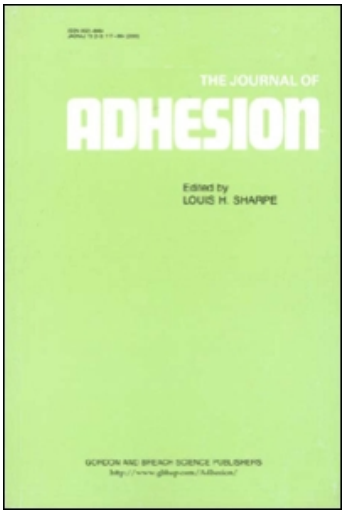

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Influence of Extractives on Bonding Properties† of White and Southern Red Oak

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White and southern red oak veneers were subjected to four methods of drying followed by five surface treatments. The four drying methods were mill drying at 350°F, laboratory drying at 350 and 212°F, and air drying. The five surface treatments were no treatment, surface scraping, soaking and dipping in 1% NaOH aqueous solution, and water extraction. Plywood panels were prepared by using a phenol-formaldehyde resin.

Even with the best drying-surface treatment combination, wood failure was only 35% for white oak and 39% for southern red oak. Overall, mill drying was the best drying method. Soaking the veneers in 1% NaOH solution significantly increased the bond quality.

Mill drying of veneers caused water-soluble extractives to migrate from the interior portions to veneer and lathe check surfaces. SEM examinations of the glue failure surface revealed that gluelines failed to adhere to the cell walls. Difficulties in bonding white and southern red oak veneers may be caused by extractive contamination.

INTRODUCTION

White and southern red oak constitute about 20.4% of the total hard-

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wood volume grown on southern pine sites.[1] These resources have not been utilized to their full potential because of their small size and low quality. Furthermore, technical difficulties in converting this material to useful products have inhibited their use by industry. In an effort to utilize southern hardwood resources, Hse[2] found that satisfactory structural exterior flakeboards could be produced from species with low density but not from species having wood density above 0.6. Although a process for manufacturing satisfactory hardwood structural flakeboards has been developed with careful control of species mix,[3] it is evident that durability of panels must be improved if a significant amount of high-density species, especially white and southern red oak, is to be used in fabricating structural panels. Craft[4] also reported that oak species grown in the Appalachian region were very difficult to glue as indicated by low wood failure values of plywood panels constructed from these oak veneers.

Plomley et al.[5] reported that contamination of the bonding surface with hydrolyzable tannins significantly reduced the bond quality of a phenol-formaldehyde resin adhesive. A light planing of the bonding surface removes surface contaminants and simultaneously exposes the highly polar secondary cell walls to which adhesives bond most efficiently.[6,7] A surface treatment with sodium hydroxide solution or neutral solvents to remove some surface contaminants has also been proven to improve bond quality.[8,9] The deleterious effect of low pH caused by extractives and other contaminants may also be eliminated by applying a proper amount of sodium hydroxide to the wood surface.[10,11]

The objectives of this study were: (1) to investigate the effect of drying on extractive migration in plywood veneers, (2) to study the effect of veneer drying methods on bond quality, (3) to improve the bond quality of white and southern red oak veneers by various surface treatments, and (4) to examine microscopic characteristics of gluelines.

MATERIALS AND METHODS

One white oak (*Q. alba* L.) and one southern red oak (*Q. falcata* Michx. var. falcata) tree approximately 15 inches in diameter at breast height were harvested and rotary-peeled at a plywood mill to produce 1/8-inch thick veneer.

Veneer Drying A portion of the veneer was mill dried at 350°F to an

average moisture content of 4%. The remaining green veneer was separated into three equal portions, which were oven dried at 350 and at 212°F and air-dried. The oven drying was done for approximately 3 hours to an average mositure content of 2%. All veneers were then cut into 12-inch by 12-inch pieces.

Surface Treatments The veneers within each drying group were subjected to 5 surface treatments: no treatment, surface scraping with a hand-held, disposable microtome knife, soaking in 1% NaOH aqueous solution for 5 minutes, dipping in 1% NaOH aqueous solution, and extraction with warm tap water in a sink for 5 days. Material scraped from the veneer surfaces was collected for extractive content determinations. Weight gain of each veneer was recorded after soaking and dipping in the 1% NaOH solution to determine the amount of NaOH absorption. The 1% NaOH solution remaining in the container after each soaking and dipping treatment was sampled for further analysis.

Extractive Content Determination To examine extractive migration, veneers were randomly sampled after drying. These veneers were cut into strips measuring 4 inches across the grain and 12 inches along the grain. These veneer strips were then planed, with the help of a push block, with a 6-inch jointer from either the tight or loose side. Thickness reduction was recorded, and the shavings were collected after each pass over the jointer until the midpoint of the veneer thickness was reached. Jointer shavings and materials collected during the surface scraping operation were milled to pass a 40-mesh screen. The milled samples were then extracted with hot water followed by a 95% alcohol extraction each for a period of 6 hours in a Soxhlet extractor.

pH Determination To study the effect of surface treatment on veneer surface pH, veneer surface shavings were obtained and milled in the same way as those for the extraction study. Wood meal was diluted with degased, distilled water in a ratio of 1 : 10 based on oven-dry weight of wood. The pH values were determined at least 2 hours after the addition of distilled water.

Plywood Fabrication and Testing All veneers were dried briefly in an oven at a low temperature (170°F) to obtain an average moisture content of 4% just before plywood fabrication. A phenol-formaldehyde resin was mixed with Furafil and wheat flour to achieve 27% resin solids and

42.5 total solids in the final mix. The glue was spread at 85 lbs/1000 ft^2 of double glueline. After a 10-minute closed assembly time, panels were pressed for 6 minutes at a temperature of 285°F and at a specific pressure of 200 psi. No post curing was performed. Three panels of each drying-surface treatment combination were prepared, thus a total of 60 panels for each species were made. Nine to twelve standard shear specimens were cut from each panel. Wet shear strength and wood failure were evaluated according to the vacuum-pressure method prescribed by PS-1-74.[12]

Electron Microscopy To examine extractive depositions near the veneer surfaces, the aged veneer surface was shallowly removed with a razor blade to eliminate possible contaminants other than extractives. The wood surface in the interior portions of veneer was also prepared by using a razor blade. Lathe check surfaces were obtained by breaking the sample along lathe checks. A direct carbon replica technique[13] was used to study microscopic characteristics of specimen surfaces. Replicas were examined with a Hitachi HU-11E-1 transmission electron microscope. Morphological characteristics of veneer surfaces and gluelines were examined with a JOEL-JSM-35 scanning electron microscope.

RESULTS AND DISCUSSION

Effect of Drying on Extractive Migration

Figures 1 and 2 illustrate the extractive distribution patterns of white and southern red oak veneers after drying. Migration of extractives is clearly shown in the mill-dried white oak veneers where both the tight and loose side surfaces accumulated higher concentrations of extractives than the interior portion (Fig. 1A). Furthermore, the loose side of the veneer accumulated a greater amount of extractives than the tight side (Fig. 1A). The same pattern of extractive migration, but at a much less extent, can also be observed for white oak veneers lab-dried at 350°F (Fig. 1B). No definite patterns of extractive distribution can be observed for other treatments.

Microscopic observations revealed that lumen walls near the veneer surfaces were heavily deposited with extraneous substances (Fig. 3) whereas lumen walls in the interior portions of the veneer were only

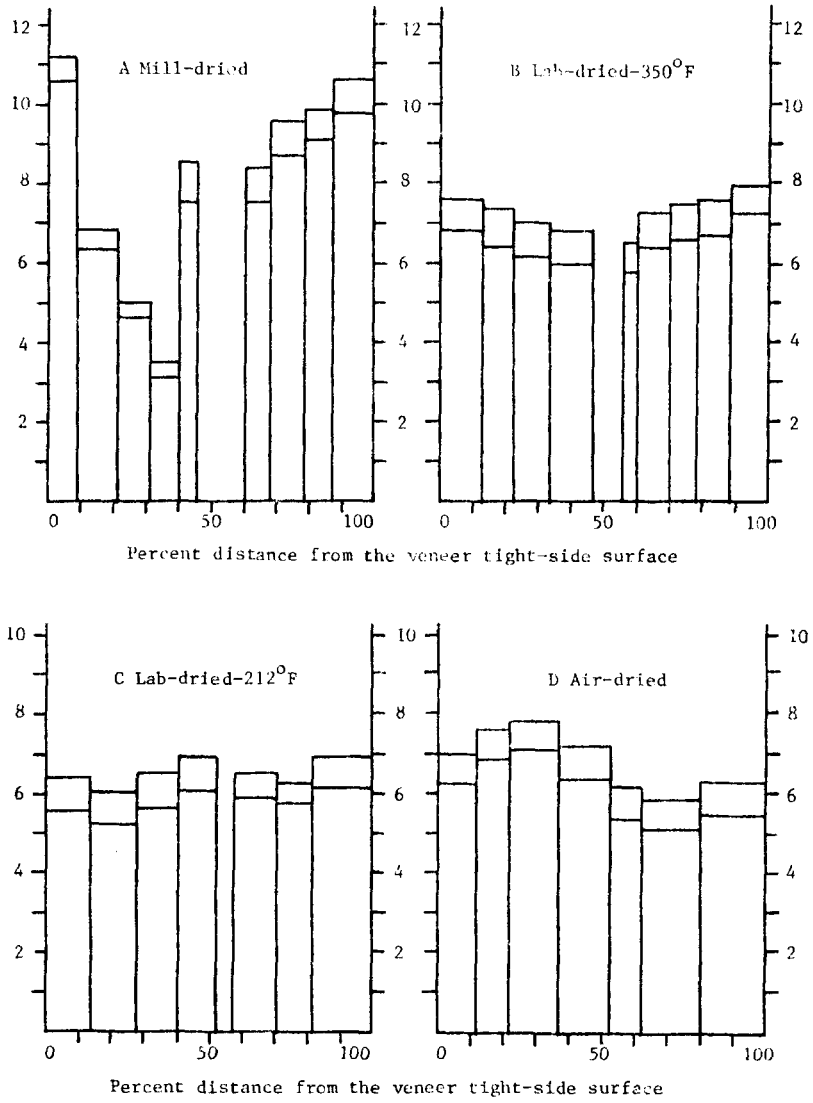

FIGURE 1 Effect of drying method on the distribution pattern of extractives in white oak veneers. The lower portion of each value is percent hot water-solubles and the upper portion is percent 95% alcohol-solubles.

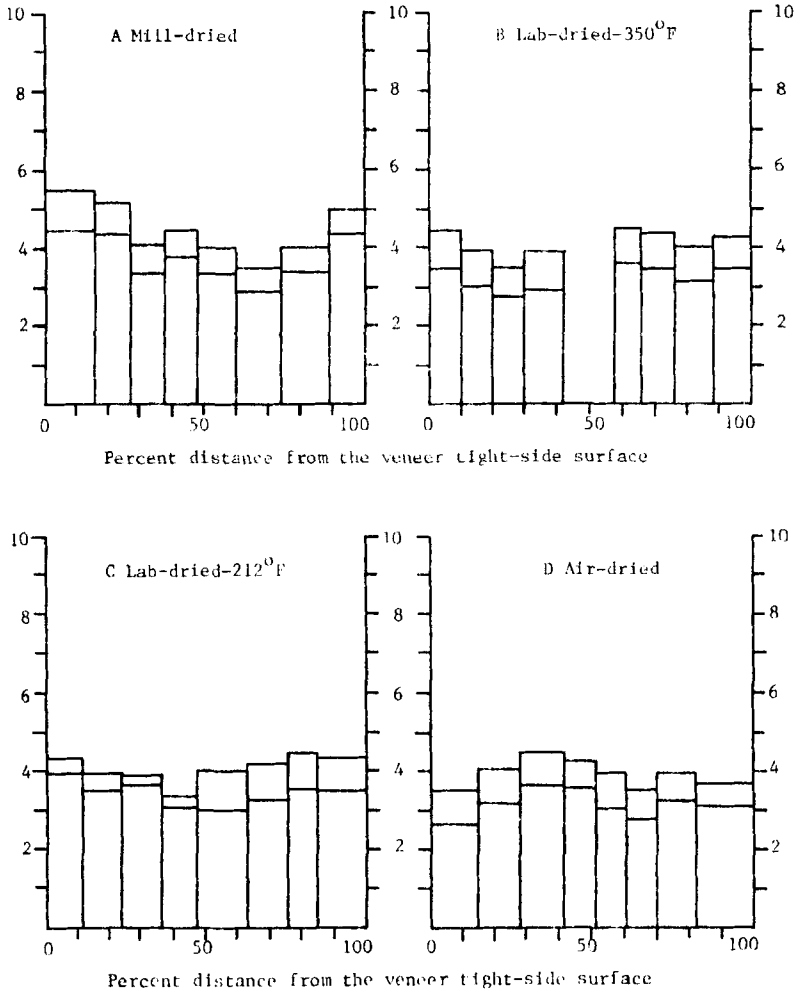

FIGURE 2 Effect of drying method on the distribution pattern of extractives in red oak veneers. The lower portion of each value is percent hot water-solubles and the upper portion is percent 95% alcohol-solubles.

slightly lined. The pit membranes in the interior portions of the veneer were also lightly encrusted. Lathe check surfaces were also heavily deposited with extraneous substances (Fig. 4). Most of the extraneous substances deposited on the cell walls could be removed by hot water extractions (Fig. 5 and 6) suggesting that these deposited substances

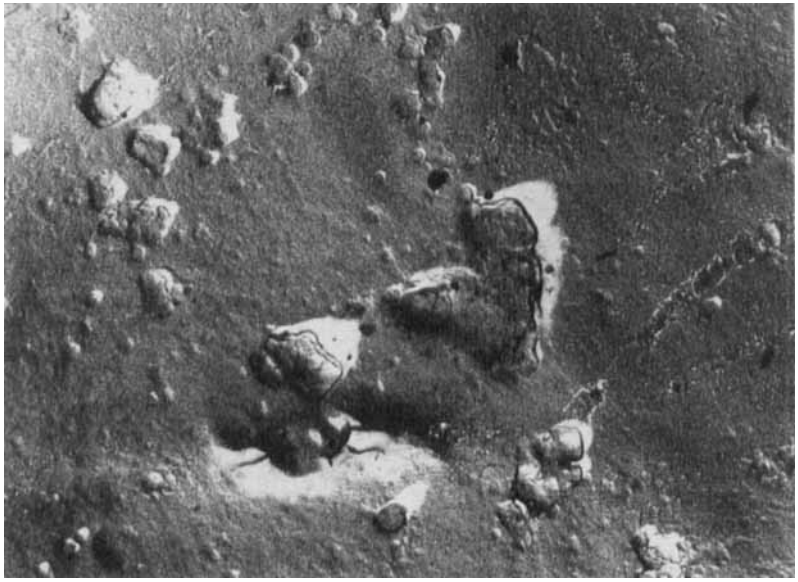

FIGURE 3 The lumen wall of a fiber tracheid on white oak veneer surface, showing heavy deposition of extractives (3,4000 ×)

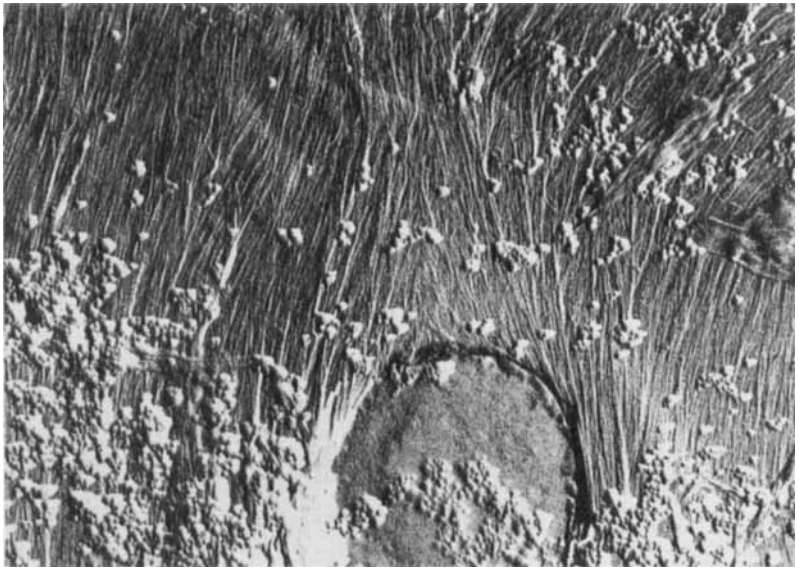

FIGURE 4 A typical lathe check surface of white oak veneer, showing extractive deposition on the pit membrane and on the exposed secondary wall near the pit (3,400 ×).

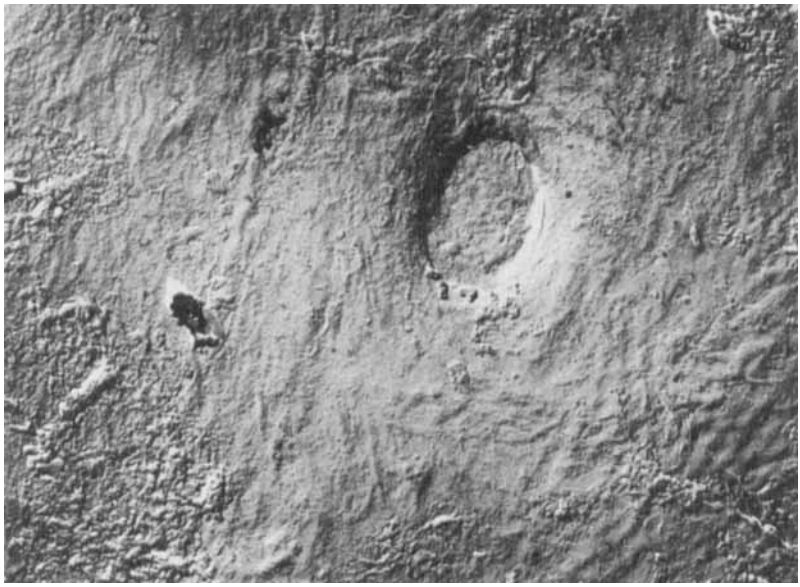

FIGURE 5 The lumen wall of a white oak fiber tracheid on the veneer surface after a hot-water extraction, showing most of extractives were removed but some resisted the solvent extraction (3,4000 ×).

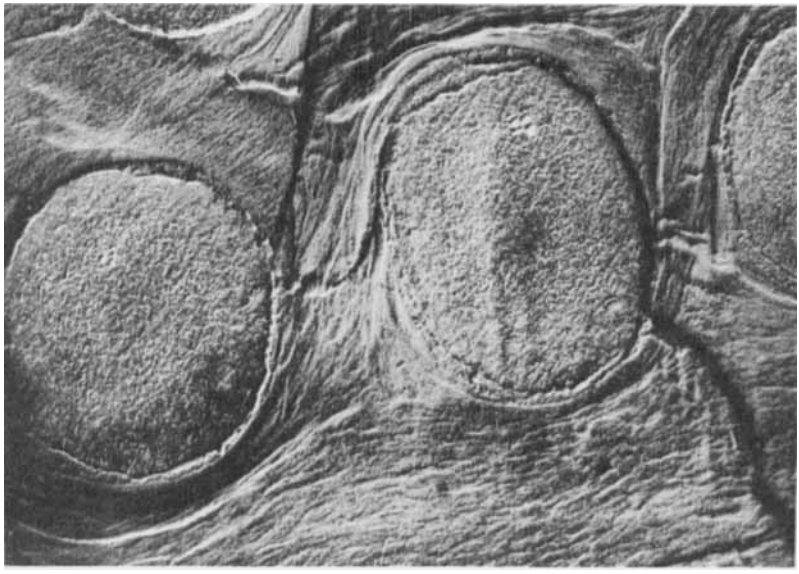

FIGURE 6 A hot-water extraction of white oak veneer also removed most of extractives deposited on the lathe check surface (4,700 ×).

TABLE I
Comparison of extractive content of white and southern red oak wood, veneer, and veneer surface scrapings

Material	White Oak			Southern Red Oak		
	Hot Water Solubles %	Subsequent EtOH Solubles %	Total	Hot Water Solubles %	Subsequent EtOH Solubles%	Total
Wood† (average)	8.66	0.84	9.50	5.05	0.83	5.88
Mill-Dried‡ Veneer (average)	7.60	0.71	8.31	3.82	0.67	4.49
Lab-Dried-350°F‡ Veneer (average)	6.49	0.83	7.32	3.15	0.91	4.06
Lab-Dried-212°F‡ Veneer (average)	5.74	0.79	6.53	3.45	0.63	4.08
Air-Dried‡ Veneer (average)	5.97	0.75	6.72	3.14	0.79	3.93
Mill-Dried§	13.10	1.15	14.25	7.32	1.01	8.33
Surface Scraping	(72.37)	(61.97)	(71.48)	(91.62)	(50.75)	(85.52)
Lab-Dried-350°F§	9.69	1.10	10.79	5.42	1.14	6.56
Surface Scraping	(49.31)	(32.53)	(47.40)	(72.06)	(25.27)	(61.58)
Lab-Dried-212°F§	8.31	0.91	9.22	5.41	0.95	6.36
Surface Scraping	(44.77)	(15.19)	(41.19)	(56.81)	(50.79)	(55.88)
Air-Dried§	8.49	0.55	9.04	5.51	0.58	6.09
Surface Scraping	(42.21)	(−26.67)	(34.52)	(75.48)	(−26.58)	(61.83)

† Contained heartwood and sapwood.

‡ Values are weighted average obtained from values shown in Figures 1 and 2.

§ An average of 0.002 inch in thickness was scraped from each veneer surface. Values in parentheses denote percent increases or decreases over the corresponding veneer average values of the same material.

were mainly water-soluble extractives. Figure 4 also shows that majority of water-soluble extractives migrated through pits and deposited on the pit membranes and on the exposed secondary cell walls in the vicinity of pits after evaporation of water. Since lathe checks also served as evaporation surfaces during drying, migration of extractives to lathe checks caused a greater accumulation of extractives in the loose side than in the tight side of the veneer (Fig. 1A).

Table I summarizes the results of the extraction study. Again, extractive migration is clearly demonstrated by higher than average extractive content values for surface scrapings. Surface scrapings collected from high-temperature-dried veneers had abnormally large amounts of water-insoluble extractives. This may indicate that during drying at high temperature water-soluble extractives migrated to the veneer surfaces and were oxidatively polymerized.

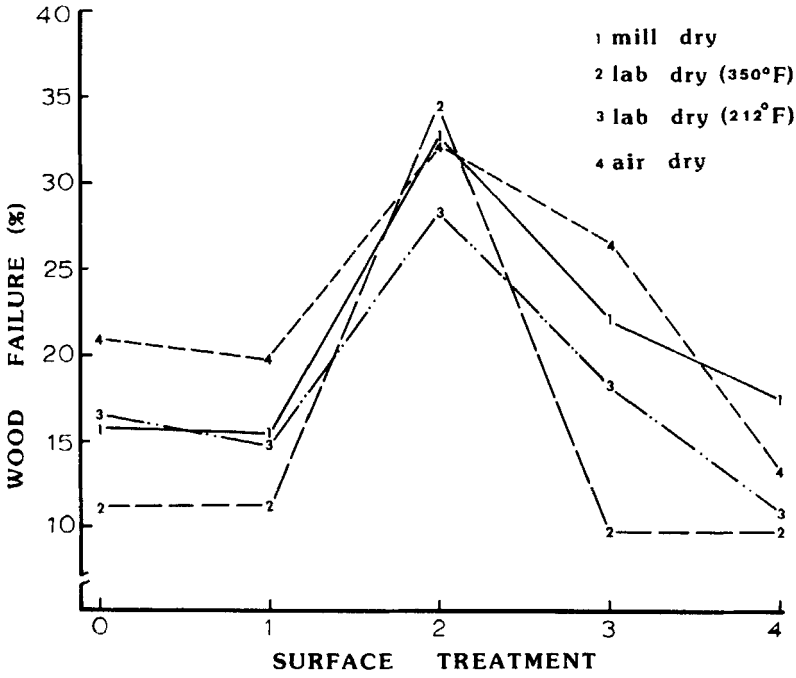

FIGURE 7 Effect of drying method and surface treatment on bond quality of white oak veneer. Surface treatment: 0-no treatment, 1-surface scraping, 2-soaking in 1% NaOH solution, 3-dipping In 1% NaOH solution, 4-water extraction.

Effect of drying method and surface treatment on bond quality

Figures 7 and 8 illustrate the effects of drying method and surface treatment on bond quality, in which some interactions between drying methods and surface treatments can be observed. The effects of drying method and surface treatment on bond quality were analyzed as an unreplicated randomized design. Thus, the interaction mean square of the factorial combination of drying method and surface treatment had to be used as the experimental error. Percentages of wood failure of nine to twelve subsamples for each treatment combination were measured, and the means were used in analyses. Duncan's multiple range test ($p = 0.05$) was used to compare the means of the four drying methods and the five surface treatments.

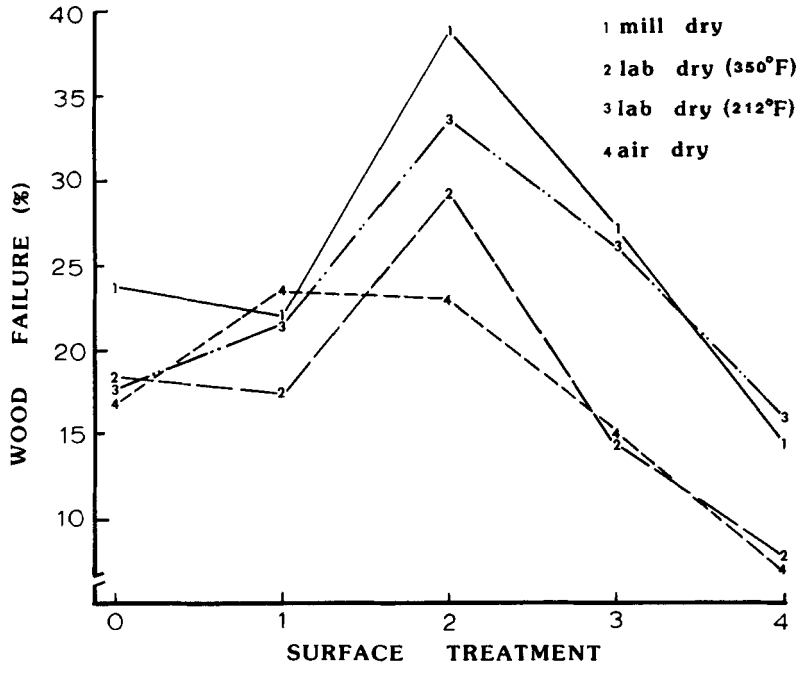

FIGURE 8 Effect of drying method and surface treatment on bond quality of southern red oak veneer. Surface treatment: 0-no treatment, 1-surface scraping, 2-soaking in 1% NaOH solution, 3-dipping in 1% NaOh solution, 4-water extraction.

TABLE II
Effect of drying method and surface treatment on the bond quality of white oak veneer

Drying Methods	Surface Treatments					
	0 No Surface Treatment	1 Surface Scraping	2 Soaking in 1% NaOH Solution	3 Dipping in 1% NaOH Solution	4 Water Extraction	Means† Drying Method
	Percent Wood Failure					
1 Mill Drying at 350°F	16	15	33	22	19	21 (A)
2 Lab Drying at 350°F	11	11	35	10	11	16 (B)
3 Lab Drying at 212°F	16	15	28	20	12	18 (AB)
4 Air Drying	20	20	33	26	13	23 (A)
Means† Surface Treatment	16 (B)	15 (B)	32 (A)	19 (B)	14 (B)	

† Means with the same letter are not significantly different at the 5% level. AB indicates the mean is not significantly different at the 5% level from means in group A and group B.

TABLE III
Effect of drying method and surface treatment on the bond quality of southern red oak veneer

Drying Methods	Surface Treatments					
	0 No Surface Treatment	1 Surface Scraping	2 Soaking in 1% NaOH Solution	3 Dipping in 1% NaOH Solution	4 Water Extraction	Means† Drying Method
	Percent Wood Failure					
1 Mill drying at 350°F	24	22	39	27	14	25 (A)
2 Lab Drying at 350°F	18	17	29	14	8	17 (B)
3 Lab Drying at 212°F	18	22	34	26	16	23 (A)
4 Air Drying	17	24	23	15	7	17 (B)
Means†	19	21	31	21	11	
Surface Treatment	(B)	(B)	(A)	(B)	(C)	

† Means with the same letter are not significantly different at the 5% level.

Tables II and III present results of the effect of drying method and surface treatment for white and southern red oak, respectively. For the four drying methods examined, it was found that mill drying and air drying of white oak veneers produced better bond quality, and mill drying and lab-drying at 212°F were the better methods for southern red oak veneers. Drying the veneers in a laboratory oven at 350°F produced inferior bonds for both species. Therefore, it seems that mill drying is consistently a better drying method in manufacturing white and southern red oak plywood even though it shows a greater magnitude of extractive migration.

Soaking the veneers in 1% NaOH solution improved bond quality but the dipping treatment did not show any effect. Analyses of the 1% NaOH solution remaining in the treating container after each soaking and dipping treatment showed that the soaking treatment of white oak veneers removed about three times the amount of extractives as the dipping treatment did. The corresponding ratio for southern red oak veneers was two to one. Extractives removed from veneers by soaking and dipping treatments were primarily located on the surfaces. Besides, as shown in Table IV, the soaking treatment enabled veneers to absorb about two times more NaOH than the dipping treatment did. Table IV also shows a positive and significant correlation between the amount of NaOH absorption and the percentage of wood failure. Plomley et al.[5] have demonstrated that an application of as little as 0.4 gram per square meter of wood extracts containing predominantly hydrolyzable tannins on veneer surfaces significantly reduced bond quality. These hydrolyzable tannins may quickly diffuse into the adhesive and adversely affect its setting property. White and southern red oak are known to contain appreciable amounts of gallo and ellogitannins.[14,15] These hydrolyzable tannins contain abundant acidic aromatic hydroxyls and would be expected to consume large amounts of NaOH in their neutralization. Therefore, the superior effect of the soaking treatment over that of the dipping treatment may be related to the combined effect of a greater magnitude of the removal of surface contaminants and the presence of a greater amount of NaOH in veneers.

Bond quality was not improved either by surface scraping or water extraction of veneers. In some cases, water extraction had an adverse effect. On the average, the water extraction of veneers caused only about 2% weight reduction. In the case of surface scraping treatment, only some extractives along with some cell wall materials were removed from

TABLE IV
Relationship between amount of NaOH absorption by each 12-inch by 12-inch veneer and percent wood failure

Species	Treatment	Mill Drying		Lab Drying at 350°F		Lab Drying at 212°F		Air Drying	
		NaOH (gram)	W. F. %	NaOH (gram)	W. F. %	NaOH (gram)	W. F. %	(gram)	%
White Oak	NaOH Soaking	0.41	33	0.29	35	0.55	28	0.38	33
	NaOH Dipping	0.28	22	0.17	10	0.32	20	0.19	26
Southern Red Oak	NaOH Soaking	0.54	39	0.35	29	0.50	34	0.44	23
	NaOH Dipping	0.31	27	0.17	14	0.28	26	0.20	15

1. Correlation between NaOH absorption and percent wood failure is significant at the 1% level.
2. The correlation coefficient is 0.7103.

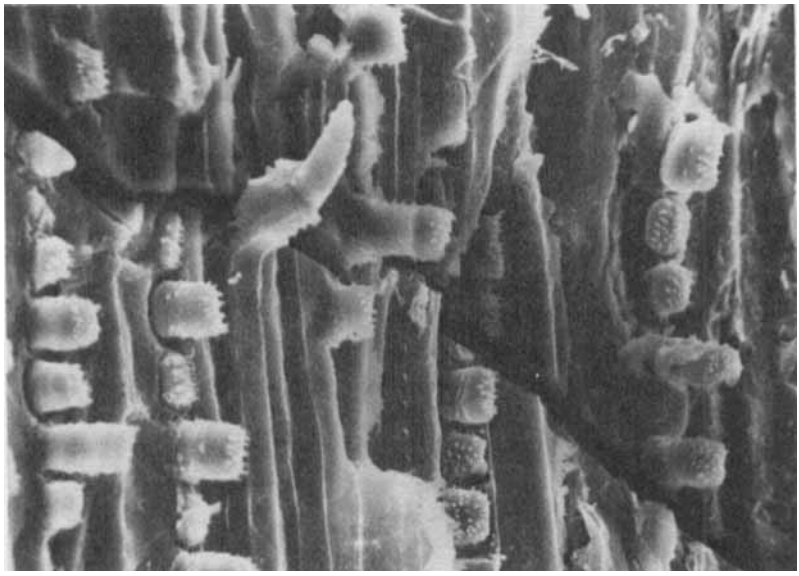

FIGURE 9 A typical glueline of white oak plywood, showing the glue molded the texture of the veneer surface and penetrated into the ray openings but failed to adhere to the cell walls (180×).

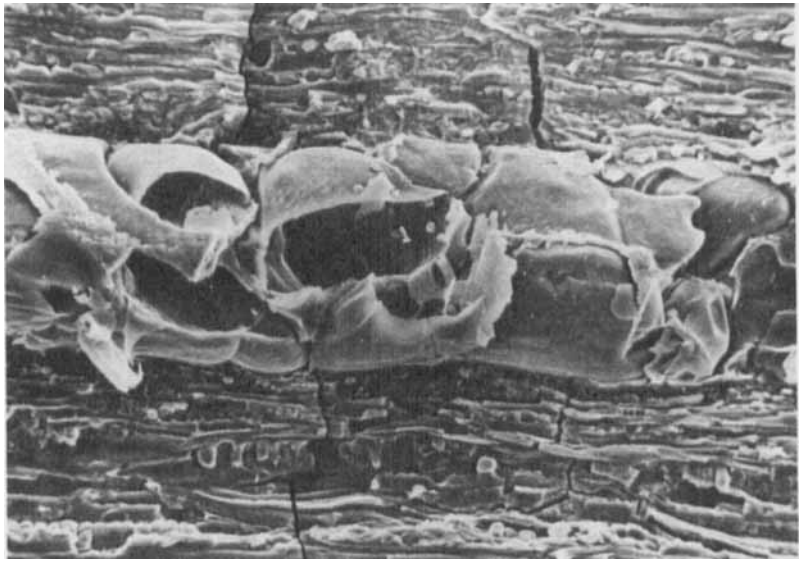

FIGURE 10 Tyloses in white oak vessels interfered with gluing (100×).

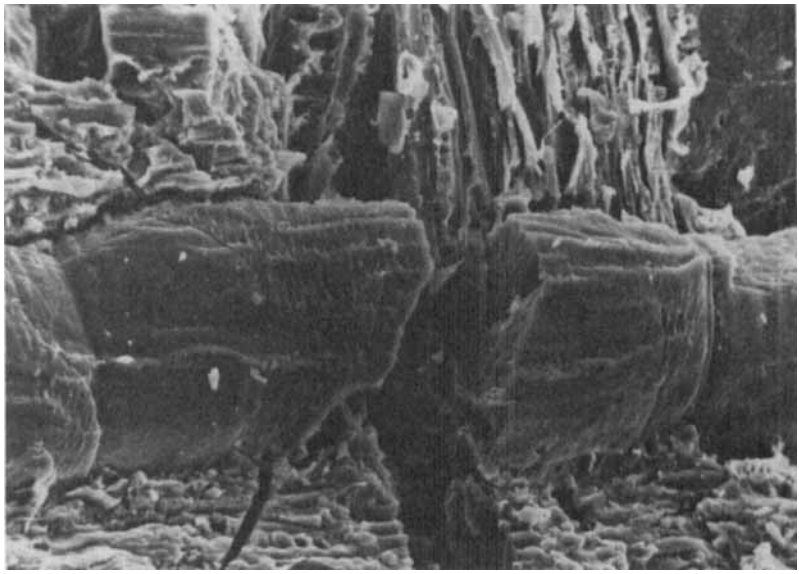

FIGURE 11 A white oak plywood glueline, showing the glue completely filled the vessel where tyloses had been removed prior to gluing (160×). The glue also failed to adhere to the cell walls.

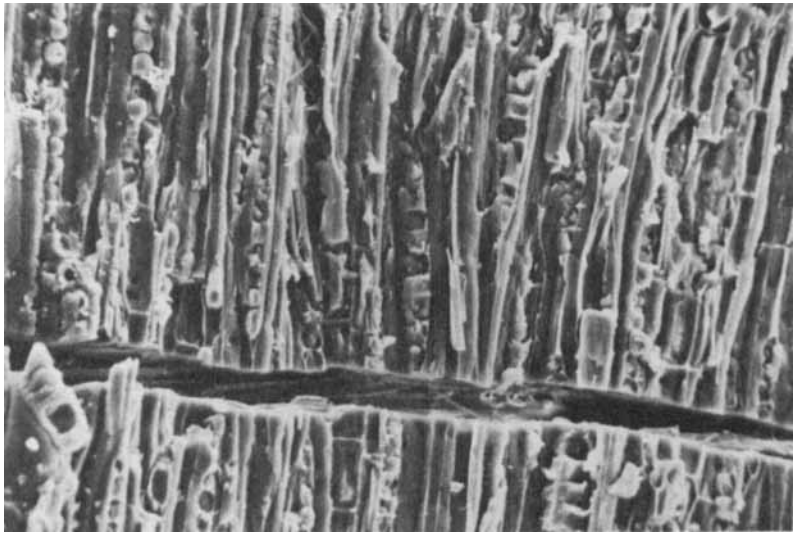

FIGURE 12 A typical southern red oak plywood glueline (160×). The glue also molded the texture of the veneer surface. The crack made during specimen preparation reveals the veneer surface beneath the glueline.

TABLE V
Effect of drying method and surface treatment on white oak veneer surface pH

Drying Methods	Surface Treatments											
	0 No Surface Treatment		1 Surface Scraping		2 Soaking in 1% NaOH Solution		3 Dipping in 1% NaOH Solution		4 Water Extraction		Means† Drying Method	
	Veneer Surface pH											
	Tight	Loose	Tight	Loose	Tight	Loose	Tight	Loose	Tight	Loose	Tight	Loose
1 Mill Drying at 350°F	4.9	4.4	—	—	6.8	6.7	—	—	—	—	—	—
2 Lab Drying at 350°F	4.5	4.2	—	—	5.6	5.7	5.6	5.9	4.8	5.0	5.1 (A)	5.2 (A)
3 Lab Drying at 212°F	4.4	4.4	—	—	5.3	5.8	5.5	6.0	4.8	4.9	5.0 (A)	5.3 (A)
4 Air Drying	4.3	4.5	—	—	5.6	6.3	5.5	6.2	5.0	5.2	5.1 (A)	5.2 (A)
Mean†	4.4	4.4	—	—	5.5	5.9	5.5	6.0	4.9	5.0		
Surface Treatment	(C)	(C)			(A)	(A)	(A)	(A)	(B)	(B)		

†Means with the same letter are not significantly different at the 5% level

TABLE VI
Effect of drying method and surface treatment on southern red oak veneer surface pH

Drying Methods	Surface Treatments											
	0 No Surface Treatment		1 Surface Scraping		2 Soaking in 1% NaOH Solution		3 Dipping in 1% NaOH Solution		4 Water Extraction		Means† Drying Methods	
	Veneer Surface pH											
	Tight	Loose	Tight	Loose	Tight	Loose	Tight	Loose	Tight	Loose	Tight	Loose
1 Mill Drying at 350°F	4.6	4.3	—	—	6.2	6.5	6.1	6.0	5.2	5.1	5.5 (A)	5.5 (AB)
2 Lab Drying at 350° F	4.1	4.1	—	—	—	—	—	—	—	—	—	—
3 Lab Drying at 212°F	4.6	4.2	—	—	6.1	6.8	5.6	6.3	4.9	5.1	5.3 (A)	5.6 (A)
4 Air Drying	4.0	4.2	—	—	5.7	6.1	6.1	6.0	4.9	4.9	5.2 (A)	5.3 (B)
Means† Surface Treatment	4.4 (C)	4.2 (D)	—	—	6.0 (A)	6.5 (A)	5.9 (A)	6.1 (B)	5.0 (B)	5.0 (C)		

† Means with the same letter are not significantly different at the 5% level. AB indicates this mean is not significantly different at the 5% level from means in group A and group B.

the veneer surfaces. Therefore, the ineffectiveness of both these two treatments in improving bond quality could be attributed to insufficient removal of deleterious extractives.

White and southern red oak veneers produced plywood panels with very poor bonds. Even with the best drying-surface treatment combination, wood failure was only 35% for white oak and 39% for southern red oak. Examination of the test specimens revealed that more than 80% had either bad transfer of the glue from the core ply to face plies or starved joints or both. Figure 9 shows a typical interfacial separation between white oak veneer and the glue. The glue molded the texture of veneer surface but failed to bond to the cell walls. Figure 9 also shows that the glue was able to flow into ray cell openings but also failed to adhere to the cell walls evidently because of the presence of extractives lining the ray cell lumen walls. Tyloses in the vessels of white oak were found to interfere with bonding (Fig. 10). The glue also failed to adhere to vessel walls even though tyloses had been removed before gluing (Fig. 11). Similar microscopic characteristics of southern red oak plywood gluelines were observed (Fig. 12).

Effect of surface pH on bond quality

Tables V and VI show the effect of drying method and surface treatment on veneer surface pH for white and southern red oak, respectively. No effect of drying method was found on veneer surface pH for either species. Soaking and dipping the veneers in 1% NaOH solution significantly increased surface pH. Extraction of the veneers with warm water also increased surface pH. However, statistical analyses showed that surface pH had no effect on bond quality. This result indicates that difficulties in bonding white and southern red oak veneers may attribute more to extractive contamination of veneer surface than to surface pH.

CONCLUSIONS

1. Mill drying of white and southern red oak veneers caused water-soluble extractives to migrate from the interior portions to veneer and lathe check surfaces.
2. White and southern red oak veneers were very difficult to glue. Even with the best drying-surface treatment combination, wood failure was only 35% for white oak and 39% for southern red oak.
3. Mill drying was consistantly the best drying method for both species,

and laboratory drying of veneers at 350°F obtained a lower bond quality.

4. Surface scraping to physically remove extractives accumulated on the veneer surfaces and water extraction of veneers did not improve bond quality. Dipping the veneers in 1% NaOH solution also did not show any effect on bond quality.
5. Removal of surface contaminants and absorption of sufficient amount of NaOH by soaking the veneers in 1% NaOH solution for 5 minutes significantly increased bond quality. This result indicates that difficulty in bonding white and southern red oak may be caused by the influence of extractives on the setting property of the glue.
6. Microscopic observation of the gluelines indicated that the glue molded the texture of veneer surfaces but failed to adhere to the cell walls.

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