DIMENSIONAL STABILITY OF FLAKEBOARDS MADE FROM ACETYLATED PINUS RADIATA HEARTWOOD OR SAPWOOD FLAKES

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ABSTRACT

Pinus radiata D. Don heartwood and sapwood flakes were acetylated with acetic anhydride in the absence of solvent or catalyst by a simple dip procedure. No difference in reagent penetration or reactivity was seen between heartwood and sapwood flakes. Acetylation weight gains of 13% to 19% were achieved with both types of flakes in 1 to 3 hours.

Flakeboards made from acetylated heartwood or sapwood flakes swelled in liquid water tests at a slower rate and to a lesser extent than control boards. The equilibrium moisture content for flakeboards made from acetylated flakes was lower at each relative humidity tested and these boards swelled less in humid air than control boards made from untreated flakes.

Keywords: acetylation; chemical modification; flakeboard; swelling; dimensional stability; equilibrium moisture content; **Pinus radiata**.

INTRODUCTION

Flakeboards made of acetylated hardwood or softwood flakes have been shown to be more dimensionally stable and more resistant to attack by brown-, white-, and soft-rot fungi and tunnelling bacteria than boards made from untreated flakes (Rowell 1984; Rowell, Tillman, & Liu 1986; Rowell, Tillman, & Simonson 1986; Rowell, Esenther, Nicholas, & Nilsson, in press; Youngquist *et al.* 1986a, b). A new procedure to acetylate wood flakes, particles, and fibres has been developed which eliminates the use of catalysts or organic co-solvents (Rowell, Tillman, & Simonson 1986, and in press). The procedure has been applied mainly to mixed heartwood and sapwood flakes of pine and aspen.

It has long been known that *Pinus radiata* heartwood is impermeable to aqueous solutions of preservatives and other chemicals. This has been attributed to the high content of resinous extractives in the heartwood as well as to the heartwood structure (Hedley 1982).

The purpose of the research reported here was to (1) determine the reactivity of *P. radiata* sapwood and heartwood flakes to acetylation and (2) make flakeboards of acetylated sapwood and heartwood flakes and determine the rate and extent of swelling in liquid water and in water vapour.

EXPERIMENTAL

Wood Flakes

Pinus radiata (100 × 100 mm) sawn timber was selected from the green chain at the Waipa Sawmill, Rotorua, and boards were identified as sapwood or heartwood by chemical spot-testing. The green sapwood had an average basic density of 446 kg/m^3 and a moisture content of 109%. The green heartwood had an average basic density of 389 kg/m^3 and a moisture content of 28%. The material was cut into shorter lengths and converted into flakes using a Bezner disc-type flaker and then screened so that material less than 26 mm in length was excluded. Flakes were air-dried and then oven-dried for 12 hours at 105°C before use. Flake size was approximately (thickness × length × width, in millimetres) 1 × 40 × random. Flake width varied from 10 to 20 mm.

Reaction of Flakes with Acetic Anhydride

The oven-dried flakes (200 g) were acetylated by a simple dip procedure as described previously (Rowell, Tillman, & Simonson 1986, and in press), dipping in acetic anhydride for 1 minute and draining for 3 minutes at 25°C. The container with the flakes was placed in a preheated (120°C) stainless steel reactor for various lengths of time. After the reaction time was completed, a vacuum was applied to the cylinder (750 mm Hg) for 2 hours at 120°C. The flakes were then oven-dried at 105°C for 12 hours. The weight percentage gain (WPG) due to acetylation was calculated based on the weight of oven-dried (o.d.) unreacted flakes.

Leaching and Acetyl Content of Acetylated Flakes

Weighed oven-dried flakes, both acetylated and untreated, were leached under two conditions: (1) 14 days in distilled water at 24°C (changing water every 24 hours), or (2) 2 hours in a Soxhlet extractor with refluxing toluene/ethanol (1/1 v/v). After re-drying, oven-dry weight loss was determined.

Acetyl content was obtained before and after leaching by gas chromatography following deacetylation of ground and mixed samples with sodium hydroxide solution. This is a standard U.S. Forest Products Laboratory procedure.

Flakeboard Preparation

Acetylated or untreated flakes (180 g o.d.) were pressed into boards approximately 12.5 × 150 × 150 mm in size with a target density of 640 kg/m^3 and a resin solids content of 6% (based on o.d. flakes). The adhesive used was a 43.5% aqueous solution of a phenol-formaldehyde resin. No catalyst or wax was added. The mat moisture content was 12% to 13%. Pressing time was 10 minutes at 177°C.

Each flakeboard was lightly sanded on both surfaces and four pieces (50 × 50 mm) were cut out, oven-dried, and weighed. The thickness was measured at the centre point of each specimen and subsequent measurements were taken at the same point.

Water Swelling Rate Tests

Each test specimen was placed in a 100 × 100-mm container, 50 mm deep. The container was on a flatbed micrometer for the thickness measurements. Water at 20°C was added to the container and the thickness was recorded as a function of time. Measurements were taken every 5 minutes for the first hour, every hour for the first 6 hours, then once a day for 5 days. All water and humidity tests were done in duplicate.

Water Soaking Tests

Cyclic water soaking tests were run as previously described (Rowell & Ellis 1978). Each of six cycles consisted of water soaking for 5 days followed by oven-drying at 105°C for 2 days. Thickness swelling was calculated as a percentage of the original thickness (o.d. board). After each cycle, the specimens were reweighed. The over-all weight loss was determined from the original and final oven-dried weights.

Humidity Tests

Oven-dried specimens were placed in constant humidity rooms at 30%, 65%, 80%, and 90% relative humidity (RH) and 27°C. After 21 days the specimens were weighed to determine the equilibrium moisture content (EMC). Previous work showed that EMC for control and acetylated boards was reached at 14 days (Rowell, Tillman, & Liu 1986).

Separate specimens were placed in a humidity room at 30% RH and 27°C. Thickness was determined after 21 days. The specimens were then placed in a humidity room at 90% RH and 27°C for another 21 days, whereafter thickness was determined. This procedure was repeated for a total of four cycles of 30% to 90% RH. The specimens were then oven-dried and thickness measured. Changes in thickness were calculated as a percentage of the original thickness (o.d. board).

RESULTS AND DISCUSSION

Weight percentage gain due to acetylation of pine heartwood and sapwood flakes as a function of time is shown in Fig. 1. Although not statistically analysed, the results indicate little difference between heartwood and sapwood flakes in the degree of acetylation for each reaction time tried. All data points seem to fit one curve.

Effects of water leaching of control and acetylated heartwood and sapwood flakes at 24°C for 14 days and refluxing for 2 hours in toluene/ethanol are shown in Table 1. Control flakes lost more weight in both tests than did acetylated flakes. There was a very small loss of acetyl in the acetylated flakes showing that whatever is extracted from the flakes has about the same acetyl content as the remaining wood.

The rate and extent of thickness swelling of pine flakeboard in liquid water is shown in Fig. 2. During the first 60 minutes, sapwood control boards swelled faster than heartwood control boards. This trend continued through 6 hours of water swelling, but at the end of 5 days both control sapwood and heartwood boards had swelled to the same extent (50% to 55%). Both sapwood and heartwood flakeboards made from acetylated flakes swelled at about the same rate and to the same extent during the 5-day test. Both boards swelled only about 7% in thickness during this time.

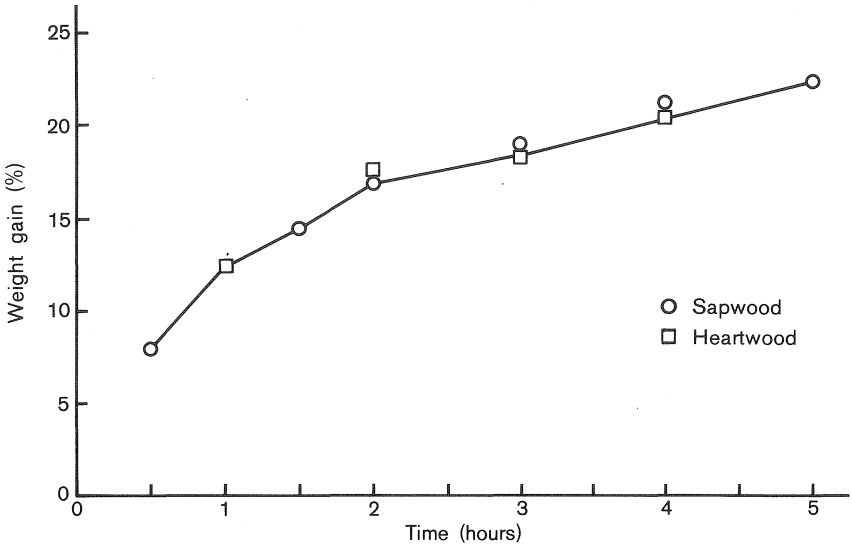

FIG. 1—Degree of acetylation of pine flakes as a function of reaction time (120°C).

TABLE 1—Weight loss and acetyl content of control and acetylated *Pinus radiata* flakes before and after leaching in various solvents*

Wood sample	Acetylation weight percentage gain	Acetyl content before leaching (%)	After leaching in			
			Water 14 days 24°C		Toluene/ethanol 1/1 v/v reflux	
			Weight loss (%)	Acetyl content (%)	Weight loss (%)	Acetyl content (%)
Sapwood						
Control	0	1.5	5.1	1.6	1.0	1.5
Acetylated	21.7	20.8	4.5	19.8	3.8	20.3
Heartwood						
Control	0	1.2	6.1	1.3	4.2	1.2
Acetylated	20.5	20.3	3.5	19.4	3.2	18.7

* Average of three specimens

Thickness changes in the repeated water soaking/oven-drying tests (Fig. 3) showed that the flakeboards made from sapwood and heartwood flakes were about the same. Both underwent maximum thickness swelling at the sixth water cycle of 64% to 67%. Considerable reductions in thickness swelling were observed in flakeboard made from acetylated heartwood and sapwood flakes. Again, very little difference was seen between acetylated heartwood and sapwood boards. Acetylated boards underwent maximum thickness swelling at the sixth water cycle of 22% to 23%. For all board specimens, the increase in thickness swelling was greatest in the first four cycles; only a small increase in thickness swelling occurred in the last two cycles.

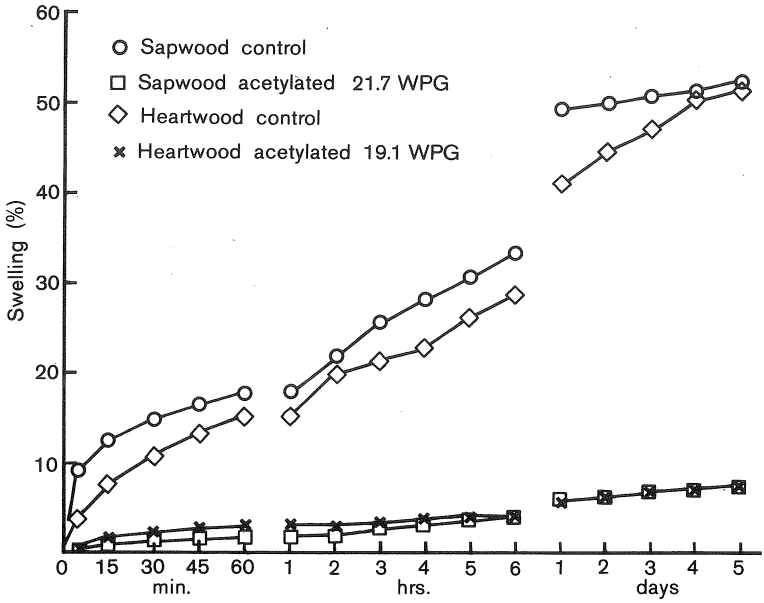

FIG. 2—Rate of swelling in liquid water of pine flakeboard made from control (untreated) or acetylated flakes.

The EMC for control flakeboards made from sapwood and heartwood flakes was the same at each RH tested (Table 2). The EMC for flakeboards made from acetylated sapwood and heartwood flakes was also about the same, and much lower than control boards. Acetylation reduced the EMC by about 40% at each RH tested.

The thickness change at 30% and 90% RH of boards made from control and acetylated flakes is illustrated in Fig. 4. Both sapwood and heartwood boards made from control flakes swelled about the same, with a maximum of about 28%. Boards made from acetylated sapwood and heartwood flakes swelled much less than control boards, with a maximum of about 6%.

CONCLUSIONS AND FUTURE WORK

No difference in reactivity of *P. radiata* sapwood or heartwood flakes was found during acetylation with acetic anhydride. Flakeboards made from acetylated sapwood or heartwood flakes showed similar rates and extents of swelling in liquid water and in humidity tests. Flakeboards made from acetylated flakes swelled much more slowly and to a lesser extent in liquid water tests and in humidity tests than untreated control boards. Equilibrium moisture content of boards made from acetylated sapwood and heartwood flakes was generally about 40% lower than that of control boards. In summary, *P. radiata* behaves similarly to other softwoods examined in earlier work.

Research is presently under way to test strength properties in larger boards made in an experimental laboratory press. Biological tests are under way to determine both weight loss and strength loss during fungal attack.

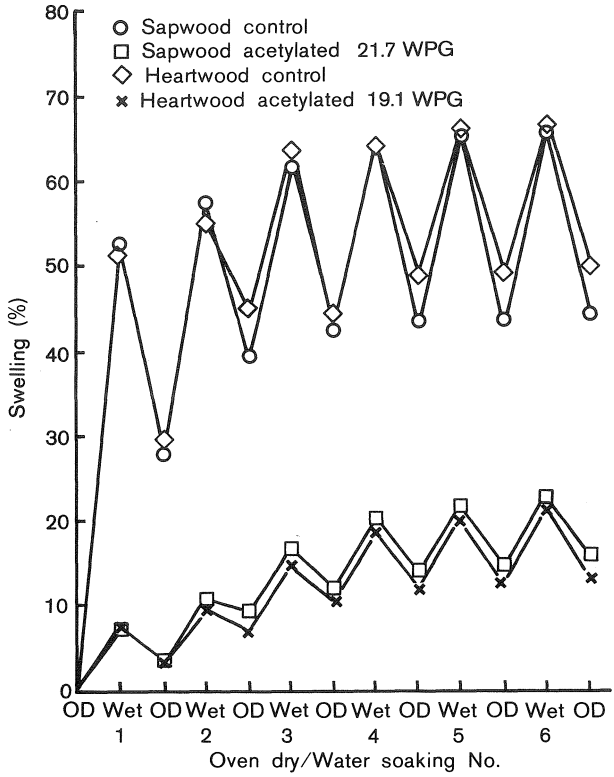

FIG. 3—Changes in thickness of pine flakeboard made from control (untreated) or acetylated flakes, during repeated water swelling tests.

TABLE 2—Equilibrium moisture content (%) of flakeboards made from control or acetylated *Pinus radiata* flakes at various relative humidities (27°C)*

Wood sample	Acetylation weight percentage gain	Equilibrium moisture content at			
		30% RH	65% RH	80% RH	90% RH
Sapwood					
Control	0	4.2	8.5	13.8	17.3
Acetylated	21.7	2.2	4.9	8.7	10.9
Heartwood					
Control	0	4.1	8.0	13.8	16.2
Acetylated	19.1	2.3	5.2	9.0	11.5

* Average of three specimens

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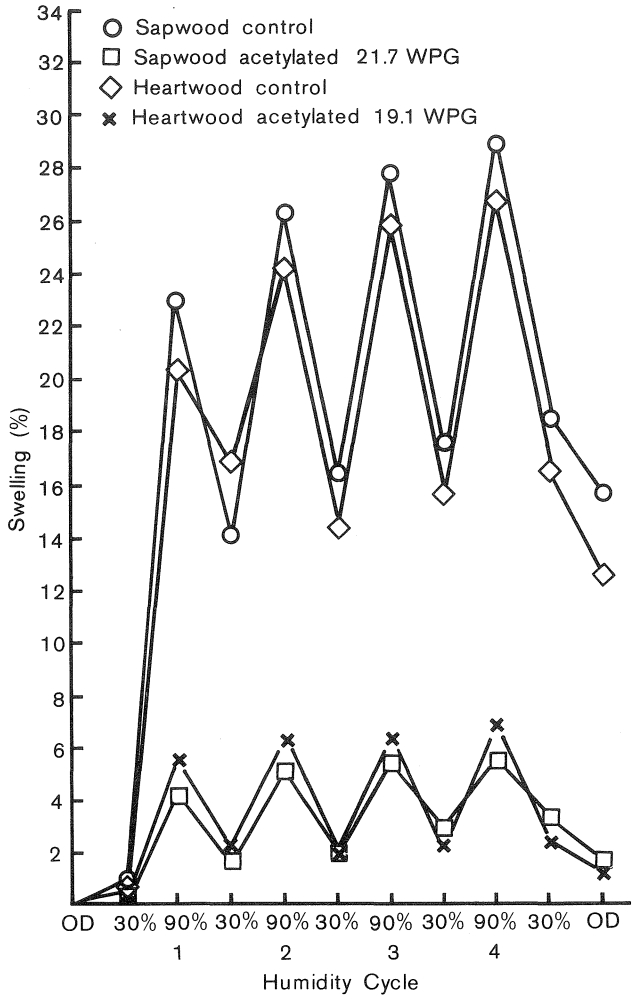

FIG. 4—Changes in thickness of pine flakeboards made from control (untreated) or acetylated flakes, at 30% and 90% RH.

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