EFFECT OF EXTRACTIVES ON MOISTURE SORPTION AND SHRINKAGE IN TROPICAL WOODS

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ABSTRACT

Samples of sixteen tropical wood species from Indonesia were selected to undergo desorption and adsorption in the unextracted and extracted form. The anisotropic shrinkage values of these samples were also determined. At high humidities, the extracted woods exhibit higher equilibrium moisture contents than the unextracted woods. However, the isotherms of extracted and unextracted woods coincide at relative humidities below 70% for both desorption and adsorption. This phenomenon indicates that the hygroscopicity of wood is affected at high humidities through the extractives bulking the amorphous region in the cell wall. The shrinkage intersection point ranges from 18.0 to 34.1%, with an average of 24.8%, which is below the predicted 30% for temperate-zone woods. The linear relationship between volumetric shrinkage and specific gravity was significant, but the correlation (r^2 = 0.40) is quite low. The removal of extractives with hot water and organic solvents caused excessive shrinkage. All the woods tested showed partial collapse, indicating the plasticization of the extractives at high temperature and high moisture content in the cell wall structure.

Keywords: Extractives, desorption, adsorption, shrinkage, moisture, tropical woods, fiber saturation point.

INTRODUCTION

Freshly cut green wood often contains a large amount of water in the cell lumen (as free water) and in the cell wall (as bound water). The natural transitional point between free and bound water, known as the fiber saturation point (FSP), is defined as the theoretical condition of wood when its cell walls are saturated with water, but no free water remains in the cell lumens. The moisture content of the wood at this point is assumed to be 30% of the dry weight of the wood, and is considered to be a constant value regardless of species. Skaar (1988) stated that the FSP is in the order of 25–30% of the dry weight of most temperature-zone woods but may be considerably lower for woods with high extractives content, such as in many tropical woods. The moisture content range below the FSP is known as the hygroscopic range, and it is at this range that the phenomena of sorption and dimensional change take place in wood. As a result, removal of this bound water often causes distortions and checks in the wood product.

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The mechanisms of sorption of water from the vapor phase by cellulosic materials have been described in the literature (Skaar 1988). The currently held concept of sorption in wood postulates the adsorption of water by three mechanisms: as monolayer water molecules in hydrate form at polar sites in the noncrystalline regions, which predominates at low relative humidities; as polymolecular water held in solid solution on the surface of cellulose crystallites at intermediate relative humidities; and as condensed water vapor in the void spaces of the cell wall, which is thought to occur at high relative humidities.

Most studies on the sorption characteristics of wood have been with temperate-zone species. These show little variation (Stamm 1964; Okoh and Skaar 1980). The few studies on tropical woods (Nearn 1955; Spalt 1957; Wangaard and Granados 1967), however, indicate some variation in the sorption properties with species, which was considered to be due to the presence of high extractives content. Spalt (1958) applied the Hailwood-Horrobin theoretical model to his sorption data for woods of high and low extractive content, and reported that the presence of extractives has little effect on sorbed monolayers but has an appreciable effect on the polylayers. Other hygroscopic properties of wood, such as fiber saturation point and shrinkage, are also affected by extractives (Choong 1969; Taylor 1974; Cooper 1974; Demaree and Erickson 1976) and by chemical composition and crystallinity of cellulose (Christensen and Kelsey 1958).

From the practical standpoint, woods of high extractive content exhibit lower equilibrium moisture content and shrinkage at room temperature but high shrinkage (and collapse) at high temperatures in kiln drying (Spalt 1979). Some tropical woods have high extractives content, but little is known how these extractives affect the hygroscopicity of wood. Therefore, the objectives of this study were (1) to evaluate the sorption and shrinkage behaviors of some tropical woods from Indonesia, and (2) to determine the effect of removing extractives on the hygroscopicity of these woods.

MATERIALS AND METHODS

Sixteen species were chosen for this study. These are as follows:

Native Name	Botanical Name
Pasang koyang	*Quercus spicata* Sm.
Keruing daun lebar	*Dipterocarpus cornutus* Dyer
Lempung ranggas	*Shorea acuminatissima* Sym.
Nyatoh II	*Palaquium ferox* H. J. Lam.
Bangkirai	*Shorea laevis* Ridl.
Merembung	*Shorea smithiana* Sym.
Kenaur	*Shorea leptoclados* Sym.
Medang lalan	*Alseodaphne umbelliflora* Hook. f.
Kiputri	*Podocarpus neriifolius* D. Don
Surian	*Toona sareni* Merr.
Banio	*Shorea platyclados* v. Slooten ex. Foxw.
Bintagus	*Calophyllum* sp.
Agathis	*Agathis labillardieri* Warb.
Palaquium	*Palaquium* sp.

Mangga hutan	*Mangifera foetida* Lour.
Jati	*Tectona grandis* L. F.

For each species, eight end-matched samples were cut to approximately 2 cm (tangential) × 5 cm (radial) in cross section from air-dried stock. Four of the samples were ½-cm in thickness and were used for sorption study; the other four were 1-cm thickness and were used for shrinkage study. Samples were prepared with true radial and tangential surfaces. For each of the two studies, the samples were divided into two groups, each consisting of two replications: Group A—untreated (i.e., unextracted condition), Group B—treated (i.e., extracted condition). Samples in Group B were steamed for 2 h inside a pressure cooker, then subjected to 6 h of hot-water extraction in a Soxhlet apparatus, and finally subjected to four cycles of 6-h toluene-ethanol extraction. Complete extraction could not be assumed by this method, especially since some species retained color, even under prolonged treatment. However, it was felt that the treatment was sufficient to satisfy the purposes of this study.

All the sorption samples were saturated between damp cloths before the start of testing and then placed in an Aminco Climate-Lab (American Instrument Co., Inc.) at 30 C where they were conditioned to successively lower equilibrium moisture contents (EMC) at nominal 92, 72, 44, 30, and 18% relative humidity (RH). A porthole in the chamber door allowed samples to be taken out and measured with minimum disturbance of the controlled environment. Samples were then oven-dried at 105 C for 24 h and then reconditioned to equilibrium at nominal 8, 34, 55, 84, and 94% RH. At each condition, the samples were weighed on an analytical balance to the nearest 0.001 g. Relative humidity was measured with a Casella mechanical psychrometer to ±0.5%. Moisture content (M or MC) was expressed on an oven-dry weight basis.

The shrinkage samples were also saturated in water and then underwent a desorption process by conditioning successively to EMC at nominal 77, 65, 46, 28, and 27% RH inside the same Aminco chamber at 30 C. At each condition, their radial and tangential dimensions were measured with a micrometer to the nearest 0.01 cm. Shrinkage values were based on swollen conditions. The specific gravity was determined by the maximum moisture content method (Smith 1955), which involved alternating vacuum and atmospheric pressure treatments in distilled water until no more air bubbles could be detected.

RESULTS AND DISCUSSION

Sorption

The sorption data were plotted for each species. The graphs (Fig. 1) illustrate the adsorption and desorption curves and the characteristic hysteresis loop in both the unextracted and extracted conditions. Since the desorption isotherm curve is invariably higher than the adsorption curve, the fiber saturation estimates (when the curve is extrapolated to 100% RH) are similarly affected. According to Urquhart and Eckersall (1930) for cotton, this difference is due to the availability of sorption sites and their relative potential energy levels for water attraction. Numerous other theories have been proposed for explaining the sorption hysteresis in wood (Skaar 1988). In these graphs, three species (merembung, medang lalan,

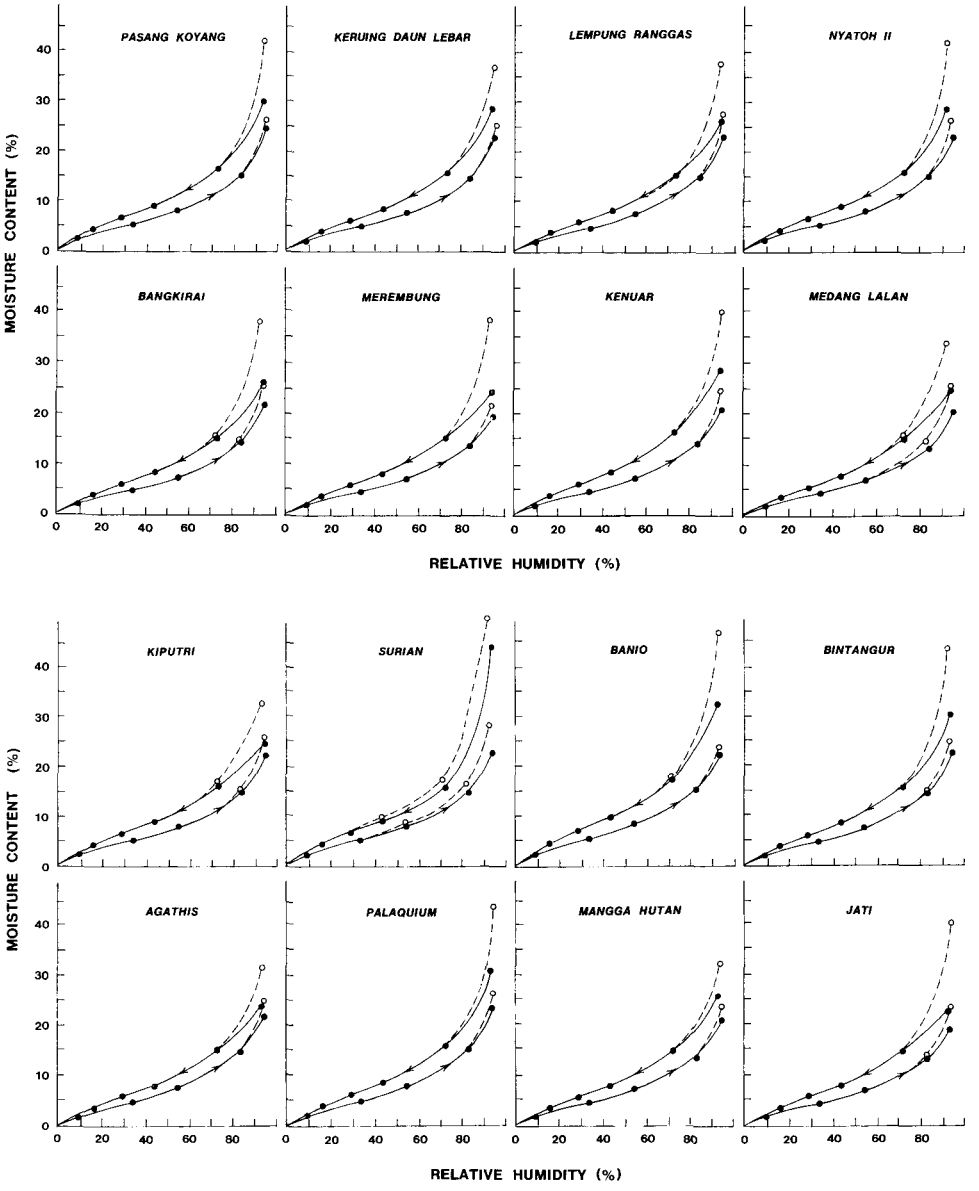

FIG. 1. Description and adsorption isotherms at 30 C. (Solid line is unextracted; broken line is extracted.)

and jati) exhibit low EMCs at high humidity range and four species (surian, banio, bintangur, palaquium) show high EMCs at high humidity range during desorption. These figures also show that when the desorption and adsorption curves of the unextracted samples are extrapolated to 100% relative humidity, they converge at around 30% MC, which is considered the FSP of wood. The values of the apparent FSP for the extracted samples are always higher than the unextracted samples, and in desorption some woods exhibit values which are over 40 percent.

TABLE 1. *Adsorption-desorption (A/D) ratio and volumetric shrinkage (S_v) in unextracted and extracted samples.*

Species	A/D ratio				Volumetric shrinkage			Extractives removed	Unextracted apparent FSP (S_v/G)
	Unextracted		Extracted						
	72% RH	44% RH	72% RH	44% RH	Unextracted (%)	Extracted (%)	Increase (%)		
Pasang koyang	0.75	0.73	0.74	0.73	19.9	31.3	57	12	25.4
Keruing daun lebar	0.77	0.74	0.79	0.75	21.0	25.9	23	10	30.5
Lempung ranggas	0.84	0.73	0.86	0.76	13.9	53.5	284	13	37.0
Nyatoh II	0.82	0.73	0.82	0.75	10.3	23.8	131	14	22.4
Bangkirai	0.82	0.71	0.80	0.74	10.7	30.5	185	9	32.2
Merembung	0.75	0.72	0.79	0.73	15.1	19.3	28	26	20.4
Kenuar	0.76	0.70	0.78	0.70	11.9	28.1	136	12	22.8
Medang lalan	0.73	0.71	0.79	0.75	11.1	22.5	103	3	29.7
Kiputri	0.80	0.73	0.78	0.74	11.4	11.8	4	7	21.6
Surian	0.80	0.72	0.79	0.73	17.9	54.9	207	2	37.0
Banio	0.75	0.70	0.71	0.70	14.5	20.9	44	2	23.9
Bintangur	0.76	0.73	0.77	0.73	15.4	27.6	79	29	26.2
Agathis	0.84	0.76	0.82	0.73	8.5	10.9	28	33	22.3
Palaquium	0.84	0.73	0.80	0.74	14.4	32.6	126	12	24.9
Mangga hutan	0.78	0.75	0.79	0.74	6.7	10.5	57	13	13.1
Jati	0.82	0.71	0.79	0.73	8.8	16.7	90	20	16.2
	0.79 ± 0.04	0.73 ± 0.02	0.73 ± 0.02	0.73 ± 0.02					

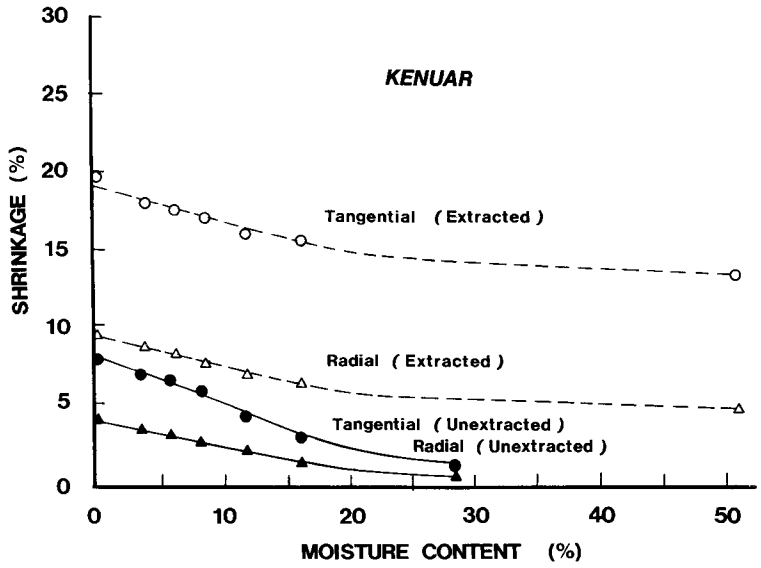

FIG. 2. Relationship of shrinkage and moisture content for kenuar.

For both desorption and adsorption, the removal of the extractives only affects the sorption characteristic at high relative humidity. The analysis of variance indicates that at 95% RH the EMCs are significantly ($P < 0.01$) different for unextracted and extraction samples. At high humidity range (Figs. 1A, B), the curves are higher for the extracted than for the unextracted samples in every species tested; but they coincide below 70%. Thus, the effect of extractives is to depress the isotherms in varying degrees depending on the individual species, but only at the high humidity range. Wangaard and Granados (1967) obtained the same result with tropical woods from Central America. They reported that only the water sorbed in polylayers, and not the monolayer, is changed with the removal of extractive material and the increase in sorbed water was greatest at relative humidities near fiber saturation. The higher EMC at high humidities and higher corresponding FSPs in the extracted wood, as compared with the unextracted wood, indicate that the nonvolatile extractives not only serve as "bulking" agent in preventing dimensional change, but also act as adsorbate in partial swollen state in the void spaces of the cell wall.

Table 1 gives the mean values of the hysteresis (A/D) ratios for unextracted and extracted samples. They are, respectively, 0.79 ± 0.04 and 0.79 ± 0.03 at 72% RH and 0.72 ± 0.02 and 0.73 ± 0.02 at 44% RH. A simple "t" test of the individual species values in both unextracted and extracted samples indicates no significant difference between these means; however, the A/D values are significantly higher at 72% RH than at 44% RH. Okoh (1976) reported the same trend, with minimum values of A/D occurring at the mid-humidity range. He gave the mean values of the A/D ratios as 0.808 ± 0.010 for the U.S. woods and 0.786 ± 0.017 for the Ghana woods over the humidity range from 35 to 85%. Spalt (1958) reported a mean A/D ratio of 0.824 ± 0.031 for eight hardwoods, seven

of which were of tropical origin. Skaar (1972) stated mean A/D ratios for 39 tropical hardwoods from the Venezuelan Guyana as being 0.803 ± 0.032 and 0.805 ± 0.028 for sapwood and heartwood at 76% RH, respectively.

Shrinkage

The radial and tangential shrinkage data were plotted over equilibrium moisture content for each species. A typical graph illustrates that the points representing averages of two samples lay almost precisely on a straight line, except for those near the horizontal and vertical axes (Fig. 2). Such deviations have been shown by Kelsey (1956) to be associated with moisture gradient and/or drying stress. These deviations were discarded in fitting the straight lines, and the five intermediate shrinkage points were used in deriving the regression equations listed in Table 2. The shrinkage intersection point (ISP), as an indicator of the FSP, was determined by extending the linear portion of the shrinkage-moisture content curve to intersect with the horizontal axis at zero shrinkage.

Tangential shrinkage invariably gave a higher intersection point than radial shrinkage, because the ratios of tangential to radial shrinkage decreased with decreasing moisture content. Hence, either the average of the two intersection points or that obtained from volumetric shrinkage (the summation of radial and tangential shrinkages) should offer a better estimate than one or the other alone. For the untreated group, the average point of intersection ranges from 18.0% for jati to 34% for surian. The average value is only 24.8%, which is below the predicted 30% for FSP reported for North American woods (Stamm 1964; Choong and Manwiller 1976). Spalt (1958), who studied sixteen North American and tropical woods, reported that their FSPs ranged from 19.8 to 30.5% in adsorption and 21.8 to 33.6% in desorption. The wide spread in values was attributed to great differences in extractive contents. For selected Indonesian woods, the ISP obtained from the volumetric shrinkage and moisture content relationship varies from 17.0% to 35.9% (Tambunan et al. 1975). A definite relationship between volumetric shrinkage and specific gravity was also reported, although the correlation coefficient was rather low. In our study, the relationship was found to be significant ($P < 0.01$); but, the correlation ($r^2 = 0.40$) was also rather low, indicating that only 40% of the variation in volumetric shrinkage was accounted by specific gravity; nevertheless, their ratio could be related to FSP as postulated by Stamm (1964), and it is shown in Table 1 for the unextracted samples.

The removal of extractives with hot water and organic solvents increases the correlation relationship between volumetric shrinkage and specific gravity in southern pine (Choong 1969) and in baldcypress and tupelo-gum (Choong et al. 1989). This has not been the case with tropical woods. Extractives, to the extent that they may be concentrated in the fine structure of the cell wall normally occupied by water, generally reduce shrinkage (Nearn 1955; Choong 1969). On the other hand, the removal of extractives material makes available additional moisture sorption sites and therefore causes an increase in shrinkage. When the shrinkage is excessive, collapse takes place. In this case, shrinkage was found to be highly negatively correlated with specific gravity, and thus the FSP obtained by this method is inapplicable.

TABLE 2. *Shrinkage equation and intersection point (ISP) for unextracted samples.*

Species	Shrinkage equation[1]	ISP	G	MX
Pasang koyang	$S_R = 7.26 - 0.26(M)$	28.1	0.79	0.85
	$S_T = 12.71 - 0.41(M)$	30.9		
Keruing daun lebar	$S_R = 7.49 - 0.29(M)$	25.7	0.69	1.11
	$S_T = 13.48 - 0.47(M)$	28.5		
Lempung ranggas	$S_R = 4.15 - 0.21(M)$	20.2	0.38	1.54
	$S_T = 9.74 - 0.37(M)$	26.1		
Nyatoh II	$S_R = 4.37 - 0.20(M)$	22.1	0.46	0.96
	$S_T = 5.90 - 0.24(M)$	24.1		
Bangkirai	$S_R = 3.59 - 0.16(M)$	22.4	0.33	1.25
	$S_T = 7.13 - 0.26(M)$	27.8		
Merembung	$S_R = 4.98 - 0.26(M)$	18.8	0.74	0.86
	$S_T = 10.11 - 0.38(M)$	27.0		
Kenuar	$S_R = 3.79 - 0.17(M)$	22.3	0.52	0.96
	$S_T = 8.08 - 0.33(M)$	24.4		
Medang lalan	$S_R = 3.36 - 0.16(M)$	20.5	0.37	1.34
	$S_T = 7.76 - 0.34(M)$	23.0		
Kiputri	$S_R = 4.51 - 0.20(M)$	23.1	0.53	0.85
	$S_T = 6.86 - 0.26(M)$	26.9		
Surian	$S_R = 6.42 - 0.21(M)$	30.5	0.48	1.06
	$S_T = 11.50 - 0.30(M)$	37.8		
Banio	$S_R = 5.12 - 0.21(M)$	24.9	0.61	0.87
	$S_T = 9.38 - 0.32(M)$	29.1		
Bintangur	$S_R = 5.76 - 0.24(M)$	23.8	0.59	0.98
	$S_T = 9.67 - 0.33(M)$	29.1		
Agathis	$S_R = 3.63 - 0.16(M)$	22.7	0.38	1.17
	$S_T = 4.87 - 0.29(M)$	31.1		
Palaquium	$S_R = 5.46 - 0.24(M)$	22.8	0.58	0.70
	$S_T = 8.92 - 0.16(M)$	31.1		
Mangga hutan	$S_R = 2.66 - 0.13(M)$	20.6	0.51	0.57
	$S_T = 4.02 - 0.16(M)$	24.4		
Jati	$S_R = 3.17 - 0.18(M)$	17.2	0.54	0.89
	$S_T = 5.68 - 0.30(M)$	18.9		

[1] The average r^2 value is 0.995. S_R, radial shrinkage; S_T, tangential shrinkage; M, moisture content; G, specific gravity (green volume basis); MX, moisture expansion coefficient.

Although the shrinkage of the extracted samples cannot be extrapolated to zero shrinkage (Fig. 2), the slope dS/dM of the unextracted and extracted samples can be compared using the five data points that correspond to the five EMCs in the graph. For a given species, the two terms were added together in each of the shrinkage equations in Table 2, for radial and tangential shrinkages, to obtain an estimate of dS/dM, the percent change in volume (based on water-soaked volume) over the percent change in moisture content. The moisture expansion coefficient, MX, is simply the ratio of dS/dM and G, where G is the green-volume specific gravity. These values (Table 2) ranged from 0.57 for mangga hutan to 1.54 for lempung ranggas, with a mean of 0.998 ± 0.243 for the unextracted samples, and 1.09 ± 0.26 for the extracted samples. The theoretical value, based on the assumption of zero change in the cell wall during shrinkage, is unity; but Skaar (1988) reported considerable variable in values, ranging from 0.6 to 1.8, among

several kinds of woods. In those for which MX is greater than 1.0, the cell cavity apparently expanded somewhat during water sorption; whereas, those with MX less than 1.0 there was some contraction in the cell cavity.

After removal of the extractives, both radial and tangential shrinkages, and hence the volumetric shrinkage, increased dramatically in some species (Table 1 and Fig. 3). In lempung ranggas and surian, the increase was more than 200%. The amount of extractives removed varied from 2 to 33%. With the increase in shrinkage, the ISP also increased to such an extent that it no longer indicates the apparent FSP (i.e., 30% MC). The resultant effect is an abnormally excessive volumetric shrinkage (collapse). In the species surian, the collapse is more evident. Kanagawa and Hattori (1984) also observed cell collapse phenomena in almost all of the 26 Indonesian woods they studied, after drying from the green condition at temperature of 60 C. In this study, even though shrinkage measurements were made after the wood had dried below the fiber saturation point, it is conceivable that the treatment of the extracted samples (i.e., steaming and soaking the wood at high temperature) caused the extractives to become more adsorbent. Spalt (1979) indicated that this treatment causes the extractives in wood to participate in sorption and to increase the overall shrinkage. There is evidence in the literature (Narayanamurti 1957) that some extractives act as plasticizers at high temperatures, which may soften the cell wall. Collapse occurs because of the degrading effect of high temperature and the large capillary pressure build up at high moisture content, especially if the cell wall is weak. However, if the liquid surface tension can be eliminated during drying (e.g., by sublimation), collapse in refractory woods may be completely eliminated (Choong et al. 1973). In work with tannin-rich redwood heartwood, Demaree and Erickson (1976) postulated that at low drying temperatures the extractives act as bulking agents, but at high temperatures the extractives become heat sensitive and make the wood cells susceptible to collapse during apparent free water loss. Alternatively, the unextracted samples show no evidence of collapse because the bulking action of the extractives in the cell wall and the occlusion of the extractives in the lumen may actually prevent or minimize collapse. According to Chafe (1987), abnormal volumetric shrinkage constituting collapse in Eucalyptus and other species is positively related to the encrusting and extraneous materials and negatively to the polysaccharide cell-wall component.

Extractives *in situ* are indeed complex material. Those found in the cell wall of heartwood are the polyphenols (e.g., flavanoids, lignans, tannins). Some have low molecular weights, and at high temperature they may be in solution with the adsorbed water and move in response to moisture content gradients during drying; therefore, they contribute to the hygroscopicity of wood. Others may accumulate in the lumens and/or occlude the pits, and may also obstruct the movement of liquid in wood. Several treatments related to the removal of extractives result in improvement in the dryability of wood. Steaming has been reported to decrease drying time (Ellwood and Erickson 1962; Kinninmonth 1973) and increase drying rate (Simpson 1975). Steaming also increases the permeability of several North American softwoods (Comstock 1965; Fogg 1968) and North American hardwoods (Ellwood and Ecklund 1961; Chen 1975), and tropical woods (Choong and Achmadi 1989). However, little information is available in the literature on how extractives contribute to improved drying. Both Comstock (1965) and Fogg (1968) reported that extraction by hot water and organic solvents tends to have the same

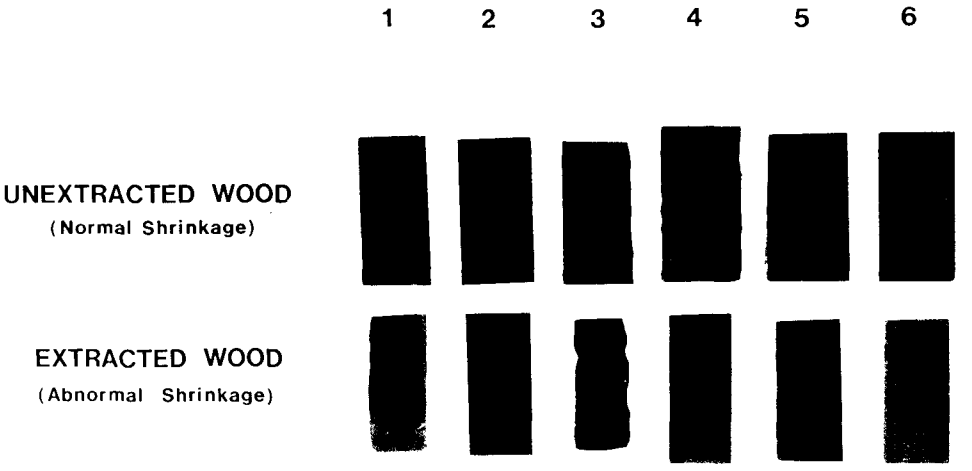

FIG. 3. Selected wood samples, showing differences between unextracted shrinkage and extracted shrinkage. (1—lempung ranggas, 2—nyatoh II, 3—surian, 4—bintangur, 5—palaquium, 6—jati.)

effects on permeability as steaming, at least in softwoods. However, we do not know whether these effects are due to removal and/or relocation of the extractives or to actual changes in the cell-wall structure itself. Also, we do not know the contribution and effect of the hemicelluloses in the removal of extractives. Structural changes as a result of steaming have been shown by Nicholas and Thomas (1968) in a softwood to be due to greater flexibility of the margo fibrils, accompanied by de-aspiration of the bordered pits. Steaming also increases the enzyme pectinase, causing degradation to the torus and to the pit membrane (Nicholas and Thomas 1967). In hardwoods, Kinninmonth (1971) observed that the materials lining cell lumina and pit areas became distorted by steaming. Kubinsky (1971) also noted disruption of the warty layer that lines the lumen walls of red oak after steaming. Moreover, at elevated temperatures, he reported that steaming increases the enzymatic susceptibility of the polysaccharides so that the hemicellulose is hydrolized and becomes soluble in hot water. Further research is needed to understand the contribution of extractives to changes in wood properties, especially during the drying process at high temperature when the wood is still wet.

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