# DIMENSIONAL STABILITY OF ACETYLATED ASPEN FLAKEBOARD<sup>1</sup>

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#### ABSTRACT

This study was conducted to determine if thickness swelling of flakeboards, which is much greater than that of plywood or solid wood, can be improved by chemical modification of the constituent wood flakes prior to board manufacture. The chemical treatment involved acetylation of oven-dry aspen ring flakes with a 50/50 mixture of acetic anhydride and xylene. Thickness swell of treated and control samples was measured by either immersion in water for periods of up to 10 days or exposure to 90% relative humidity for periods of up to 20 days. Treated samples exhibited one-sixth to one-seventh the thickness swelling of controls for the water-soak test. Similar trends were observed for humidity exposure.

Keywords: Flakeboards, dimensional stability, thickness swell, chemical treatment, physical property.

#### INTRODUCTION

Flakeboards generally have poorer dimensional stability than plywood or solid wood. This precludes the use of these composite boards where they are exposed to weather or high relative humidity—house siding and trim, garage doors, concrete forming, and farm buildings, for example. If flakeboards with properties similar to those of plywood could be obtained with reasonable economy, these major markets would be available for these products. This research was undertaken to determine if the dimensional stability properties of flakeboards, as measured by thickness swell and water absorption, can be improved by chemical treatment of the constituent wood flakes.

The chemical treatment we studied was acetylation of dry flakes with a 50/50

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mixture (weight percent) of acetic anhydride and xylene. This treatment is usually done in a high-pressure reactor with the wood completely immersed in the treating solution. There are several drawbacks to this procedure. The treatment schedule is slow, and large volumes of chemicals, relative to the volume of wood, are required. Additionally, the size of the treating cylinder available for use at our laboratory precluded treating enough flakes so that a statistically significant number of treated flakeboards could be manufactured and tested. Because of these drawbacks, a secondary concern of this work was to determine if an atmospheric pressure/vapor-phase treatment schedule could be shown to give results equivalent to those of the liquid-phase treatment.

### BACKGROUND DISCUSSION AND PAST RESEARCH

All wood products are hygroscopic, and shrink and swell when subjected to environmental conditions that cause desorption or absorption of water. Wood is dimensionally stable when the moisture content is above the saturation point and changes dimension as moisture is gained or lost below that point.

The magnitude of the dimensional change of flakeboards is much greater in the thickness direction than would be expected from the normal shrinking and swelling of solid wood.

The additional thickness swelling that occurs when flakeboards are exposed to moisture—greater than that normally expected for wood material—is due to the release of residual compressive stresses imparted to the board during the pressing of the mat in the hot press. It is known that compressive failure of at least a portion of the wood particles is required to produce particleboard. The moisture content reduction while the mat is restrained in the hot press reduces the plasticity of the wood and results in a "set" of these compressive stresses. At some future date when the moisture content increases, the additional moisture will plasticize the wood and permit these stresses to be relieved, allowing expansion in the thickness direction. Subsequent redrying will result in thickness shrinkage equal only to the shrinkage of the particles; none of the compressive stress release will be recovered (Beech 1975; Neusser et al. 1965). Additional wetting and drying cycles will also result in further irrecoverable thickness swelling, but at a decreasing rate.

Approaches to the improvement of the dimensional stability properties of flakeboards have included the study of both optimizing various manufacturing processes and modifying wood chemically to minimize moisture sensitivity.

## Dimensional stabilization of flakeboards through process optimization (Kelly 1977)

A number of researchers have studied the water absorption and thickness swelling of particleboards and flakeboards as influenced by various processing parameters.

Density. — The effect of board density on the thickness swelling of particleboard is confounded with the springback behavior. Higher density boards will possess more compressive set than lower density boards when both are made with the same wood furnish. Because of this, thickness swelling normally is reported to increase with increasing board density (for example, Gatchell et al. 1966; Halligan and Schniewind 1972; Hse 1975). However, other authors have found either an increase or no change in thickness swelling with increasing board density (Kelly 1977; Lehmann and Hefty 1973; Vital et al. 1974).

*Particle configurations.*—The available literature appears to be practically unanimous in that better thickness stability is obtained with boards produced from thin particles than from thick particles. The lower wood mass in each particle and the increased number of particle–particle interfaces possibly allows better dispersion of the hygroscopic swelling into the interparticle voids. Consequently, this swelling into the macroscopic board voids, not internal swelling within the wood particles, results in less thickness swelling.

Much less agreement is found regarding the influence of particle length on thickness swelling. In general, it appears that increasing particle length improves the thickness stability.

*Resin and wax.*—Increasing the resin content in a given particleboard will result in improved interparticle bonding, which should also improve the thickness stability. However, in all probability there is a level at which additional resin will no longer enhance this bonding and serves only to impregnate or coat the component particles. This is the basis upon which impregnating resins have been used to limit the hygroscopicity of the material, but the economics do not presently justify this treatment.

The use of a paraffin wax is widespread in the particleboard industry for both urea-formaldehyde- and phenol-formaldehyde-bonded boards. Contradictory claims appear in the literature as to the value of the wax in reducing the dimensional changes of particleboard (Kelly 1977).

Other methods. — Other proposals to reduce the hygroscopicity and consequently improve the dimensional stability of particleboard are heating the unbonded particles or heating the finished particleboards, with or without steam (Burmester 1973; Halligan and Schniewind 1972; Heebink and Hefty 1969; Hujanen 1973; Suchsland and Enlow 1968). Post-steaming appears to be the most effective method for improving dimensional stability, but the additional costs of this treatment have prevented its acceptance by flakeboard manufacturers.

Overall, the dimensional stabilization of flakeboards through process adjustments or changes does not provide enough improvement in thickness swell or water absorption properties to provide entry for this product into new markets.

# Dimensional stabilization of flakeboards through chemical treatments

Traditionally, wood has been treated with chemicals to increase its decay, fire, and/or moisture resistance. These chemical treatments can be applied as surface coatings, as bulking agents within the swollen structure of the wood fibers, or by reaction of a constituent of wood with a chemical to form a new moisture-insensitive chemical component.

Wood is composed essentially of polysaccharides (cellulose and hemicellulose) and lignin in proportions of about 70% and 30%, respectively. Past research indicates that the water-attracting property of wood is due in large measure to the hydroxyl groups located on the polysaccharide chains and to a lesser extent on lignin (Tarkow et al. 1950). This finding suggested the possibility of stabilizing wood by replacing some of the hydroxyl groups with less hygroscopic groups by a method that would leave unchanged the residues to which the groups are attached. It would not be necessary to replace all the hydroxyl groups, as x-ray investigations have shown that when water enters wood or other cellulosic materials the crystalline portions within cellulose do not come in contact with the moisture (Tarkow et al. 1950). Only those hydroxyl groups located on the surface of the micelles or in the amorphous regions of wood come in contact with water and, presumably, only these need to be replaced.

Many types of reactions are known to involve hydroxyl groups. One of the most practical of these is the interaction of a hydroxyl group with an acid anhydride, called esterification. In particular, when acetic anhydride is used, the reaction is called acetylation. The acetic anhydride replaces the relatively hygroscopic hydroxyl groups in the wood with the less hygroscopic acetyl groups, and leaves the wood in a swollen condition because of the bulking action of the acetyl groups within the cell walls.

A method of acetylating solid wood with acetic anhydride and pyridine was developed at the Forest Products Laboratory (Tarkow et al. 1950). This treatment resulted in a substantial improvement in dimensional stability properties. Over the years, extensive research has been conducted on the chemical approach to the dimensional stabilization of solid wood. Little effort has been made to apply these chemical modification principles to the constituent components of a reconstituted wood product.

Rowell et al. (1984) completed a baseline study in which aspen, Douglas-fir, and southern pine sawdust, flakes, and solid wood were acetylated with uncatalyzed acetic anhydride at 120 C, 150 lb/in.<sup>2</sup> pressure for various reaction times to give levels of bonded chemicals up to 25 weight percent gain. These modified flakes were then made into flakeboards and dimensional stabilization was determined. Results were encouraging. For example, wood particles acetylated to 15 to 20 weight percent gain before board formation resulted in a particleboard that absorbed 50% less liquid water and reduced thickness swelling up to 70% as compared to untreated controls.

#### EXPERIMENTAL PROCEDURE

# Material preparation

Aspen ring-cut flakes, screened to eliminate material passing a  $\frac{1}{4}$ -inch screen, were oven-dried at 105 C for 12 hours for control boards. Flakes to be chemically treated were oven-dried at 105 C for 12 hours prior to acetylation.

#### Flake treatment

The oven-dry flakes were treated in a glass vessel using atmospheric pressure. Two chemical treatments were used. One involved complete immersion of flakes in the treating solution of acetic anhydride/xylene. The other consisted of exposing the oven-dried flakes to vapors of acetic anhydride/xylene for a period of 4 hours. Reaction of the flakes with a 1/1 (V/V) solution of acetic anhydride in xylene at 140 C produced approximately a 15 weight percent gain of added acetate groups after 4 hours. Excess chemical was then drained from the reactor and a vacuum of 750 mm Hg was applied for 1 hour at room temperature. The treated flakes were removed from the reactor and oven-dried for 24 hours at 105 C.

Flakes immersed in the acetic anhydride/xylene solution gained 14.6% bonded chemical compared to a gain of 15.1% via the vapor-phase treatment.

#### Flakeboard manufacture

Acetylated and control flakes (180 grams) were sprayed with a 44 weight percent aqueous solution of phenol-formaldehyde resin to give a 6% adhesive content based on the oven-dry weight of wood. The flakes, at 10% moisture content, were then formed into a 6-inch-square randomly oriented mat and pressed at an average of 600 lb/in.<sup>2</sup> for 10 minutes between platens heated to 180 C to produce a  $\frac{1}{2}$ -inch-thick flakeboard with a density of 40 lb/ft<sup>3</sup>. No catalyst or wax was used. Four untreated controls, four liquid-phase, and two vapor-phase acetylated boards were made. The final trimmed boards measured  $\frac{1}{2}$  by 4 by 4 inches.

Each trimmed flakeboard was cut into four samples  $\frac{1}{2}$  by 2 by 2 inches, lightly sanded, and oven-dried at 105 C for 18 hours. Of each set of four samples, one was used for the water-soak, one for the humidity, and one for the internal bond tests, and one was unused.

The samples were weighed and measured. Thicknesses were taken at five locations (the four corners and the center) of each sample with a flatbed micrometer. Because the flakes that were treated by immersion had essentially the same weight gain as those that were vapor-phase treated, the samples made from these flakes were grouped together for the water-soak, humidity, and internal bond tests.

#### TESTS CONDUCTED

#### Water-soak test

Specimens were immersed in water at 20 C for various lengths of time. After each soaking period, the specimens were wiped of excess water, measured for thickness, and weighed. The percent weight gain, thickness swelling, and water absorption were determined on the basis of initial oven-dry measurements. Samples were measured after 2, 4, 6, and 8 hours and after 1, 2, 3, 5, 7, and 10 days of water soaking.

#### Humidity tests

Control and treated liquid phase specimens were placed in a 90% RH, 27 C room for varying lengths of time. Weights and thickness measurements were taken after 2, 4, and 8 hours and after 1, 2, 3, 5, 7, 10, 14, and 20 days. Again, the percent weight gain, thickness swelling, and water absorption were determined based on the initial oven-dry measurements.

#### Internal bond test

To determine whether or not the treatments had any effect on adhesive bond strength, internal bond tests were conducted on specimens conditioned to 65% RH and 22 C for 6 days, according to ASTM Standard D 1037 (1978).

#### RESULTS AND DISCUSSION

Because of the limited number of specimens per individual test, no statistical treatment of the data was possible. The size of the treating vessel used for acetylation of the flakes allowed for the treatment of enough material for one 6- by 6-inch flakeboard sample per treating schedule for the vapor-phase treatment, and for two 6- by 6-inch samples for the liquid-phase treatment. The results presented here should be considered as indicative of trends that a larger, statis-

Board parameter measured		Increase over soaking times of										
	Flake variation		Ho	ours		Days						
		2	4	6	8	1	2	3	5	7	10	
Thickness swell	Control <sup>2</sup> Acetylated <sup>3</sup>					42.2 6.6						
Water absorption	Control <sup>2</sup> Acetylated <sup>3</sup>					79.0 41.6						

TABLE 1. Thickness swell and water absorption for control and acetylated flakeboard samples immersed in water for varying lengths of time.

<sup>1</sup> Based on initial oven-dry measurements.

<sup>2</sup> Based on an average of four specimens per measurement.

<sup>3</sup> Based on an average of six specimens per measurement.

tically valid, experiment should confirm. They are similar to those obtained with solid wood products treated in a similar manner (Rowell 1975).

### Thickness swell

Thickness swelling of treated and control specimens was compared by either immersion in water or exposure to 90% RH for specific times. After selected exposure times, the samples were checked for thickness or water absorption. Changes based upon comparisons to initial oven-dry readings are listed in Tables 1 and 2, and are presented graphically in Figs. 1 and 2.

The water-soak test shows dramatic differences in thickness swell properties for treated and control specimens (Table 1, Fig. 1). Control boards showed six to seven times the swelling of the treated boards.

For the specimens exposed to 90% RH, trends were similar to those observed for the water-soak tests.

Thickness swell of the controls was 2.3 times more than that of acetylated samples after 2 hours storage, and 4.4 times more for 20 days storage (Table 2, Fig. 2). The acetylated samples appear to stabilize after about 5 days of exposure to 90% RH, whereas the controls continue to expand or swell through the 20-day storage period.

Board parameter measured		Increase over storage times of											
	Flake variation	Hours			Days								
		2	4	8	1	2	3	5	7	10	14	20	
Thickness swell	Control <sup>2</sup>	1.4	2.2	3.5	7.0	10.4	13.3	17.0	18.3	19.8	20.7	21.3	
	Acetylated <sup>3</sup>	0.6	0.7	0.9	1.9	2.5	3.1	3.7	4.1	4.3	4.6	4.8	
Water absorption	Control <sup>2</sup>	2.6	3.5	4.9	8.9	12.0	14.2	16.7	17.7	18.5	18.9	19.1	
	Acetylated <sup>3</sup>	2.3	3.3	4.5	7.6	9.7	11.5	13.3	14.2	15.0	15.3	15.3	

 TABLE 2.
 Thickness swell and water absorption for control and acetylated flakeboard samples stored at 90% relative humidity for varying lengths of time.

Based on initial oven-dry measurements.

<sup>2</sup> Based on an average of four specimens per measurement.

<sup>3</sup> Based on an average of six specimens per measurement.

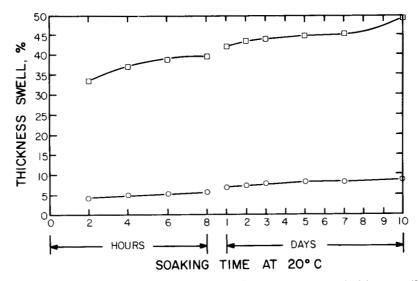


FIG. 1. Thickness swell of control and acetylated flakeboards when soaked in water (20 C) for varying lengths of time.  $\Box$  = control; O = acetylated.

# Water absorption

Data are presented in Tables 1 and 2 for the water absorption percentages for the water-soak and humidity-storage tests, respectively. The trends observed in water absorption are similar to those obtained with the thickness swell test. The flakeboards made with untreated flakes absorbed  $1\frac{1}{2}$  to  $2\frac{1}{2}$  times more water than

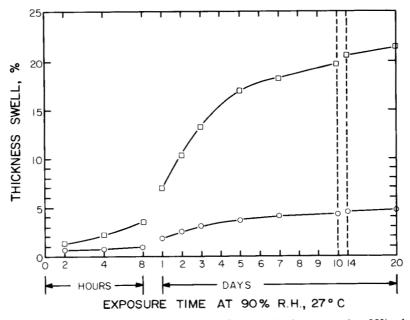


FIG. 2. Thickness swell of control and acetylated flakeboards when exposed to 90% relative humidity (27 C) for varying lengths of time.  $\Box$  = control;  $\bigcirc$  = acetylated.

did the boards with acetylated flakes (Table 1). The water-soak test was more sensitive than the humidity exposure test (Table 2) in showing the differences between the stability of the treated and control flakeboards.

#### Internal bond

In the internal bond test, the six samples with acetylated flakes averaged 98 lb/ in.<sup>2</sup> with a range from 84 to 111 lb/in.<sup>2</sup>. The four control samples averaged 85 lb/in.<sup>2</sup> with a range from 69 to 100 lb/in.<sup>2</sup>. The American National Standards Institute Specification ANSI A208.1 (1979) requires that phenol-formaldehydebonded flakeboards and waferboards have a minimum average internal bond strength of 50 lb/in.<sup>2</sup>. Both the treated and control samples exceeded this value. These results indicate that the chemical treatment did not adversely affect the adhesive bond strength.

#### CONCLUSIONS

The dimensional stability of aspen flakeboards, as measured by the thickness swell and water absorption tests, can be greatly improved by acetylating the flakes with acetic anhydride.

Vapor-phase acetylation of aspen flakes gave permanent weight gains equal to those obtained when liquid-phase acetylation was used.

The internal bond strengths of the acetylated samples were at least as good as control values, and both exceeded minimum requirements of the Specification ANSI A208.1 (1979) for mat-formed particleboard.

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