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Biological Durability of Oil Heat Treated Alder Wood

Biološka otpornost johovine termički modificirane u ulju

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ABSTRACT • *The article presents preliminary results of the biological durability of oil heat treated (OHT) alder wood (Alnus glutinosa) against pure cultures of rot fungi (Postia placenta and Trametes versicolor) in lab conditions. The modification was performed by heating of specimens immersed in soya oil. There were four heating regimes of different duration (6 and 10 hours) at final temperature of 180 and 200 ºC. The increase in mass (MI) caused by modification and mass loss of wood caused by fungal decay (ML) were determined. In addition, the natural durability of alder wood was determined and compared to the natural durability of beech wood as the reference wood species.*
After modification of alder wood at 200 °C, MI was lower than after treatment at 180 °C. MI was also lower after 10 hours of treatment than after 6 hours of treatment. The results showed significantly increased biological durability of modified alder wood against both tested fungi. The effect of OHT on increasing the biological durability of alder wood was higher against the fungus P. placenta. It seems that the fungus T. versicolor favours the remained oil after modification causing higher mass loss. The results showed that alder wood, thermally modified in soya oil by testing regimens, is not suitable for applications in use classes 3-5.

Key words: *durability class, Postia placenta, soya oil, Trametes versicolor, use class*

SAŽETAK • *U radu su prezentirani preliminarni rezultati biološke otpornosti drva johe (Alnus glutinosa) termički modificiranoga u sojinu ulju protiv čistih kultura gljiva truležnica (Postia placenta i Trametes versicolor) u laboratorijskim uvjetima metodom mini blok prema CEN TS 15083-1. Modifikacija je izvedena zagrijavanjem uzoraka uronjenih u sojino ulje sobne temperature. Četiri načina termičke modifikacije razlikovala su se po trajanju držanja drva (6 i 10 sati) na konačnoj temperaturi (180 i 200 ºC). Usto je određena i prirodna otpornost johovine u usporedbi s prirodnom otpornosti bukovine kao referentne prirodno slabo otporne vrste drva. Mjereno je povećanje mase modifikacijom (DMM) i gubitak mase djelovanjem spomenutih gljiva (GMG). Nakon modifikacije pri višoj temperaturi DMM drva johe bio je manji nego nakon modifikacije pri nižoj temperaturi. Slično tome, dulji je tretman rezultirao nižim DMM-om nego kraći tretman. Rezultati su potvrdili povećanje biološke otpornosti modificirane johovine protiv obje testirane gljive truležnice. S povećanjem temperature modifikacije znatno se povećava biološka otpornost protiv obje gljive, dok produljenje vremena zagrijavanja ima blagi učinak povećanja biološke otpornosti. Utjecaj modifikacije na povećanje biološke otpornosti veći je protiv gljive smeđe truleži P. placenta. Utvrđeno je da je veći gubitkom mase modificiranih uzoraka djelovanjem gljive T. versicolor (u usporedbi s gubitak mase djelovanjem gljive P. placenta) najvjerojatnije nastao zbog razaranja preostalog ulja*

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u drvu nakon modifikacije. Dokaz tome je crvena boja mikroskopskih preparata modificiranih uzoraka (postojanje lignina) te lokalna plava obojenost u lumenima traheja (nedostatak lignina). U lumenima traheja ostalo je najviše ulja nakon modifikacije, pa se tu ujedno i najjače razvio micelij gljive bijele truleži T. versicolor, koji je, osim razgradnje ulja, djelomično uništio i lignin. Rezultati su pokazali da johovina termički modificirana u biljnom ulju, prema testiranim režimima, nije primjerena za upotrebu u razredima opasnosti 3 i višima.

Ključne riječi: *Postia placenta, razredi opasnosti, razred trajnosti, sojino ulje, Trametes versicolor*

1 INTRODUCTION
1. UVOD

Wood is a material that can be used for different purposes. However, it can be degraded by xylophagous microorganisms, xylophagous insects, UV rays, etc. (Hasan, 2010; Beyzar, 2012). It is flammable and hygroscopic, and its dimensions change depending on its moisture content. The above mentioned properties are undesirable and limit the application of wood in comparison to other new materials. There are many processes that can reduce/eliminate the undesired properties of wood. Hill (2006) mentioned some of the first scientists (Tiemann, 1915; Stamm and Hansen, 1937; Stamm *et al*., 1946; etc.) who have introduced various wood modification processes. The general goal of wood modification is to get wood with desirable properties during its service life, not toxic and not releasing any toxic substances (Hill, 2006). Among many modification processes, some chemical and thermal modifications have been the most investigated, but only some of them are commercialized.

Chemical modification implicates etherification or esterification between some chemical and OH groups of cellulose, hemicelluloses and lignin (Militz, 1993). The following parameters are important for successful chemical modification: temperature, type of chemical, processing time, type of catalyst and wood species (Hill, 2006; Hasan, 2010).

Thermal modification is a process where the wood cell wall polymers are destructed to the radicals that repolymerise with OH groups of wood cell wall compounds only by heating. Thermal modification is mostly conducted in operating cylinder at the temperature between 150 and 260 °C without the presence of oxygen (Leithoff and Peek, 1998; Rapp and Sailer, 2001a, 2001b; Rep and Pohleven, 2001; Yildiz *et al.*, 2003; Hill, 2006; Beyzar, 2012). The type of heating medium, period of heating, final temperature and wood species are the most important parameters of thermal modification processes. By any such modification process, the dimensional stability and resistance of wood against rot-fungi are improved (eg. Rapp and Sailer, 2001a, 2001b; Hill, 2006 mentioned Tiemann, 1915, Stamm and Hansen, 1937 and Stamm *et al*., 1946; Hasan *et al*., 2008; Beyzar, 2012), but some mechanical properties are decreased (Bengtsson *et al.*, 2002; Ladner and Halmschlager, 2002; Patzelt *et al.*, 2002; Bak and Nemeth, 2012). Resistance against fungi increases at increasing the degree of modification as well as at increasing the oil uptake (Rapp and Sailer, 2001b; Sidorova, 2009; Bazyar, 2012). Wood thermally modified in vegetable oils has greater durability against rot fungi than when modified in air atmosphere (Rapp and Sailer, 2001a; Despot *et al.*, 2008; Hasan *et al*., 2008; Hasan, 2010). Thermal modification of wood in air atmosphere at a final temperature ranging between 140 and 180 °C does not significantly increase biological durability compared to non-modified wood (Rapp and Sailer, 2001b; Hasan *et al.*, 2006, 2007; Hasan, 2010; Despot *et al.*, 2008). Feist and Sell (1987) reported that thermally modified wood was still sound without any sign of biodeterioration after 8 months of field testing. They explained that paucity of bluestain's and mould's mycelium on the surface of modified wood ensures reduced discoloration, and that durability against moulds and bluestain also increases with the increase of modification degree. They stated that the difference in discoloration becomes most significant between 8 and 14 months of field testing because the amount of simple carbohydrates decreases and chemical structure of parenchyma cells contents change during modification, so fungal enzymes become less effective (Feist and Sell, 1987).

Latest research reports that thermally modified wood is not resistant against staining fungi, and in some cases it becomes even more susceptible to staining fungi.

Sidorova (2009) modified spruce, pine heartwood, pine sapwood and aspen in rapeseed oil at 180, 210 and 240 °C for 0.5, 1.0 and 1.5 hours. One set of specimens were taken away from the oil immediately after treatment and cooled in air atmosphere while the other set of specimens were cooled in the oil. Her results are similar to the results of Bazyar (2012). He also modified aspen wood in linseed oil at temperatures of 190, 205 and 220 °C for 4.5 and 6 hours. He reported that the WPG was about 83.9 to 86.2 %, and that the WPG was not significantly affected by time or temperature of modification. After the main heating stage, the oil was removed from the operating cylinder and specimens were vacuumed to remove surplus and remaining oil from the specimens. This is contrary to the results and procedure of Sidakova (2009).

Spear *et al*. (2006) reported the WPG of 89.9 and 87.4 % in Corsican pine wood and below 20 % in Norway spruce wood after oil heat treatment in linseed oil at 190 and 200 °C, respectively, at decreased pressure. Bazyar (2012) cited similar WPG data of Sailer and Rapp (2001). The increase of mass of wood after oil heat treatment was about 42 to 51 % for pine wood and 10 to 18 % for spruce wood, depending on the immersion time of wood in oil during the cooling phase.

Bazyar (2012) explained high values of WPG in his study as a result of the small size of specimens. He cited Jones *et al*. (2005), who reported that longer sam-

ples of sitka spruce have lower WPGs. Also, aspen is a hardwood with wide cells and it is permeable (Rowel 1984). He also explained that the high level of WPG could be related to anatomical changes of samples citing Hietala *et al*. (2002). Also Boonstra *et al*. (1998) reported that hardwood species such as beech and poplar are sensitive to the collapse of vessels and deformation of the libriform fibres near the vessels.

Oil heat treatment has been performed mainly on non-durable wood species such as spruce, fir and pine sapwood as well as on beech, alder and aspen (Bazyar, 2012; Sidorova, 2009; Hasan *et al*., 2008; Yildiz *et al.*, 2003; Bengtsson *et al.*, 2002; Patzelt *et al.*, 2002; Rapp and Sailer, 2001a, 2001b; Feist and Sell, 1987). Alder wood is a fast growing hardwood species and it covers a wide area of Europe (Kajba and Gračan, 2003; Prpić and Milković, 2005). Technical properties of alder wood are poor and it is a non-durable wood species. The idea of the authors is to try to increase some properties of alder wood through modification and to increase its commercial importance. The article presents preliminary results of the mass increase (MI) and improved biological durability (in lab conditions) of oil heat treated alder wood against rot-fungi.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

Home-grown alder wood (*Alnus glutinosa*, L.) was used in the oil heat treatment and durability experiment. Beech wood (*Fagus sylvatica* L.) was used only untreated for comparison of the biological durability against the chosen test rot fungi, since it is reference species for natural durability rating due to its very low natural durability.

Lattices were sawn from the region close to the bark of an air-dried and afterwards kiln-dried (below 60 °C) plank for each wood species. Specimens were cut to dimensions 15×5×30 ± 0.2 mm (R×T×L). They were selected and marked successively according to CEN TS 15083-1 (2005) (Tab. 1).

Since Bak and Nemeth (2012) reported no significant influence of oil type on tested mechanical and physical properties, the cheapest oil on the market was used for this experiment. Soya oil was used as a heating medium, and the modification was performed in an open cylinder at ambient pressure.

2.1 Modification procedure
2.1. Postupak modifikacije

All specimens were oven dried at 103 ± 2 °C for 48 hours to constant mass, weighed (m_1), and then conditioned under the standard conditions (20 °C and 65 % relative air humidity) to constant mass. Each group of specimens modified at the same regime (48 specimens) was immersed into 1 l of fresh soya oil at room temperature. Then the oil was heated together with specimens. When the oil temperature of 180 and 200 °C, respectively, was reached, the groups of specimens were boiled for further 6 and 10 hours, respectively. Immediately after modification, specimens were removed from the oil and cooled in air atmosphere over the silica gel and weighed again (m_2).

2.2 Determination of mass increase and natural durability
2.2. Izračunavanje povećanja mase modifikacijom i određivanje biološke otpornosti

Increase of mass (MI) of modified specimens was calculated as a ratio of difference of oven-dried mass after modification (m_2) and oven-dried mass before modification (m_1) and m_1 (1).

$$MI = \frac{m_2 - m_1}{m_1} \cdot 100 \qquad (1)$$

Diological durability of oil heat treated alder wood was determined according to CEN TS 15083-1 (2005). White rot fungus *Trametes versicolor* (L.: Fr.) Pilat. and brown rot fungus *Postia placenta* (Fr.) M.J. Larsen & Lombard were chosen. "Potato dextrose agar (PDA)" by OXOID was used as a nutrient medium. Specimens were placed on the inert plastic network (1 mm thick) over the fungal mycelium in 90 mm Petri dishes and incubated for 9 weeks at 24 ± 1 °C and 70 ± 5 % relative humidity.

Mass loss of specimens caused by fungal decay (ML) was calculated by dividing the difference of oven-dried mass of specimens after fungal decay (m_3) and starting mass before decay (m_2) with starting mass before decay (m_2) (2).

Table 1 Distribution and number of specimens
Tablica 1. Raspored i broj uzoraka

Fungus species / *Vrsta gljive*	**Modification type** / *Postupak modifikacije*	**No. of specimens** / *Broj uzoraka*
Trametes versicolor (L.: Fr.) Pilat.	Oil heat treated alder wood at 180 °C, 6 h (OHT-18/6)	12
	Oil heat treated alder wood at 180 °C, 10 h (OHT-18/10)	12
	Oil heat treated alder wood at 200 °C, 6 h (OHT-20/6)	12
	Oil heat treated alder wood at 200 °C, 10 h (OHT-20/10)	12
	Alder wood control (AC)	24
	Beech wood control (BC)	24
Postia placenta (Fr.) M.J. Larsen & Lombard	Oil heat treated alder wood at 180 °C, 6 h (OHT-18/6)	12
	Oil heat treated alder wood at 180 °C, 10 h (OHT-18/10)	12
	Oil heat treated alder wood at 200 °C, 6 h (OHT-20/6)	12
	Oil heat treated alder wood at 200 °C, 10 h (OHT-20/10)	12
	Alder wood control (AC)	24
	Beech wood control (BC)	24

$$ML = \frac{m_2 - m_3}{m_2} \cdot 100 \quad (2)$$

This mass loss percentage ML (%) is the unit that shows the durability of specimens. ML will be smaller when the wood is more durable, and vice versa. The durability was ranked as proposed in CEN TS 15083-1 (2005).

2.3 Examination of decay pattern using light microscopy
2.3. Istraživanje mehanizma biološke razgradnje uz pomoć svjetlosnog mikroskopa

About 20 μm thin sections were cut from alder wood specimens decayed by *T. versicolor* using Reichert-Shandon microtome. Sections were stained in safranin and astrablue solution. Cellulose in wood cell walls stained blue, while lignin stained red.

Stained slides were examined using Leitz Wetzlar light microscope, and photographs were taken at 100×, 280× and 400× magnification.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Mass increase (MI)
3.1. Povećanje mase modifikacijom

It is known that wood specimens lose their mass during thermal modification due to the evaporation of extractives and of volatile compounds formed during thermolysis mainly of hemicelluloses and partly of lignin (Hill, 2006 quoted Shafizadeh and Chin, 1977 and Sudo *et al*., 1985; Rapp and Sailer, 2001b quoted Sandermann and Augustin, 1963, Kollmann and Fengel, 1965, Topf, 1971 and Tjeerdsma *et al*. 1998; Sidorova, 2009). Although mass increase during OHT process is the actual result of mass loss of wood and oil uptake in wood, many authors reported this mass increase as WPG.

In this experiment, it was impossible to remove the remained oil from the specimens with the available equipment, so the mass loss of specimens could not be determined. The oil remained in the specimens, so they gained mass. The results of mass increase (MI) indicate that by increasing either modification temperature or modification duration, MI of specimens significantly decreases (Fig. 1).

The obtained values of MI are similar to the results of Sidorova (2009). The only difference is that the mass increase of her specimens, which were cooled in the air, had the tendency to increase by increasing the time of modification at 180 °C, while at higher temperatures of modification, mass increase tends to decrease by increasing the modification duration.

3.2 Biological resistance against rot fungi
3.2. Biološka otpornost protiv gljiva truležnica

The results of this research confirmed significant increase of biological durability of modified alder wood compared to control specimens of both alder and beech wood. The biological durability of all modified alder wood specimens against both tested fungi was significantly higher than the controls. The effect of modification had greater impact on biological durability against *P. placenta* than against *T. versicolor*. By increasing the temperature, biological durability against both tested fungi increased. The only exception to this rule was found in ML caused by *T. versicolor* between OHT-18/10 and OHT-20/10 with no significant difference. Extended period of modification at both temperatures slightly affected, but not significantly, the increasing of biological durability (Fig 2 and 3). According to Rapp and Sailer (2001a, b), by increasing the mass loss of specimens during OHT, biological durability also increases.

Biological durability against both tested fungi of beech wood is slightly higher than that of alder wood. This can be explained by the difference in wood density between these two wood species (Fig 2 and 3).

Similar results of ML were obtained by Bayzar (2012) with aspen thermally modified in oil. Dirol and Guyonnet (1993) studied the effects of wood heat treatment at temperatures between 205 and 260 °C of three

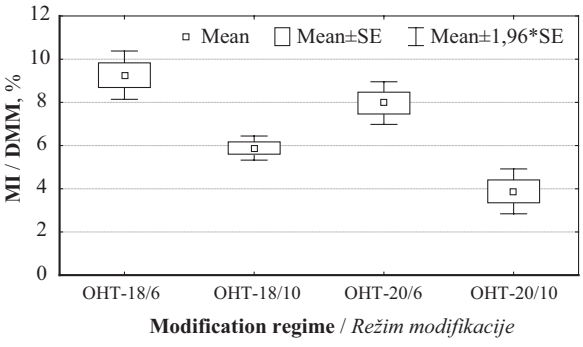

Figure 1 Mass increase of alder wood, MI after different modification regimes (OHT-18/6 = 180 °C, 6 h; OHT-18/10 = 180 °C, 10 h; OHT-20/6 = 200 °C, 6 h; OHT-20/10 = 200 °C, 10 h).
Slika 1. Povećanje mase uzoraka johovine (DMM) nakon različitih režima modifikacije

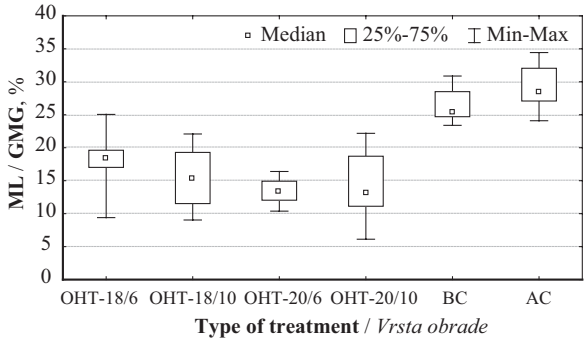

Figure 2 Mass loss of specimens caused by fungus *Trametes* versicolor (ML) of differently modified and non modified alder and beech wood (OHT-18/6 = 180 °C, 6 h; OHT-18/10 = 180 °C, 10 h; OHT-20/6 = 200 °C, 6 h; OHT-20/10 = 200 °C, 10 h, AC-alder wood controls, BC-beech wood controls; n = 24 control specimens, n = 12 for OHT specimens).
Slika 2. Gubitak mase uzoraka djelovanjem gljive *T. versicolor* (GMG) različito modificiranih uzoraka johovine i bukovine i nemodificiranih uzoraka

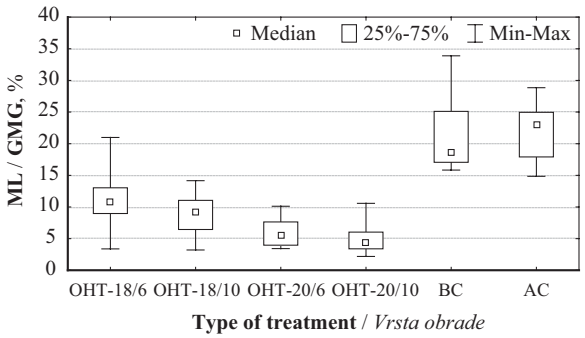

Figure 3 Mass loss of specimens caused by fungus *Postia placenta* (ML) of differently modified and non modified alder and beech wood (OHT-18/6 = 180 °C, 6 h; OHT-18/10 = 180 °C, 10 h; OHT-20/6 = 200 °C, 6 h; OHT-20/10 = 200 °C, 10 h, AC-alder wood controls, BC-beech wood controls; n = 24 for control specimens, n = 12 for OHT specimens).
Slika 3. Gubitak mase uzoraka djelovanjem gljive *P. placenta* (GMG) različito modificiranih uzoraka johovine i bukovine i nemodificiranih uzoraka

non-durable wood species including poplar wood on resistance to several rot fungi including *T. versicolor* and *Coniophora puteana*. They reported mass loss of less than 1 % for all modifications compared to mass loss of controls of 40 %. Rapp and Sailer (2001a, b) reported that spruce and pine sapwood oil heat treated at 190 to 220 °C have improved resistance to the fungus *C. puteana*. They reported the increase of mass loss from 48 and 40 % to about 11 and 5.5 % in pine sapwood and spruce, respectively. Welzbacher and Rapp (2002) showed that oil heat treatment of spruce and pine sapwood can improve durability against *T. versiclor* and *C. puteana*. Leithoff and Peek (2001) reported the temperatures above 170 °C to be effective for increasing biological resistance of two bamboo species.

The modification temperature of 180 °C, used in this research, does not significantly increase the durability class against white-rot fungus *T. versicolor*, while the modification at 200 °C increases the durability from class 4 to class 3. On the other hand, the temperature of 180 °C increases the durability against brown-rot fungus *P. placenta* from class 4 to durability class 2-3, while the modification temperature of 200 °C resulted in an increase of durability of OHT alder wood against brown-rot fungus *P. placenta* from class 4 do class 1-2.

Taking into account mass losses of both tested fungi, only the treatment at 200 °C can improve durability class of alder wood from class 4 to durability class 3 and hence broaden the application of alder wood.

3.3 Decay pattern
3.3. Mehanizam biološke razgradnje

Figure 4 shows the slides of non-decayed specimen that was oil heat treated at 200 °C for 10 hours. A lot of red colour and thickness of the wood cell double walls of the whole OHT slides prove completely non degraded wood (Fig. 4a and 4b).

Although OHT specimens of alder wood decayed by *T. versicolor* had greater ML compared to specimens decayed by *P. placenta*, the overall degree of wood degradation was very similar. Light microscopy examination shows that wood cell walls of OHT specimens decayed by *T. versicolor* were not as severely degraded as control specimens. The majority of blue colour of control slides proves the lack of lignin (great lignin degradation; Fig. 5a, 5b), while OHT slides are mainly red (proof of presence of lignin) with very local blue coloured regions (proof of lack of lignin; Fig. 5c, 5d). These very limited regions of blue colour are mainly in the vessels lumens. As the mycelium of the fungus *T. versicolor* was the most developed in the vessels lumens in OHT specimens, where the majority of oil remained (Olsson *et al*., 2001; Hill, 2006; Bazyar, 2012), it can be concluded that the fungus favours the presence of oil in wood (Fig. 5c, 5d, 6). This leads to the conclusion that the tested fungus *T. versicolor* mainly utilised the oil remained in vessels lumens causing grater ML, and also partially degraded lignin in the inner layer of the cell walls (blue colour; Fig. 5d). Another proof of poor and local lignin degradation in OHT specimens is the red colour of the whole OHT slides, which proves the presence of lignin, although *T.*

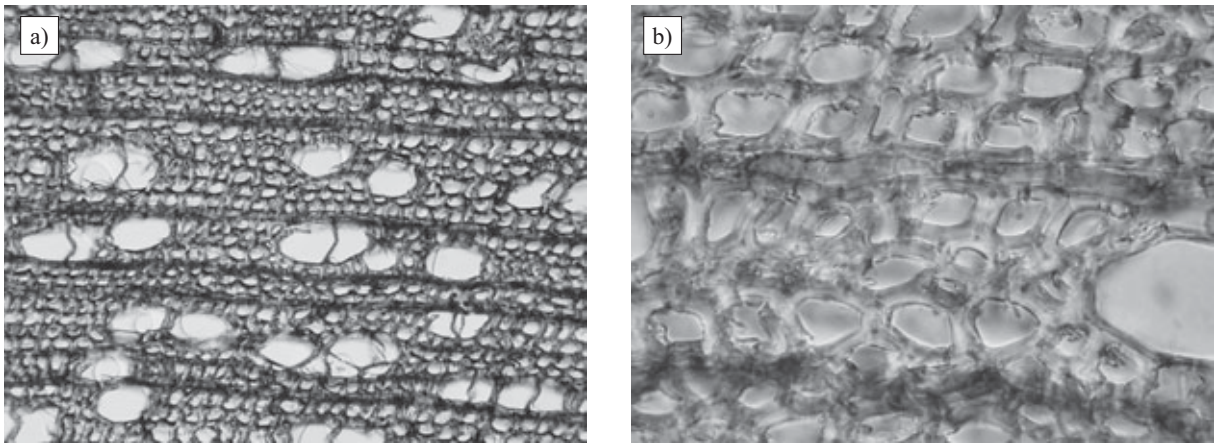

Figure 4 Cross section of non-decayed alder wood: a) OHT at 200°C for 10 h, 100×; b) OHT at 200°C for 10 h, 400×
Slika 4. Poprečni presjek nerazorenoga modificiranog uzorka johovine: a) OHT pri 200 °C za 10 h, povećanje 100 puta; b) OHT pri 200 °C za 10 h, povećanje 400 puta

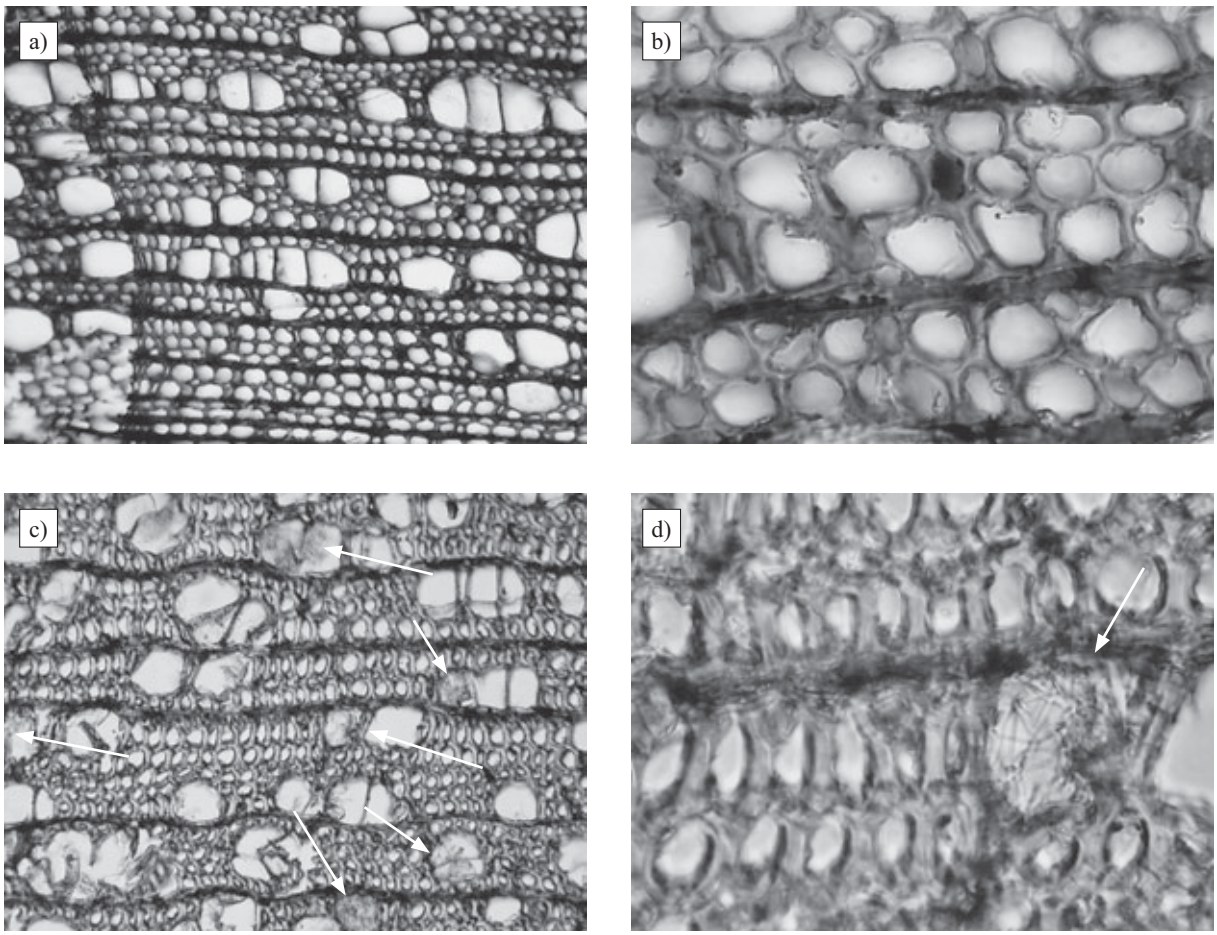

Figure 5 Cross section of alder wood decayed by white-rot fungus *Trametes versicolor*: a) control, 100×; b) control, 400×; c) OHT at 200°C for 10h, 100×; d) OHT at 200°C for 10h, 400×.
Slika 5. Poprečni presjek uzorka johovine razorenoga gljivom bijele truleži *T. versicolor:* a) kontrolni uzorak, povećanje 100 puta; b) kontrolni uzorak, povećanje 400 puta; c) OHT pri 200 °C za 10 h, povećanje 100 puta; d) OHT pri 200 °C za 10 h, povećanje 400 puta

versicolor is white-rot fungus and predominantly degrades lignin (Fig. 5c and 6a). Comparing the thickness of wood cell double-walls of modified non-decayed specimen (Fig. 4b), of control decayed specimen (Fig. 5b) and of modified decayed specimen (Fig. 5d), it is visible that the thickness of cell double-walls of modified non-decayed specimen and modified decayed specimen are very similar. At the same time, the thickness of cell double-walls of control decayed specimen is visibly thinner and lumens are larger.

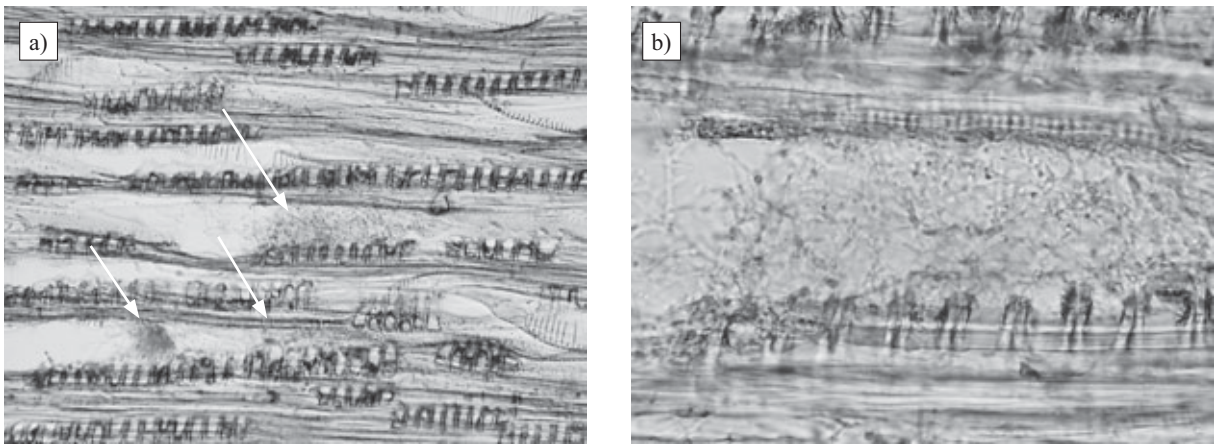

Figure 6 Tangential section of alder wood oil heat treated at 200 °C for 10 h decayed by white-rot fungus *Trametes versicolor*: a) magnification 100×; b) magnification 400×.
Slika 6. Tangentni presjek uzorka johovine modificiranog na 200 °C 10 sati i razorenoga gljivom bijele truleži *T. versicolor*: a) povećanje 100 puta; b) povećanje 400 puta

4 CONCLUSION
4. ZAKLJUČAK

The increase in mass of modified specimens decreased by increasing the duration of oil heat treatment modification at both tested temperatures. The results indicated that the increase of temperature as well as the increase of modification duration had a positive effect on biological durability of alder wood against tested fungi. The tested modification regimes were not adequate for the application of modified alder wood in use classes 3 to 5.

Light microscopy showed that the mycelium of the fungus *T. versicolor* was the most developed in the vessels lumens of modified specimens. Greater mass loss of modified alder wood, decayed by this fungus, can be explained by the fungal utilisation of the oil remained in vessels lumens.

Oil heat treatment is a simple and environmentally friendly method by which biological durability against wood rot fungi can be relatively easily improved. However, it is very important to put in mind that such modification can produce significant decrease in mechanical properties. Further research will show the actual effect of OHT on mechanical properties of alder wood.

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