

# Fungal Resistance of Pine Particle Boards Made from Various Types of Acetylated Chips

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## Keywords

Acetylation  
Particle board  
Fungal Resistance  
Fungus Cellar Tests  
*Pinus sylvestris*

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## Summary

Four different non-catalyzed methods were used to acetylate pine wood chips to various levels of acetyl weight gains; vapor phase acetylation using acetic anhydride, vapor phase acetylation with ketene gas, liquid phase acetylation using refluxing xylene/acetic anhydride, and liquid phase acetylation using a limited amount of acetic anhydride. Particle boards made from the different types of chips were evaluated with respect to their resistance to fungal attack when placed in unsterile soil in a fungus cellar. White, brown and soft rot fungi, and tunneling bacteria were present in the soil. Boards made from chips acetylated in a liquid phase procedure, using either a limited amount of acetic anhydride or a refluxing mixture of xylene and acetic anhydride, were the most resistant to attack. At acetyl weight gains of about 15% of the chips, both types of boards were not attacked after 12 months. When chips were acetylated using vaporous acetic anhydride or ketene gas, the resulting boards were less resistant.

## Introduction

A large amount of research has been conducted to modify wood by chemical means and thereby improve such properties as dimensional stability, biological resistance, color stability and hardness, and decrease water sorption (cf. Rowell 1984). Acetylation is a chemical treatment that has been much investigated, and different acetylation procedures have been reported. Many require a catalyst which complicates the recovery of chemicals and may cause wood degradation.

Four different non-catalyzed methods used to acetylate pine wood chips have been investigated. The methods were: vapor phase acetylation using acetic anhydride (Rowell *et al.* 1986a, 1986d), liquid phase acetylation using a refluxing mixture of xylene and acetic anhydride (Rowell *et al.* 1986c), vapor phase acetylation with ketene gas (Rowell *et al.* 1986f), and liquid phase acetylation using a limited amount of acetic anhydride (Rowell *et al.* 1986b, 1986e). Particle boards made from acetylated chips prepared by the four methods showed greatly improved dimensional stability in both water soaking tests and when sub-

jected to humid air. The fungus cellar tests on these particle boards have now been completed.

While much research has been done on resistance to fungal attack of acetylated solid wood, very little has been done on reconstituted wood products. In 1978, Bekere *et al.* showed that fiber boards made from acetylated fibers were resistant to attack by *Coniophora cerebella*. Arora *et al.* (1981) found particle boards resistant to “commonly occurring Indian wood destroying fungi.” The particles were acetylated with acetic anhydride using pyridine as a catalyst. Yin and Wang (1983) reported that poplar plywood made from veneers acetylated with vaporous acetic anhydride, the reaction being catalyzed with potassium acetate, showed no attack by *Lenzites trabea*. More recently, Nishimoto and Imamura (1985) made particle boards from mixtures of unmodified and acetylated spruce chips. Soil block tests with *Tyromyces palustris* or *Coriolus versicolor* resulted in very slow decay in boards containing 50% by weight of acetylated chips, and no decay in boards made from acetylated chips only.

The purpose of the research reported here was

twofold: first, to determine if the standard fungus cellar test used for solid wood blocks could be used directly on particle boards made with melamine-urea-formaldehyde adhesive, without the adhesive having a toxic effect on the soil micro-organisms. Second, to then use the fungus cellar test to evaluate the rot resistance of particle boards made from different types of acetylated chips.

## Experimental Procedures

### Wood Chips

Commercial pine wood chips (*Pinus sylvestris*) which contained a small amount of bark were used. Before use, the chips were passed over a 3 × 3 mm screen to remove fines, and oven-dried at 105°C. The chips had an average thickness of about 1 mm and a maximum length of 25 mm.

### Acetylation of Chips and Particle Board Making

Four different procedures described earlier were used to acetylate oven-dried pine wood chips to various levels of acetyl weight gains: vapor phase acetylation using acetic anhydride (Rowell *et al.* 1986a, 1986d), liquid phase acetylation using refluxing xylene/acetic anhydride (Rowell *et al.* 1986c), vapor phase acetylation with ketene gas (Rowell *et al.* 1986f), and liquid phase acetylation using a limited amount of acetic anhydride (Rowell *et al.* 1986b, 1986e). The weight gain was calculated as a percent by weight based on oven-dried, unmodified chips.

Particle boards 10 × 175 × 175 mm in size were made from unmodified (control) chips as well as from each type of acetylated chips using 8% melamine-urea-formaldehyde adhesive as described earlier (Rowell *et al.* 1986a). Each board had 20 mm removed from all sides, was lightly sanded and cut into 10 × 60 × 30 mm pieces.

### Fungus Cellar Tests

Fungus cellar tests were run as previously described (Nilsson and Rowell 1982). Solid pine wood blocks (10 × 25 × 50 mm) and control particle boards were incubated at approximately 25°C in moist unsterile soil. The specimens were inspected each month and the degree of attack was rated. Among the micro-organisms present in the soil were white, brown and soft rot fungi, and tunneling bacteria.

Particle boards made from acetylated chips and control boards were also tested in the fungus cellar and inspected periodically. After the test was completed (12 months), sections were cut for microscopic examination.

## Results and Discussion

Fungus cellar tests were conducted to determine if the melamine-urea-formaldehyde adhesive would have a toxic effect on the soil micro-organisms and thereby prevent or delay the decay of the wood particles compared to that of a solid wood block. From Table 1 it can be seen that both solid pine wood blocks and unleached control particle boards were attacked at the same rate. Both kinds of samples were destroyed in 6 months. The result also indicates that it is not necessary to water-leach particle board specimens prior to the fungus cellar test.

Table 2 shows the results of a 12 month fungus cellar test on particle boards made from control and various

Table 1. Fungus cellar test on solid pine wood and particle boards made from pine chips and 8% melamine-urea-formaldehyde adhesive.

Sample type	Rating <sup>a)</sup> after months				
	2	3	4	5	6
Solid wood	1	2	3	3	4
Particle board	S/1	S/2	S/2	S/3	S/4

<sup>a)</sup> Rating system: 0—no attack, 1—slight attack, 2—moderate attack, 3—heavy attack, 4—destroyed, S—swollen. Average of three specimens

types of acetylated chips, respectively. In all particle boards it was observed that no biological attack occurred before swelling of the specimen. It is not known if the swelling is a result of water wetting or the initial enzymatic attack. In all specimens that were degraded, tunneling bacteria were the first organisms to attack. Specimens which were heavily attacked were degraded by tunneling bacteria and brown and soft rot fungi.

Table 2 also shows that at low levels of vapor phase acetylation with acetic anhydride (6.7% weight gain), the acetylated board was degraded faster than was the control board. This may be due to the presence of sugars which were generated during acetylation by hydrolysis and not leached out (Rowell *et al.* 1986a). A similar result can also be noticed for the boards made from ketene acetylated chips, where the board made from chips with 8.5% weight gain was degraded at a rate comparable to that found for the controls

Particle boards made from chips impregnated and acetylated with a limited amount of acetic anhydride were resistant to attack at weight gains of about 18%. No swelling or evidence of attack could be seen at these high levels of acetylation.

Boards made from chips acetylated in refluxing acetic anhydride/xylene with 12% weight gain showed results similar to those found for boards made from vapor phase acetylated chips with 18.3% weight gain. At 23% weight gain, boards made from liquid phase (acetic anhydride/xylene) acetylated chips were not attacked during the test.

Boards made from ketene acetylated chips, even at the highest levels of acetylation, were not resistant to attack. These boards were made from Southern pine wood using 6% phenol-formaldehyde adhesive. Southern pine and *Pinus sylvestris* are degraded at the same rate in the fungus cellar, and the type of adhesive used does not seem to influence the fungal degradation (Rowell *et al.* 1987). Lack of resistance may be due to the fact that even though the weight gain was 17% (cf. Table 2), chemical analysis of the chips

Table 2. Fungus cellar test on pine particle boards made from unmodified and acetylated chips with 8% melamine-urea-formaldehyde adhesive.

Type of acetylation	Weight gain %	Rating <sup>a)</sup> after months							
		2	3	4	5	6	8	10	12
None (control)	0	S/1	S/2	S/3	S/3	S/4	-	-	-
Ketene <sup>b)</sup>	8.5	S/1	S/1	S/2	S/2	S/3			S/4
	17.0	0	0	S/1	S/1	S/2			S/3
Vapor phase acetic anhydride	6.7	S/0	S/3	S/3	S/4	-	-	-	-
	10.9	0	0	S/0	S/2	S/2	S/2	S/3	S/4
	18.3	0	0	0	0	0	0	S/0	S/0
	21.1	0	0	0	0	0	0	0	0
Limited amount of of liquid acetic anhydride	6.3	S/0	S/0	S/1	S/1	S/2			S/2
	10.7	S/0	S/1	S/1	S/1	S/2			S/2
	13.8	0	S/0	S/0	S/0	S/0			S/0
	18.2	0	0	0	0	0			0
	22.0	0	0	0	0	0			0
Refluxing acetic anhydride/xylene	12.0	0	0	0	0	0	0	S/0	S/0
	23.0	0	0	0	0	0	0	0	0

<sup>a)</sup> See Table 1 for rating system.

<sup>b)</sup> Southern pine, 6% phenol-formaldehyde adhesive.

showed an acetyl content of only 10.3% (Rowell *et al.* 1986f).

Particle boards made from chips impregnated and acetylated with a limited amount of acetic anhydride were resistant to attack at weight gains of about 18%. No swelling or evidence of attack could be seen at these high levels of acetylation.

The mechanism of biological resistance in acetylated wood is not completely understood. However, the resistance is believed to be due to two factors; greatly decreased moisture sorption in the cell wall and blocking of the hydroxyl groups of the wood component polymers in such a way that the enzymes cannot recognize them as attackable substrates (Rowell 1982, 1984). The equilibrium moisture content of acetylated chips is much lower than that of the control chips. Comparing the various types of acetylated chips, the equilibrium moisture contents (EMC) for specimens with equivalent weight gains were in the order; EMC of chips acetylated with ketene >> EMC of chips acetylated with vaporous acetic anhydride > EMC of chips acetylated with a limited amount of liquid acetic anhydride > EMC of chips acetylated in refluxing acetic anhydride/xylene (Rowell *et al.* 1986a-f).

The acetyl content determined by analysis of the various types of acetylated chips at equivalent weight gains vary in reversed order. That is, chips acetylated with ketene have a much lower acetyl content than the weight gain would indicate while chips acetylated with vaporous acetic anhydride have an acetyl content corresponding to the weight gain, and liquid phase acety-

lated chips have a slightly higher acetyl content than what could be expected from the weight gain (Rowell *et al.* 1986a-f). Additionally, the cell wall regions and polymers reacted in the four different acetylation methods might differ, resulting in different properties of the chips and the boards.

The carbohydrate polymers are the most susceptible to biological attack with the hemicelluloses the most accessible and hygroscopic of the cell wall polymers. If the first step in fungal degradation of wood is attack of the hemicelluloses, acetylation of this fraction may be the key to protection against biological degradation by chemical modification.

#### Conclusions

- Of the four methods of acetylation studied, pine particle boards made from ketene modified chips were the least resistant to biological attack in the fungus cellar.
- Vapor phase acetylation with acetic anhydride is more effective than ketene modification but not as effective as either acetylation with a limited amount of liquid acetic anhydride or liquid phase acetylation with refluxing xylene/acetic anhydride.
- Acetylation with a limited amount of liquid acetic anhydride is the preferred method of acetylation since no catalysts or organic cosolvents are used and the biological resistance of the resulting boards is nearly equivalent to that for boards of chips treated with the more complicated method using refluxing xylene/acetic anhydride.

## Acknowledgements

The authors wish to thank Byggelit-KP, Sweden for supplying the pine chips and for the use of their experimental particle board press. The Swedish National Board for Technical Development and the Swedish Council for Building Research are gratefully acknowledged for financial support.

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