

Effective protection of pinewood against fungal attack

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Abstract. This paper gives an overview about an effective protection technology of pinewood. The new impregnation emulsion based on rapeseed-oil and boron compounds was developed and tested. To compare the efficiency of new impregnate widely used ‘Tanalith E’ wood preservative was used. The obtained results demonstrate that the new impregnation agent ensures effective protection against wood rotting fungi and discolouring fungi. Bio testing according to the EN113 at Latvian State Institute of Wood Chemistry revealed that the new preservative ensures effective protection against wood rotting fungi (mass loss less than 3%) and discolouring fungi (mould).

Keywords: Pinewood, rapeseed oil-based wood preservatives, protection against wood rotting fungi

Introduction

When impregnated wood is exposed to surface water during its service life, toxic substances in wood preservatives like chromium and arsenic can leach and harm the environment. Therefore the use of chromium and arsenic containing preservatives (CCA) was restricted due to environmental concerns and other systems were introduced. Also, nonbiocidal techniques for wood protection have become more and more important in the last few years. Different impregnation processes (Royal process, etc) are used to protect pinewood (*Pinus silvestris*) against fungal attack (Schultz, 2007). Solutions of Wolmanit CX-8 and Tanalith are used (Kängsepp, 2011) and some alternative preservative systems for pinewood are developed (Liibert, 2011). Both water-based and solvent-based products qualify for Scots pine sapwood preservation purpose and among the water based products the 100% active micro-emulsions based on organosilicons can be used (Vetter, 2009).

In water environment inorganic constituent chemicals easily diffuse into wood cell wall regions and protect wood against aggressive decay. Drying vegetable oil – in this case rapeseed oil – forms on the internal surfaces of wood cells and inside micro capillaries hydrophobic polymerised film that decreases the washing-out of active ingredients.

Before the impregnation tests timber must be dried approximately to 30% residual humidity. The best method is vacuum drying which makes the internal canals in pinewood easily permeable compared to other drying methods. The timber which was

used in this study was vacuum dried pine (*Pinus silvestris*) roundwood and sawn timber.

For active ingredients in emulsion for impregnation tests were chosen boron compounds – borax and boric acid – and quaternary ammonium salts (here dioctylmethyl ammonium chloride). Boron compounds ensure the potential bio protection against wood destroying fungi and QUATS against discolouring fungi. Boric acid (BA) and borax (BX) are also the most commonly used fire retardants in wood preservation industry (Baysal, 2007). The main problem is how to exclude leaching of boron compounds from impregnated wood. Some studies have shown that leaching of boron from spruce wood impregnated with preservative solutions based on boric acid can be significantly reduced (Islam, 2008 and Obanda, 2008). The new impregnation emulsion in this study was tested against the emulsion prepared from widely used ‘Tanalith E’ wood preservative.

Materials and methods

The experiments were made with samples of pine roundwood with outside dimensions 1,000 mm in length and about 200 mm of diameter and with sawn timber of different cross-section.

The density (oven dry) of used Estonian pinewood was in the range of 410...480 kg m⁻³ and bait about 5...8 rings per 1cm. To prevent the liquid penetration in the longitudinal direction, cross sections of the samples were sealed with nitrolaquer. The emulsion used in impregnation tests was rapeseed oil in water. The optimal concentration of rapeseed oil in water emulsion was determined experimentally. The impregnating agent was carried into the wood in one technological step. Dissolved in water phase active inorganic constituent chemicals diffuse into wood cell wall regions and protect wood against aggressive decay.

To stabilize the emulsion oleic acid and the by-product of the rapeseed oil production (partially de-esterified rapeseed oil, so-called acid oil) were used. Emulsification was carried out in a specific pulsation apparatus with the pulse frequency of 900 Hz. Obtained emulsion was stable for 24 hours.

Two different methods were used for testing of leaching. Method M1 is based on analysing copper and boron content after 24h leaching in water at 20–22°C. Firstly, the specimens were cut from impregnated roundwood in radial direction with dimensions 40 x 25 x 25 mm. Then the specimens were dried and all sections were covered with polyvinyl acetate glue, only outside tangential surface was not covered. Then the specimens were soaked in water at 20–22°C for about 24 hours.

Method M2 was based on imitation of rainy conditions to the impregnated wood. The specimens were cut from impregnated roundwood in stem direction with dimensions 60 x 30 x 30 mm, dried and all sections were covered with polyvinyl acetate glue, except outside tangential surface. Then, on the specimens was dropped 100ml water with speed about 8–12 drops per minute.

In collected leaching water the content of copper and boron was analysed. The copper content was analysed by atomic absorption spectroscopy (AAS). The boron content in leached-out water was analysed via ICP (Inductively Coupled Plasma) technique.

Bio testing of impregnated pinewood specimens was carried out according to standards EN113:2000, EN73 and EN84:2000 at Latvian State Institute of Wood Chemistry. From impregnated sapwood were cut out blocks $25 \times 25 \times 50$ mm for testing. Average density of test specimens was 485 kg m^{-3} . Wood specimens were sterilized, conditioned and exposed to fungi according to the EN113 (1996) using fungi *Coniophora puteana*, *Gleophyllum trabeum*, *Poria placenta*. The specimens were placed in sterilized plastic film bags and exposed to fungi using brown-rotting fungi. Plastic film bags were placed into climate chamber, where they were kept at 23°C and 75% of relative humidity.

Results and discussion

Impregnation test results

The impregnating agent was carried into the wood in one technological step. The used technological regime for the impregnation was following:

- preliminary vacuum treatment at residual pressure 100 mbar for 0.5 hours;
- pressure impregnation at 12.5 bar for 1 hour;
- final vacuum treatment at residual pressure 100 mbar for 0.5 hours.

To evaluate the penetration efficiency determination of the retention of the agent, average depth of penetration, leaching-out of active components, and bio-durability were carried out. After the impregnation tests, the colour characteristics of the wood impregnated with new agent were similar to those of untreated wood. To compare the leaching test results with aqueous solutions and emulsions, the leachability of active agents for emulsions was reduced about 1.5 times.

The series of impregnation tests were repeated with the same emulsion. It was concluded that the working emulsion is reusable. In impregnation tests with developed oil in water emulsions were also compared aqueous impregnates. On the Figs 1–2 are compared penetration of Tanalith E aqueous impregnant with specially colored boron-based emulsion. The absorption of emulsion in pine sapwood was in similar range with oil based and water based emulsions. The oil in water emulsion, where the maximum concentration of the rapeseed oil concentration was 4% of the emulsion, gave the best retention results. In the case of higher concentrations of oil, the permeability of the sapwood decreased and at the same time the hydrophobic effect increased.

Results in Table 1 show great difference between radial and tangential directions of penetration with Tanalith E (T27) and Boron-based rapeseed-oil emulsions (U25).

Table 1. Impregnation results of roundwood

Impregnation agent	Wood properties			Impregnation results	
	Density, $\text{kg} \cdot \text{m}^{-3}$	Annual rings $\text{no} \cdot \text{cm}^{-1}$	Moisture, %	Radial direction	Tangential direction
				Retention of emulsion, kg m^{-3}	Retention of emulsion, kg m^{-3}
T27	447	6.5	24.7	188	103
U25	464	7	26.7	207	112

Table 2. Impregnation results of sawn timber

Impregnation agent	Sawn timber crosssection, mm	Properties of timber			Retention of emulsion, Kg m ⁻³
		Density, Kg m ⁻³	Annual rings no·cm ⁻¹	Moisture, %	
T27	100 x 100	453	8	27.3	238
	22 x 100	420	6.5	22.6	270
	38 x 100	416	5	24.5	254
U25	100 x 100	446	8	26.8	220
	22 x 100	413	6	25.6	228
	38 x 100	450	7	23.8	244

In Table 2 are presented results of sawn timber impregnation with Tanalith E and Boron-based rapeseed-oil emulsions. As it follows from the results presented in Table 1 and Table 2, the better retention of impregnation agent was achieved with sawn timber. It can be explained with greater specific surface of sawn timber. By analysing the absorption depth in sawn timber the problem was to determine the amount of sapwood in cross section. Used impregnation regime allows penetrating sapwood up to 95–100%.

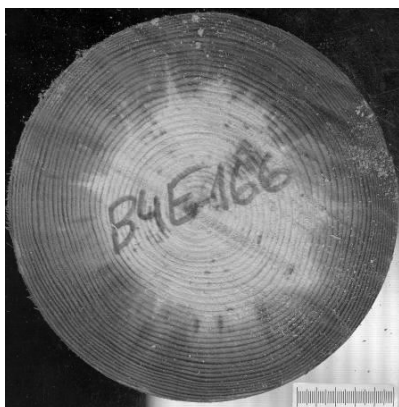


Fig. 1. Results of impregnation tests of pine roundwood with Tanalith-E agent.



Fig. 2. Results of impregnation tests of pine roundwood with coloured rapeseed oil emulsion.

Results of biological durability test

The sterilized and conditioned specimens (25 × 25 × 50 mm) were held in contact with wood destroying fungi about 8 weeks. Then the distance between the bottom surface and upper surface with rotting fungi was measured and the fungal growth of the upper surface was determined in percent of the total height of the specimen. As it follows from Fig. 3, the pine wood specimens without impregnation are almost 100% covered with rotting fungi after 8 weeks testing in climate chamber.

As it follows from the Table 3, the bio testing results at Latvian State Institute of Wood Chemistry (EN113, EN73 and EN84) revealed, that the new penetration agent

ensures effective protection against wood destroying fungi (mass loss less than 3%) and discoloring fungi (mould).

After leaching, the bio tests were performed to analyze the decay resistance effect of both impregnation agents (Table 4). The results of bio test study showed permanent decay resistance against fungi *Coniophora puteana*, *Gleophyllum trabeum*, *Poria placenta* of pine wood impregnated with rapeseed oil and boron compounds based emulsion. Tanalith E showed decreasing of fungal decay resistance in bio protection tests, which were performed after leaching of impregnation agents.



Fig. 3. Results of the biological durability test with *Coniophore puteana* of pine wood specimens with and without impregnation.

Table 3. Results of the wood biological durability tests without preliminary leaching

Impregnation agent used	<i>Coniophora puteana</i>		<i>Gleophyllum trabeum</i>		<i>Poria placenta</i>	
	Retention, kg m ⁻³	Mass loss, %	Retention, kg m ⁻³	Mass loss, %	Retention, kg m ⁻³	Mass loss, %
Engrained impregnation, bio-testing without leaching						
Rapeseed oil emulsion+B	611	0.7	802	0	714	0.2
Rapeseed oil emulsion+B	633	1.7	740	0.6	794	0
Boron compounds aqueous solution	722	0	731	0	675	0.7
Tanalith E	718	1.1	759	0	772	0.4
Tanalith E, aqueous solution	769	0.3	784	0	564	0.2
Rapeseed oil emulsion	708	37.7	770	21.3	776	23.6

Table 4. Results of the biological durability tests after preliminary leaching

Impregnation agent used	<i>Coniophora puteana</i>		<i>Gloeophyllum trabeum</i>		<i>Poria placenta</i>	
	Retention, kg m ⁻³	Mass loss, %	Retention, kg m ⁻³	Mass loss, %	Retention, kg m ⁻³	Mass loss, %
	Surface impregnation, bio-testing after leaching					
Rapeseed oil emulsion+B	744	0.1	691	0.9	755	0
Rapeseed oil emulsion+B	691	0	661	2.5	596	0.9
Boron compounds aqueous solution	700	0	664	0.2	675	0.1
Tanalith E	692	6.2	726	0.5	731	19.9
Tanalith E, aqueous solution	616	0	595	0	644	0.1
Rapeseed oil emulsion	750	48	694	24.7	757	27.3

Conclusions

1. The most effective emulsion against wood rotting fungi had the composition of 3.5% boron compounds, 2% rapeseed oil, 2% acid oil. Mass-loss caused by fungal decay was less than 3% (according to EN113).

2. The obtained results from bio testing of impregnated pine wood specimens demonstrated that the new impregnation emulsion ensures effective protection against wood rotting fungi and discolouring fungi.

3. Bio testing at Latvian State Institute of Wood Chemistry (EN113, EN73 and EN84) revealed that the new penetration agent ensures effective protection against wood rotting fungi (mass loss less than 3%) and discolouring fungi (mould).

4. The results of the bio test study showed permanent decay resistance against fungi *Coniophora puteana*, *Gloeophyllum trabeum*, *Poria placenta* of pine wood impregnated with rapeseed oil and boron compounds based emulsion. On the basis of this scientific research a utility model 'Method of preservation of pinewood' has been received (EE0068641).

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References

Baysal, E., Altinok, M., Colak, M., Toker, H. 2007. Fire resistance of Douglas fir (*Pseudotsuga menziesii*) treated with borates and natural extractives, *Bioresource Technology*, **98**, Issue 5, pp. 1101–1105.

Humar, M., Žlindra, D., Pohleven, F. 2007. Improvement of fungicidal properties and copper fixation of copper-ethanolamine wood preservatives using octanoic acid and boron compounds. *Holz Roh Werkst*, **65**: 17–21.

Islam, M.N, Ando, K., Yamauchi, H., Kobayashi, Y., Hattori, N. 2008. Comparative Study between Full Cell and Passive Impregnation Method of Wood Preservation for Laser Incised Douglas Fir Lumber, *Wood Science and Technology*, **42**, 4, pp. 343–350.

Kängsepp, K., Larnøy, E., Meier, P. 2011. The Influence of Sample Origin on the Leachability of Wood Preservatives, *Materials science*, **17**, 3, pp. 282–286.

Liibert, L., Treu, A., Meier, P. 2011 The Fixation of New Alternative Wood Protection Systems by Means of Oil Treatment, *Materials Science*, **17**, 4, pp. 402–406.

Obanda, D.N., Shupe, T.F., Barnes, H.M. 2008. Reducing leaching of boron-based wood preservatives – A review of research, *Bioresource Technology*, **99**, Issue 15, pp. 7312–7322.

Schultz, T.P., Nicholas, D. D., Preston, A. F. 2007. Perspective a brief review of the past, present and future of wood preservation. *Pest Management Science*, **63**, p. 784–788.

Vetter, L. De et al. 2009. Fungal decay resistance and durability of organosilicon-treated wood, *International Biodeterioration & Biodegradation* **63**, pp. 130–134.