Abstract: The possibility of improving the resistance of beech wood (Fagus moesiaca) to two dangerous wood decaying fungi: Trametes versicolor – white rot agent and Coniophora puteana – brown rot agent, was investigated under laboratory conditions. The heat treatment of 200°C/5 hours, vacuum impregnation with boric acid (5%) and the combination of the heat treatment and subsequent impregnation were used. Thermal modification of wood (TMW) and impregnation with boric acid (B) increase the resistance of wood to the investigated fungi. Weight loss of the samples impregnated with boric acid (B) was approximately the same for both tested fungi. Impregnation of thermally modified samples (TMW + B) increased the resistance to Trametes versicolor in comparison to the samples of wood that were only thermally treated (TMW), but the resistance of these samples was lower than the samples of wood impregnated with boric acid (B). Thermal modification + impregnation with boric acid (TMW + B) showed lower resistance to Coniophora puteana fungus than these treatments (TMW or B) separately.

Keywords: Trametes versicolor, Coniophora puteana, Fagus moesiaca, natural resistance, decay, mass loss
битак масе узорака импрегнисан борном киселином (B) приближно је исти за обе ис-питиване гљиве. Импрегнација борном киселином претходно термично третираних узорака (TMW + B) резултирала је већом отпорношћу против гљиве Trametes versicolor у поређењу са узорцима који су третирани само топлотним третманом (TMW), али је отпорност ових узорака смањена у поређењу са отпорношћу узорака импрегнираних борном киселином (B). Термичка обрада + импрегнација борном киселином (TMW + B) показала је мању отпорност против гљиве Coniophora puteana од одвојених третма-на (TMW или B).

Кључне речи: Trametes versicolor, Coniophora puteana, Fagus moesiaca, природна отпорност, трулеж, губитак масе

1. INTRODUCTION

Beech is one of the broadleaved species whose wood is highly sensitive to wood decaying fungi. However, if dried properly, its wood provides one of the most beautiful materials for furniture and parquet as well as for a wide range of wooden products in everyday human life.

In order to improve the quality of wood in use, non-toxic modification of wood that involves the use of chemical, physical and biological methods of improving the properties of this material, such as dimensional stability, greater resistance to wood decaying fungi, insects or weathering, etc. aroused deeper interest in the 20th century. It became of essential importance to look for non-toxic and environmentally-friendly substances and agents.

Although thermal modification of wood has been a widely used method in the last two decades, it is still being developed. The method of thermal modification of wood was known in the early twentieth century, but it has been in commercial use only for the last ten years (Hill, 2006). In the first half of the twentieth century, there were attempts to develop the methods of furfurilation and acetylation, but without any significant results for commercial use. However, during the 70’s and 80’s thermal modification of wood came into the focus again and the first commercial method was developed in Finland (Hill, 2006).

The method of thermal modification is mainly aimed at solving the problem of dimensional stability of wood which would allow the application of wood in all conditions, from the interior (floor) to the exterior conditions and the conditions of increased humidity. However, this method does not provide complete protection of wood against insects and fungi, especially in outdoor conditions with increased humidity. This has been confirmed by a number of studies (Rapp and Sailer, 2000; Jämsä and Viitanemi, 2001; Vermais, 2000; Syrjänen et al. 2000; Miltiz and Tjeerdsmma, 2000; Mayes and Oksanen, 2002). The solution to these problems would allow the use of TMW in conditions with a high probability of wood infection.

The purpose of this method is primarily to improve wood characteristics of certain tree species, such as beech which is very widespread in Serbia but shows great dimensions instability with the changes in humidity. Moreover, beech accounts for more than a third of the total wood supply in Serbia.

The use of the heat treatment would increase the value of beech wood products, such as flooring, furniture, window and door components, exterior construction
wood, garden furniture, various linings, facades, pergolas, noise-control elements, as well as constructions and elements in contact with the ground etc.

On the other hand, the method of wood impregnation with boric acid has been used since World War II and the first tests of its preservation qualities in the timber industry were carried out in 1937 (Drysdale, 1994). Since then, boric acid has been increasingly used in wood preservation, and its properties and advantages have been confirmed in numerous scientific papers (Carr, 1959; Barnes et al. 1989; Dickinson and Murphy, 1989; Nunes, 1997; Graf, Manser and Lanz, 1998). Besides its toxicity to insects and fungi, the benefits of boric acid as a protective agent include environmentally-friendly characteristics, low cost and low toxicity to mammals. However, boron is unstable in outdoor wood as a dissolvable substance in contact with atmospheric water. Currently, boron, as a protective agent, is most widely used for protection of wooden constructions and objects in the United States, Australia and New Zealand where it has been in use for more than 60 years (Vincken, 1990).

2. MATERIAL AND METHOD

Wood samples were prepared from beech trees - Fagus moesiaca (Domin, Mally / Czeczott.) originating from the mountain of Goč in Serbia. The samples of 25×12.5×5 mm were dried in a laboratory oven at the temperature of 103 ± 2 ºC to the absolutely dry state and measured with an accuracy of 0.01 g. The samples were sorted in four testing series as follows: control (C), impregnated with boric acid (B), thermally treated (TMW), and thermally treated with subsequent treatment with boric acid (TMW + B). Each series contained 32 samples and the results were calculated as the arithmetical average for each series. Each series was exposed to wood decaying fungi: white rot fungus Trametes versicolor (Fr.) Quel, and brown rot fungus Coniophora puteana (Schum.:Fr.) Karst. for 4, 8 and 12 weeks. A set of 4 samples was placed on developed dikaryotic mycelia, in 90 mm Petri dishes, containing cca 20 ml of 2% Malt- 2% Agar. The samples exposed to mycelia were then incubated at a temperature of 21±1ºC for 4, 8 and 12 weeks. After this period, the samples were cleaned of surface mycelia, dried to absolutely dry mass and measured. The mass loss due to the attack of decaying fungi was determined using the following formula:

\[ G = \left( \frac{m_1 - m_2}{m_1} \right) \times 100 \% \]

\( G \) = mass loss [%]
\( m_1 \) = absolutely dry mass of samples before the fungal attack [g];
\( m_2 \) = absolutely dry mass of samples after the fungal attack[g];

The series of TMW and TMW + B samples were exposed to heat treatment at 200ºC for 5 hours in a laboratory oven under anaerobic conditions using the method suggested by Welzbabeler (2007). Before the heat treatment, all the samples were wrapped in Al foil to prevent contact with air - oxygen and possible burning. After the heat treatment, the samples were measured and the mass loss caused by the procedure was calculated as a difference between the mass before
and after the treatment.

Some samples were impregnated with 5% boric acid (B and TMW + B groups) under lab conditions. Vacuum impregnated samples were impregnated for 2 h and then left in the solution for the next 12 h. The samples were then dried to absolutely dry mass and measured to calculate the soaked boron during the impregnation.

3. RESULTS AND DISCUSSION

3.1. Mass loss of beechwood control series (C)

Regarding the control samples, approximately the same mass loss was recorded for both fungi after 12 weeks (Table 1), which coincides with the data of other authors (Podgorski et al., 2008; Goktas 2007; Reinprecht, 2007). The values of the control samples for *Trametes versicolor* were within the expected range. The loss amounted to 15.90% after 4 weeks, 26.67% after 8 weeks and up to 31.52%. After 12 weeks. The greatest loss caused by this fungus was recorded in the first 4 weeks (over 50%), while it decreased in the next two periods of measurement.

**Table 1** Mass loss (%) of control samples (C) of beech wood exposed to fungi *Trametes versicolor* and *Coniophora puteana* after 4, 8 and 12 weeks

<table>
<thead>
<tr>
<th>fungus</th>
<th>4 weeks</th>
<th>8 weeks</th>
<th>12 weeks</th>
</tr>
</thead>
<tbody>
<tr>
<td><em>Trametes versicolor</em></td>
<td>15.90%</td>
<td>26.67%</td>
<td>31.52%</td>
</tr>
<tr>
<td><em>Coniophora puteana</em></td>
<td>2.70%</td>
<td>22.22%</td>
<td>31.41%</td>
</tr>
</tbody>
</table>

Regarding *Coniophora puteana*, the mass loss was similar to the loss by *Trametes versicolor* after 12 weeks (31.41%), but after 4 weeks the mass loss was as low as 2.70%. The mass loss was the highest after 8 weeks and amounted to 22.22%. Between the 4th and the 8th week, the samples lost some 60% of the total mass loss. *Trametes versicolor* was initially more aggressive to beech wood compared to *Coniophora puteana*.

**Table 2** Mass loss (%) of thermally-modified beech wood (TMW) caused by *Trametes versicolor* and *Coniophora puteana* fungi after 4, 8 and 12 weeks

<table>
<thead>
<tr>
<th>fungus treatment</th>
<th><em>Trametes versicolor</em></th>
<th><em>Coniophora puteana</em></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>After…weeks</td>
<td>After…weeks</td>
</tr>
<tr>
<td></td>
<td>4 8 12</td>
<td>4 8 12</td>
</tr>
<tr>
<td>control</td>
<td>15.90% 26.67% 31.52%</td>
<td>2.70% 22.22% 31.41%</td>
</tr>
<tr>
<td>TMW <em>(T=200°C / 5h)</em></td>
<td>5.12% 8.90% 13.95%</td>
<td>0% 0.46% 1.00%</td>
</tr>
</tbody>
</table>
After 4 weeks, there was no mass loss in TMW samples exposed to *Coniophora puteana* fungus. It was 0.46% after 8 weeks and of 1.00% after 12 weeks. The mass loss was lower compared to *Trametes versicolor* due to different nutritional requirements of the tested fungi. *Coniophora puteana* as a brown rot agent breaks down cellulose, which, as noted earlier, is usually more easily decomposed by heat treatment than lignin which *T. Versicolor* mainly feed on. Therefore, the food source of *C. puteana* is not in the form appropriate to be consumed by this fungus.

When exposed to heat treatment, wood polymers, mostly hemicellulose, and lignin to a lesser extent, degrade and reducing the number of free OH groups reduce the number of places to which water can bind.

This reduces moisture absorption and improves dimensional stability, *i.e.* reduces shrinkage and swelling. If, however, the process of heating is performed with the presence of oxygen, it damages cellulose and deteriorates certain wood properties, particularly bending strength. Besides hemicellulose, a part of lignin will also decompose at higher temperatures. As the cellulose and lignin slowly break down, hemicellulose loses its binding role and lignin obtains its thermoplastic properties (Feist, Sell, 1987).

### 3.2. Mass loss of beechwood samples impregnated with boric acid (B)

All wood samples were measured in a dry state before the impregnation. They were then impregnated, dried and measured again in order to calculate the increased mass of wood that contained salt. An average value of increased mass of impregnated samples in an absolutely dry state was around 1.2 %.

A decrease in the mass loss from 31.52% in the case of control samples after 12-week exposure to *T. versicolor* fungus to as low as 1.56% points to the extraordinary good protective effect. It was a similar case with *C. puteana* where the mass loss in the control series was 31.41%, while the impregnated samples lost on average as much as 1.32%.

<table>
<thead>
<tr>
<th>Table 3</th>
<th>Mass loss (%) of beech wood impregnated with 5 % boric acid (B) caused by <em>Trametes versicolor</em> and <em>Coniophora puteana</em> fungi after 4, 8 and 12 weeks</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>fungus</strong></td>
<td><strong>Trametes versicolor</strong></td>
</tr>
<tr>
<td>treatment</td>
<td>After...weeks</td>
</tr>
<tr>
<td></td>
<td>4</td>
</tr>
<tr>
<td>Control (C)</td>
<td>15.90 %</td>
</tr>
<tr>
<td>Boric acid (B)</td>
<td>0 %</td>
</tr>
</tbody>
</table>

Mass loss of beech wood exposed to the attack of test-fungi *T. versicolor* and *C. puteana* was not uniformed since the former is well-known to be more aggressive.
to beech wood, causing white rot, in comparison to the latter one - *C. puteana* which prefers conifer wood (Mirić, Popović, 1993).

### 3.3. Mass loss of thermally modified beechwood samples impregnated with boric acid (TMW + B)

Decreasing of the mass loss is obviously significant in treated samples in comparison with the control samples.

**Table 4** Mass loss (%) of thermally modified beech wood impregnated with boric acid (TMW + B) caused by *Trametes versicolor* and *Coniophora puteana* fungi after 4, 8 and 12 weeks

<table>
<thead>
<tr>
<th>Treatment</th>
<th><em>Trametes versicolor</em></th>
<th><em>Coniophora puteana</em></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>After...weeks</td>
<td>After...weeks</td>
</tr>
<tr>
<td></td>
<td>4 8 12</td>
<td>4 8 12</td>
</tr>
<tr>
<td>Control</td>
<td>15.90 % 26.67 % 31.52 %</td>
<td>2.70 % 22.22 % 31.41 %</td>
</tr>
<tr>
<td>Heat / boric acid (TMW + B)</td>
<td>0 % 2.26 % 3.08 %</td>
<td>1.55 % 1.70 % 1.99 %</td>
</tr>
</tbody>
</table>

Differences in the mass loss of thermally-treated beechwood samples impregnated with boric acid caused by the attack of *Trametes versicolor* and *Coniophora puteana* fungi after 4, 8 and 12 weeks reveal that *Trametes versicolor* causes greater mass loss of thus modified wood. Looking at the results of all tested treatments for fungus *Trametes versicolor* we can see that the control samples had the greatest mass loss, which was an expected result. The samples treated with boric acid had high resistance to the test fungi, what means that boric acid could be used as a substance for increasing natural resistance of beech wood to wood decaying fungi.

**Table 5** Mass loss (%) of treated beechwood samples caused by *Trametes versicolor* and *Coniophora puteana* fungi after 4, 8 and 12 weeks

<table>
<thead>
<tr>
<th>Treatment</th>
<th><em>Trametes versicolor</em></th>
<th><em>Coniophora puteana</em></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>After...weeks</td>
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</tr>
<tr>
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</tr>
<tr>
<td>Heat (TMW): T=200 °C / 5h</td>
<td>5.12 % 8.90 % 13.95 %</td>
<td>0 % 0.46 % 1.00 %</td>
</tr>
<tr>
<td>boric acid (B)</td>
<td>0 % 1.34 % 1.56 %</td>
<td>0 % 0.88 % 1.32 %</td>
</tr>
<tr>
<td>TMW/boric acid (B)</td>
<td>0 % 2.26 % 3.08 %</td>
<td>1.55 % 1.70 % 1.99 %</td>
</tr>
</tbody>
</table>
Graph 1 Mass loss (%) of the treated beechwood samples caused by Trametes versicolor and Coniophora puteana fungi

Grafikon 1. Gubitak mase (%) tretiranih uzoraka bukovog drveta izazvan napadom gljiva Trametes versicolor i Coniophora puteana
Boric acid provides very efficient protection against lignicolous fungi in the interior. If the material treated with boric acid is exposed to extreme outdoor conditions, it will surely over time lose a certain amount of preservative and the protective effect will be reduced. In addition, the wood might receive moisture in such conditions, which will threaten the dimensional stability of wood. The wood treated with a combined treatment such as heat treatment combined with boric acid could perhaps be a solution (Graph 1).

In the case of fungus *Coniophora puteana*, the highest mass loss was obtained in the control samples.

4. CONCLUSIONS

Based on the performed investigations, the following conclusions can be drawn:

- After 12 weeks of the exposure to fungi, mass loss of control samples (C) was greater than the loss of all tested groups and reached almost 32% for both fungi;
- The mass loss of TMW exposed to *Coniophora puteana* mycelia was lower than the mass loss of TMW exposed to *Trametes versicolor* mycelia;
- TMW and impregnation with boric acid (TMW + B) increased the resistance of wood to the tested fungi;
- Mass loss of the samples impregnated with boric acid (B) was approximately the same for both tested fungi;
- Impregnation of previously thermally treated samples (TMW + B) increased the resistance of wood to *Trametes versicolor* fungus in comparison to the samples of wood that were only thermally treated (TMW), but the resistance of these samples was lower compared to the samples of wood impregnated with boric acid (B);
- Thermal modification + impregnation with boric acid (TMW + B) showed lower resistance to *Coniophora puteana* fungus than these treatments (TMW or B) separately.

To make the obtained results applicable in practice, it is necessary to examine the influence of these treatments on changes of mechanical properties of beech and other wood species used for the thermal modification.

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ПОБОЉШАЊЕ ОТПОРНОСТИ ДРВЕТА БУКВЕ Fagus moesiaca (Domin, Mally / Czeczott.) НА ГЉИВЕ ТРУЛЕЖНИЦЕ Trametes versicolor (Fr.) Quel. и Coniophora puteana (Schum.: Fr.) Karst. КОНТРОЛИСАНИМ ТОПЛОТНИМ ТРЕТМАНОМ И НАКНАДНИМ ТРЕТИРАЊЕМ ЕКОЛОШКИ ПРИХВАТЉИВИМ ПРЕПАРАТОМ

Мilenko Mirić
Vladan Jelkić
Mimica Stefanović
Tamara Tešić

Сажетак

Могућност повећања отпорности буковог дрвета (Fagus moesiaca Domin, Mally/Czeczott.) против две опасне гљиве трулежнице: Trametes versicolor – изазивача беле трулежи и Coniophora puteana – изазивача мрке трулежи, испитивана је у лабораторијским условима. Испитан је температурни третман од 200 °C / 5 часа, као и вакуумска импрегнација борном киселином (5%). Комбиновани третман коришћењем температурног третмана и накнадне импрегнације, такође је испитан. После 12 недеља излагања гљивама, губитак масе контролних узорака био је највећи у поређењу са свим испитаним групама и достигао скоро 32% за обе гљиве. Губитак тежине термично модификованог дрвета (TMW) изложеног мицелији гљиве Coniophora puteana био је нижи него губитак масе TMW изложеног мицелији гљиве Trametes versicolor. Термички третман дрвета (TMW) и импрегнација борном киселином (B) повећава отпорност дрвета против испитиваних гљива. Губитак тежине узорака импрегнираних борном киселином (B) је приближно исти за обе испитане гљиве. Импрегнација претходно термички третираних узорака (TMW + B) узроковала је бољу отпорност против гљиве Trametes versicolor у поређењу са узорцима дрвета који су само термички третирани (TMW), али и смањену отпорност узорака који су само импрегнисани борном киселином (B). Топлотни третман + импрегнација борном киселином (TMW + B) показала је нижу отпорност против гљиве Coniophora puteana него одвојени третмани (TMW или B). Да би добијени резултати били примењиви у прaksi, неопходно је испитати утицај ових третмана на промене механичких својстава букве и других врста дрвета који се користе за термичку модификацију.