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THE RESISTANCE OF PINUS *SYLVESTRIS* L. WOOD MODYFIED WITH FURFURYL ALCOHOL FOR DESTRUCTION BY *RETICULITERMES LUCIFUGUS VAR. SANTONENSIS* (DE FEYTEAUD)

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ABSTRACT

The aim of the research was to determine the changes in susceptibility of Scots pine (*Pinus sylvestris* L.) wood to feeding by subterranean termites *Reticulitermes lucifugus var. santonensis* conducted in accordance with the ASTM D 3345-08: 2009 The sapwood after modification in furfuryl alcohol at concentration 50% with the addition citric acid at concentration 1%, natural sapwood and heartwood experiment as a compulsory reference material were used for the tests. The blocks of Scots pine wood were used to create seasoned samples of 7% ± 1% moisture content. The blocks with dimensions of 25.4 x 25.4 x 6.4 mm were made from three trees. Each variant was represented by 5 blocks. All wood blocks were freeze-dried before starting the experiment in order to measure the dry weight. After 4 weeks of termite feeding visually rates of wood destruction were: 9 (light attack) for modified sapwood, 2.8 (heavy attack/failure) for natural sapwood and 8.2 (light attack/moderate attack) for natural heartwood. The loss of wood weight of blocks was: 0.01 g for modified sapwood, 0.071 g for natural sapwood and 0.21 g for natural heartwood. The mortality of the termites in the case of modified sapwood was complete. The termite mortality in natural sapwood has been classified as slight, and in natural heartwood as slight to complete.

KEYWORDS: Biodeterioration, modified wood, subterranean termite.

INTRODUCTION

Scots pine (*Pinus sylvestris* L.) is the predominate species in Poland apart from the mountainous area, where the other species like spruce, fir and beech have a greater rate. Pine consists 58% of the forest area (Zajączkowski et al. 2019), providing most of the wood used in construction. Pine wood is not resistant to biological destruction therefore it seems to be a material predestined for modification (EN 350: 216).

Wood modification is defined as the process of joining a simple chemical compound to the reactive center of a cell wall polymer, with or without a catalyst, to form a covalent bond between them. Significant developments in the area of wood modification have been achieved during the last three decades. A number of wood modification techniques such as chemical and impregnation modifications or heat treatments have been introduced, and some of these technologies have reached the industrial level (Mantanis 2017). The furfurylation might be the answer to optimize the utilization of some tree species. Various catalysts can be used here (Sejati et al. 2017). Furfurylated wood has many positive qualities: is more hard and rigid, has an excellent tropical-timber-like appearance and texture, and it is better suited for applications like decking (Mantanis 2017). Several factories produce furfurylated wood flooring and exterior wall cladding. Furfurylated wood, in addition to good physical and chemical properties and resistance to biological degradation factors, has an attractive appearance comparable to the sought-after exotic species (Ratajczak et al. 2011). In last years, furfurylation studies have been undertaken for several Pinus species in different ranges: *P. sylvestris* L. (Baysal et al. 2004, Hadi et al. 2005, Bartkowiak and Doczekalska 2017), *P. pinaster* Aiton (Esteves et al. 2011), *P. radiata* D.Don (Gascón-Garrido et al. 2013), *P. massoniana* Lamb Thermal wood modification is competitive with furfurylation and acetylation among wood modification methods (Albrektas et al. 2020).

The problem of termites has received considerable attention in Europe, following their persistent persistence in some cities and the spread and effectiveness of control methods (Becker 1970, Becker et Kny 1977, Seelensschlo 1988, Ferrari et al. 2011). The Atlantic regions of France and Portugal, Italy and the Mediterranean regions of Spain and France, regions of the Adriatic and Black Sea coasts of several countries are home to termites in Europe. So far, 5 species of termites have been found in Europe (Dominik and Starzyk 2004). Recently, the spread of soil termites in Great Britain is of particular importance (Laine 2002).

Hence the interest in the natural resistance of wood of native species (Schultze-Dewitz 1958, Krajewski et al. 2015, Krajewski et al. 2016, Krajewski et al. 2019) and tree species introduced in Europe (Schultze-Dewitz 1958) to the foraging of various species of termites. Becker and Petrowitz (1971) found that pine wood has ketones that attract insects and that aldehyde repels various species of termites. In addition, different termite species react differently to furfural.

The results of furfurylated wood tests carried out according to various procedures in relation to dry wood termites (*Cryptotermes cynocephalus* Light), subterranean termites (*Macrotermes gilvus* Hage) (Hadi et al. 2005) and some Mediterranean termites (Reticulitermes spp.) have been published. According to the EN 350: 2016 standard, the natural pine heartwood is rated as "unstable (S)" in relation to termite feeding. Pine white is even more susceptible to destruction. The research was undertaken in order to answer the question: what is the resistance of pine whiteness modified with furfuryl alcohol to destruction caused by *Reticulitermes lucifugus var. santonensis* (De Feyteaud).

MATERIAL AND METHODS

The tests were conducted out in accordance with the ASTM D 3345-08: 2009 standard, which aims to test the resistance of wood and wood-based materials to subterranean termites. The natural sapwood, natural heartwood and furfurylated sapwood of Scots pine (*Pinus sylvestris* L.) were used in the research. The blocks of each category of wood came from three trees, about 70 years old. The test blocks had dimensions 25.4 x 25.4 x 6.4 mm and a moisture content of 7%. In order to determine the dry weight of the wood prior to the experiment, all blocks were freeze-dried. The average density of dry natural sapwood was 0.60 $g \cdot cm^{-3}$.

The wood samples modified with furfuryl alcohol were prepared as follows. To 200 ml solution of furfuryl alcohol in aqueous concentration of 50% was added 2 g of citric acid as a catalyst. Then the measured and weighed samples were placed in a beaker, loaded with a glass stopper and poured with the solution. All samples were completely immersed in the liquid. After pouring, the vessel was placed in a vacuum desiccator. The vessel was depressurized to value 0,02 MPa. The pump was stopped after approximately 20 min, when air bubbles ceased to be emitted from the samples. The amount of the absorbed solution and furfuryl alcohol are given in Tab. 1.

Tab. 1: The uptake of absorbed solution and furfuryl alcohol.

Sample number	The absorbed solution (g)	The absorbed furfuryl alcohol (g)
1	2.84	1.62
2	2.84	1.62
3	2.90	1,65
4	2.87	1.64
5	2,46	1.40
average size	2.78	1.59

After about an hour of impregnation, the samples were removed from the solution and wrapped in aluminium foil to prevent volatilization of the furfural. These samples, in aluminium foil, were sealed in an oven at 120°C for 24 hours. The furfurylated sapwood samples were then removed from the foil and placed back in the oven at 120°C for a further 24 hours. In order to determine the dry weight of the furfurylated wood, the blocks were again freeze-dried. Following these steps, the samples were measured and weighed and the density of blocks was calculated. The density of blocks was also calculated. Average density of modified wood with furfuryl alcohol was 0.67 $g \cdot cm^{-3}$. The blocks after modified wood with furfuryl alcohol had the moisture content of 2% at the start of the biological test.

During the biological test, each block was placed individually on the bottom of a glass vessel and covered with 200 g of sand which was sieved, rinsed and heat sterilized. The volume of the vessel was 450 ml. The amount of water used to moisten the sand in the test vessel was then reduced by 7% of the sand's saturation point. Then each vessel was filled with 1 ± 0.05 g of termites. Pseudergates include for over 90% of the individuals in each vessel. The test vessels with wooden blocks and termites were placed in an incubator at 27°C for 4 weeks. The water content of the test vessels was replenished weekly.

The approximate termite mortality rate was determined after a period of four weeks, according to the ASTM D 3345-08: 2009 procedure on a scale of: slight (0 - 33%), moderate (34 - 66%), heavy (67 - 99%) and complete/total (100%). After the test was completed, the wooden blocks were weighed after removing termites and sand, and then freeze-dried in order to determine the final dry weight. The blocks were photographed in order to determine the extent of the damage. The degree of damage of the wood blocks was determined visually according to the scale recommended in ASTM D 3345-08: 2009, i.e. 10 – sound, surface nibbles permitted, 9 – light attack, 7 – moderate attack, penetration, 4 – heavy, 0 – failure. In ambiguous cases, the intermediate value was written: 9/7=8, 4/0 = 2.

For each category of wood, the average degree of block destruction was calculated. The blocks' weight losses and the average weight losses for each wood category were also calculated. The significance of the difference in the obtained mean results was statistically verified using the Chebyshev inequality (reference) (Eq.1). Each time the absolute value of the difference between the arithmetic means of wood damage levels in two variants of the experiment was higher than or equal to the triple value of the standard error of the difference between the average wood damage levels:

$$|\overline{x_1} - \overline{x_2}| \geq 3 \cdot \varepsilon(\overline{x_1} - \overline{x_2}) \tag{1}$$

where: $\bar{x}_1$ - arithmetic average of wood damage level of natural wood or arithmetic average of weight loss of natural wood (g), $\bar{x}_2$ - arithmetic average of wood damage level of modified wood or arithmetic average of weight loss of modified wood (g), $\varepsilon(\bar{x}_1 - \bar{x}_2)$ - value of the standard error of the difference between of wood damage level or the average loss of wood weight (g). Then the difference of average values was recognized as statistically significant. Otherwise, it was recognized as accidental.

RESULTS AND DISCUSSION

The colour of pine wood modified with furfuryl alcohol was strongly darkened. The moisture content of the wood blocks at the end of the bioassay were: for natural sapwood 39% and for modified sapwood with furfuryl alcohol 56%. The results of the experiments involving visual estimation of the degree of wood destruction, loss of wood mass and termite mortality are shown in Tab. 2.

Tab. 2: Visually rates of wood destruction, loss of wood weight (g) and termite mortality.

Pinus sylvestris	**N° of block**	**Visually rates of wood destruction**	**Loss of wood weight (g)**	**Termite mortality** ASTM D 3345-08: 2009
Sapwood modified with furfuryl	1	9	0.00	complete
	2	9	0.02	complete
	3	9	0.04	complete
	4	9	0.01	complete
	5	9	0.00	complete
	average	**9**	**0.01**	-

Natural sapwood	1	4	0.54	slight
	2	0/4=2	0.56	slight
	3	4	0.82	slight
	4	0/4=2	0.81	slight
	5	0/4=2	0.83	slight
	average	**2.8**	**0.71**	-
Natural heartwood	1	9	0.10	complete
	2	9	0.12	complete
	3	7	0.35	slight
	4	9/7=8	0.19	slight
	5	9/7=8	0.30	slight
	average	**8.2**	**0.21**	-

The average weight loss of the modified pine sapwood was 0.01 g, while that of the natural sapwood was 0.71 g. The visually rates of wood destruction were also clearly different - 2.8 and 9, respectively. An example of the condition of the wood blocs after 4 weeks of termite feeding is presented in Fig. 1

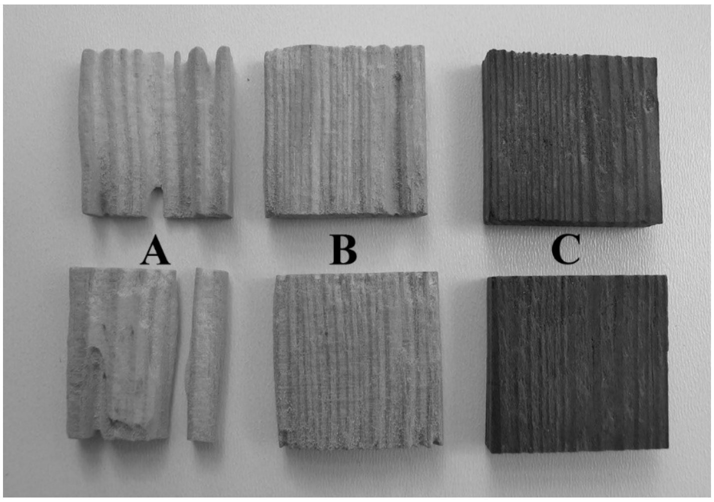

Fig. 1: An example of the wood condition (Pinus sylvestris) after 4 weeks of termite feeding: A – natural sapwood, B – natural heartwood, and C – sapwood modified with furfuryl alcohol.

The obtained absolute difference of arithmetic means of loss of wood weight in the tested variants was greater than three times the value of the standard error of the difference of mean losses of wood weight, which is illustrated in Tab. 2.

Tab. 3: Statistical verification of the difference in weight loss of wood samples (Chebyshev inequality).

Variants of the experiment	$\lvert x_1 - \bar{x}_2 \rvert$	$3 \cdot \varepsilon (x_1 - \bar{x}_2)$	**Evaluation**
Natural sapwood - natural heartwood	0.50	0.25	difference significant
Natural sapwood - modified sapwood	0.70	0.2	difference significant
Natural heartwood - modified sapwood	0.20	0.15	difference significant

Using the criterion of visually rates of destruction, modified sapwood was also significantly more resistant to termite feeding than natural heartwood. The modified sapwood was slightly attacked. Visually rates of destruction for modified sapwood of *Pinus sylvestris* were 9 and for natural heartwood 8.2. The obtained absolute difference in arithmetic means of visually rates of wood destruction in the studied variants was greater than three times the value of the standard error of the difference in mean losses of wood mass, which is illustrated in Tab. 3.

Tab. 4: Statistical verification of the difference in degrees of visual damage assessment of wood samples (Chebyshev inequality).

Variants of the experiment	$\lvert \bar{x}_1 - \bar{x}_2 \rvert$	$3 \cdot \varepsilon (\bar{x}_1 - \bar{x}_2)$	Evaluation
natural sapwood - natural heartwood	5.40	1.85	difference significant
natural sapwood - modified sapwood	6.20	2.01	difference significant
natural heartwood - modified sapwood	0.80	1.12	difference significant

Termites of the genus *Reticulitermes* (Isoptera: Rhinotermitidae) occur in Europe in the Mediterranean zone, but their presence in urban areas extends northwards beyond their natural range. The local distribution of the occurrence sites of these species suggests a connection with human activity (Dominik et Starzyk 2004, Uva 2006). The observations of the impact of climate change on phytophagous insects also suggest that the role of thermophilic species is currently increasing. This is mainly due to the shifting of their range to the north and to higher altitudes (Jaworski et Hilszczański 2013). Therefore, one can expect a shift to the north of termite distribution sites in Europe. This justifies undertaking research on the resistance of native wood species to termite feeding and on ways to protect wood against these insects.

Sapwood of Scots pine appeared in many publications on the possibility of termite feeding only as a control variant (Krajewski et al. 2015, 2016, 2019). At the same time, it was completely immune to the attack of soil termites However, it is much more susceptible to termite destruction (light attack / moderate attack) than natural heartwood, which is rated as susceptible (S) (PN-EN 350: 2016). The termite feeding susceptibility of individual blocks of natural heartwood is more variable than the susceptibility blocks of modified sapwood with furfuryl. Visually rates of natural sapwood destruction were also quite variable and rated only at 1.6; 1.6 and 3.2 (heavy/failure). The present test showed increased hydrophobicity of pine sapwood and reduced susceptibility to biological destruction, which is expected after wood modification (Ratajczak et al. 2011). The humidity of furfurylated and unmodified wood samples at the end of the biological test differed by as much as 17%. A relatively high concentration of furfuryl alcohol (50%) was used here.

It is difficult to compare the protective effect of fyrfuryl alcohol on pine sapwood with other publications, as it has been used in various concentrations, with the addition of various substances, on various species of pine and against various factors of wood degradation (Baysal et al. 2004, Esteves et al. 2011, Bartkowiak and Doczekalska 2017). The high efficiency of protection of *Pinus pinaster* sapwood was also obtained using a 70% solution of furfuryl alcohol against the fungi *Postia placenta* and *Coniophora puteana* (Esteves et al. 2011).

Contrary to thermal modification, modification with furfuryl alcohol resulted not in a decrease in weight, but in an increase in the weight of the wood. Modification of Scots pine wood with furfuryl alcohol resulted in an increase in sapwood mass by 15% and heartwood mass by 12% in this study. For comparison, modification of Scots pine wood with the aqueous solution of furfuryl alkohol with concentration of 5% w/w with the addition of maleic anhydride in an amount 1% caused weight percent gain (WPG) o 8.2% (Bartkowiak et Doczekalska 2017). In studies (Estevans et al. 2004) the weight percent gain (WPG) of *Pinus pinaster* was on average 38%.

It is interesting to compare the resistance to feeding of very durable wood species with pine wood modified with furfuryl alcohol. Grace et al. (1994), Grace et al. (1996) and Kard et al. (2007) during field tests with *Coptotermes formosanus* Shiraki found that *Cryptomeria japonica* (sugi, Taxodiaceae), *Cordia subcordata* (kou, Boraginaceae), *Calophyllum inophyllum* (kamani, Gutiferae), *Thespesia populnea* (milo, Malvacae) and *Eukalyptus microcorys* (Thallowood, Myrthaceae) were very resistant to termite feeding. *Pandanus tectorius* (hala, Pandanaceae) was moderately resistant and the resistance of *E. microcorys* was reduced to equivalent after 3 years of external exposure. *Acacia koa* (koa, Leguminosae), *Metrosideros polymorpha* (ohia Lehua, Myrtaceae) and *Eukalyptus robusta* (Robusta, Myrtaceae) were slightly resistant to termite attack, by contrast *Eukalyptus deglupta* (bagras eukaliptus), *Cardwellia sublimis* (silky oak) and *Albizia falcataria* (Molucca albizia, Leguminosae) were very susceptible. Visually rates heartwood destruction was 10 for heartwood of *Erythrophleum fordii* and for *Hopea pierrei*, and for European oak (*Quercus robur* L.) (Krajewski et al. 2019). Thus, the modification of Scots pine sapwood increases the wood resistance in relation to termite feeding to the level of the heartwood of oak. Due to the different species of termites used in the tests, it is of course difficult to say unequivocally whether the furfuryl alcohol-modified pine white will be equally resistant to termite feeding *Reticulitermes grassei* and *Coptotermes formosanus*, as it was in the case with *Reticulitermes lucifugus* var. santonensis. However, it seems that the difference should not be significant.

The low-furfurylated Scots pine wood was poorly protected against attack by some species of termites. In opposite, medium- and high-furfurylated specimens (too *P. sylvetris*) were highly resistant to attack by dry wood termites (*Cryptotermes cynocephalus* Light) and subterranean termites (*Macrotermes gilvus* Hage) (Hadi et al. 2005). The resistance of two *Pinus* species were tested in laboratory no-choice test following the standard EN-117 with different technologies against Mediterranean termites (*Reticulitermes* spp.). Furfurylated wood can improve the resistance to termites, but only without leaching (Gascón-Garrido et al. 2013). Therefore, further research is needed to determine the minimum amount of furfuryl alcohol in pine wood that protects against R. *lucifugus var. santonensis*

CONCLUSIONS

(1) The Scots pine sapwood modified with furfuryl alcohol in aqueous concentracion of 50% with the addition of citric acid as a catalyst obtained increased resistance to destruction by termites. The average degree of wood destruction is rated light attack, while natural sapwood is rated heavy/failure. The modification of the wood in this way resulted in complete termite mortality. *(2)* The modification of Scots pine sapwood with furfuryl alcohol provides greater resistance to termite feeding than the natural heartwood of Scots pine.

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