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BIOLOGICAL DURABILITY OF SCOTS PINE (*PINUS SYLVESTRIS* L.) SAPWOOD MODIFIED WITH SELECTED ORGANO-SILANES

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ABSTRACT

Paper presents the decay and mould resistance of the Scots pine sapwood specimens modified with four organo-silanes, the methyltrimethoxysilane (MTMS), the vinyltrimethoxysilane (VTMS), the propyltrimethoxysilane (PTMS), or the 3-aminopropyltrimethoxysilane (APTMS), and some of them additionally painted with the transparent acrylic coating (Balakryl-Dixol). Before biological tests, the treated specimens were not or were artificially aged in water and Xenotest, respectively. The organo-silanes without $-NH_2$ group (MTMS, VTMS and PTMS) had only a slight anti-decay efficiency against the brown-rot fungus *Coniophora puteana* and the white-rot fungus *Trametes versicolor*. Their anti-mould effect against the *Aspergillus niger* was none, and against the *Penicillium brevicompactum* only a mild. On the other hand, the anti-decay and anti-mould effects of the APTMS were evidently higher. The transparent coating significantly increased the biological durability of the modified pine sapwood only in some cases. Owing to the artificial ageing the decay resistance of the modified or the modified and painted pine sapwood decreased.

KEYWORDS: Pine, organo-silanes, coatings, ageing, decay fungi, moulds.

INTRODUCTION

Wood is a natural biopolymer and therefore not-seldom it is subjected to degradation processes resulting from its molecular and anatomical structure. Chemical protection of wood using the toxic biocides has many opponents today, especially from perspective of environmental contaminations. Nowadays the durability and service life of wooden products exposed in moist conditions can be increased by using of thermally modified wood or wood treated with non-toxic modification chemicals. Within the frame of chemical modification of wood have been searched many chemicals, but only few of them found application in practice, e.g. the acetic anhydride for acetylation – Titan wood, the furfurylalcohol for furfurylation – Kebony wood, and the dimethyloldihydroxyethyleneurea for eterification – Belmadur wood (Hill 2006).

Organo-silanes belong also to prospective substances for industrial chemical modification of wood. These substances have one organic group attached directly to the silicon atom (-R-Si). This group may be inert (methyl, n-propyl, etc.), or may participate in chemical reactions either with polymers of wood (vinyl, 3-isocyanatepropyl, amine, glycidyloxy, etc.), further with other silane molecules, or with a co-additive (Mai and Militz 2004). Chemical reaction between the reactive organic group (R) of organo-silane and the hydroxyl group (-OH) of wood can start in presence of a suitable initiator or catalyst (Tingaut et al. 2005). Reactive organo-silanes are able to react with hydroxyl groups of wood, create bridges between these groups, and reduce hygroscopic character of wood. Organo-silanes have also three alkoxy (methoxy, ethoxy, etc.) groups linked to the silicon atom (-Si-OR). These groups after pre-hydrolysis with water molecules create silanol groups (-Si-OH), which have a high affinity for other reactions in a wet wood. The silanol groups may be involved either in reactions with hydroxyl groups of the polysaccharide-lignin substance of wood (Wood-OH) which generate bonds (-Si-O-Wood), or in their mutual cross-condensation reactions which incur siloxane polymer networks (-Si-O-Si-) (Mai and Militz 2004; Salon et al. 2007; Xie et al. 2011).

The aim of this work was to search: 1) the anti-decay and anti-mould efficiency of selected organo-silanes used for wood modification; 2) promoting anti-decay effect of one transparent acrylic coating painted on modified wood; 3) weakening of the anti-decay and anti-mould effects of used organo-silanes and coating after ageing of treated wood in water or Xenotest.

MATERIAL AND METHODS

Wood

From sapwood of the Scots pine (*Pinus sylvestris* L.) boards, dried on a moisture app. 12 %, were prepared specimens without biological damages, knots and growth inhomogeneities. Their dimensions were: 25x25x3 mm (LxRxT) for the decay test, and 50x10x5 mm (LxRxT) for the mould test. All specimens were firstly sterilized 6 hours at 103±2°C and then conditioned in sterile climatic room at 23±2°C and 50±5 % RH until a constant EMC app. 8 % ($m_{before\text{-}mod.}$).

Organo-silanes

Four types of organo-silanes, the MTMS (methyltrimethoxysilane), the VTMS (vinyltrimethoxysilane), the PTMS (propyltrimethoxysilane) and the APTMS (3-aminopropyltrimethoxysilane), were provided from the Sigma-Aldrich Chemie Steinheim – Germany. Wood modification was performed with their 10 % (by mass) aqueous solutions. The MTMS was used also in 5, 20, or 30 % (by mass) aqueous solutions.

Modification of wood with organo-silanes

The modification of the pine sapwood with the organo-silanes was performed by dipping method at a temperature of 20°C, during 3 minutes. The modified specimens were immediately putted on a filter paper for 1 minute, conditioned 1 day under ambient conditions in sterile room with UV light, subsequently dried 6 h at 60°C, and finally conditioned until a constant EMC app. 8 % ($m_{after\text{-}mod.}$).

There, only a lower drying temperature of 60°C, instead of 103±2°C used usually for co-sterilization of specimens, was chosen in accordance with the work of Van Acker et al. (2003).

This was proposed on the basis of a knowledge related to lower boiling points of some organo-silanes, and also to fear of loss of the organo-silanes from modified specimens before their artificial ageing or fungal attacks.

The retentions of the organo-silanes into the pine sapwood specimens were determined as Weight-Percent-Gain (WPG_{Silane}) in percentage (%) by the Eq. 1:

$$WPG_{Silane} = \frac{(m_{after-mod.} - m_{before-mod.})}{m_{before-mod.}} \times 100 \quad (1)$$

Painting of wood surfaces

The transparent acrylic coating Balakryl-Dixol (Trilak festékgyártó Kft. Budapest – Hungary) was painted on one half of the modified specimens used for the decay test – on all they surfaces in an amount of 2 x 100 $g.m^{-2}$. The painted specimens were conditioned until a constant EMC app. 8 % ($m_{after-mod.\& paint.}$).

Artificial ageing

The aim of the artificial ageing (leaching in water and exposition in Xenotest, respectively) was to find out an anti-weathering stability of the organo-silanes in the treated wood before testing its biological durability against the decay fungi and moulds. The artificially aged specimens were conditioned until a constant EMC app. 8 % ($m_{after-ageing}$).

Accelerated leaching of the treated and reference specimens was performed in distilled water according to the standard EN 84 (1997). Their accelerated weathering was performed in Q-SUN Xe-1-S Xenotest (Q-Lab Corporation, USA), using 1-week long exposure cycle according to the partly adapted standard EN 927-6 (2006) in the following sequence: 1^{st} Step → 24 h at 45 ± 3°C; 2^{nd} Step → 48 sub-cycles each lasting 3 h (2.5 h = UV irradiance 0.55 $W.m^{-2}$ from a 1800 W xenon lamp at 340 nm; and 0.5 h = water spray at 20 ± 1°C).

Decay test

The decay test of the reference, modified, and modified & painted specimens was performed according to the partly adapted standard EN 113 (1996), applying these changes: - a smaller dimension of specimens, 25x25x3 mm (LxRxT) instead of 50x25x15 mm (LxRxT); - another treatment process of specimens, dipping instead of vacuum impregnation; - a shorter time of decay similarly with Chittenden and Singh (2011), only 6 weeks instead of 16 weeks. The decay of specimens was performed with the brown-rot fungus *Coniophora puteana* (Schumacher) *P.* Karsten and the white-rot fungus *Trametes versicolor* (L.) Pilát., respectively. At the end of the decay test the specimens were pulled out from Petri dishes, carefully cleaned from the fungal mycelium, air-dried in a laboratory during 4 days, and finally conditioned until a constant EMC app. 8 % ($m_{after-decay}$).

The decay resistance of the specimens was valued on the basis of their mass losses caused at action of fungi (Δm) in percentage (%) – by the Eqs. 2a and 2b for the artificially un-aged ones, and by the Eq. 3 for the artificially aged ones:

$$\Delta m = [(m_{after-mod.} - m_{after-decay}) / m_{after-mod.}] \times 100 \quad (2a)$$

$$\Delta m = [(m_{after-mod.\alpha\, paint.} - m_{after-decay}) / m_{after-mod.\alpha\, paint.}] \times 100 \quad (2b)$$

$$\Delta m = [(m_{after-ageing} - m_{after-decay}) / m_{after-ageing}] \times 100 \quad (3)$$

Mould test

The growth activity of moulds (GAM), the *Aspergillus niger* Tiegh. and the *Penicillium brevicompactum* Dierckx, on the top surfaces of the reference and modified pine specimens was tested during 28 days at temperature of 24 ± 2°C and RH of 90 – 95 % according to the standard EN 15457 (2007). The values of the GAM were evaluated in the scale from 0 to 4, where: 0 is no growth, 1 is growth ≤ 10 %, 2 is growth ≤ 30 %, 3 is growth ≤ 50 %, and 4 is growth ≤ 50 % on the top surface of the specimen.

RESULTS AND DISCUSSION

The retentions of the four organo-silanes into the pine sapwood were mutually comparable (Tab. 1). The WPG_{Silane}, using their 10 % aqueous solutions, were in a narrow range from 3.1 % (APTMS) to 3.6 % (VTMS). Similarly, with rising concentration of the MTMS from 5 to 30 %, the $WPG_{Silane\text{-}MTMS}$ increased quite evenly from 1.9 to 7.8 % .

Tab. 1: WPG_{Silane} of organo-silanes in the Scots pine sapwood specimens 25x25x3 mm.

Organo-silane	Concentration (%)	WPG_{Silane}(%)
MTMS	5	1.9 (0.3)
MTMS	10	3.4 (0.6)
MTMS	20	5.2 (0.9)
MTMS	30	7.8 (1.6)
MTMS	10	3.4 (0.6)
VTMS	10	3.6 (0.6)
PTMS	10	3.2 (0.8)
APTMS	10	3.1 (0.7)

Means values are from 72 replicates.
Numbers in the parentheses are the standard deviations.

Decay resistance

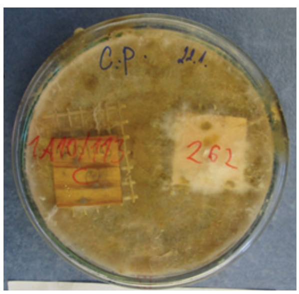
The tested organo-silanes, except of the APTMS, had not a more notice able positive impact on the decay resistance of the pine sapwood (Tab. 2). The MTMS, VTMS and PTMS (applied in 10 % aqueous solutions) although significantly reduced the mass losses of the pine sapwood at decay – confirmed by the Duncan's test on 99.9 % significance level (Tab. 2), but their anti-decay effect was shown only as a weak for practical usage. For example – without previous artificial ageing – these organo-silanes decreased the mass losses of the pine specimens exposed to *C. puteana* from 15.6 to 9.2 – 11.1 % and to *T. versicolor* from 14.4 to 6.4 – 9.1 %, respectively (Tab. 2). Similarly, for the VTMS (vinyltrimethoxysilane) a weak anti-decay efficiency reported Hill et al. (2004).

On the other hand, the pine sapwood modified with the APTMS (3-aminopropyltrimethoxysilane), having $-NH_2$ group, apparently well resisted to decay processes (Fig. 1). The mass losses of the pine sapwood modified with the APTMS – without previous ageing – were below 2 % (Tab. 2). Similarly, Mai et al. (2005) reported increase of the decay resistance of the pine wood after its modification with a silicone-oligomer system containing amino groups.

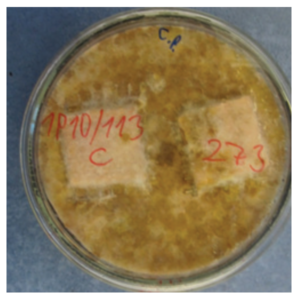
Tab. 2: Mass losses (Δm) of the reference and the modified pine sapwood caused by decay fungi.

Organo-silane	Without ageing		Ageing – Leaching (EN 84)		Ageing – Xenotest (adapted EN 927-6)	
	Coniophora puteana Δm (%)	*Trametes versicolor* Δm (%)	*Coniophora puteana* Δm (%)	*Trametes versicolor* Δm (%)	*Coniophora puteana* Δm (%)	*Trametes versicolor* Δm (%)
Reference	15.6 (3.2)	14.4 (2.3)	15.0 (2.4)	13.7 (2.7)	15.2 (4.1)	12.3 (3.0)
5 % MTMS	12.7 (2.1)^{c}	10.5 (2.0)^{b}	13.2 (2.0)^{d}	10.9 (0.9)^{b}	14.3 (1.0)^{d}	10.7 (0.9)^{d}
10 % MTMS	11.1 (2.0)^{a}	9.1 (3.1)^{a}	12.7 (2.0)^{c}	10.4 (3.3)^{b}	14.1 (2.6)^{d}	9.6 (1.9)^{c}
20 % MTMS	7.6 (1.7)^{a}	7.5 (1.2)^{a}	11.1 (2.0)^{b}	8.3 (0.9)^{a}	11.1 (1.4)^{b}	8.9 (1.0)^{b}
30 % MTMS	6.6 (1.2)^{a}	7.3 (1.1)^{a}	10.6 (1.1)^{b}	8.2 (1.1)^{a}	10.8 (1.7)^{b}	8.4 (0.8)^{a}
10 % MTMS	11.1 (2.0)^{a}	9.1 (3.1)^{a}	12.7 (2.0)^{c}	10.4 (3.3)^{b}	14.1 (2.6)^{d}	9.6 (1.9)^{c}
10 % VTMS	9.2 (1.1)^{a}	6.4 (1.0)^{a}	11.1 (2.2)^{b}	8.9 (2.9)^{a}	10.5 (1.1)^{b}	8.7 (1.8)^{b}
10 % PTMS	9.6 (1.5)^{a}	6.9 (0.8)^{a}	11.2 (2.9)^{b}	7.4 (1.7)^{a}	12.8 (3.1)^{d}	9.2 (0.9)^{c}
10 % APTMS	1.7 (0.9)^{a}	1.1 (0.3)^{a}	4.8 (1.4)^{a}	2.7 (0.9)^{a}	5.1 (0.9)^{a}	3.8 (1.0)^{a}

Mean values are from 6 modified replicates, or from 48 reference replicates.
Numbers in the parentheses are the standard deviations.
Duncan's tests of significance are performed in relation to the reference: a) 99.9 % significance level, b) 99 % significance level, c) 95 % significance level, d) < 95 % significance level.



a)



b)

Fig. 1: Activity of the Coniophora puteana mycelium on specimens was more suppresed by the APTMS (a = 1A10/113) than by the PTMS (b = 1P10/113). Reference specimens No. 262 and 273 are on the right.

The modified pine sapwood through additional treatment with the transparent acrylic coating obtained a partly higher decay resistance (Tab. 3). The MTMS, VTMS and PTMS in combination with the acrylic coating – without previous ageing – decreased the mass losses of the pine sapwood exposed to *C. puteana* from 15.6 to 4.4 – 6.4 % and to *T. versicolor* from 14.4 to 4.5 – 6.2 %, respectively. A positive anti-decay effect of the final coating was observed also for the APTMS when the mass losses of the complexly treated pine sapwood – without its previous ageing – were below 1 % (Tab. 3).

Tab. 3: Mass losses (Δm) of the reference and the modified & painted pine sapwood caused by decay fungi.

Organo-silane & Coating	Without ageing		Ageing – Leaching (EN 84)		Ageing – Xenotest (adapted EN 927-6)	
	Coniophora puteana Δm (%)	*Trametes versicolor* Δm (%)	*Coniophora puteana* Δm (%)	*Trametes versicolor* Δm(%)	*Coniophora puteana* Δm (%)	*Trametes versicolor* Δm(%)
Reference	15.6 (3.2)	14.4 (2.3)	15.0 (2.4)	13.7 (2.7)	15.2 (4.1)	12.3 (3.0)
5 % MTMS	6.7 (1.2) a	6.7 (1.6)a	9.9 (0.8) a	7.3 (2.2) a	8.9 (0.9) a	7.9 (1.1) a
10 % MTMS	6.4 (1.0) a	6.2 (0.9) a	9.0(1.5) a	7.0 (2.0) a	8.2 (1.8) a	6.6 (1.2) a
20 % MTMS	6.2 (1.0) a	5.9 (0.8) a	8.8 (1.3) a	6.7 (1.4) a	8.1 (1.0) a	6.3 (0.8) a
30 % MTMS	5.6 (0.6) a	5.7 (0.8) a	7.4 (0.8) a	6.3 (1.3) a	6.3 (1.4) a	6.0 (1.2) a
10 % MTMS	6.4 (1.0) a	6.2 (0.9) a	9.0 (1.5) a	7.0 (2.0) a	8.2 (1.8) a	6.6 (1.2) a
10 % VTMS	5.8 (1.3) a	4.5 (1.7) a	10.1 (1.2) a	5.8 (1.1) a	6.5 (0.8) a	5.6 (0.9) a
10 % PTMS	4.4 (0.4) a	5.1 (0.6) a	9.2 (1.8) a	5.7 (1.9) a	8.5 (1.9) a	5.4 (1.6) a
10 % APTMS	0.4 (0.4) a	0.8 (0.2) a	4.0 (1.6) a	2.5 (0.8) a	2.8 (1.3) a	2.7 (0.6) a

Mean values are from 6 modified replicates, or from 48 reference replicates.
Numbers in the parentheses are the standard deviations.
Duncan's tests of significance are performed in relation to the reference: a) 99.9 % significance level, b) 99 % significance level, c) 95 % significance level, d) < 95 % significance level.

Owing to the ageing impacts on the treated wood – leaching in water or exposure in Xenotest – the initial anti-decay efficiency of the organo-silanes used alone or in combination with the transparent acrylic coating decreased. At the decay processes the mass losses of the treated and aged pine specimens were higher in comparison to the mass losses of the treated and un-aged pine specimens (Tabs. 2 and 3). The previous artificial ageing had the greatest negative impact on the decay resistance of wood modified with the APTMS, when the mass losses caused by rot increased from 0.4 – 1.7 % (previously un-aged specimens) to 2.5 – 5.1 % (previously aged specimens). However, it does not automatically mean a weaker fixation of the APTMS in wood in comparison to other tested organo-silanes. Here, by our opinion, may be formulated this hypothesis: "If the initial anti-decay efficiency of organo-silanes is only a slight (e.g., the MTMS, VTMS, PTMS), it at leaching of modified wood in water or ageing in Xenotest cannot be decreased so apparently as of the most anti-decay effective organo-silanes having similar chemical-physical properties – polarity, etc. (e.g., the APTMS).

Goethals and Stevens (1994) reported a better decay resistance of wood modified with the PTMS (propyltrimethoxysilane) against the white-rot fungus *Trametes versicolor* as against the brown-rot fungus *Coniophora puteana*. Their knowledge was a partly confirmed in this experiment. For example, in a situation when the final coating was not applied, the ratio of mass losses caused by *C. puteana* and *T. versicolor* was a slightly higher in presence of the PTMS: 1) without previous ageing, the ratio was 1.08 for the reference pine and 1.39 for the pine modified with the PTMS; 2) with previous leaching in distilled water, the ratio was 1.09 for the reference pine and 1.51 for the pine modified with the PTMS; 3) with previous ageing in Xenotest, the ratio was 1.24 for the reference pine and 1.39 for the pine modified with the PTMS (Tab. 2).

More authors reported that the anti-decay effect of the silicones increases with their amount in wood. Hill et al. (2004) and De Vetter et al. (2009) this knowleadge determined for organo-silanes, or Reinprecht et al. (2013) for the methyl-tripotassiumsilanol. In this experiment, the concentration growth of the MTMS in aqueous solutions from 5 to 30 % (Tab. 1 – see increased WPG_{Silane}) had significant positive effect on a better decay resistance of the modified pine sapwood (Tab. 2, Fig. 2). However, due to a previous leaching in water or exposure in Xenotest the positive concentration effect of the MTMS on a higher decay resistance of the modified pine sapwood decreased, more evidently at testing its resistance against the brown-rot fungus *Coniophora puteana* (Tab. 2, Fig. 2).

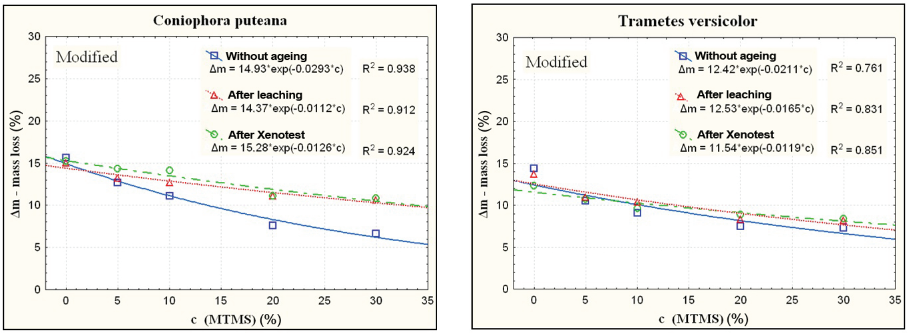


Fig. 2: Mass losses of the modified pine specimens at the decay test with the fungi Coniophora puteana and Trametes versicolor – influenced by the concentration of the MTMS and by the previous ageing processes.

The transparent acrylic coating generally increased resistance of the modified specimens against decay fungi. Afterwards, the concentration effect of the MTMS was not so dominant – and the mass losses of specimens were in a narrower range (compare Tab. 3 with Tab. 2).

Mould resistance

From the tested organo-silanes only the 3-aminopropyltrimethoxysilane (APTMS) slightly suppressed growth activity of the *Aspergillus niger*, and more apparently growth activity of the *Penicillium brevicompactum* (Tab. 4). Gosh et al. (2009) determined also a positive anti-mould effect of $-NH_2$ group in silicone compounds when modified wood with macro-emulsion of amino-silicones. Other types of tested organo-silanes, the MTMS, VTMS and PTMS, had none anti-mould efficiency (Tab. 4).

Tab. 4: Growth activity of moulds (GAM) on the top surfaces of the reference and the modified pine sapwood.

Organo-silane	Without ageing		Ageing – Leaching (EN 84)		Ageing – Xenotest (adapted EN 927-6)	
	Aspergillus niger GAM (0-4)	*Penicillium brevicomp.* GAM (0-4)	*Aspergillus niger* GAM (0-4)	*Penicillium brevicomp.* GAM (0-4)	*Aspergillus niger* GAM (0-4)	*Penicillium brevicomp.* GAM (0-4)
Reference	4	4	4	4	4	4
5 % MTMS	4	4	4	4	4	4
10 % MTMS	4	4	4	4	4	4
20 % MTMS	4	4	4	4	4	4
30 % MTMS	4	4	4	4	4	4
10 % MTMS	4	4	4	4	4	4
10 % VTMS	4	4	4	4	4	4
10 % PTMS	4	4	4	4	4	4
10 % APTMS	3.5	2	4	3	4	3

Mean values are from 4 replicates.

CONCLUSIONS

- This study showed that pine sapwood treated with the MTMS, VTMS and PTMS organo-silanes, or also additionally painted with the transparent acrylic coating, is accessible for fungal decay and mould attacks.
- The concentration rise of the MTMS from 5 to 30 % had a positive effect on the decay resistance of the pine sapwood exposed to action of *C. puteana* and *T. versicolor*, respectively, but only if the modified wood before action of these decay fungi was not artificially aged in water or Xenotest.
- The APTMS (3-aminopropyltrimethoxysilane), containing $-NH_2$ group, had a significant anti-decay efficiency. However, it was apparently reduced due to artificial ageing.
- Improvement of the fixation of the biologically effective organo-silane APTMS in wood, e.g. by suitable catalysts, should be searched in future experiments.

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