wood, there was an increase in uptake from all surfaces but especially from the radial surfaces. Lower extractive contents, disruption of epithelial and ray parenchyma cells, and alteration of the condition of bordered pits were also associated with pre-steaming. It was therefore possible to classify flow paths in radiata pine heartwood five ways, according to uptake values and wood anatomical features.

Key words Flow pathway · Impregnation · Pre-steaming · Radiata pine · Ray parenchyma · Resin canal · Uptake

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Introduction

It is well known that the sapwood of radiata pine (*Pinus radiata* D. Don) is easily impregnated with preservative chemicals after drying. With heartwood, however, it can be more difficult to achieve consistent penetration to the specified levels. Several studies have described the usual flow pathways for radiata pine sapwood. For example, McQuire[1] found that during high-pressure preservative treatment the solution penetrated the ray parenchyma. Rupture of the pit membranes of half-bordered pit pairs subsequently led to infiltration of tracheids. Bamber and Burley[2,3] identified interstitial spaces created by the collapse of thin-walled parenchyma during drying. These spaces improved conduction through the ray and resin canal networks. A likely model for liquid flow in sapwood would involve most liquid first entering the resin canals and then flowing into the adjoining network of ray parenchyma and finally into tracheids via conductive pits.[4,5] What is lacking is a clear understanding of the factors responsible for variation in liquid uptake by radiata pine heartwood. Also, it is not precisely known how industrial treatments, such as pre-steaming green lumber and high-temperature drying, lead to improvements in treatability.

There are relatively few established theories explaining liquid flow in heartwood. Matsumura et al.[6] stated that the removal of resin increased the permeability of Japanese larch heartwood. They found that there were significant correlations between the permeability of samples and the methanol-soluble extractive content. There was also a relation found between permeability and the number of resin canals. According to Cown,[7] the resin content of radiata pine sapwood does not vary greatly from 1.5% (by weight) of substances soluble in methanol. On the other hand, the resin content of heartwood commonly ranges from 2% to 10%. Resin content may therefore be a significant factor determining liquid uptake by heartwood. High resin content in the resin canals would be an obvious obstruction to liquid flow, particularly when the resin canals are probably also a primary flow pathway in heartwood.

In addition, occlusion or encrustation of non-aspirated bordered pits would further restrict flow between tracheids.[8]

The purpose of this study was to clarify the effects of pre-steaming and drying on liquid uptake by radiata pine heartwood and sapwood. We used vacuum treatment, not pressure treatment, because the pressure itself can cause changes to wood microstructure. Matsumura et al.[9] used a combination of fluorescent dye and confocal microscopy to observe the anatomical flow paths through wood. Using this technique and ethanol extraction of resin in this study, we further describe the relations between flow path and liquid uptake for radiata pine heartwood and the changes associated with pre-steaming and drying.

Experiments

Wood samples

Three radiata pine (*Pinus radiata*) trees that were 30 years old and approximately 50 cm diameter breast height (DBH) were felled in Kaingaroa Forest in the Central North Island of New Zealand. Short logs were obtained from below breast height (1.4 m) of each tree, and each log was sawn into four boards symmetrically about the pith, each 60 cm long and 5 cm thick. The average green moisture contents for the heartwood and sapwood were 39% and 130%, respectively.

Drying methods and pre-steaming

The boards were end-coated with paint and dried using one of three drying schedules, with some boards undergoing a pre-steaming treatment before drying (Table 1). The drying schedules were for high-temperature kiln drying 120°/70°C wet/dry bulb for 20 h followed by 4 h of reconditioning at 100°/100°C; for conventional-temperature kiln drying 70°/60°C for 7 days followed by 4 h of reconditioning at 100°/100°C; and for air drying room temperature for 3 months under cover with plenty of air circulation. When pre-steaming was performed, the conditions began with steaming for 1 h 45 min at a temperature of 122°–126°C and pressure of 140–150 kPa. The conditions were then returned to atmospheric pressure for 30 min before applying a vacuum of 20 kPa for another 30 min. It is important to note that the drying and pre-steaming treatments were applied in various combinations to the boards from each tree (Table 1).

Table 1. Combination of drying methods and pre-steaming used to treat radiata pine boards containing mixed heartwood and sapwood

Billet no.	Board 3	Board 4	Board 5	Board 6
1	Air	HT, 120°/70°C	CT, 70°/60°C	St, HT
2	CT, 70°/60°C	HT, 120°/70°C	Air	St, CT
3	CT, 70°/60°C	HT, 120°/70°C	St, HT	St, Air

HT, high-temperature kiln drying; CT, conventional-temperature kiln drying; Air, air drying; St, pre-steaming

Impregnation

From each of the dry boards, end-matched wood samples (4 × 4 × 10 cm long) were prepared from pure heartwood and pure sapwood. The samples were also sawn to provide true flat-sawn and quarter-sawn orientation of the growth rings (i.e., not cross-sawn). The samples were then stored in a controlled environment room at 12% moisture content.

To study the direction of liquid uptake, some of the samples were treated with liquefied silicone in a way that left only selected surfaces unsealed. These treatments included sealing all surfaces except the transverse ends (2L, for the study of liquid uptake primarily through longitudinal flow pathways), sealing all surfaces except the tangential faces (2R, for the study of liquid uptake primarily through radial flow pathways), and sealing of all surfaces except the radial faces (2T, for the study of liquid uptake primarily through tangential flow pathways).

Samples were impregnated using a vacuum treatment method. For heartwood, it involved immersion in liquid for 60 min at 20 kPa and then 40 min at atmospheric pressure. For sapwood, the periods were 30 and 20 min, respectively. In an earlier experiment, no rupturing of pit membranes was found to be caused by these vacuum schedules. The solutions used for impregnation were deaerated, double-distilled water (ddH_2O), 0.5% toluidine blue dye in ddH_2O, 0.01% fluorescein ($C_{20}H_{12}O_5$) in ddH_2O, and a mixture of 0.01% fluorescein and 0.5% toluidine blue dye in ddH_2O. Uptake (grams per cubic centimeter) was calculated by dividing the difference in weight before and after impregnation by the sample volume.

Extractives

The samples that had been impregnated with ddH_2O and whose uptake had already been determined were soaked in ethanol (EtOH) at room temperature. After 2 weeks the samples were dried; the oven-dried weights were recorded, and the EtOH-soluble extractive contents were calculated by weight loss. Earlier experiments indicated that soaking in EtOH for 2 weeks gave equivalent EtOH-soluble extractive content determinations compared to 24 or 48 h of Soxhlet extraction. The former method was adopted because it was deemed to be safer and more convenient.

Observation on flow paths

Flow paths made visible with blue dye were observed using a common light microscope. Fluorescent dyes were observed using a Leica TCN/NT laser scanning confocal microscope.[9] Detailed wood anatomical observations were made by scanning electron microscopy (JOEL 5600 LV).

Results and discussion

Liquid uptake by sapwood

Liquid uptake by sapwood averaged 0.565 g/cm^3 and was not significantly related to pre-steaming of the green wood (Table 2). Also there was little effect of the drying methods on liquid uptake, ranging from 0.558 g/cm^3 for high-temperature drying to 0.579 g/cm^3 for air drying. The moisture content after impregnation averaged 125% ± 4.1% (SE, or standard error), which was almost the same as the original green moisture content for the sapwood (130.0% ± 4.8%).

Pathways of liquid flow in sapwood were consistent with results in an earlier report;[9] that is, the penetration was due predominantly to longitudinal and radial flow. When transverse faces (2L) or tangential faces (2R) were unsealed, 80% of the uptake by unsealed sapwood samples was achieved. On the other hand, uptake from the radial surfaces was only 20% of the uptake achieved when specimens were completely unsealed. This suggests that tangential flow pathways in sapwood are comparative minor. Anatomically, the tissues that were found to take part in conduction (resin canal network, parenchyma network) all extend in the radial and axial directions, so it is not surprising that the conduction in the tangential direction was less. Limited conduction was observed between tracheids.

Liquid uptake by heartwood

Liquid uptake by heartwood samples was variable, with a coefficient of variation of 98% (Table 3). Largely, this was due to the difference in uptake between pre-steamed heartwood (0.211 g/cm^3) and heartwood dried without pre-steaming (0.082 g/cm^3). There was little variation in liquid uptake between billets (representing between tree variation) when pre-steaming was not done (Table 3). However, only two of the three billets (nos. 1 and 2) showed a large increase in uptake between samples dried with and without pre-steaming. Billet 3 had average liquid uptakes of 0.069, 0.077, and 0.069 g/cm^3, respectively, for samples dried using high-temperature conditions, high-temperature conditions with pre-steaming, and air drying with pre-steaming. The lack of response to pre-steaming by samples from billet 3 suggests that the effect of pre-steaming on liquid uptake by heartwood is variable among trees. It may be caused by differences in the original resin content or the frequency of resin canals, because resin canals are a primary flow pathway.

Ratios of uptake by sealed heartwood samples that were high-temperature dried to unsealed samples are shown in Fig. 1. These ratios for samples with and without pre-steaming are comparable to those reported for radiata pine dried with conventional kiln schedules.[9] Uptake by heartwood without pre-steaming was mainly from the transverse (2L) and tangential (2R) surfaces; and compared to

Table 2. Liquid uptake by radiata pine sapwood after vacuum treatment

Billet no.	HT	St, HT	CT	St, CT	Air	St, Air	No St	St	All
1	0.579	0.552	0.580		0.547		0.563	0.552	0.562
	[0.023]	[0.007]	[0.021]		[0.033]		[0.030]	[0.007]	[0.027]
2	0.575		0.583	0.586	0.611		0.595	0.586	0.593
	[0.054]		[0.025]	[0.006]	[0.009]		[0.029]	[0.022]	[0.026]
3	0.520	0.538	0.537			0.545	0.528	0.539	0.533
	[0.012]	[0.008]	[0.007]			[0.013]	[0.013]	[0.013]	[0.013]
Mean	0.558	0.545	0.567	0.586	0.579	0.545	0.569	0.555	0.565
STD	0.040	0.016	0.027	0.006	0.041	0.013	0.036	0.022	0.033
CV (%)	7.2	2.9	4.8	1.1	7.0	2.3	6.4	4.0	5.9

HT, high-temperature kiln drying; CT, conventional-temperature kiln drying; Air, air drying; St, presteaming; STD, standard deviation; CV, coefficient of variation

Results are in grams per cubic centimeter. The numbers in brackets are all the standard deviations

Table 3. Liquid uptake by radiata pine heartwood after vacuum treatment

Billet no.	HT	St, HT	CT	St, CT	Air	St, air	No-St	St	All
1	0.063	0.425	0.110		0.074		0.081	0.425	0.157
	[0.004]	[0.029]	[0.009]		[0.013]		[0.022]	[0.029]	[0.153]
2	0.063		0.081	0.438	0.076		0.073	0.438	0.164
	[0.007]		[0.008]	[0.022]	[0.009]		[0.011]	[0.022]	[0.166]
3	0.069	0.077	0.148			0.069	0.095	0.073	0.082
	[0.009]	[0.018]	[0.012]			[0.004]	[0.026]	[0.013]	[0.030]
Mean	0.066	0.193	0.113	0.438	0.075	0.069	0.082	0.211	0.130
STD	0.007	0.180	0.031	0.022	0.010	0.004	0.026	0.183	0.128
CV (%)	11	94	27	5	13	6	25	87	98

See Table 2 for explanations and definitions

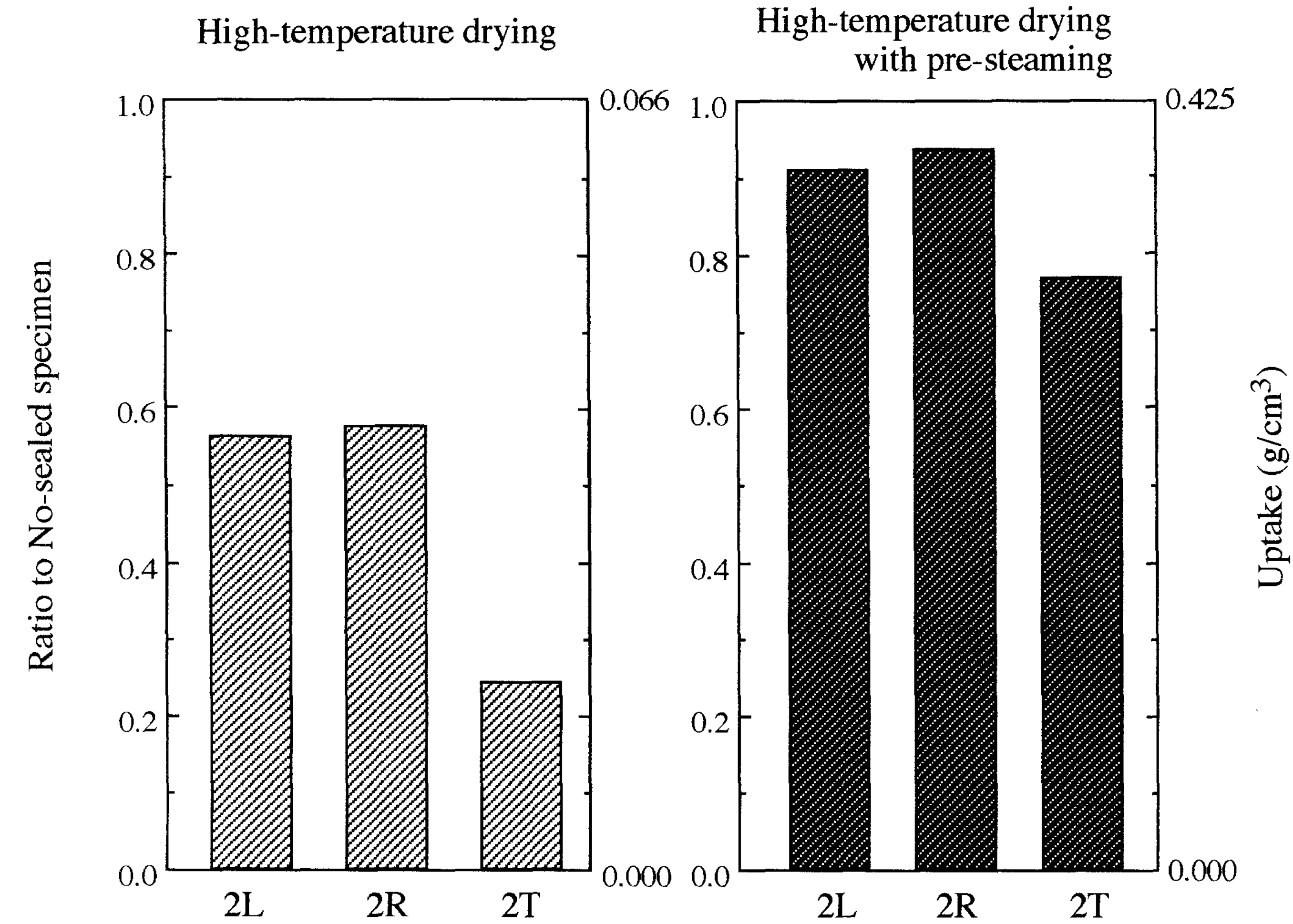

Fig. 1. Uptake of liquid by radiata pine heartwood after selected surfaces were sealed with silicone. *2L*, *2R*, and *2T* refer to the longitudinal, radial, and tangential directions of primary liquid flow, made possible by leaving unsealed only the transverse surfaces (L), tangential surfaces (R), and radial surfaces (T)

samples without sealed surfaces, about 60% of uptake was achieved. Without pre-steaming, the liquid uptake from radial surfaces (2T) was minor (<30%).

Effect of steaming on heartwood

When samples were steamed before drying, 90% of the liquid uptake was achieved from only the transverse surfaces (2L) or the tangential surfaces (2R), compared to unsealed specimens. This ratio for the radial surfaces (2T) was 77% (Fig. 1). Pre-steaming of heartwood therefore resulted in increased penetration of all surfaces, but especially the radial surfaces. Greater conduction through bordered pits has already been shown to be one of the consequences of pre-steaming, and it is believed to increase tangential flow pathways.[9] Other anatomical changes noted in this study were damage to epithelial and ray parenchyma cells in samples that had been pre-steamed (Fig. 2). The resin canals in radiata pine heartwood have thin-walled epithelial cells without secondary wall structure.[10] As a result of the disruption of parenchyma cells by pre-steaming, cavities for liquid conduction were increased.

Removal and redistribution of resin was found to also be associated with the increased liquid uptake of pre-steamed heartwood. In Fig. 3a it is shown that the samples with the lowest ethanol (EtOH)-soluble extractive contents also had the greatest uptake. Samples from billet 3, whose uptake was not increased by pre-steaming, had high extractives in comparison with samples whose uptake was increased by pre-steaming. Anatomical changes, mentioned above, were also not found. These results suggest that when pre-steaming leads to anatomical changes, such as damage to bordered pits, destruction of epithelial and ray parenchyma cells, and the removal of resin, liquid uptake then increases markedly.

A significant correlation between uptake by heartwood without pre-steaming and EtOH-soluble extractive content is shown in Fig. 3b ($r = -0.68$). However, we were not able to detect a clear relation between liquid uptake and severity of drying. In contrast, Booker and Evans[11] found that the radial permeability of radiata pine boards could be increased by increasing the rate of drying. It is also recognized by industry that high-temperature drying improves heartwood treatability. We conclude that without significant change of wood structure, as occurs with pre-

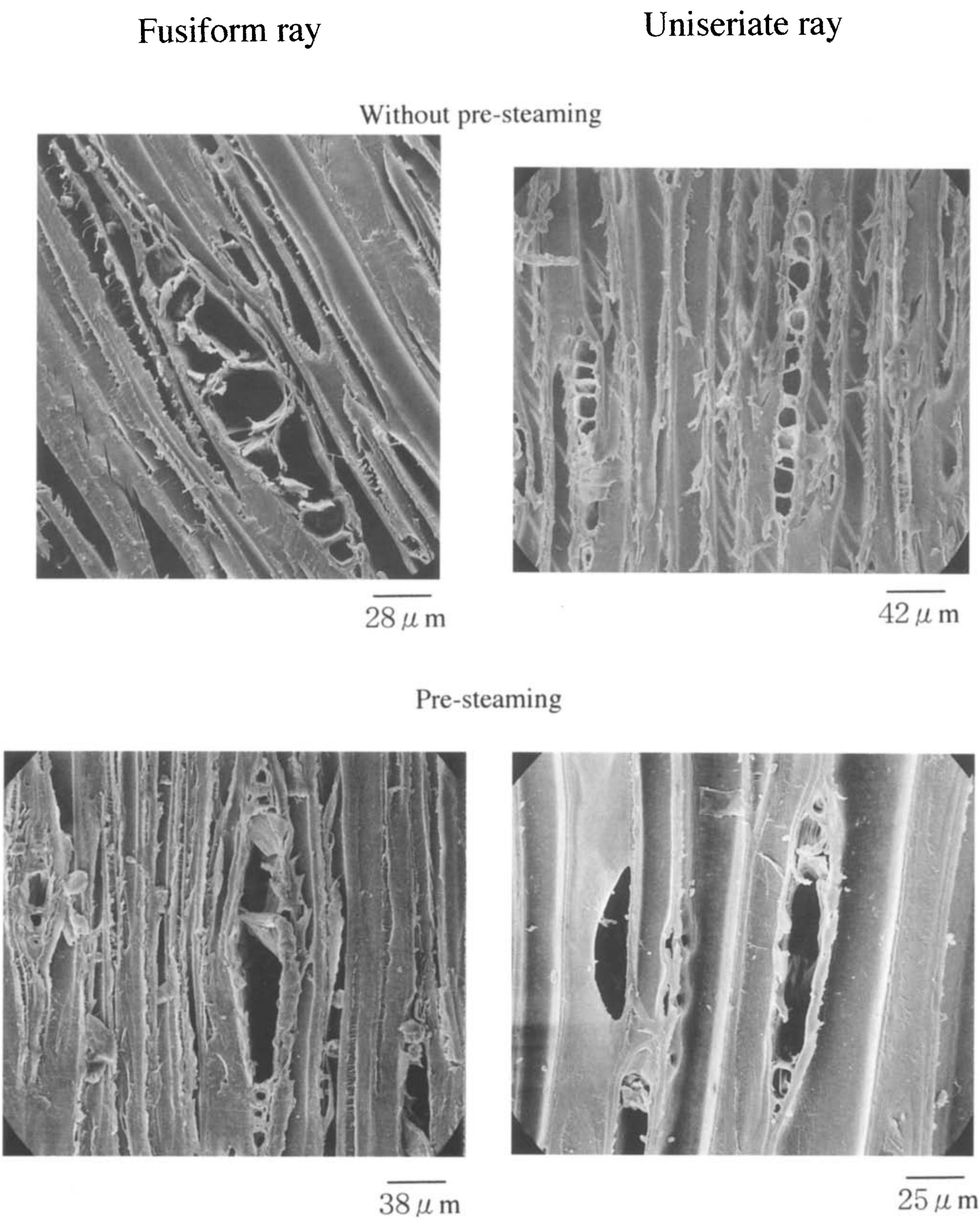

Fig. 2. Damage to fusiform and uniseriate rays was noticed in samples that had been pre-steamed. This was not the case for samples without pre-steaming

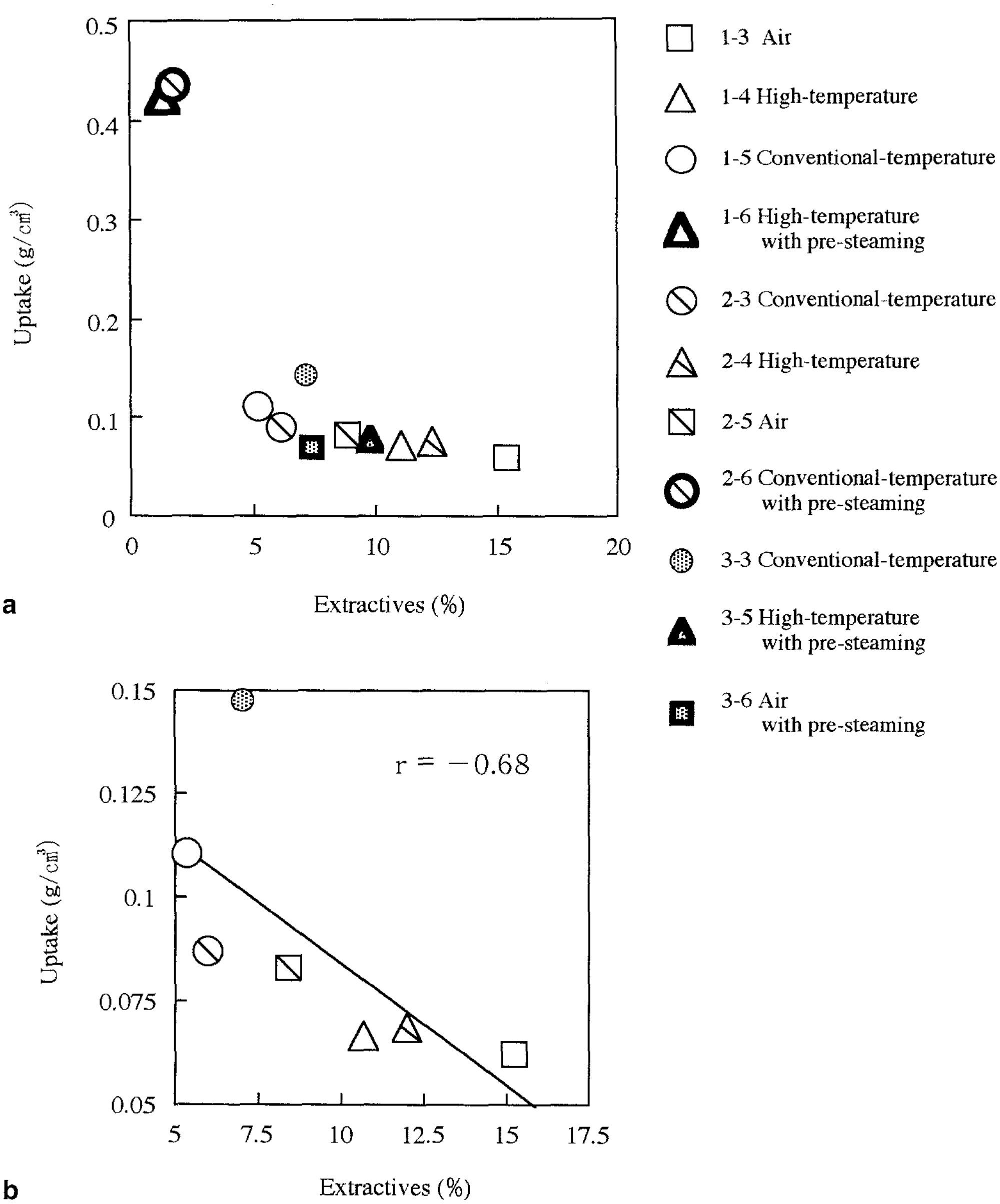

Fig. 3. Relation between liquid uptake by radiata pine heartwood and the extractive concentration. **a** Heartwood samples from boards dried by high-temperature, conventional-temperature, and air drying, with or without pre-steaming. **b** Heartwood samples that were not pre-steamed

steaming, the resin remaining in heartwood is a factor determining liquid uptake.

Flow path and liquid uptake in heartwood

A summary of liquid uptake in radiata pine heartwood in relation to drying method and pre-steaming is shown in Fig. 4. This diagram shows five classifications for liquid uptake that can be related to operating flow pathways in the wood and various wood anatomical features.

Class 1: having a liquid uptake less than 0.065 g/cm³ and no penetration into wood cells except at the surfaces.

Class 2: having liquid uptake of 0.065–0.080 g/cm³. Dye can be seen inside resin canals, but the flow is interrupted by resin.

Class 3: having liquid uptake of 0.075–0.100 g/cm³. Dye can be seen at the intersections of axial and radial resin canals indicating that the resin canal network is acting effectively in conduction. Ray parenchyma are also conductive (i.e., uniseriate and fusiform rays are conductive), but ray tracheids are generally not conductive.

Class 4: having liquid uptake of more than 0.100 g/cm³. Most resin canals and rays are conductive, and there is some liquid movement between adjacent tracheids.

Class 5: having liquid uptake of more than 0.400 g/cm³. Flow pathways are similar to those in sapwood. This level of liquid uptake is a characteristic of pre-steamed heartwood, where there has been resin removal and significant damage to bordered pits and epithelial and ray parenchymal cells.

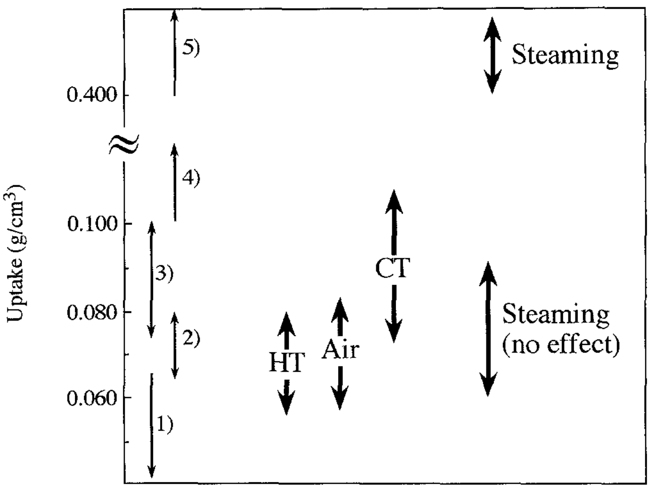

Fig. 4. Flow paths in radiata pine heartwood classified five ways according to pre-treatment. Anatomical characteristics are described for each class in the text. *HT*, high-temperature kiln drying; *CT*, conventional-temperature kiln drying; *Air*, air drying

Conclusions

Radiata pine boards, containing heartwood and sapwood, were dried using a high-temperature kiln schedule, a conventional-temperature kiln schedule, and air drying. Pre-steaming was selectively applied to some boards. Wood samples sawn from these boards were then impregnated with water (containing various dyes) using vacuum pressure to study the relations between liquid uptake, flow pathways, and wood anatomical features. The following conclusions were reached for radiata pine sapwood.

1. There was little difference in liquid uptake between high-temperature, conventional-temperature, and air dried samples and between samples treated with and without presteaming.
2. The predominant flow pathways were longitudinal and radial. More than 80% of the uptake by unsealed samples was achieved when only transverse or tangential surfaces were not sealed with liquefied silicone.
3. Resin canals and rays were found to be effective liquid conduits, providing little apparent resistance to liquid flow.

For radiata pine heartwood, we concluded that:

1. Uptake was usually strongly influenced by pre-steaming.
2. Penetration was predominantly by longitudinal and radial flow pathways. Pre-steaming increased liquid uptake from all surfaces, but especially from the radial surfaces (tangential flow pathway), from less than 30% to almost 80% of the uptake by unsealed samples.
3. Samples having increased uptake after pre-steaming were also found to have lower extractive contents, disruption of epithelial and ray parenchyma cells, and an increase in conduction through the bordered pits of adjacent tracheids.
4. The liquid uptake levels were classified five ways according to the drying treatments, flow pathway, and wood anatomical features.

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