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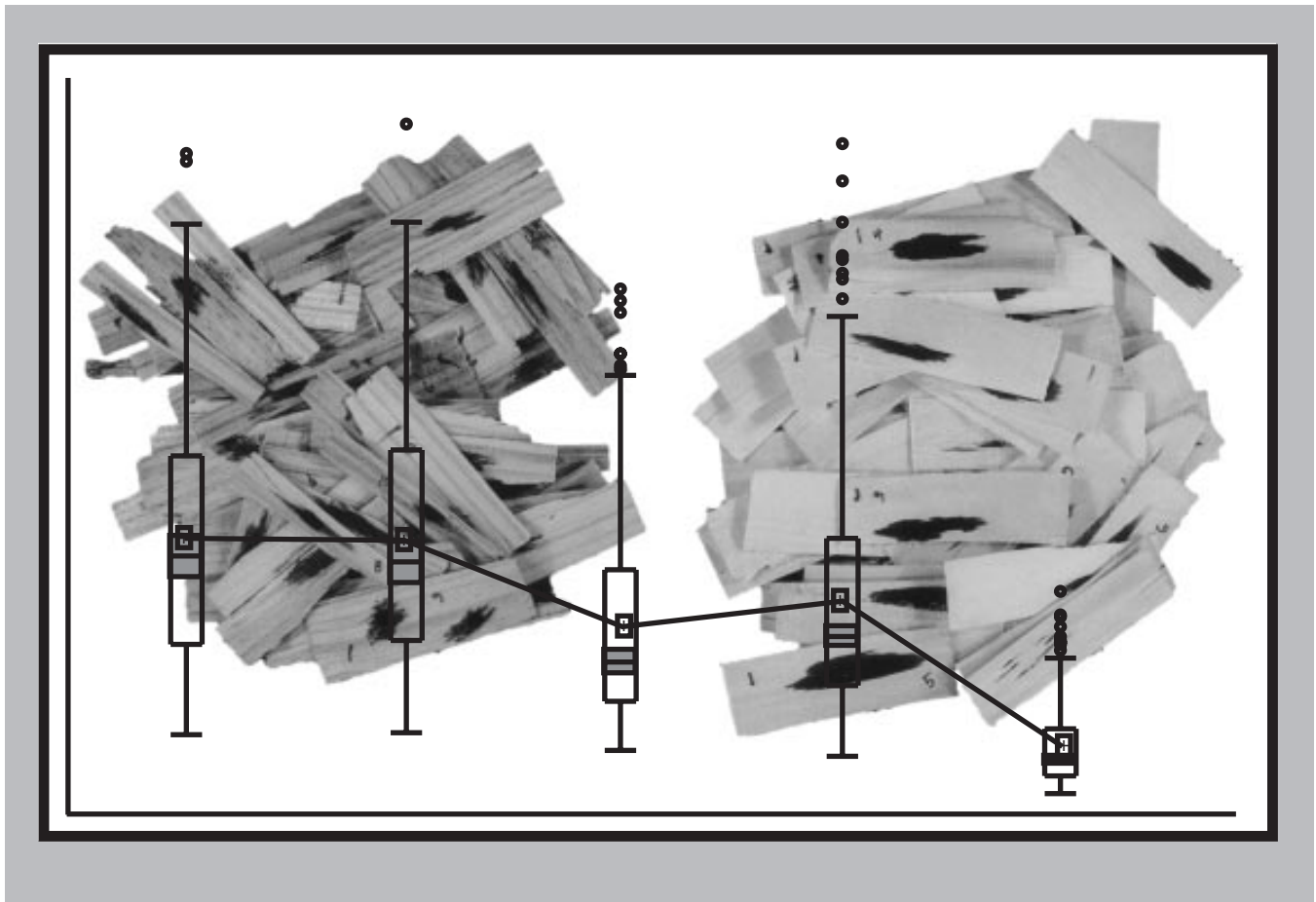
Forest
Products
Laboratory

Research
Note
FPL–RN–0267



Flake Storage Effects on Properties of Laboratory-Made Flakeboards

Charles Carll



Abstract

Aspen (*Populus gradidentata*) and loblolly pine (*Pinus taeda*) flakes were prepared with tangential-grain and radial-grain faces on a laboratory disk flaker. These were gently dried in a steam-heated rotary drum dryer. Approximately 1 week after drying, surface wettability was measured on a large sample of flakes using an aqueous dye solution. Three replicate boards of each combination of flake species and face grain were then fabricated. Each of the three replicate boards was made with a different adhesive resin. The three adhesives were urea-formaldehyde, phenolic, and neat isocyanate. The procedure (wettability measurements and board fabrication) was repeated at 1 month, 6 months, and 12 months after flake preparation. Surface wettabilities were reduced as the flake surfaces aged. Tension strengths of boards perpendicular to their faces (internal bond strengths) were also reduced as flake surfaces aged, and reductions in internal bond strengths and surface wettabilities appear to be related. Bending properties were, in general, not influenced by flake age. The observed reductions in internal bond strengths suggest that laboratory storage of flakes for much longer than 6 months is not a good practice. However, no conclusive statements can be drawn from this work concerning the influence of flake aging on board properties.

Keywords: Flake surfaces, flakeboard, flake aging, surface wettability, internal bond

May 1998

Carll, Charles. 1998. Flake storage effects on properties of laboratory-made flakeboards Res. Note FPL–RN–267. Madison, WI: U.S. Department of Agriculture, Forest Service, Forest Products Laboratory. 10 p.

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Flake Storage Effects on Properties of Laboratory-Made Flakeboard

Charles Carll, Research Forest Products Technologist
Forest Products Laboratory, Madison, Wisconsin

Introduction

At laboratories, flakes may be stored for extended periods of time before being fabricated into boards. The influence of this practice on board properties is unknown. Aged surfaces are not expected to be as wettable by adhesives as are freshly cut surfaces. Gray (1962) showed that the wettability of planed wood surfaces was reduced by mere hours of aging. Stumbo (1964) showed that storage of wood blocks in clean and constant-humidity conditions for a period of 5 months resulted in substantial reduction in strength of casein or phenol-resorcinol gluebonds, as evaluated by tension tests perpendicular to the glue line. Chen (1970) showed a distinct correlation between surface wettability of wood surfaces and glued-block shear strength. However, it is not at all clear that these findings are applicable to flakeboard fabrication.

Although flake aging could plausibly influence properties of laboratory-made boards, the effect of such aging has not been investigated. The influence of flake or particle drying temperature might plausibly be similar to the effect of flake or particle aging (by influencing surface wettability). The influence of drying temperature on properties of wood-based boards has been investigated (Bryant 1968, Plagemann and others 1984, Roffael 1987), with mixed results. None of these researchers reported surface wettability, although Bryant (1968) attempted to measure contact angle of adhesive droplets on flake surfaces (unsuccessfully, he reported). Hse (1972) and Wellons (1980) made wettability measurements on veneer surfaces and attempted to relate wettability measurements to plywood bond quality. Hse (1972) found fairly weak correlations between surface wettability and wet shear strength, while Wellons (1980) reported no correlation. More recently, Christiansen (1994) found no correlation between surface wettability of sawn (and surfaced) veneer surfaces and bond shear strengths of plywood made from the veneer. Carroll and McVey (1962) found that flakeboards made with a phenolic resin that readily wetted wood surfaces had poor internal bond strength but excellent flexural properties. They

suggested that excessive glue penetration occurred in core layers but not in surface layers, in which the adhesive was more quickly cured and thereby rendered immobile. Although the works cited in this paragraph frequently present contradictory results, altogether, they do not suggest that flake storage would significantly degrade properties of boards made from them. Since these previous works (concerning board products) did not always show consistent results and were not based on aging of surfaces, I performed an empirical study concerning aging of flakes.

Objectives

The purposes of this study were to determine if storage of flakes, as sometimes occurs in laboratories, influences their surface wettability and to determine if this could be related to properties of boards made from them.

Methodology

Experimental Design

Aspen and loblolly pine flakes were prepared with tangential-grain faces and with radial-grain faces. These were dried in a steam-heated rotary drum drier. Approximately 1 week after drying, surface wettability was measured on a sample of flakes. Three replicate boards of each combination of flake species and face grain were then fabricated, each with a different one of three adhesive resins: a urea-formaldehyde (UF) resin, a phenolic (PF) resin, and an isocyanate (MDI) resin. The procedure was repeated at 1 month, 6 months, and 12 months after flake preparation. The procedure was also repeated in part at 30 months flake age; surface wettability measurements were made and replicate UF-bonded boards were fabricated. No PF-bonded or MDI-bonded boards were fabricated at 30 months flake age because there were not enough flakes. The flakeboards were evaluated for internal bond, static bending, and thickness swelling as induced by water immersion.

Flake Preparation, Drying, and Storage

Flakes were prepared from sawn billets on a laboratory disk flaker. This permitted production of flakes with very close to true radial-grain and true tangential-grain surfaces. Billets were well in excess of fiber saturation and measured 76 mm along the grain and 25 by 25 mm. Knife projection was set at 0.64 mm, and rake angle was 60 degrees. The flakes had sufficient curvature to permit easy determination of the “tight” or concave side from the “loose” or convex side. The flakes were dried (each combination of species and anatomical face separately) in a steam-heated rotating drum dryer to a target moisture content of 4%. Temperature in the rotating drum remained below 100°C. Compared with flakes dried in industrial direct-fired driers, these flakes were gently dried. Their drying was representative of laboratory drying at the USDA Forest Service, Forest Products Laboratory (FPL).

After drying, flakes were stored in sealed polyethylene bags in a storage area that was heated in cold weather but not cooled during hot weather. Humidity control was not provided in the storage area. The lack of humidity control resulted in seasonal fluctuation of flake moisture content (MC) (Table 1). Flake storage in an area without humidity control is common laboratory practice.

Wettability Measurements

Wettability measurements were made by dispensing droplets of a 1% aqueous solution of safranin dye from a micropipette on flake surfaces and allowing the dye to spread. The dye was allowed to reach its ultimate spread; dye-applied flakes were generally left for a period of between 5 h and 14 h to allow water in the dye droplets to dissipate. The longest chord of dye that spread parallel to the wood grain was measured with a sonic digitizer. The red color of the dye permitted rapid visual identification of spread. This permitted a reasonably large sample of wettability measurements

(usually in excess of 200) to be made at each combination of species, anatomical face, and time since flake preparation.

Flakes for wettability measurements were taken directly from storage bags the day before boards of that flake type were fabricated. Samples of flakes consisted of flakes taken from at least three locations in each storage bag. One droplet of dye was carefully placed on the tight side of each flake in the sample. Dye application was performed at room temperature.

Flakeboard Fabrication

As indicated previously, each board was fabricated using one of three types of adhesives. The PF resin used was a liquid flakeboard resin at 43.5% resin solids. It was the most commonly used resin in our laboratory, specifically chosen for its consistency from batch to batch. The amounts needed were drawn from recently purchased containers that were normally maintained in our lab in refrigerated storage. The MDI adhesive was neat (100% resin solids). The adhesive manufacturer claimed that during storage, the adhesive could react with atmospheric moisture in the container to form a skin similar to that found on paint. With the exception of this reaction with atmospheric moisture, the manufacturer indicated that the adhesive was essentially unaffected by storage. The MDI adhesive was stored under refrigeration in a small container, with air purged from the container with dry nitrogen gas. No skin formed on its surface during the year between first and last use in this study, suggesting that the adhesive did not change during this study. The UF resin used was a liquid particleboard resin of 65% resin solids. This was also stored under refrigeration. A viscosity check at 6 months showed negligible change, so the same batch used for previously fabricated boards was used at that time. New batches of this adhesive were obtained for boards fabricated after 12 months and after 30 months of flake storage. The resin supplier indicated that these two batches differed slightly in formulation from the first batch and from each other. Although it is unlikely that this slight change in formulation was significant, it was a potential confounding variable.

Table 1—Flake moisture contents at different flake ages

Species and face	Flake moisture content (%)				
	1 week	1 month	6 months	12 months	30 months
Aspen					
Radial	4.2	4.6	7.6	5.7	5.3
Tangential	3.1	4.5	8.2	5.7	6.4
Pine					
Radial	5.7	7.8	7.6	6.5	7.6
Tangential	4.2	5.9	7.6	6.1	7.6

Table 2— Board fabrication variables

Adhesive	Adhesive content (%)	Press temperature (°C)	Compaction time (s)	Press time (s)
Urea-formaldehyde	5	149	60	300
Phenolic	5	177	70	430
Isocyanate	3	177	60	270

Boards measured 355 by 305 by 11 mm. Target oven-dry board density was 1.3 times wood species density as cited in the Wood Handbook (Forest Products Laboratory 1987). The boards did not contain wax. Flakes were not aligned. Resin content levels, press temperatures, compaction times, and press times are shown in Table 2. Target mattress (matt) moisture content into the press was 10% (total solids basis). Target MC was usually exceeded but generally by 1% or less. In a few cases, MC into the press was as high as 12.4%. Moisture content into the press was influenced by MC of the flakes as retrieved from storage and by ambient laboratory conditions during blending and matt formation. Although matt MC (for any given combination of species, anatomical face, and adhesive) varied with fabrication time, the range never exceeded 2.4% MC. The range was usually less than 1.2% MC. As will be discussed later, some data were later deleted to narrow the range of matt MC. The press was computer controlled. Different press control programs were used for the boards made with different adhesive types. For boards of the same adhesive type, the press control program was constant throughout the study.

Flakeboard Evaluation

Boards were tested for static bending, tensile strength perpendicular to the surface (internal bond), and thickness swelling by water immersion according to ASTM standard methods (ASTM 1986). Water immersion was performed with specimens in horizontal orientation. Thickness measurements were made at specimen edges (four measurements per specimen). Time between board fabrication and testing ranged from 4 to 23 months. Testing was done in two phases: first on boards made of flakes aged for 6 months or less, then 21 months later on boards made of flakes aged for 12 months or more.

Across the range of matt moisture contents within a combination of species, flake face, and adhesive, there sometimes was a discernible influence of MC on the ratio of modulus of rupture (MOR) in static bending to internal bond (IB) strength, suggesting that matt MC had influenced through-the-thickness (vertical) density distribution and was thus a

confounding variable. By deleting data from 6 of the 52 combinations of species, flake face, adhesive, and flake age, I held the range in matt MC with age for any combination of the other variables to between 0.1% and 1.4% and essentially eliminated any discernible influence of matt MC on MOR/IB ratio.

Watersoak tests were performed at room temperature and were done in batches but always by the same technician. Variation in room temperature caused variation in water temperature. This variation was as much as 3.3°C between batches and 3.3°C within a batch. In summary, the watersoak tests were probably not as well controlled as were the mechanical tests. Some of the watersoak data were deleted from the data set due to apparent recording errors.

Results and Discussion

Wettability Measurements

In general, dye spread was reduced as flake age increased. Within the samples of flakes at each combination of species, face, and age since cutting, there was substantial variation in dye spread. The maximum dye spread within a sample was from 4 to 10 times as great as the minimum. Observations were not normally distributed within a sample; the samples showed consistent skewness with a few dye spreads of much greater than average value. Results of a nonparametric analysis of variance (ANOVA) and a Tukey multiple comparison test showing the differences between dye spread median values are shown in Table 3. Despite substantial variation in dye spread within samples, statistically significant differences in spread with age were discernible, with a general trend of reduced wettability with increasing age.

The large variation in dye spread seems to agree with previously published results of wettability measurements. Bryant (1968) reported what he termed inconsistent results in an attempt to measure contact angle, which may in fact indicate variability. Hse (1972) reported high variability, but he did not quantify variability within samples. Christiansen (1994) found reasonably large variability in water drop absorption times on planed wood surfaces prepared from the same short board and dried in an identical manner. Richter and others (1994) made contact angle measurements of drops of water and of exterior stain and paints on wood surfaces. They made no quantitative statement regarding variability of their measurements but stated that it was high.

A word of caution is in order concerning the wettability measurements. Wettability measurements at different points in time were on surfaces at different moisture contents; flake moisture contents with time are shown in Table 1. Wellons (1980) indicated that surface wettability of wood with aqueous NaOH increases with increasing wood moisture content

Table 3—Median values of dye spread

Species and face	Spread (mm) at age ^a				
Aspen					
Radial	<u>23.6</u>	<u>20.3</u>	16.3	14.0	5.1
	1 wk	1 mo	12 mo	6 mo	30 mo
Tangential	<u>37.8</u>	<u>34.3</u>	30.6	27.4	24.9
	1 mo	1 wk	30 mo	6 mo	12 mo
Pine					
Radial	19.1	<u>16.1</u>	<u>15.0</u>	<u>12.4</u>	<u>12.3</u>
	1 wk	1 mo	12 mo	30 mo	6 mo
Tangential	<u>25.4</u>	<u>24.1</u>	<u>23.2</u>	20.6	13.9
	1 mo	12 mo	1 wk	6 mo	30 mo

^aMedian values underlined by a common solid line were not significantly different from each other at $\alpha = 0.05$ by Tukey multiple comparison test. Median values underlined by a common dashed line were not significantly different from each other at $\alpha = 0.10$ by Tukey multiple comparison test.

(independent of heat exposure or aging). Table 1 indicates that for the most part, flake moisture increased with age. Therefore, reduction in wettability with age was probably greater than suggested by the values in Table 3.

Mechanical Properties of Boards

In general, IB strengths decreased as flake age increased. Results of ANOVA and Tukey multiple comparison tests are presented in Table 4. For most combinations of species, face, and adhesive, IB strength at 12 months was substantially reduced relative to its level at 1 week or 1 month. Comparison of Figures 1 and 2 suggests that although IB strengths and surface wettabilities both tended to decrease with flake age, the relationship between IB strength and surface wettability with time was not particularly strong.

In contrast to the fairly consistent influence of flake age on IB strength, flake age generally did not have a consistent effect on static bending strength or stiffness (Tables 5 and 6). In only one combination of species, flake face, and adhesive type did flake age have a statistically significant influence on bending properties.

I can find no empirically supportable argument for why IB and static bending showed different degrees of response to flake age. I can only conjecture that the conditions within core layers of boards are never as conducive to bond formation as they are in surface layers and thus that IB strength is more sensitive to factors that adversely affect flake bonding than is static bending.

Thickness Swelling

The poorer control over test conditions for thickness swelling than for the mechanical tests leads me to view results of the watersoak tests with caution. Flake age had no consistent influence on thickness swelling induced by water immersion, but thickness swelling at 12 months of flake age was in many cases less than at other flake ages. Table 7 indicates that 24-h thickness swell values for flakes aged 12 months were significantly ($P < 0.05$) less than those at 1 month or less of flake age for 5 of the 12 combinations of species, flake face, and adhesive type; for 2-h thickness swell values, the difference was significant for 4 of the 12 combinations. The less thickness swelling at 12 months of flake age cannot be dismissed on the basis of variability in the test procedure that resulted in a lesser amount of water absorption; boards fabricated from 12-month-old flakes frequently showed greater water absorption than did boards fabricated from other flakes. The trend of superior performance in thickness swell tests at 12 months flake age was contradictory to what was observed in IB testing.

Conclusions

1. Flake aging showed a general trend of reducing surface wettability of aspen and pine flakes. The method used to measure surface wettability (spread of aqueous dye solution) permitted rapid collection of data on a property that is notoriously variable.
2. Flake aging generally had a negative impact on IB strength of flakeboards bonded with commonly used adhesives. Reduction in IB strength at 12 months of flake age was in some cases substantial. This reduction in IB strength appears to be related to loss of flake wettability. This result was not expected in light of previously published literature, much of which showed no relation between loss of surface wettability and degradation of bond in hot-pressed wood-based products.
3. Flake aging generally did not affect static bending properties, although there were a few exceptions.
4. Flake aging for 12 months seemed to have a beneficial influence on thickness swelling as induced by water immersion.

Table 4—Mean values of internal bond strength of flakeboards for various combinations of species, flake face, and adhesive type by flake age at time of board fabrication. Values are arranged in decreasing order of magnitude (left to right)

Species and face	Adhesive ^a	Internal bond strength (kPa) at age ^b				
Aspen						
Radial	UF	548	524	486	394	327
		1 mo	6 mo	1 wk	30 mo	12 mo
		<hr/>				
	PF	698	608	580	437	
		1 wk	1 mo	6 mo	12 mo	
		<hr/>				
	MDI	904	781	741	734	
		1 wk	1 mo	6 mo	12 mo	
		<hr/>				
Tangential	UF	717	609	590	587	
		1 mo	6 mo	12 mo	30 mo	
		<hr/>				
	PF	767	692	573		
		1 mo	1 wk	12 mo		
		<hr/>				
	MDI	1,105	1,071	901	687	
		6 mo	1 mo	1 wk	12 mo	
		<hr/>				
Pine						
Radial	UF	852	761	584	559	
		1 wk	6 mo	12 mo	30 mo	
		<hr/>				
	PF	871	781	710		
		6 mo	1 wk	12 mo		
		<hr/>				
	MDI	948	934	861		
		1 wk	1 mo	12 mo		
		<hr/>				
Tangential	UF	964	895	703	626	
		1 wk	1 mo	6 mo	30 mo	
		<hr/>				
	PF	921	893	845	799	
		6 mo	1 wk	1 mo	12 mo	
		<hr/>				
	MDI	1,234	1,129	1,102	968	
		6 mo	1 wk	1 mo	12 mo	
		<hr/>				

^aUF, urea-formaldehyde; PF, phenolic; MDI, isocyanate.

^bMean values underlined by a common solid line were not significantly different from each other at $\alpha = 0.05$ by Tukey multiple comparison test.

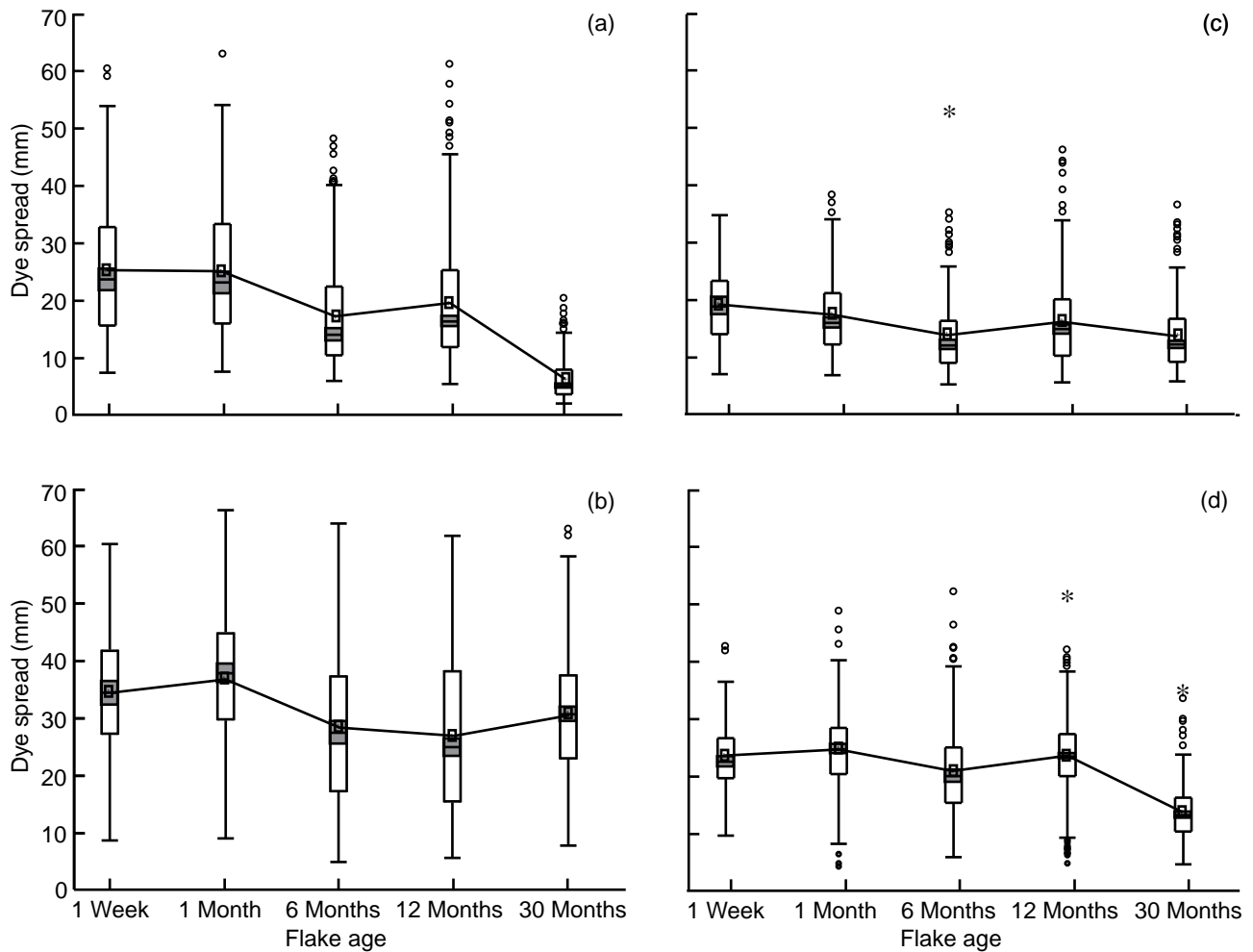


Figure 1—Box plots of dye spread as a function of flake age for the four combinations of flake species and anatomic face: (a) aspen radial, (b) aspen tangential, (c) pine radial, (d) pine tangential. Boxed areas enclose the two quartiles about the mean. Upper and lower horizontal lines include the entire range of data, with the exception of outliers. Circles outside the upper and horizontal lines are data points greater than 1.5 times the interquartile range away from the median (outliers). Asterisks are data points greater than 3.0 times the interquartile range away from the median (extreme outliers). The line between boxes connects mean values. The shaded area in the middle of each boxed area represents the 95% confidence interval about the median value.

5. In light of Conclusion 2, it appears that storage of flakes for much longer than 6 months is not a good practice. However, in light of Conclusions 3 and 4, no conclusive statement can be made concerning the overall influence of flake age on board properties.

Acknowledgments

The author acknowledges the laboratory assistance of Copeland Francis (deceased), William Kreul (retired), Sandra Lange, and James Wood (retired). James Evans, statistical mathematician, provided substantial advice for data analysis. Victoria Herian, statistician, and Cheryl Hatfield,

statistician, performed statistical analyses and prepared figures for this manuscript.

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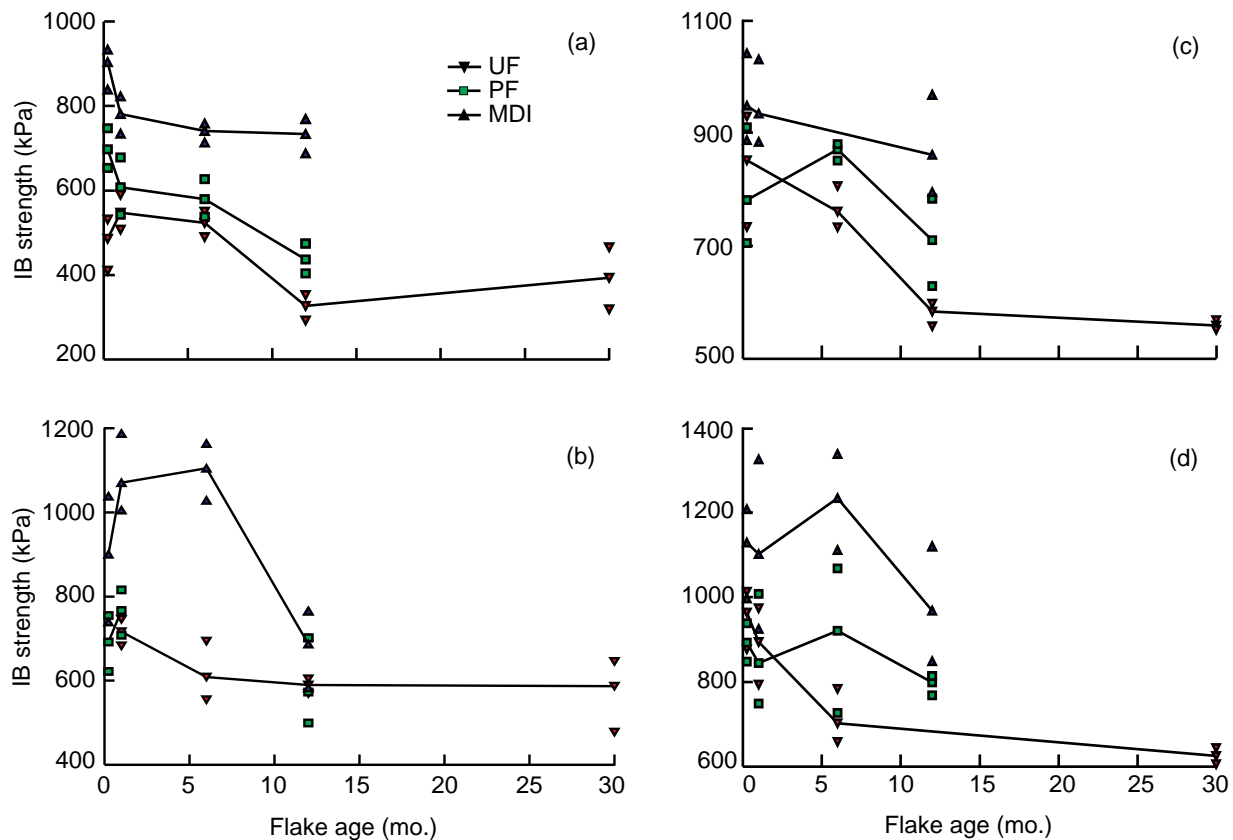


Figure 2—Internal bond (IB) strengths compared with flake age for the four combinations of flake species and anatomical face: (a) aspen radial, (b) aspen tangential, (c) pine radial, (d) pine tangential. Symbols not connected by lines indicate the range of board average values. UF, urea-formaldehyde resin; PF, phenolic resin; MDI, isocyanate resin.

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Table 5—Mean values of static bending strength of flakeboards for various combinations of species, flake face, and adhesive type by flake age at time of board fabrication. Values arranged in decreasing order of magnitude (left to right)

Species and face	Adhesive ^a	Mean bending strength (MPa) at age ^b					
Aspen	Radial	UF	39.3	39.3	38.9	37.1	31.6
			<u>6 mo</u>	<u>1 mo</u>	<u>1 wk</u>	<u>30 mo</u>	<u>12 mo</u>
			37.6	37.6	33.0	26.8	
				<u>12 mo</u>	<u>1 mo</u>	<u>1 wk</u>	<u>6 mo</u>
	Tangential	MDI	42.4	40.2	37.4	33.6	
			<u>12 mo</u>	<u>6 mo</u>	<u>1 wk</u>	<u>1 mo</u>	
			41.9	40.0	38.0	30.9	
				<u>6 mo</u>	<u>30 mo</u>	<u>1 mo</u>	<u>12 mo</u>
	Tangential	PF	34.9	33.0	31.9		
<u>12 mo</u>			<u>1 mo</u>	<u>1 wk</u>			
54.0			39.0	37.5	35.5		
			<u>6 mo</u>	<u>1 mo</u>	<u>1 wk</u>	<u>12 mo</u>	
Pine	Radial	UF	39.8	39.3	37.6	36.8	
			<u>1 wk</u>	<u>30 mo</u>	<u>12 mo</u>	<u>6 mo</u>	
			44.2	42.2	37.4		
				<u>6 mo</u>	<u>12 mo</u>	<u>1 wk</u>	
	Tangential	MDI	50.7	45.0	42.7		
			<u>1 wk</u>	<u>1 mo</u>	<u>12 mo</u>		
			46.3	46.1	42.5	42.2	
				<u>1 mo</u>	<u>6 mo</u>	<u>30 mo</u>	<u>1 wk</u>
	Tangential	PF	50.1	47.8	46.6	46.0	
<u>6 mo</u>			<u>1 mo</u>	<u>1 wk</u>	<u>12 mo</u>		
57.0			51.2	49.7	48.4		
			<u>6 mo</u>	<u>12 mo</u>	<u>1 mo</u>	<u>1 wk</u>	

^aUF, urea-formaldehyde; PF, phenolic; MDI, isocyanate.

^bMean values underlined by a common solid line were not significantly different from each other at $\alpha = 0.05$ by Tukey multiple comparison tests.

Table 6—Mean values of static bending stiffness of flakeboards for various combinations of species, flake face, and adhesive type by flake age at time of board fabrication. Values arranged in decreasing order of magnitude (left to right)

Species and face	Adhesive ^a	Mean bending stiffness (GPa) at age ^b				
Aspen						
Radial	UF	6.14	6.12	5.85	5.72	4.96
		1 wk	1 mo	30 mo	6 mo	12 mo
	PF	5.40	5.29	5.23	4.93	
		1 mo	12 mo	1 wk	6 mo	
	MDI	6.16	6.05	5.30	5.14	
		12 mo	6 mo	1 wk	1 mo	
Tangential	UF	6.28	6.13	6.12	4.62	
		6 mo	30 mo	1 mo	12 mo	
	PF	5.24	5.20	4.81		
		12 mo	1 wk	1 mo		
	MDI	6.92	5.65	5.54	5.13	
		6 mo	1 mo	1 wk	12 mo	
Pine						
Radial	UF	5.64	5.60	5.34	5.14	
		1 wk	30 mo	12 mo	6 mo	
	PF	5.98	5.94	5.89		
		12 mo	6 mo	1 wk		
	MDI	6.31	5.94	5.91		
		1 wk	1 mo	12 mo		
Tangential	UF	6.74	6.41	6.33	6.19	
		1 mo	6 mo	30 mo	1 wk	
	PF	6.56	6.45	6.12	6.01	
		6 mo	1 mo	1 wk	12 mo	
	MDI	7.01	6.90	6.60	6.03	
		6 mo	12 mo	1 wk	1 mo	

^aUF, urea-formaldehyde; PF, phenolic; MDI, isocyanate.

^bMean values underlined by a common solid line were not significantly different from each other at $\alpha = 0.05$ by Tukey multiple comparison tests.

Table 7—Mean values of 24-h thickness swelling of flakeboards for various combinations of species, flake face, and adhesive type by flake age at time of board fabrication

Species and face	Adhesive ^a	24-h thickness swell (%) at age ^b					
Aspen	Radial	UF	37.5	37.4	37.4	34.5	26.6
			6 mo	30 mo	1 mo	1 wk	12 mo
			27.6	25.4	24.6	21.3	
	Tangential	PF	6 mo	1 mo	1 wk	12 mo	
			21.5	20.4	16.3	12.6	
			1 mo	6 mo	1 wk	12 mo	
		UF	31.9	25.8	19.2		
			1 mo	30 mo	12 mo		
			21.6	20.1	16.7		
	MDI	1 mo	1 wk	12 mo			
		17.9	15.8	15.4	12.3		
		1 wk	6 mo	1 mo	12 mo		
Pine	Radial	UF	49.7	48.3	30.9		
			6 mo	30 mo	12 mo		
			41.3	27.0	26.9		
		PF	6 mo	1 wk	12 mo		
			38.5	32.0			
			1 mo	12 mo			
	Tangential	UF	51.3	40.7	40.0	30.3	
			6 mo	1 wk	30 mo	1 mo	
			42.4	37.5	24.6	22.7	
		PF	6 mo	1 wk	12 mo	1 mo	
			34.2	32.7	28.9	24.8	
			1 wk	6 mo	12 mo	1 mo	

^aUF, urea-formaldehyde; PF, phenolic; MDI, isocyanate.

^bMean values underlined by a common solid line were not significantly different from each other at $\alpha = 0.05$ by Tukey multiple comparison test.

