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ORIGINAL ARTICLE

Understanding decay resistance, dimensional stability and strength changes in heat-treated and acetylated wood

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Abstract
Reductions in hygroscopicity, increased dimensional stability and decay resistance of heat-treated wood depend on decomposition of a large portion of the hemicelluloses in the wood cell wall. In theory, these hemicelluloses are converted to small organic molecules, water and volatile furan-type intermediates that can polymerize in the cell wall. Reductions in hygroscopicity and improved dimensional stability of acetylated wood depend on esterification of the accessible hemicelluloses in the cell wall reducing hydrogen bonding with water and bulking the cell wall back to its green volume. Stability is not 100% since the water molecule is smaller than the acetyl group so water can access hydroxyl sites even when the wood is fully acetylated. The cell-wall moisture content is too low in acetylated wood to support fungal attack so the initial enzymic attack starting the colonization does not take place. Strength properties are reduced in heat-treated wood owing to the degradation of the cell-wall matrix resulting from the hemicellulose loss. Strength properties are not significantly changed in acetylated wood and acetylation results in greatly improved wet strength and wet stiffness properties.

Keywords: *Acetylation, acetyl content, brown-rot fungus, equilibrium moisture content, heat treatment, mechanism, sugar analysis, weight loss, wood.*

Introduction

With an increased awareness of the fragility of our environment and the need for durability in wood products, new technologies have been developed to increase the service life of wood materials without the use of toxic chemicals. There are issues of sustainability, carbon sequestration and performance converge in this search for environmentally friendly methods of wood preservation.

Heat treatments of wood and chemical modification using acetic anhydride to acetylate wood have been studied for many years and are now used commercially. Both methods result in increased decay resistance and improved dimensional stability to different degrees. Strength properties of the wood are also affected, mainly by heat treatments.

Processes

Heat treatments

Heating wood to improve performance dates back many thousands of years. The Kalvträsk wooden ski was bent using heat by the Saami and used in northern Sweden over 5200 years ago (Åström, 1993; Insulander, 1999). Hieroglyphic pictures of furniture in Egypt between 2500 and 3000 BC show chairs made using heat-bent wooden members (Rivers & Umney, 2005). The Egyptians also bent wood for bows using hot water, around 1900 BC, as shown on tomb paintings (Ostergard, 1987). In the eleventh century, Viking shipbuilders also bent wood using heat for parts of the ship (Olsen & Crumlin-Pedersen, 1967). In the Norwegian stave churches, large beams were bent using heat and joined (Bugge,

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1953; Holan, 1990). Finally, wooden cask-makers knew how to heat-bend wood 2000 years ago (Twede, 2005). A picture of a wooden cask is shown on the wall of an Egyptian tomb from 2690 BC (Kilby, 1971). Wooden casks were also made by the Romans to store wine (Twede, 2005).

In the early part of the twentieth century it was found that drying wood at high temperature increased dimensional stability and reduced hygroscopicity (Tiemann, 1915; Stamm & Hansen, 1937). Later, it was found that heating wood in molten metal at temperatures between 140 and 320°C reduced swelling in Sitka spruce by 60% and also increased resistance to microbiological attack (Stamm *et al.*, 1946). They also found that the increase in stability and durability also increases brittleness and loss in some strength properties including impact toughness, modulus of rupture and work to failure. The treatments usually cause a darkening of the wood and the wood has a tendency to crack and split.

Burmester (1973) studied the effect of temperature, pressure and moisture on wood properties. He found that the optimum conditions for pine were 160°C, 20–30% moisture and 0.7 MPa pressure. He reported high dimensional stability and resistance to brown-rot fungi attack and minimal loss of strength. He named his process FWD (Feuchte-Wärme-Druck).

More recently, Norimoto *et al.* (1993) and Ito *et al.* (1998) heated wet wood to improve dimensional stability. Inoue *et al.* (1993) heated wet wood to fix compression set in wood.

Several names have been given to the various heat-treated products and treatments for wood, including Staypak and Stabwood in the USA (using dry wood), Lignostone and Lignofol in Germany (using dry wood), Jicwood and Jablo in the UK, Thermo-Wood in Finland (using water vapour), Plato in The Netherlands (using water), Perdure in Canada (using steam), Oil Heat Treated (OHT) in Germany (using oil) and New Option Wood/Retification in France (using nitrogen) (Hill, 2006).

Acetylation

The acetylation of wood was first performed in Germany by Fuchs (1928), using acetic anhydride and sulfuric acid as a catalyst. Fuchs found an acetyl weight gain of over 40%, which meant that he decrystallized the cellulose in the process. He used the reaction to isolate lignin from pine wood. In the same year, Horn (1928) acetylated beech wood to remove hemicelluloses in a similar lignin isolation procedure. Suida and Titsch (1928) acetylated powdered beech and pine using pyridine or dimethylaniline as a catalyst to yield an acetyl weight gain of 30–35% after 15–35 days at 100°C. Tarkow (1945) first demonstrated that acetylated balsa was resistant to decay. Tarkow (1946) first described the use of wood acetylation to stabilize wood from swelling in water. Since the 1940s, many laboratories around the world have looked at acetylation of many different types of woods and agricultural resources.

Acetylation of wood using acetic anhydride has mainly been done as a liquid phase reaction (Rowell *et al.*, 1986; Rowell, 2005). The early work used acetic anhydride catalysed with either zinc chloride or pyridine. Over the years, many other catalysts have been tried with both liquid and vapor systems. Some of the catalysts used include urea–ammonium sulfate, dimethylformamide, sodium acetate, magnesium persulfate, trifluoroacetic acid, boron trifluoride and gamma-rays. The reaction has also been done without a catalyst and using an organic co-solvent. Gas-phase reactions have also been reported using both ketene and acetic anhydride; however, the diffusion rate is very slow so this technology has only been applied to thin veneers (Rowell, 2005). Most acetylation reactions today do not use a catalyst.

Acetylated wood is now sold by Titanwood in Arnhem, The Netherlands. It is sold under the name of Accoya® and is available for several species of wood in Europe, North America and Asia.

Chemistry

Heat-treated wood

Various thermal modification processes have been developed. The results of the process depend on several variables including time and temperature, treatment atmosphere, wood species, moisture content, wood dimensions and the use of a catalyst (Stamm, 1956; Millett & Gerhards, 1972). Temperature and time of treatment are the most critical elements and treatments in air result in oxidation reactions not leading to the desired properties of the treated wood. Wood degrades more rapidly when heated in either steam or water compared with dry conditions (MacLean, 1951, 1953; Millett & Gerhards, 1972; Hillis, 1975). Stamm (1956) showed that wood heated in the presence of oxygen degraded more rapidly that wood heated in an oxygen-free atmosphere. As the treatment time increases, embrittlement of the wood increases (Yao & Taylor, 1979; Edlund, 2004). In general, weight loss occurs to a higher extent in hardwoods than in softwoods, which may be due to the higher content of acetyl groups in hardwoods releasing more acetic acid during heat treatment contributing

to acid hydrolysis (MacLean, 1951, 1953; Millett & Gerhards, 1972; Hillis, 1975).

As the wood is heated, the first weight loss is due to the loss of water, followed by a variety of chemistries that produce degradation products and volatile gases (Shafizadeh & Chin, 1977). As the temperature increases, wood cell-wall polymers start to degrade. Pyrolysis of the hemicelluloses takes place at about 270°C, followed closely by cellulose. Lignin is much more stable at high temperatures (Stamm, 1956). In the early stages of degradation, 83% of the weight loss is water (Mitchell *et al.*, 1953). The hemicellulose polymers are the first to degrade, followed by cellulose, resulting in the formation of furans such as furfural and hydoxymethyl furfural (Figure 1). The hemicelluloses and cellulose are also degraded to internal ethers and other rearrangement products. The optimum amount of water that can be lost is three molecules of water from two anhydroglucose units, or 16.7% of the carbohydrate polymers or 12% of the wood (Stamm & Baechler, 1960).

Table I shows the weight loss resulting from heating wood at different temperatures in the presence of moisture. As the temperature increases, so does the weight loss.

Table II shows the sugar analysis before and after treating aspen with high-pressure steam (220°C) for 8 min. The data show that in the very early stages of weight loss, the hemicellulose polymers are breaking down and cellulose remains unchanged. Over half of the arabinan and rhamnans are lost during this initial heating.

Table I. Weight loss due to heating wood in the presence of moisture.[a]

Heating temperature (°C)	Time (h)	Weight loss (%)
120	2	2–3
120	10	5
180	10	10–12
190	2	4–7
190	12	5–10
200	10	10–17
220	1	6–7
235	4	25
240	0.5	7

Note: [a]Data taken from several sources.

Acetylated wood

The reaction of acetic anhydride with wood results in esterification of the accessible hydroxyl groups in the cell wall, with the formation of acetic acid as a by-product. The by-product acid must be removed from the product as the human nose is quite sensitive to the odour of acetic acid. While this is easily done in the case of wood particles and fibre, it is somewhat difficult to do in solid wood.

Acetylation is a single-addition reaction, which means that one acetyl group is on one hydroxyl group with no polymerization:

$$\mathrm{WOOD-OH + CH_3-C(=O)-O-C(C=O)-CH_3 \rightarrow WOOD-O-C(=O)-CH_3 + CH_3-C(=O)-OH}$$

Acetic anhydride — Acetylated wood — Acetic acid

Thus, all the weight gain in acetyl can be directly converted into units of hydroxyl groups blocked. This is not true for a reaction where polymer chains are formed (epoxides and isocyanates, for example). In these cases, the weight gain cannot be converted into units of blocked hydroxyl groups.

Isolated lignin reacts more quickly with acetic anhydride compared with hemicelluloses and holocellulose (Kumar & Agawal, 1983; Rowell *et al.*, 1994). Kumar and Agawal (1983) reported that at an acetyl weight percentage gain of 13.5, 86.4% of the lignin was acetylated, 21.6% of the hemicelluloses and 9.3% of the cellulose. Reacting wood at 120°C with acetic anhydride and no catalyst, at an acetyl weight gain of 16–19%, theoretically about 90% of the lignin is esterified, and 25% of the holocellulose (Rowell, 1982). It is assumed that 100% of the hemicellulose hydroxyl groups are substituted and no cellulose hydroxyl substituted.

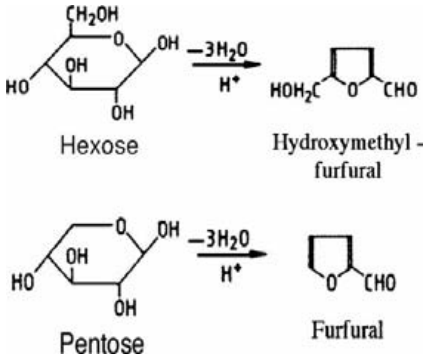

Figure 1. Formation of hydroxymethyl furfural from hexoses and furfural from pentoses as a result of heat treatments of wood.

Table II. Sugar analysis of aspen before and after heating at 220°C for 8 min.[a]

Weight loss (%)	Total carbohydrate (%)	Arabian (%)	Galactan (%)	Rhamnan (%)	Glucan (%)	Xylan (%)	Mannan (%)
0	61.8	0.56	0.68	0.30	42.4	16.3	1.6
2.2	59.2	0.20	0.58	0.14	42.2	14.6	1.5

Note: [a]New data from the authors not published before.

A small amount of hydroxyls may be esterified on the surface hydroxyls in the amorphous regions of cellulose. This conclusion is based on the observation that pure cotton cellulose cloth can be used to hold wood fibre and no weight gain is observed in the cotton cloth after several acetylation reactions.

Moisture and dimensional stability

Heat-treated wood

Because of the loss of hygroscopic hemicellulose polymers during heat treatment, the equilibrium moisture content (EMC) is reduced. Dimensional stability of heat-treated wood was first thought to be due to a cross-linking of cellulose chains (Stamm & Hansen, 1937). The furan intermediates (Figure 1) that form from the decomposition of the hemicelluloses have poor hygroscopicity and have little effect on the EMC.

Table III shows a summary of recent results from various authors on EMC resulting from heat treatments. In most cases, a 50% reduction in EMC is observed. In most examples given, the process was done in a vapor atmosphere between 190 and 220°C with a relative humidity of 35–65%.

Table IV shows the reduction in swelling of wood that has been heat treated.

Acetylated wood

Acetylation of wood reduces the number of hydroxyl groups that can sorb moisture by hydrogen bonding so the EMC and fibre saturation point (FSP) are reduced and the dimensional stabilization increases with increasing weight gain due to acetylation (Rowell, 2005). Table V shows the reduction in EMC and FSP and the antishrink efficiency (ASE) as a result of acetylation. Table V shows the reduction in EMC and Table VI shows the antishrink efficiency for different species.

Decay resistance

Heat-treated wood

As dimensional stability increases and hygroscopicity decreases in heat-treated wood, resistance to decay also increases (Stamm *et al.*, 1946; Stamm & Bacchler, 1960). Table VII shows the data of Stamm *et al.* (1946). A reduction in swelling over 40% results in no weight loss in the decay test. Similar results were obtained when the test fungus was *Lenzites trabea* 517 (Stamm & Bacchler, 1960).

Table VIII shows fungal resistance of two types of heat-treated wood (Rapp & Sailer, 2001). Pine (*Pinus sylvestris*) and spruce (*Picea abies*) were heated in oil or air at three different temperatures and the treated blocks were tested for decay for 19 weeks in the EN 113 (European Committee for Standardization, 1996) decay test using *Coniophora puteana* fungus. The data show that heating in oil is more effective at reducing attack by fungi than heating in air. This may be due to the presence of the oil in the samples.

Welzbacher and Rapp (2007) compared different types of industrially heat-treated products using several different fungi in laboratory tests and in different field and compost conditions. Table IX shows the weight loss during an EN 113 test using three different fungi. The heat treatment in oil was the most effective but the effect of the oil in the decay test is not known (Welzbacher & Rapp, 2007).

Resistance to decay in heat-treated wood is probably due to the loss of hemicellulose polymers in the cell wall. As shown earlier, the more weight loss in the heating process, the more durable the heat-treated wood.

Table III. Reduction in equilibrium moisture content (EMC) as a result of heating wood.

Wood species	Conditions	Reduction in EMC (%)	Reference
Pinus pinaster	190°C, 8–24 h	50	Esteves *et al.* (2006)
Pinus sylvestris	220°C, 1–3 h	50	ThermoWood Handbook (2003)
Eucalyptus globulus	190°C, 2–24 h	50	Esteves *et al.* (2006)
Pinus sylvestris	Plato process	50–60	Tjeerdsma (2006)

Table IV. Dimensional stability of various woods as a result of heat treatment.

Wood species	Conditions	ASE (%)	Reference
Pinus pinaster	170°C WL 2%	50	Esteves *et al.* (2006)
Eucalyptus globulus	190°C WL 2%	77	Esteves *et al.* (2006)
Eucalyptus globulus	200°C WL 10%	60	Esteves *et al.* (2006)
Pinus sylvestris	220°C	40	Rapp & Sailer (2001)

Note: WL = weight loss; ASE = antishrink efficiency.

Table V. Reduction in equilibrium moisture content (EMC) as a result of acetylation.

Species	WPG	Reduction in EMC	Reference
Pinus sylvestris	20	50	Minato *et al.* (2003)
Pinus elliotti	20	50	Hillis (1984)
Populus tremula	20	50	Rowell (2005)
Pinus sylvestris	20	70	Epmeier *et al.* (2004)

Note: WPG = weight percentage gain.

Acetylated wood

Over the years, several mechanisms have been put forward to explain the resistance to brown-rot fungal attack provided by acetylated wood. The earliest ideas centred around the modification of the conformation and configuration of the substrate such that the specific enzymic attack could not take place (Stamm & Baechler, 1960; Takahashi *et al.*, 1989*a*, *b*). Another theory is based on the bulking effect of the covalently bonded acetyl group (Forster *et al.*, 1997; Forster, 1998). Alternatively, enzyme penetration is prevented by physical blocking of the cell-wall micropores (Papadopoulos & Hill, 2002; Hill, 2002; Hill *et al.*, 2005). Highley *et al.* (1994) showed that the smallest enzyme of a brown-rot fungus is too large to penetrate the cell wall. The average size of a cellulitic enzyme is about 5 nm and the smallest pore size in wood is < 3.8 nm. Mohebby (2003) speculated that there are very small regions in the cell wall that are not acetylated owing to the size of the acetate group but that are accessible to free radicals produced by the fungi.

Table VI. Antishrink efficiency of several types of acetylated wood.

Species	WPG	ASE	Reference
Pinus elliotti	15	60	Rowell (1983)
Pinus sylvestris	20	70	Epmeier *et al.* (2004)
Pinus strobus	22	70	Rowell (2005)

Note: WPG = weight percentage gain; ASE = antishrink efficiency.

Table VII. Relationship between reduced swelling and decay resistance of white pine using *Trametes serialis* fungus (Stamm *et al.*, 1946).

Reduction in swelling (%)	Weight loss due to decay (%)
30–33	12
33–38	4.5
≥ 40	0

Table VIII. Fungal resistance of pine and spruce heated in oil or air.[a]

	Oil heated		Air heated	
Temperature (°C)	Pine WL (%)	Spruce WL (%)	Pine WL (%)	Spruce WL (%)
Control	40	48		
180	13	15	25.0	31.2
200	1.9	13.1	15.8	26.7
220	2.0	0.0	11.0	5.5

Note: [a]Data taken from several sources.
WL = weight loss.

Table X shows the results of a fungal cellar test using control and acetylated flakeboard samples at increasing levels of acetyl content (Rowell *et al.*, 1987; Nilsson *et al.*, 1988).

The present authors have postulated that the mechanism of decay resistance in acetylated wood is based on moisture exclusion owing to the fact that the EMC of a highly modified wood is too low to support fungal attack, i.e. there are no water molecules at the

Table IX. Weight loss (%) of different woods and different heat-treating processes due to attack by *Poria placenta*, *Coriolus versicolor* and *Coniophora puteana*.

Material	*P. placenta*	*C. versicolor*	*C. puteana*
Pine (control)	31.0	5.1	47.5
Douglas fir (control)	14.0	2.6	27.4
Oak (control)	0.8	14.3	3.9
Plato (heat treated)	10.0	6.8	3.7
Premium (heat treated)	16.0	9.0	1.9
NOW (heat treated)	13.3	7.8	12.2
OHT (heat treated)	7.4	5.6	3.4

Note: Plato = heat treated (The Netherlands); premium = heat treated (Finland); NOW = New Option Wood; OHT = Oil Heated Wood.

Table X. Fungal cellar tests[a] of aspen flakeboards made from control and acetylated flakes.[b]

WPG	Rating at intervals (months)[c]							
0	2	3	4	5	6	12	24	36
7.3	S/2	S/3	S/3	S/3	S/4	–	–	–
11.5	S/0	S/1	S/1	S/2	S/3	S/3	S/4	–
13.6	0	0	0	0	S/0	S/1	S/2	S/4
16.3	0	0	0	0	0	0	0	0
17.9	0	0	0	0	0	0	0	0

Note: [a]Non-sterile soil containing brown-, white- and soft-rot fungi and tunneling bacteria; [b]flakeboards bonded with 5% phenol–formaldehyde adhesive; [c]rating system: 0 = no attack; 1 = slight attack; 2 = moderate attack; 3 = heavy attack; 4 = destroyed; S = swollen. WPG = weight percentage gain.

site of a glycosidic bond, which the fungal enzymes need for hydrolysis (Rowell, 1983; Ibach & Rowell, 2000; Ibach *et al*., 2000; Rowell *et al*., 2007). Another idea is that the mechanism is based on the modification of the hemicellulose fraction in the cell wall. Maybe even more specific is the modification of the arabinose sugar in the hemicelluloses, considering that this is an L-pentose sugar in the hemicelluloses and it is the only sugar in a strained five-membered ring. Why is this sugar present in this strained ring form? Could it be the trigger that nature uses as a point of attack to start the entire degradation process? Is the mechanism of resistance as simple as protecting arabinose from hydrolysis?

It is interesting to note that all woods contain acetyl groups: softwoods from 0.5 to 1.7% and hardwoods from 2 to 4.5%. Why they are present is unknown. Acetylation of wood to an acetyl level of about 17–20% renders the wood resistant to attack by brown-rot, white-rot and soft-rot fungi.

Table XI shows the loss of carbohydrates in the wood fibre. The non-acetylated fibre loses 85.8% carbohydrate, with major weight loss in all sugars except for the galactans. The acetylated sample at 15% acetyl shows only 13.2% total carbohydrate loss, no loss of cellulose (glucans), but major losses of arabans and rhamnans.

Strength

Heat-treated wood

Stamm *et al*. (1946) were the first to report a loss of mechanical properties as a result of heat treatments of wood. They heated wood at 320°C for 1 min or 150°C for 1 week and found a 17% reduction in modulus of rupture (MOR) when heated in molten metal and 50% when heated in air. Under the same conditions, there was less loss of modulus of elasticity (MOE).

Mitchell (1988) found that losses in strength properties were much less when wood was heated dry as opposed to wet, with a greater loss in MOR than in MOE. Rusche (1973) found that strength losses were related to rate and extent of mass loss when heating beech and pine in the presence and absence of air at temperatures ranging from 100 to 200°C. Losses in maximum strength and work to maximum load were greater for compression than for tension loads. Loss in MOE was only significant when the mass loss was greater than 8–10% and was the same for both species.

Rapp and Sailer (2001) heated pine and spruce at 180 to 220°C for various times in air and in oil and determined MOR and MOE in a three-point bending test. The highest MOE was 11,000 N mm^{-2} in oil heating and there was little loss in MOE in either air or oil heating. MOR, however, decreased 30% in oil heating and impact bending strength decreased and the wood became brittle. Oil-heated wood lost about 50% and air-heated wood lost over 70% of the impact strength compared with controls.

Table XII summarizes other data on strength changes in heat-treated wood. In all cases, there was only a small decrease in MOE but major changes in MOR depending on temperature, time and atmosphere.

Table XI. Carbohydrate analysis after brown-rot degradation of wood fiber.

WPG	Weight loss (%)	Total carbon lost (%)	Araban lost (%)	Galactan lost (%)	Rhamnan lost (%)	Glucan lost (%)	Xylan lost (%)	Mannan lost (%)
0	51.7	85.8	87.9	71.9	90.0	83.8	90.6	92.5
15	1.4	13.2	89.0	55.2	70.0	0	38.3	42.0

Note: WPG = weight percentage gain.

Table XII. Change in MOR as a result of heat treatment of various woods.

Species	Treatment	MOR (%)	Reference
Betula pendula	Vapour, 200°C	−43	Johansson & Moren (2005)
Beech	Vapour, 200°C	−50	Yidiz *et al.* (2006)
Pinus sylvestris	OHT, 220°C	−30	Rapp & Sailer (2001)
Pinus pinaster	O_2, 180–200°C	−6–25	Esteves *et al.* (2006)

Note: MOR = modulus of rupture.

Acetylated wood

Narayanamurti and Handa (1953) acetylated some woods from India and found a slight decrease in MOE of acetylated wood compared with controls. The Koppers Company found that acetylated southern and ponderosa pine showed no loss of impact bending strength compared with controls (1961). Dreher *et al.* (1964) studied the strength properties of acetylated pine, oak and maple boards. Compressive strength, hardness, fibre stress at proportional limit and work to proportional limit were higher in acetylated boards than in controls. MOR increased by acetylation in pine but decreased in oak and maple. MOE was unchanged while shear strength parallel to the grain was decreased in all three acetylated species. Table XIII shows the bending stiffness (MOE) and bending strength (MOR) of acetylated radiata pine. The table shows that strength properties are retained even after acetylation.

Finite span tensile strength of thin strips of pine and lime showed no significant difference in tensile strength between control and acetylated wood (Rowell & Banks, 1987).

Conclusions

Reductions in hygroscopicity, increased dimensional stability and decay resistance of heat-treated wood depend on decomposition of a large portion of the hemicelluloses in the wood cell wall. Strength properties are reduced in heat-treated wood as a result of the degradation of the cell-wall matrix resulting from the hemicellulose polymer degradation. In acetylated wood, reductions in hygroscopicity are due to substitution of hydroxyl groups with an acetyl group which is less hydroscopic than the hydroxyl group. Increased dimensional stability in acetylated wood is due to the bulking of the cell wall back to its original green dimensions, so the cell wall cannot expand very much more (because of the elastic limit of the cell wall). Decay resistance of acetylated wood is due to the cell-wall moisture content becoming too low to support fungal attack, so the initial enzymic attack does not take place. The critical sugar in the hemicelluloses that may be the trigger for fungal attack is arabinose, as it is the only sugar in a less stable five-member ring compared with sugars in a stable six-membered ring. Strength properties are not significantly changed in acetylated wood and acetylation results in greatly improved wet strength and wet stiffness properties.

Table XIII. Bending stiffness [modulus of elasticity (MOE)] and bending strength [modulus of rupture (MOR)] of control and acetylated radiata pine.

WPG	Density (kg m^{-3})	Moisture (%)	MOE (N mm^{-2})	MOR (N mm^{-2})
0	417	12.2	9664	43
20	492	5.2	8788	39

Note: WPG = weight percentage gain.

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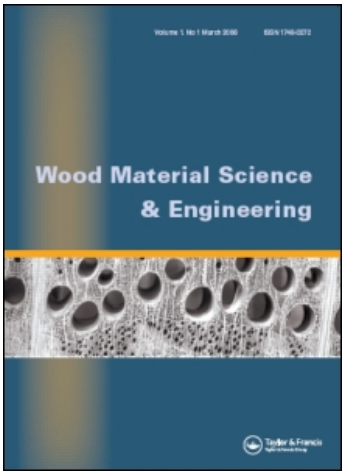

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Understanding decay resistance, dimensional stability and strength changes in heat-treated and acetylated wood

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