PUBLICATION II

# Resistance of pine and spruce heartwood against decay – The effect of wood chemical composition and coating with water-borne wood oil product

Doc. No. IRG/WP 06-10597.
International Research Group on Wood Preservation.

**IRG/WP 06-10597**

**THE INTERNATIONAL RESEARCH GROUP ON WOOD PROTECTION**

**Section 1** **Biology**

# Resistance of pine and spruce heartwood against decay

## The effect of wood chemical composition and coating with water-borne wood oil product

Hannu Viitanen [1], Sini Metsä-Kortelainen [1], Tapio Laakso [2]

[1] VTT Technical Research Centre of Finland
P.O. Box 1000
FI-02044 VTT, Finland

[2] Finnish Forest Research Institute (Metla), Vantaa Research Unit,
P.O. Box 18, 01301 VANTAA, Finland

Paper prepared for the 37th Annual Meeting
Tromsø, Norway
18-22 June 2006

**IRG SECRETARIAT**
**SE-100 44 Stockholm**
**Sweden**
**www.irg-wp.com**

# Resistance of pine and spruce heartwood against decay

## The effect of wood chemical composition and coating with water-borne wood oil product

**Hannu Viitanen [1], Sini Metsä-Kortelainen [2], Tapio Laakso[3]**

[1] VTT, P.O. Box 1000, FI-02044 VTT, Finland, hannu.viitanen@vtt.fi

[2] VTT, P.O. Box 1000, FI-02044 VTT, Finland, sini.metsa-kortelainen@vtt.fi

[3] Metla, Vantaa Research Centre, P.O. Box 18, FI-01301 Vantaa, Finland, tapio.laakso@metla.fi

**ABSTRACT**

Natural durability of wood has been widely studied, but the combination of the natural durability and different treatments has not been the focus of many studies. The durability of wooden products is mainly based on the water permeability and the resistance against organisms. In this study, the water absorption and decay resistance of sapwood and heartwood of Scots pine and Norway spruce were examined. The effects of the wood origin and the coating with water-borne wood oil were also observed. The wood oil is penetrating treatment which is recommended for decking and garden furniture. The results were compared to the chemical composition of wood. The water absorption of untreated pine sapwood was significantly higher than that of pine heartwood or spruce. Decay resistance of pine heartwood was relatively high. However, the decay resistance was widely varied among different pine heartwood samples and in some cases this variation was significantly higher than that of pine sapwood or spruce. The effect of wood oil coating on decay resistance of pine heartwood was more significant than on pine sapwood. Elevated decay resistance was also found among coated spruce samples, but no significant difference between sap and heartwood was found. Decay resistance was comparable with water permeability and pinosylvin content of wood.

**Keywords:** Brown rot, Chemical composition, Decay resistance, Durability, Pine, Spruce, Water absorption, Wood oil treatment.

**1. INTRODUCTION**

According to the European standard EN 350-1, 2 (1994) based on laboratory and field test results using different wood species and according to experiences of using different wood material as a building material, heartwood is naturally more durable than sapwood. The better natural durability of heartwood in many wood species is mainly originated from the wood microstructure and chemical composition (Rayner and Boddy 1988). In many wood species, however, wide variation has been found between individual trees and within the individual logs. The variation of durability or decay resistance has been compensated by using chemical treatments, preservation, modifications or different kinds of surface treatments of wood (Zabel and Morrell 1992).

The heartwood of Scots pine (*Pinus sylvestris*) is classified to natural durability classes 3 - 4 (moderately to slightly durable) and the heartwood Norway spruce (*Picea abies*) belongs to the natural durability class 4 (slightly durable) according to the standard EN 350-2 (1994).

Discussion about the durability of wooden products and service life has been activated, and the exploitation of the natural durable wood material is in focus. However, there are problems concerning the using of results of biological tests for service life prediction of wooden products. There are several methods for testing the biological durability of impregnated wood material, but very few of them are suitable for testing the durability of wooden products (CEN 2005).

The natural durability of wood has been widely studied, but the combination of the natural durability and different treatments has been less studied. In this study, the water permeability and the decay resistance of coated wood against brown rot fungus were determined by using modified standard tests. Also main chemical characteristic for wood biological durability were studied.

## 2. MATERIALS AND METHODS

The sample logs of Scots pine (*Pinus sylvestris*) and Norway spruce (*Picea abies*) were sawn from different trees felled from the southern parts of Finland. The origin of trees is mentioned by using codes. The trees were from four different places: Pine trees were from Puumala (U) and Ruokolahti (R) and spruce trees were from Parikkala (P) and Heinola (H). Logs from the trees are numbered using tree code (first number) and log code (last number, 0 = butt log, 1 = middle log, 2 = top log). The logs were sawn to planks, which were as clear sapwood or heartwood as possible, and these planks were dried very carefully at temperature of 60°C. After drying and conditioning (RH65%, 20°C) to constant mass, test specimens were sawn out of the planks. For the mini decay test (Bravery 1979, Venäläinen et al. 2003), 10 replicate specimens per test material were sawn. Half of the specimens were sawn out from sapwood and half from heartwood planks, and half of the specimens were coated by dipping with water-borne primer and thereafter with water-borne pigmented wood oil. The dried matter contents of the primer and the wood oil were 16 vol-% and 11 vol-%, respectively. The wood oil is recommended to be used in wood decking and garden furniture. Pine specimens were collected from eight different logs and spruce specimens from six logs. The size of the specimen was 5 x 20 x 35 $mm^3$. The samples were dried at 60°C for 48 hours, after which they were cooled in a desiccator and weighed to an accuracy of 1 mg. Then the samples were sterilised by using a radiation dose of between 25 kGy and 50 kGy of radioisotope $^{60}Co$ source. The samples were subjected to mini decay test on agar using *Coniophora puteana* (Bam Ebw 15) as a test organism. The incubation times were 6 and 10 weeks, and after incubation the moisture content and mass loss were analysed according to the standard EN 113 (1996).

The water uptake to the uncoated and coated samples was studied by using modified EN 927-5 (2000) method. The sample size was 15 x 40 x 320 $mm^3$and three samples were used per test material. Half of the samples were coated with a brush throughout with same primer and wood oil as the mini test samples. The specimens were conditioned to constant mass at RH65% and 20°C and then weighed. Then, the specimens were floated outer face downwards in a water basin and weighed again after 72 hours floating.

The extractive content of wood samples was analysed by determining the methanol extract profile. 50 mg finely powdered sapwood and heartwood samples were sonicated with 2 ml methanol (p.a.) for 30 min. 0,4 mg diethylstilbesterol (Sigma D 4628) and 0,2 mg m-erythritol (Fluka 45670) were used as internal standards. After centrifugation 1 ml extract solution was evaporated dry under nitrogen. Before GC/MS analysis, dried samples were silylated with 0,5 ml 20 % TMSI-pyridine mixture (TMSI=1-(trimethylsilyl)imidazole). The GC/MS analyses were performed using a HP 6890 GC-system equipped with mass selective detector 5873 and HP-5 capillary column (30m x 0,25 mm i.d., 0,25 um film thickness). Helium was used as carrier gas, flow 1,5 ml/min. The chromatographic conditions were as follows: initially temperature 180°C; temperature rate, 5°C/min; final temperature 300°C for 5 min; injector temperature 280°C and split ratio 1:20. MS-interface temperature was 300°C and ion source temperature 230°C. Mass spectra were obtained by electron impact (EI mode) ionization energy 70 eV. Stilbenes (PS and PSM) and total resin acids were quantified from chromatograms using internal standards and pure reagents (Arbonova) for stilbenes and 75 % abietic acid (Fluka 00010) for resin acids.

## 3. RESULTS

### 3.1 Uptake of wood oil, water uptake and wood chemical composition

The uptake and spreading rates of wood oil during dipping and brushing treatments are presented in the table 1. Results of chemical composition of wood are shown in the figures 1 and 2. Water uptake in the floating test (EN 927-5) is presented in the figure 3.

Table 1: Average results of spreading rate of primer and wood oil by dipping and brushing ($g/m^2$).

| Wood Material | Dipping rate for decay samples | | Spreading rate for floating samples | |
|---|---|---|---|---|
| | Water based primer | Water based pigmented wood oil | Water based primer | Water based pigmented wood oil |
| Pine sapwood | 129.7 | 75.8 | 81.5 | 55.9 |
| Pine heartwood | 91.2 | 61.0 | 65.9 | 54.0 |
| Spruce sapwood | 124.7 | 75.3 | 80.6 | 70.5 |
| Spruce heartwood | 102.0 | 72.3 | - | - |

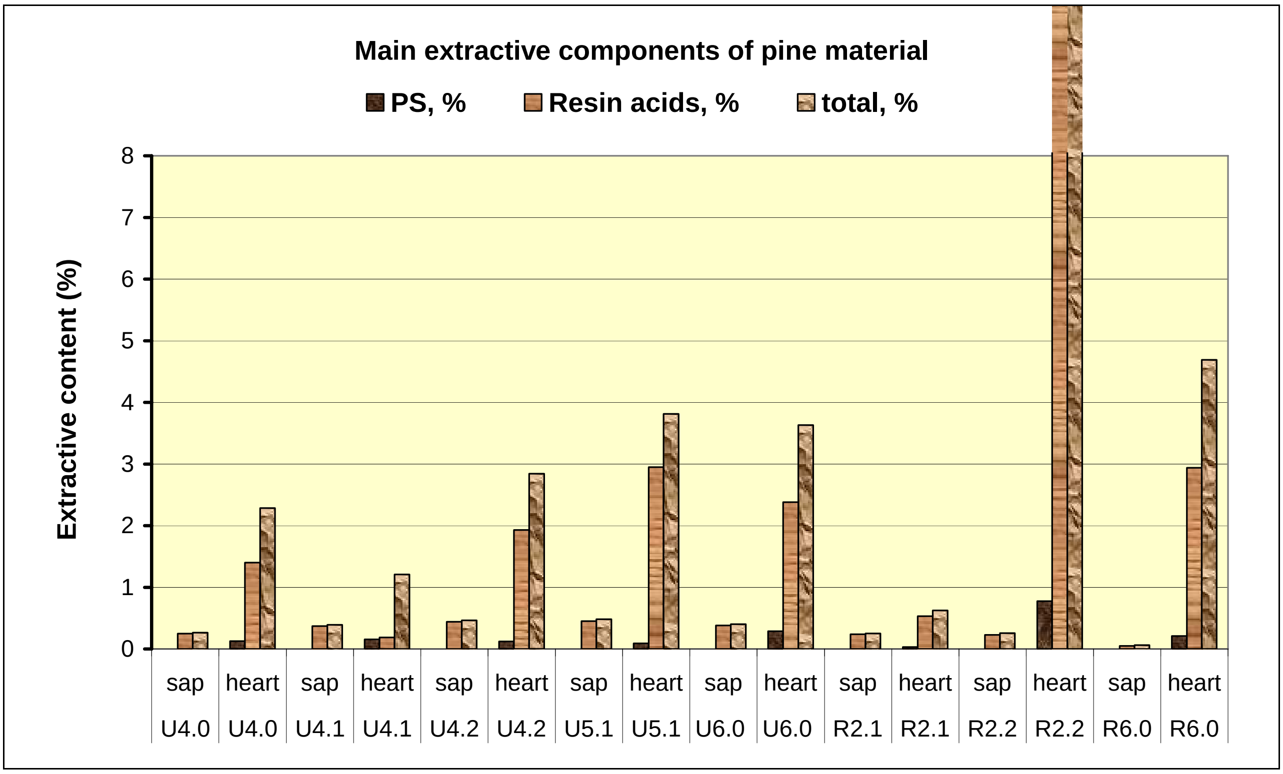



Figure 1. Analyse results of extractive contents (%) of Scots pine wood (pinosylvin, resin acids and total extractive content).

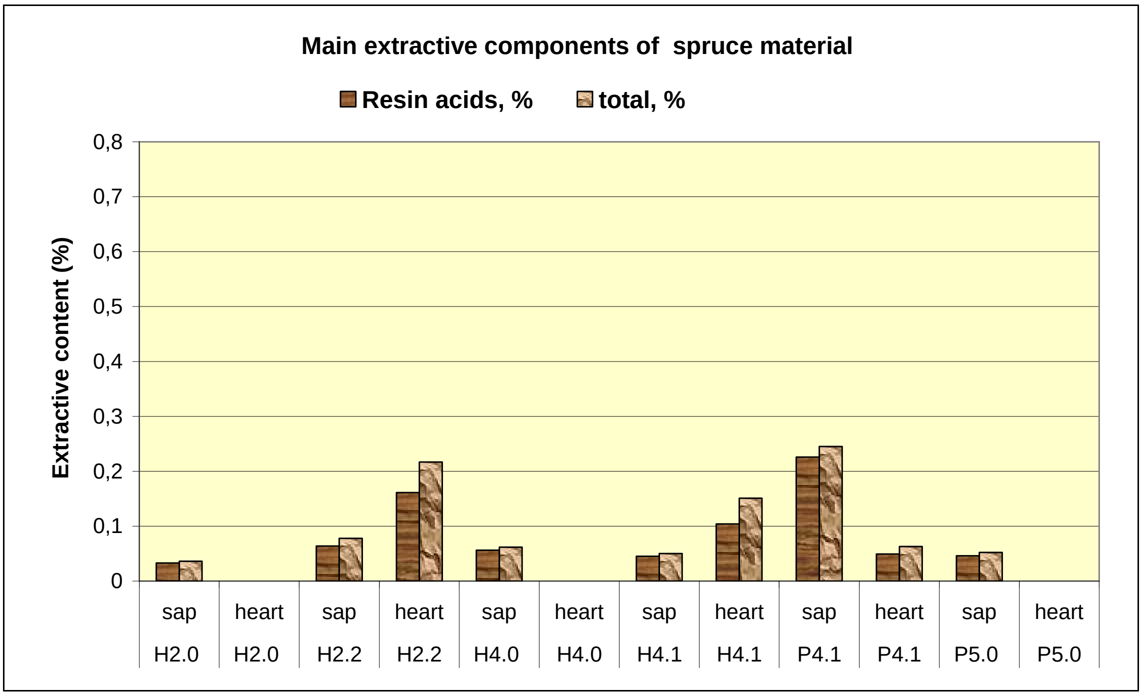



Figure 2. Analyse results of extractive contents (%) of Norway spruce wood (resin acids and total extractive content).

.

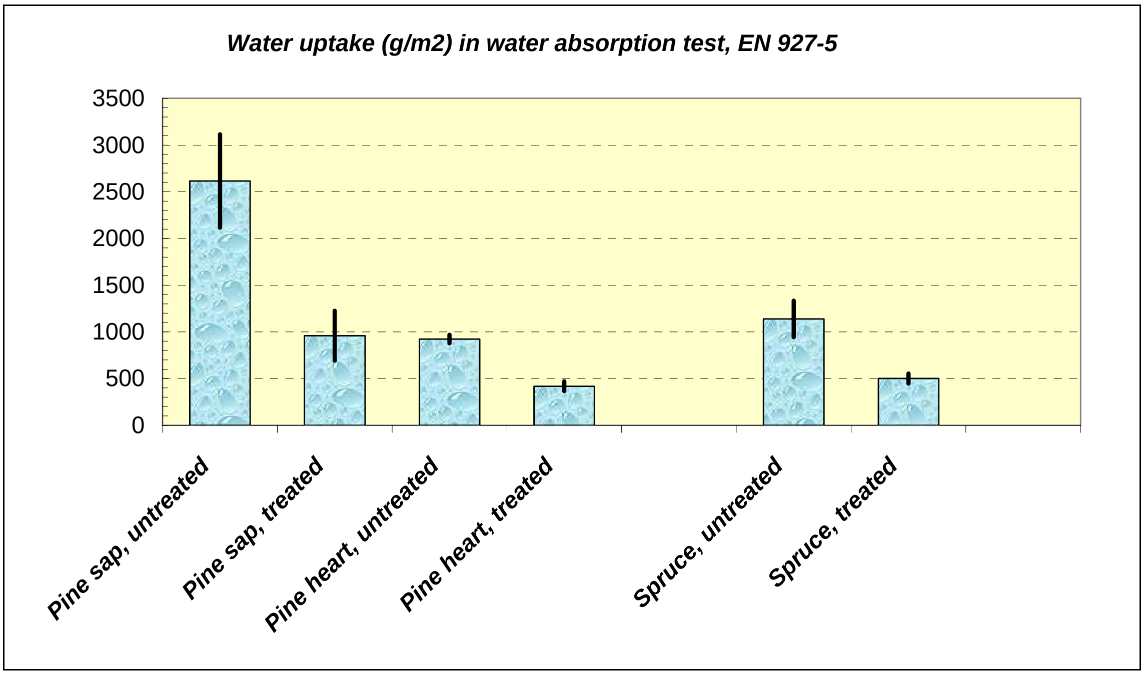



Figure 3. Water uptake of treated and untreated pine and spruce in a modified EN 927-5 test. Average results of different trees and logs.

The average water uptake of the untreated pine sapwood samples was high, which means high water permeability. The water uptake of wood oil coated pine sapwood was significant lower, as well as that of un-treated pine heartwood and untreated spruce. The water uptake was very low into the wood oil coated pine heartwood and spruce samples. The samples were not weathered prior to the water permeability test.

## 3.2 Decay resistance

Mass losses of untreated and treated pine and spruce sapwood are shown in the figures 4 and 5. The origin of wood material is mentioned by using short codes.

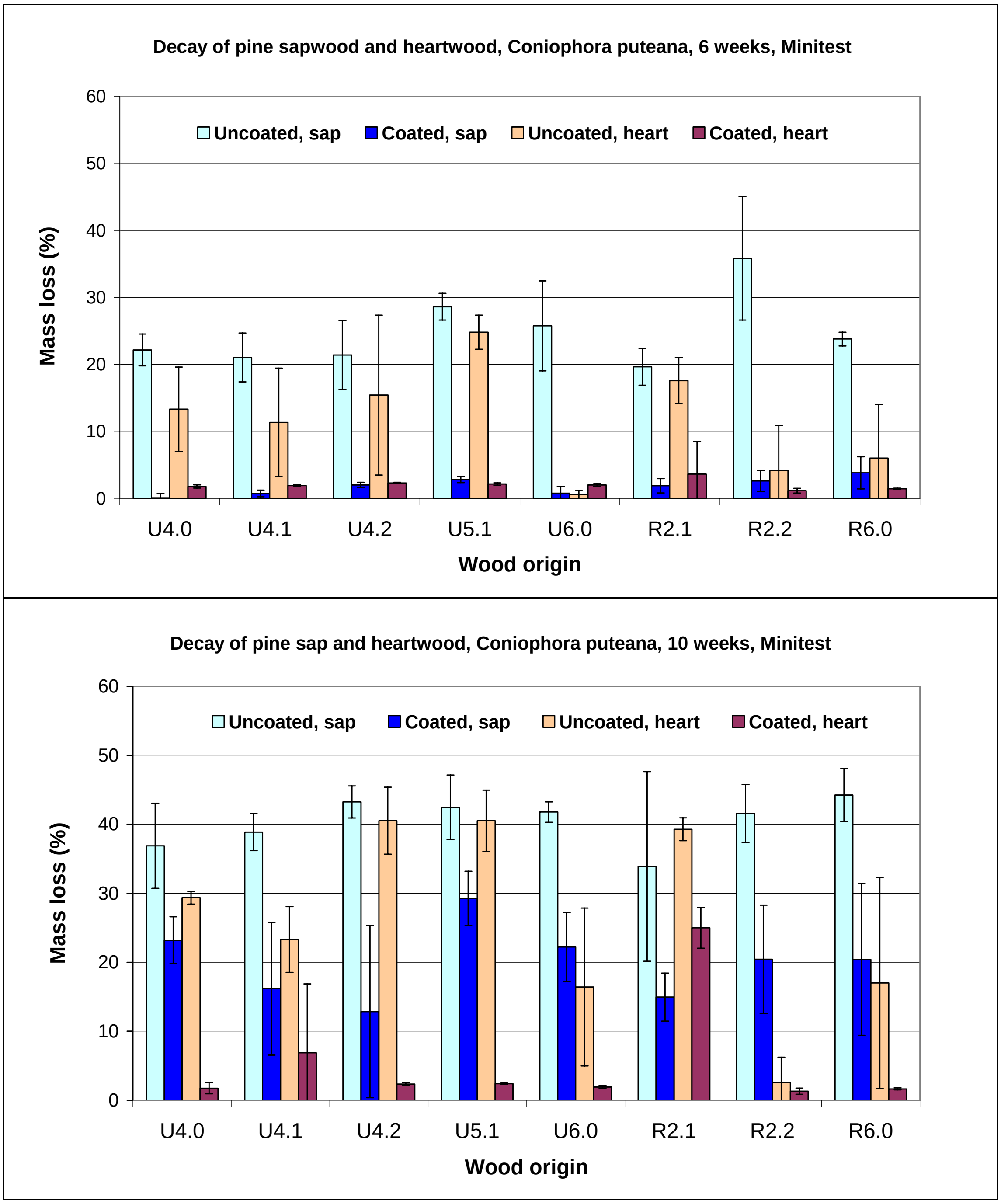
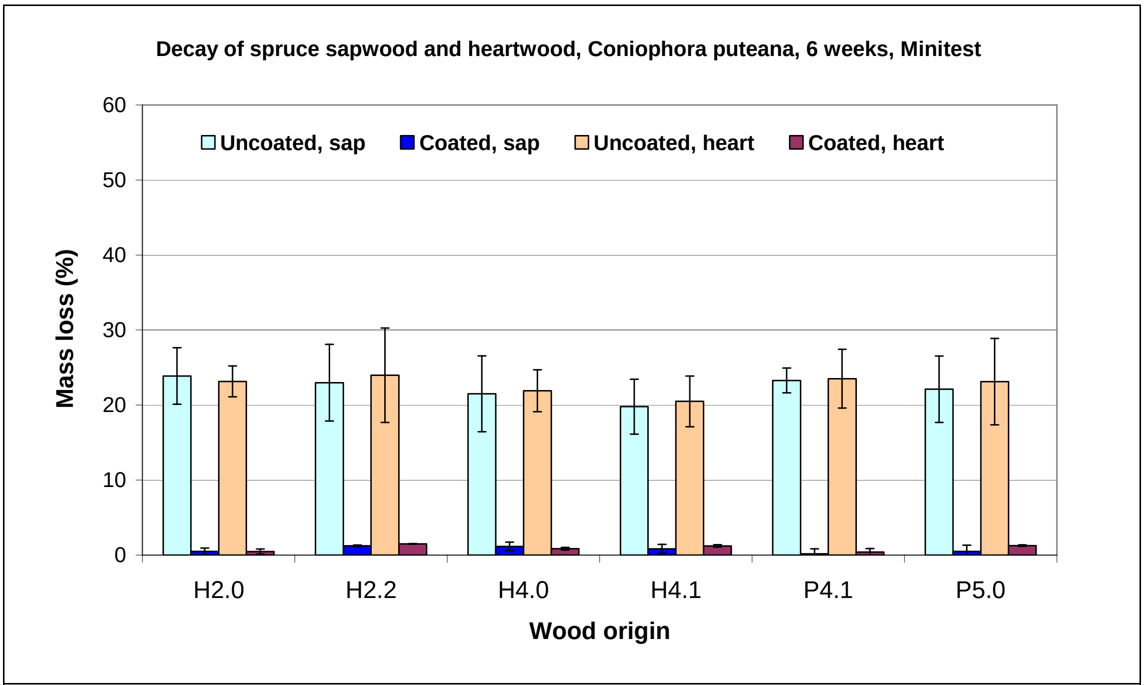



Figure 4: Mass loss of untreated and treated pine sap and heartwood after incubation of 6 and 10 weeks in decay test with *Coniophora puteana* on agar.



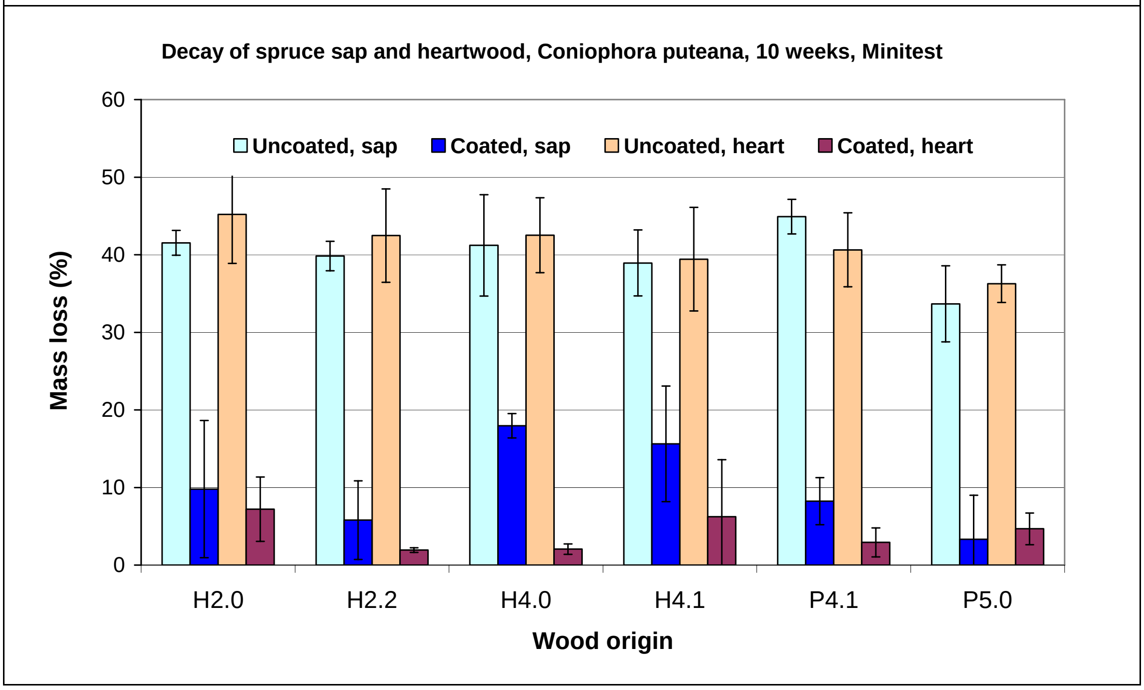




Figure 5: Mass loss of untreated and treated spruce sap and heartwood after incubation of 6 and 10 weeks in decay test with *Coniophora puteana* on agar.

The results of decay resistance in the mini test using *Coniophora puteana* correlated with the water permeability (Figure 6), but also with the wood chemical composition, especially with pinosylvin content of pine heartwood (Figure 7). The average mass loss of untreated pine sapwood after exposure of 6 weeks varied between 20 and 35 % and after 10 weeks between 35 and 45 %. There was a very high variation among mass losses of untreated pine heartwood after both exposure times. After 6 weeks, the lowest average mass loss of pine heartwood was around 4 % and the highest around 25 %, and the mass loss of samples treated with wood oil was very low for both sap and heartwood. After 10 weeks exposure, the mass losses of all untreated heartwood samples were higher than after 6 weeks, but samples from one log (R2.2) showed especially low mass loss reflecting high a extractive content. However, the mass loss of the samples from R2.1 (from the same tree as R2.2) was significantly higher reflecting a very low extractive content of wood. The results of the chemical analyses of the heartwood sample R 2.1 in the figure 1 show a relatively low extractive content, which may refer to possibility of having sapwood or equal than sapwood in this heartwood sample.

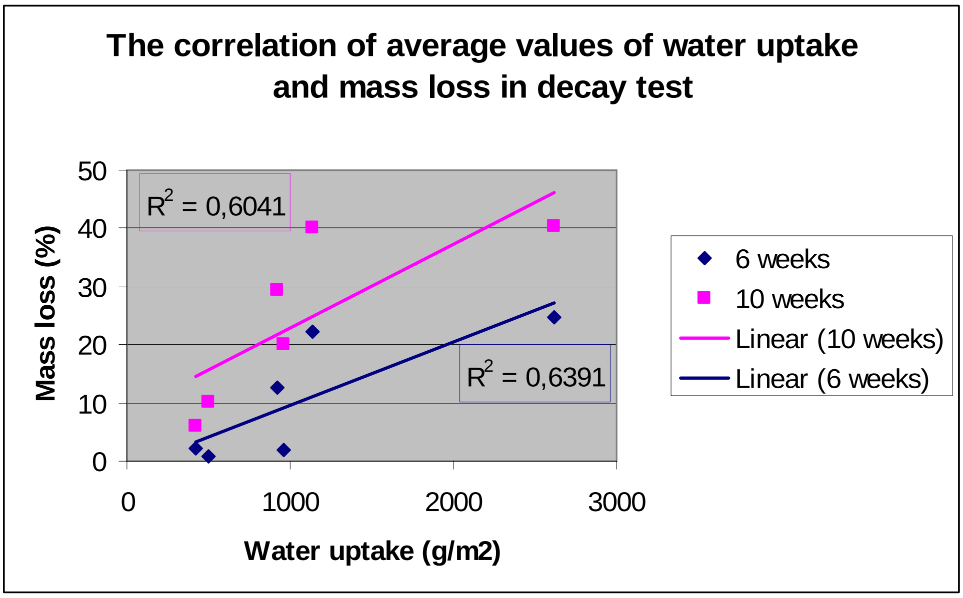



Figure 6. The correlation of results of water absorption and decay tests.

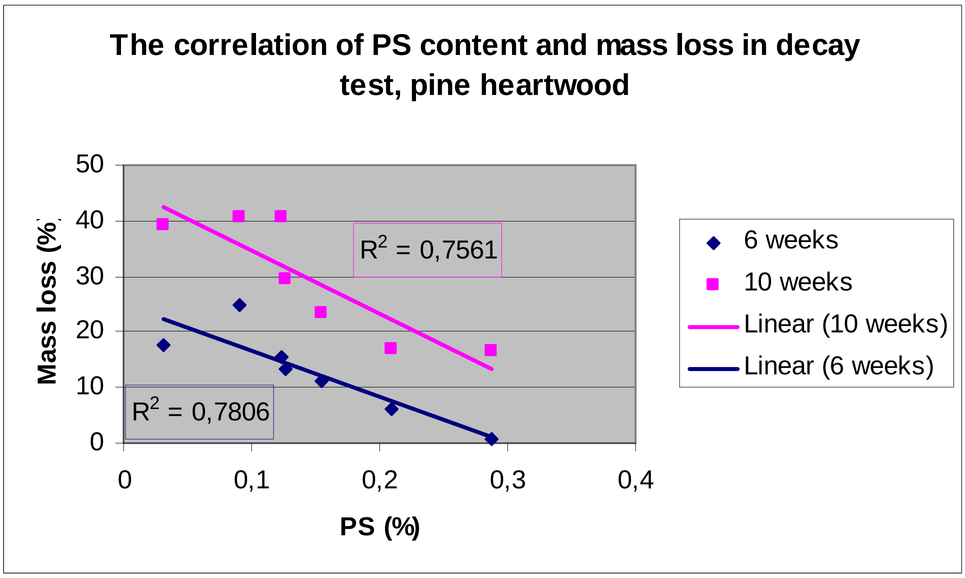



Figure 7. The correlation of pinosylvin content and mass loss in decay test, pine heartwood.

## 4. DISCUSSION

The results of the decay test of spruce differed notably from the results of pine. There was not any significant difference between mass loss of untreated spruce sap and heartwood, and the mass loss of spruce heartwood was significantly higher than that of pine heartwood. The mass loss of wood oil treated sapwood of spruce was significantly lower than that of untreated sapwood. Also, the decay resistance of wood oil treated spruce heartwood was in average around the same level than that of treated pine heartwood. Water permeability of spruce was significantly lower than that of pine sapwood. This may add the resistance and durability of spruce in use condition, where moisture stress is not too severe, e.g. claddings, fences (EN 335-1). The results of the present screening test showed, that coated and selected pine heartwood would be reliable material for wooden products also in high moisture exposure conditions (e.g. applications in use class 3 like decking and garden furniture in this case). Wide variation was found within the decay resistance of pine heartwood even among the limited test material used in the present study. Variation in decay resistance of pine heartwood has been found in many studies (van Acker et al. 1999, Harju et al. 2002, Venäläinen et al. 2003). In this study, pinosylvin seemed to be the most important chemical compounds for the decay resistance against brown rot. The effect of pinosylvin content on decay resistance has been found also in many other studies (e.g. Harju et al. 2003, Venäläinen et al. 2003).

The spruce could be an optional wood species for standardized tests for measuring resistance of coated wood products against decay, because the effect of the wood part (sapwood / heartwood) is not so important than with pine. This would be argued also due to the use of spruce as a material for coated building products. For screening tests of biological durability of wooden products, it would be useful to have several exposure time periods during a test. This would help to evaluate the effect of resistance of wooden products for preliminary evaluation of life time in different exposure conditions. Also water permeability tests should be included. In this context, the screening test system should be as convenient, reliable, fast and cheap as possible.

## 5. CONCLUSIONS

The chemical composition of wood seemed to be an important factor effecting on the decay resistance of wood. Especially the pinosylvin content of pine heartwood was a significant factor. The difference of decay resistance between pine heartwood and spruce heartwood reflected very well with the chemical composition of the wood. Surface treatment using water-borne wood oil, however, affected on the water permeability and also on the decay resistance of wood. The samples were not preconditioned (weathered, leached or evaporated) prior to the exposure to decay fungus. In the next phase, the decay resistance after 4 and 10 months natural weathering will be studied.

## 6. ACKNOWLEDGEMENTS

This work is a part of the consortium study ‘Optikesto - Optimising durability and standardization of resistance of wood products, to attack of discolouring and decay fungi’ financed by TEKES, Industry, VTT and Metla. The practical work for wood drying, working and sample preparation at VTT was performed by Holger Forsen, Veikko Tarvainen, Pentti Ek and Heikki Murto. The practical work for decay test was performed by Liisa Seppänen, Hellevi Botska and Pauliina Saurus. The sample preparation for chemical analyses at Metla was performed by Irmeli Luovula.

## 7. REFERENCES

Bravery A F (1979). A miniaturised wood-block test for rapid evaluation of wood preservative fungicides. IRG/WP 2113. International Research group of Wood Preservation.

CEN/TC 38 N 1618 (2005). Report of the COST E 37 Task Force. Performance-based classification in wood durability and protection: a proposal to CEN TC 38 for future progress.

European standard EN 113 (1996). Determination of toxic values of wood preservatives against wood destroying Basidiomycetes cultured on agar medium. Brussels. European Committee for Standardization. 24 p.

European standard EN 335-1 (1992). Durability of wood and wood based products – Definition of hazard (use) classes – Part 1: General. Brussels. European Committee for Standardization.

European standard EN 350-1 (1994). Wood and wood based products - Natural durability of wood - Part 1: Principles of testing and classification of the natural durability of wood. Brussels. European Committee for Standardization. 14 p.

European standard EN 350-2 (1994). Wood and wood based products - Natural durability of wood - Part 2: Natural durability and treatability of selected wood species of importance in Europe. Brussels. European Committee for Standardization. 37 p.

European standard EN 927-5. (2000). Paints and varnishes – Coating materials and coatings systems for exterior wood – Part 5: Assessment of the liquid water permeability. Brussels. European Committee for Standardization. 6 p.

Harju AM, Kainulainen P, Venäläinen M, Tiitta M, Viitanen H (2002) Differences in resin acid concentration between brown-rot resistant and susceptible Scots pine heartwood. Holzforschung. 56 (2002) 479-486

Harju AM, Venäläinen M, Anttonen S, Viitanen H, Kainulainen P, Saranpää P., Vapaavuori E (2003). Chemical factors affecting the brown rot decay resistance of Scot pine heartwood. Trees. 17: 263-268.

Rayner ADM, Boddy L (1988) Fungal decomposition of wood. Its biology and ecology. Portsmouth, John Wiley, Sons.

Van Acker J, Militz H, Stevens M (1999) The significance of accelerated laboratory testing methods determining the natural durability of wood. Holzforschung 53: 449–458.

Venäläinen M, Harju AM, Anttonen S, Kainulainen P, Viitanen H. Nikulainen H. (2003). Variation in the decay resistance and its relationship with other wood characteristics in old Scots pines. Annales of Forest Sciences: 60: 409-417.

Zabel RA, Morrell JJ (1992) Wood Microbiology: Decay and its Prevention. Academic Press Inc., California.