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Processes and properties

Performance of acetylated wood in aquatic applications

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ABSTRACT

Acetylation of wood to enhance its resistance against wood decaying fungi and insects has been studied extensively under both laboratory and terrestrial field trials. Also several studies are investigating the performance of acetylated wood in marine and fresh water exposures. This paper updates nine year **marine** tests with acetylated radiata pine in Hejlsminde (Denmark) and presents data on 3 year field tests at 3 marine locations in Italy (Follonica, Genova, Venice) and one in Oregon (USA) as well as an oyster bed test at 2 locations in Australia. Data are also presented on the performance of acetylated Scots pine, radiata pine, and poplar as sheet piling installed in **fresh water** in 1995 or 2000 in the Netherlands.

Keywords: acetylated wood, field tests, fresh water, marine borers

1. INTRODUCTION

Acetylation of wood to enhance its resistance against wood decaying fungi, as well as improving its dimensional stability under varying moisture conditions, has been extensively studied (Hill 2006, Homan and Jorissen 2004, Rowell and Dickerson 2014). There is general agreement that acetylated wood shows marked resistance to attack by most wood destroying fungi when the wood is treated to acetyl weight percent gains (WPG) greater than 15 to 20 % (Alexander *et al.* 2014, Alfredsen *et al.* 2013). Acetylated solid wood also appears to be resistant although not completely immune to termite attack (Bongers *et al.* 2015, Imamura and Nishimoto 1986, Papadopoulos *et al.* 2008a).

The resistance of acetylated wood to marine borers has been previously investigated in a number of studies. Johnson and Rowell (1988) tested southern pine (*P. taeda* L., *P. palustris* Mill., *P. elliotii* Engelm.) with a 22% WPG for three years at Key West Bight where both limnoriid and teredinid borers are active. The acetylated wood had an average rating of 8 (10: no attack; 0: destroyed) and was performing better than wood treated with coal-tar creosote (320 kg/m³), but not as well as CCA treated wood (38 kg/m³). One of the five acetylated samples, however, was destroyed by gribbles. Larsson-Brelid *et al.* (2000) reported test results of an EN 275 marine test with acetylated Scots pine exposed at the Kristineberg Marine Biology Station on the west coast of Sweden, 100 km north of Gothenburg. Even the highly acetylated samples (21.2% WPG) were severely attacked by shipworms and gribbles. Westin *et al.* (2006) reported results of another test with acetylated Scots pine (22% WPG) at Kristineberg Marine Biology Station after five years of exposure. The samples were slightly attacked. After 10 years, the acetylated Scots pine samples were moderately attacked (Larsson-Brelid and Westin 2010), and severely attacked after 16 years of exposure (Westin *et al.* 2016). In another test in the same location acetylated southern yellow pine (SYP) samples of lower acetyl content (18%) failed after 3 years. Highly acetylated SYP (24% acetyl content) were completely sound or only slightly attacked after 11 years of exposure (Westin *et al.* 2016).

Papadopoulos *et al.* (2008b) investigated the resistance of pine wood modified with linear chain carboxylic acid anhydrides to the crustacean wood borer *L. quadripunctata* by measuring faecal pellet production and mortality following feeding over three weeks in a rapid laboratory test developed by Borges *et al.* (2003). The type of anhydride used had little impact on the resistance. Highest resistance was obtained with high WPG of around 24%. The average number of faecal pellets produced by gribbles feeding on the wood decreased from ca. 45 to 5 pellets per animal per day due to acetylation. Using the same assay, Klüppel *et al.* (2010) found comparable numbers for Scots pine controls and Accoya. Faecal pellet production on radiata pine controls (ca. 75 per day) was even higher than on Scots pine controls.

Fresh water, in some ways, represents a less hazardous environment because low oxygen availability limits fungal attack, but continuous immersion increases the potential for loss of active ingredients in traditional biocidal protection schemes. This becomes less of an issue with acetylated wood, but fresh water exposures do present a greater risk of bacterial degradation. The marine environment presents special challenges because the wood is still subjected to continuous leaching, but there are also marine organisms that have evolved to either use wood as a habitat or food source. As a result, chemical loadings required for performance of traditional wood preservatives are typical several times higher than those used in terrestrial applications. Acetylated wood presents a different pathway to protection and it is unclear whether modifying the wood will be able to prevent marine borer attack.

This paper provides an update of active marine and fresh water tests of acetylated wood.

2. MATERIAL AND METHODS

There are ongoing marine trials in Australia, Denmark, Italy and the United States. The trials involve different materials and marine borer risks (see Table 1). Two fresh water sheet piling trials with acetylated wood are currently running in the Netherlands (see paragraph 2.5).

Table 1: Materials exposed in ongoing marine water trials at various locations.

Wood Material	Australia	Denmark	Italy			United States
			Ligurian sea	Tyrrhenian sea	Adriatic Sea	
Radiata pine sapwood		X	X	X	X	X
Scots pine sapwood		X	X	X	X	-
Beech		-	X	X	X	X
Southern pine sapwood		-	-	-	-	X
Douglas-fir heartwood		-	-	-	-	X
Western juniper (<i>Juniperus</i> spp)		-	-	-	-	X
Accoya radiata pine	X	X	X	X	X	X
Acetylated beech*		-	X	X	X	X
ACC treated Scots pine		X	-	-	-	-
Acetylated southern pine*		-	-	-	-	X
Chestnut (<i>Castanea sativa</i>)		-	X	X	X	-
European oak (<i>Quercus robur</i>)		-	X	X	X	-
Okoume plywood		-	X	X	X	-
Mediterranean Tricoya		-	X	X	X	-
Ekki (<i>Lophira alata</i>)		-	-	-	-	X
Greenheart (<i>Chlorocardium rodiei</i>)		-	-	-	-	X
ACZA Treated Douglas-fir		-	-	-	-	X

*Since the acetylation of beech / SYP is not commercially released by Accsys Technologies, it is called acetylated beech / SYP rather than Accoya beech / SYP

2.1 Marine trial Denmark

The test set-up was described by Klüppel *et al.* (2010). Ten test specimens measuring 200 x 75 x 25 mm³ (longitudinal x radial x tangential) of Accoya[®] Radiata pine (RP), radiata pine (*Pinus Radiata* D. Don) sapwood, Scots pine (*P. sylvestris* L.) sapwood, and CC-treated (0.6% and 2.5% concentration based to the standard EN 275). Scots pine sapwood were installed in a marine field trial in the Baltic Sea (Hejlsminde, Denmark) in May 2008. The CC treatment was performed using acid copper chromate composed of 30% (m/m) CuO and 70% (m/m) CrO₃ with concentrations as defined in EN 275.

The racks are exposed 2-4 m below medium high tide. Salinity at the test site ranges between 15 PSU and 25 PSU (practical salinity unit; corresponds to 1.5-2.5%). Water temperature varies between 0-5 °C in January and 20-25 °C in August.

The samples have been visually examined and x-rayed annually and rated according to EN 275 (Table 2). Five new untreated Scots pine sapwood controls are placed each year.

Table 2: Rating system for attack by teredinids according to EN 275.

Rating	Classification	Condition and appearance of test specimen on the x-ray film
0	No attack	No sign of attack
1	Slight attack	Single or a few scattered tunnels covering not more than 15% of the area of the specimen
2	Moderate attack	Tunnels covering not more than about 25% of the area of the test specimen
3	Severe attack	Tunnels covering between 25% and 50% of the area of the specimen
4	Failure	Tunnels covering more than 50% of the area of the specimen

2.2 Marine trial Italy

EN 275 field tests were started in April 2015 at three different Italian sites:

1. Ligurian Sea, Genova, experimental marine station of CNR ISMAR in Genova.
2. Tyrrhenian Sea, Follonica, supporting laboratory: CNR IVALSA in Firenze.
3. Adriatic Sea, Venice Lagoon, CNR ISMAR in Venezia.

Wood species included in the study are 1) Accoya radiata pine, 2) acetylated beech¹, 3) untreated radiata pine sapwood, 4) Scots pine sapwood, 5) untreated beech (*Fagus sylvatica* L.), 6) untreated chestnut (*Castanea sativa* Mill.), 7) untreated European oak (*Quercus robur* L.), 8) exterior grade Okoume (*Aucoumea klaineana* Pierre) plywood of 25 mm thickness, and 9) Medite Tricoya[®] Extreme of 18 mm thickness. The temperature in the Venice Lagoon is varying from around 7 °C in winter up to 26 °C in summer. Salinity is around 30-32 PSU. Full details of the test set-up are described by Palanti *et al.* (2017).

2.3 Marine trial Oregon USA

A marine test with 1) Accoya radiata pine, 2) acetylated beech, 3) acetylated SYP, 4) ekki (*Lophira alata* Banks *ex* Gaertn.), 5) greenheart (*Chlorocardium rodiei* (M.R.Schomb.) Rohwer *et al.*), 6) untreated radiata pine sapwood, 7) untreated SYP (*Pinus* spp), 8) untreated Douglas fir (*Pseudotsuga menziesii* (Mirb.) Franco), 9) untreated juniper (*Juniperus communis* L.) and 10) Ammoniacal Copper Zinc Arsenate (ACZA) treated Douglas fir was started at the Yaquina River near Newport Oregon (USA) on 15 July 2015. Salinity varies widely at this site between 1.5 and 3.0‰ because salt water can intrude quite far upstream during low summer stream flows. *Teredo navalis* L. is the primary wood destroying agent present at the site, but *Limnoria tripunctata* Menzies, *L. quadripunctata* Holthuis and *L. lignorum* Rathke are also present.

The samples were set on racks that were sunk to a depth of 2.5 to 3 m, but the test site is a floating dock and tidal ranges are large, so they are sometimes as shallow as 1 meter at minus tides.

The samples are evaluated twice per year with regard to the extent of fouling and end grain erosion and rated according to AWWA Standard E5 (see Table 3). Inspections were done on 15 February 2016 (7 months of exposure), 28 July 2016 (12 months) and 31 July 2017 (24 months).

¹ Since the acetylation of beech is not commercially released by Accsys Technologies, it is called acetylated beech rather than Accoya beech

Table 3: Rating system Oregon USA marine test.

Rating	Description AWWPA E5	Overall rating
10	No more than trace attack	No damage
9.5		up to 5% cross-sectional damage
9	Light attack	5-10 % cross-sectional damage
8		10-20% cross-sectional damage
7	Moderate attack	
6		20-40% cross-sectional damage
4	Heavy attack	Over 40% cross-sectional damage, not failed
0	Destroyed by attack	Failure of samples

2.4 Oyster bed trial- Australia

In June 2015 eight stakes of *Accoya Radiata* pine of 77 x 53 mm cross-section and 3 metres in length were installed in Mooney Mooney Bay at various locations along the Hawkesbury river estuary in New South Wales, Australia. The area is known for attack by marine borers (especially during spring), primarily the shipworms (Teredinids; Family: Teredinidae Rafinesque) and the crustacean genus *Limnoria* Leach, and potentially the genera *Sphaeroma* Bosc. and *Martesia* Sowerby I. The salinity in the Hawksbury estuary varies between around 26-30 psu (daily fluctuations due to tides). The objective of the trial was to determine the suitability of *Accoya radiata* pine for oyster farming and comparing versus H4 treated radiata pine. One acetylated stake was removed from the field trial after 12 months' continuous exposure in the marine environment, and a second stake was removed from the field trial (7th June 2017) after 24 months' continuous exposure. The condition of the stakes was determined from visual examination.



Figure 1: Oyster farm system deployed in the Hawkesbury river estuary, Mooney Mooney, NSW, Australia (left), and one of the eight *Accoya Radiata* pine stakes installed in the field trial (right).

2.5 Fresh water trials the Netherlands

Two fresh water sheet piling trials with acetylated wood are currently running in the Netherlands. The oldest trial was established in 1995 and consists of both untreated and acetylated Scots pine and poplar (*Populus* spp.), as well as Scots pine impregnated with a copper-chromium-(CC)-preservative. A detailed description of the test set-up can be found in Beckers *et al.* (1995).

In September 2000 a trial with acetylated *Radiata* pine was started, together with six durable hardwood species including ekki, three thermally modified wood products (Perdure, Plato

Radiata pine and Stellac) as well as untreated Norway spruce (*Picea abies* (L.) H. Karst.) and larch (*Larix* spp).

The samples in the field tests were visually assessed from the water and their condition determined by probing with a sharp pointed knife and a Pilodyn-like apparatus in August 2015.



Figure 2: Fresh water test (canal lining) in the Netherlands (wood sheets installed on left and right position of the water way).

3. RESULTS

3.1 Marine trial Denmark

Shipworm attack on untreated controls (Scots pine sapwood) was extensive. Specimens were always destroyed within one year of exposure with the exception of the 2012 season, but these specimens rated at 3 (severely attacked) and after an additional year all were completely destroyed. Each year five new controls were added. After nine years of exposure, the Accoya radiata pine test specimens show no visual signs of degradation. X-ray measurements (see Fig. 3) confirm the visual assessments.

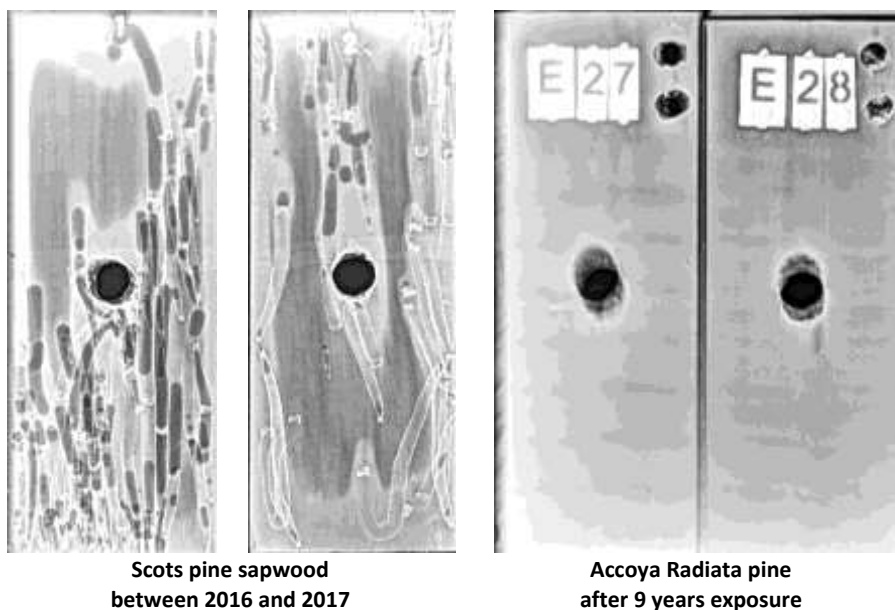


Figure 3: X-ray images of test specimens in the marine trial in Denmark.

3.2 Marine trial Italy

The results after one and two years of exposure are described by Palanti *et al.* (2017). All untreated wood species (chestnut, beech, oak, radiata pine and Scots pine) were destroyed (rating 4) by teredinids in all three Italian exposure sites after one year of exposure. Slight attack due to crustaceans was also observed in some untreated samples. Okoume plywood was slightly to moderately attacked by teredinids after one year of exposure, and severely attacked after two years. All acetylated samples, including the Tricoya were completely sound at all three test sites after two years of exposure. Illustrating X-ray images are shown in Fig. 4.

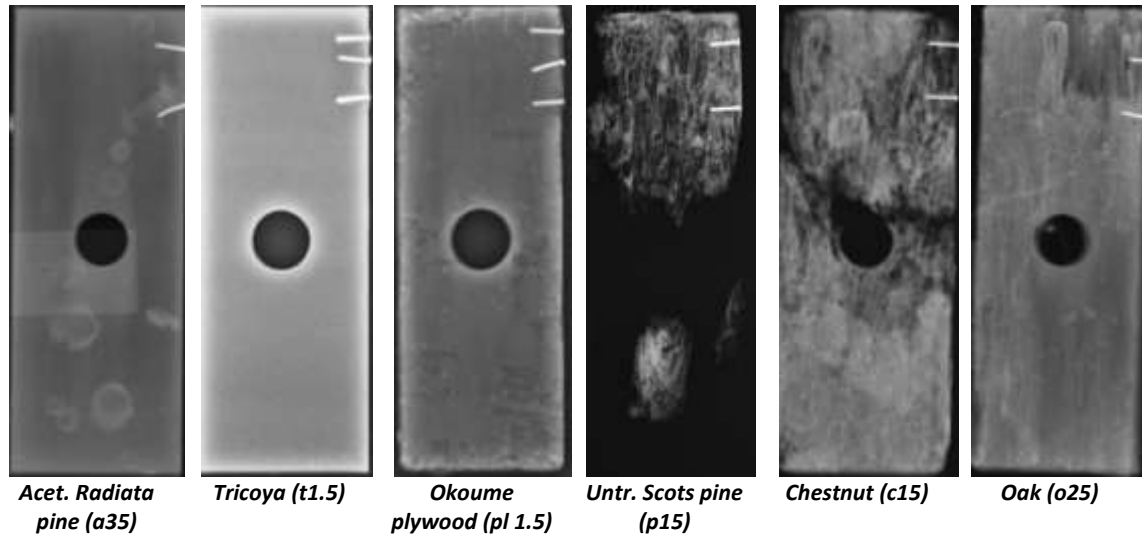


Figure 4: X-ray images of test specimens in marine trials in Italy. Scots pine, chestnut and oak after one year of exposure. Images for other materials after two years of exposure. White/light indentations in top right of each specimen are the nails from metal tags.

3.3 Marine trial Oregon USA

The results from visual inspections after seven, 12 and 24 months of exposure are shown in Table 4. None of the acetylated samples have been attacked after two years of exposure. Similar results have been observed for ekki and greenheart. Most of the untreated radiata pine and SYP test specimens failed after two years.

Table 4: Overall rating of samples in marine tests Oregon at different exposure times.

Wood species	Time of exposure [months]*		
	7	12	24
Acet. radiata pine	10	10	10
Acet. beech	10	10	10
Acet. SYP	10	10	10
Ekki	10	10	10
Greenheart	10	10	10
Untr. radiata Pine	9.4 (8-10)	5.7 (0-10)	0.4 (0-4)
Untr. SYP	9.5 (9-10)	5.5 (0-9.5)	0
Untr. Douglas fir	9.8 (9-10)	7.8 (4-10)	3.4 (0-9)
Untr. juniper	10	10 (9.5-10)	9 (6-10)

*average overall rating (min – max)

3.4 Oyster bed trial Australia

Examination of sections of the *Accoya radiata* pine stakes removed after one and two years of exposure showed no evidence of attack by marine organisms (see **Fel! Hittar inte referenskölla.**).

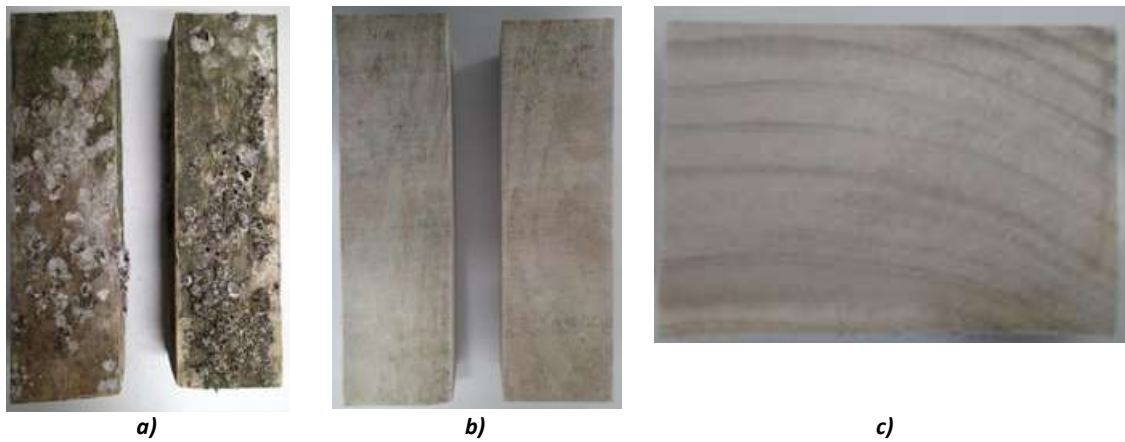


Figure 5: Condition of sections of *Accoya radiata* pine after two years of exposure, a) after partial cleaning of the adhering marine organisms, and b) after complete removal of the adhering marine organisms, c) cross section.

3.5 Fresh water trials the Netherlands

The thickness of the less durable untreated timbers (spruce, larch, Scots pine and poplar) was considerably reduced in both trials and their surfaces are strongly grooved due to erosion of especially early wood by a combination of soft rot/bacterial decay and mechanical wearing by water movement. Both acetylated Scots pine and acetylated poplar are performing almost equally as well compared to the CC-treated Scots pine in the oldest trial (20 years' exposure). Loss in thickness was negligible and their surface hardness has been maintained. The high quality ekki is performing the best in the second trial (15 year exposure), with some of the other hardwoods and the acetylated radiata pine exhibiting similar performance.

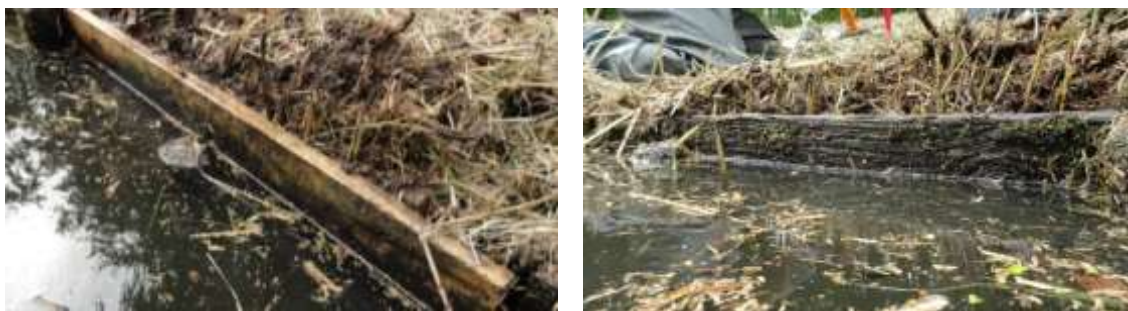


Figure 6: Acetylated radiata pine (left) and untreated spruce (right) after 15 years' exposure in fresh water (sheet piling, in the Netherlands).

4. DISCUSSION

4.1 Bio-deterioration in fresh and marine water

The types of potential bio-deterioration in timber vary in fresh and marine water. Wood is subject to fungal and insect attack above and in ground as well as above water. White-, brown-, or soft-rot fungal decay can significantly reduce timber strength properties. Fungi also cause surface moulding and other discolorations. The rate and type of fungal decay depends on many factors but wood moisture content, oxygen content, temperature and the wood chemistry (pH, metals, extractives) are most critical. Other factors that affect growth and reproduction of wood-decay fungi and their capacity to degrade wood include osmotic concentrations, atmospheric pressure, sound vibrations, gravitational forces, radioactivity and light intensity (Zabel and Morrell 1992). Most wood-inhabiting fungi are obligate aerobic organisms and require moderate amounts of oxygen for respiration. The higher tolerance of soft-rot fungi to low-oxygen concentrations, compared to the white- and brown-rot groups, may explain their prevalence as decay agents in more water saturated woods. Wood that is permanently water-saturated in fresh water is resistant to fungal and insect attack and will perform for many years. More tolerant organisms, including bacteria, may begin to degrade the wood, but their impact on wood properties is much slower (Zabel and Morrell 1992). Tunnelling bacteria (TB) are active in the presence of oxygen. Erosion bacteria (EB) can tolerate conditions with extremely low levels of oxygen, and wooden objects in water-saturated conditions in anoxic conditions are predominantly attacked by erosion bacteria. Although bacterial degradation of wood is markedly slower compared to that of the wood-decaying fungi, the unique features of the bacteria enable degradation of even wood species which are highly resistant to fungal decay such as preservative-treated wood and timbers containing highly toxic heartwood extractives (Singh *et al.* 2016).

Distinct differences in types of degradation can also be found in timber above and below the water level in salt water applications. Fungal decay is likely to occur above water (use class 3, EN 335), while damage below water (use class 5; EN 335) is primarily associated with marine borers. Marine borer is the collective term used for the many invertebrates that damage wood exposed to ocean or brackish waters. The two major phyla involved are the Mollusca (molluscs) and Crustacea (crustaceans).

The two major shipworm genera are *Teredo* and *Bankia*. *Teredo navalis* is found in warmer waters compared to *Bankia setacea*, and tolerates lower salinity. Shipworms begin their life as free-swimming larvae. When a larva encounters a piece of wood, it settles on it and metamorphoses. Although shipworms grow to lengths of 0.3 to 1.5 m, the only external sign of infestation is a small hole (< 0.30 cm in diameter). Adult shipworms release hundreds of thousands of larvae that disperse very effectively on ocean currents. They are able to very quickly colonise new environments under suitable conditions. The level of attack of marine borers, however, varies; and is often concentrated near the mud line or around joints, where the marine borers are more protected. The major ecological factors for growth, reproduction and distribution are water temperature and salinity, with the optimum and range being different for each species (Klüppel *et al.* 2015, Zabel and Morrell 1992).

Marine structures are also attacked by gribbles (*Limnoria* Leach) mainly in the highly oxygenated zone between the mud-line and the high-tide-line.. They are typically 3-4 mm long and chew small tunnels that penetrate only a short distance into the wood (2-10 cm). Extensive attack by gribbles weakens the wood to such an extent that the surface layers are eroded away by wave action. This erosion causes *Limnoria* to burrow continually deeper, thereby creating the typical hourglass shape of marine poles (Klüppel *et al.* 2015, Zabel and Morrell 1992).

4.2 Acetylated wood in fresh water

Moisture and oxygen levels in fresh water applications may fluctuate, creating different patterns of degradation. Mixed soft rot and bacterial attack are often found in situations with adequate moisture and oxygen. Generally, there is good agreement amongst researchers that wood acetylated to levels above acetyl WPG of 15-20% shows marked resistance to attack by wood destroying fungi. High resistance to soft-rot attack has been reported at lower acetylation levels of 10% WPG (Beckers *et al.* 1994, 1995). Larsson-Brelid *et al.* (2000) found that acetyl contents of around 15% resulted in high resistance in different soil tests. A higher percentage of acetyl (18.5%) was needed to prevent attack by brown and white rot. An acetyl content higher than 20.9% seems to be required to prevent attack by tunnelling bacteria. Mohebbi and Militz (2010) found that bacterial attack of highly acetylated exposed in soil was limited to pits.

The low amount of decay found in the acetylated wood in the different fresh water tests agrees with the results from laboratory tests.

4.3 Acetylated wood in marine water

The severity of a test site depends on different parameters including the quantity of marine borers present and the water temperature (Şen *et al.* 2009). The severity can also fluctuate over time, and even with the use of control samples, it can be difficult to compare results from different trials. Long term testing is also necessary since degradation of natural woody materials by marine borers, even highly durable samples rich in extractives and minerals such as silica and calcium crystals, is inevitable (Şen *et al.* 2009).

From the published literature (Larsson-Brelid *et al.* 2000, Larsson-Brelid and Westin 2010, Westin *et al.* 2016) it is evident that the resistance of wood against marine borers increases with increasing level of acetylation.

Klüppel *et al.* (2015) discussed the mode of action of using resin to increase resistance to marine borers. One possible explanation for the resistance was increased hardness. But, it was concluded that hardness cannot be the sole protection mechanism of modified wood since acetylation does not increase cell wall hardness (Jakes *et al.* 2011). The protective effect of acetylation may be either micropore blocking or enzyme non-recognition of the chemically altered wood polymers. Westin *et al.* (2006) suggested that non-recognition of acetylated wood was a mode of action. Post-treatment with methylated melamine resin (MMF) treatment, further improved the resistance to marine borer attack for acetylated wood. This enhancement was suggested to be caused by physical blocking of the cell wall by polymerizing agents. Both studies concluded that more research was needed in order to develop a deeper understanding of the protection mechanisms of acetylated wood. However, the mode of action does not seem biocidal.

5. CONCLUSIONS

A variety of studies demonstrate that marine borer resistance of wood increases with increasing level of acetylation. Tests with *Accoya radiata* pine, even after nine years exposure in Denmark, show no evidence of attack. Erosion of the top surface is evident in fresh water exposures, but the acetylated sheet piling is still almost completely intact after 20 years of exposure.

The marine and fresh water tests will be continued to gain more information on the suitability of acetylated wood in aquatic conditions. Further research into suitability in brackish water and into applications as engineered acetylated wood products is advised.

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