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Combustion and Decay Resistance Performance of Scots Pine Treated with Boron and Copper Based Wood Preservatives

Otpornost prema gorenju i propadanju borovine zaštićene sredstvima na bazi bora i bakra

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ABSTRACT • *Boron compounds in the form of boric acid, borax or disodiumoctaborate tetrahydrate have been used as insecticide, fungicide and fire retardant in wood preservatives industry for decades. Also, copper is the most commonly used component in most of relatively modern preservatives as it is highly effective against fungi. The objective of this study was to investigate the combustion and decay resistance of boron-copper based solutions which were developed by our group. These solutions contain boric acid, sodium borate decahydrate, copper hydroxy carbonate, ethanolamine, quaternary ammonium compound (benzalkonium chloride), and/or organic acid (octanoic acid). Scots Pine (Pinus sylvestris) woods were treated with the preservatives according to ASTM D1413-07 standard by vacuum-pressure impregnation system, which was developed by our group. Decay resistance performances against white and brown rot fungi were determined according to EN 113 standard and combustion tests were performed with respect to ASTM E160-50 standard. All the impregnated wood samples were found highly resistant to both white (Trametes versicolor L.) and brown (Coniophora puteana L.) fungi. Besides, they gave better results than the control samples in terms of combustion tests.*

KEYWORDS*: wood preservatives; decay resistance; boron compounds; combustion resistance filler*

SAŽETAK • *Spojevi bora poput borne kiseline, boraksa ili dinatrijeva oktaborata tetrahidrata već se desetljećima u industriji sredstava za zaštitu drva proizvode kao insekticidi, fungicidi i usporivači gorenja. Usto, bakar je najčešća komponenta u većini relativno modernih zaštitnih sredstava jer je visoko učinkovit u zaštiti od gljiva. Cilj ove studije bio je istražiti otpornost prema gorenju i propadanju drva bora zaštićenog otopinama na bazi bora i bakra koje je razvila naša grupa. Te otopine sadržavaju bornu kiselinu, boraks, bakrov(II) karbonatni hidroksid karbonat, etanolamin, kvaterni amonijev spoj (benzalkonijev klorid) i/ili organsku kiselinu (oktansku kiselinu). Uzorci borovine (Pinus sylvestris) obrađeni su zaštitnim sredstvima prema standardu ASTM D1413-07 postupkom impregnacije pod vakuumom koji je razvila naša grupa. Otpornost prema gljivama bijele i smeđe truleži određena*

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je prema standardu EN 113, a ispitivanje gorenja provedeno je prema standardu ASTM E160-50. Svi impregnirani uzorci drva pokazali su se vrlo otpornima i na bijele (Trametes versicolor L.) i na smeđe (Coniophora puteana L.) gljive. Osim toga, pri ispitivanju gorenja pokazali su bolje rezultate od kontrolnih uzoraka.

KLJUČNE RIJEČI: *sredstva za zaštitu drva; otpornost prema propadanju; spojevi bora; otpornost prema gorenju*

1 INTRODUCTION
1. UVOD

Wood is both an ecological and sustainable natural resource. It is frequently preferred both indoors and outdoors because it is easy to maintain and repair, aesthetically pleasing, durable and has a wide range of use. In addition to its light and durable structure, it provides good thermal insulation and is therefore an ideal construction material. However, if the necessary care and conditions are not provided, deterioration occurs in wood. Materials that are in direct contact with weather conditions in the external environment are subject to more deformation. Biological pests such as bacteria, fungi, insects, termites, adverse weather conditions, exposure to humidity and sunlight are among the factors that accelerate the deterioration of wood. Thermal stability and durability are significant for the wood material. Therefore, wood preservatives are used to kill fungi, bacteria or insects directly, or to provide higher thermal stability (Ramage *et al.,* 2001; Bekhta and Niemz 2003).

Wood preservation is based on the impregnation of wood with biocides such as creosote, arsenic, zinc, copper, boron, chromium, etc. to prevent degradation of wood and to eliminate the appropriate nutrient environment for the growth of microorganisms. In the wood preservation industry, the main change in the process that has been developing since the beginning of the twentieth century has occurred in wood preservatives rather than in wood preservation methods. In recent years, growing environmental concerns have led to drastic modifications in the active ingredients of wood preservatives in many countries, such as eliminating or restricting the use of creosote and chromate copper arsenate (CCA). Alternatively, wood preservatives containing copper, boron and organic biocides as active ingredients are used in many countries. In this context, recent prohibitions on the use of these toxic impregnants have led the wood protection industry to use and develop wood preservatives based on organic or inorganic compounds such as alkali copper quaternary ammonium compounds (ACQ-1, ACQ-2), copper azole and copper-HDO. ACQ wood preservatives are classified into two types based on their composition: ACQ-1 contains copper and benzalkonium chloride (BAC) and ACQ-2 contains copper and didecyldimethyl ammonium chloride (DDAC) (Koski, 2008; Humar *et al*., 2005; Tomak, 2011).

Copper is the most commonly used component in most of the relatively modern preservatives. The copper cation has been reported to be adsorbed or to form complexes with phenolic groups of lignin or cellulose (Richardson, 2003; Lebow, 1996). Recently, amines have been frequently used to prevent the leaching of copper from wood. Therefore, amines that act like a ligand and thus affect the stability, polarity and solubility of copper amine complexes appear very efficient in fixing copper into wood (Humar *et al.,* 2001).

The use of boron compounds, known as environmentally friendly impregnating agents, has an important place in this respect and their importance is increasing day by day. Borates (borax, boric acid, disodium octaborate tetrahydrate) are inorganic, colorless and odorless boron-based biocides that are noncorrosive to metal fasteners and readily soluble in water. Boron compounds exhibit both fungicidal and insecticidal properties against wood destroying insects and fungi (Tomak *et al.,* 2011; Freeman, 2008; Terzi *et al.,* 2017). In addition, when boron compounds are exposed to heat, they form a glassy structure in the wood, reducing the rate and spread of flammable gases and preventing the movement of thermal decomposition products. Boric acid reduces combustion in the form of ember, but does not completely prevent the spread of the flame (Freeman, 2008; Townsend and Solo-Gabriele, 2006; Yamaguchi, 2003). Borax prevents the spread of flame. In different studies, boric acid and borax were used together and it was determined that wood material had higher burning resistance (Baysal *et al.,* 2003).

Boron compounds are considered to be more effective preservatives than copper and zinc compounds due to their wide fungicidal and insecticidal effects. The reason why copper and zinc perform better is not their natural fungicidal activities, but their fixation in wood (Obanda *et al.,* 2008). However, the boron is susceptible to rapid leaching particularly in outdoor exposure. The boron compounds are well diffusible substances to the wood and as a result, they can be leached easily when in contact with water. The reason for easy diffusion and leaching of boron in wood is that the molecules cannot be fixed to the cell wall. Boron does not react with the cell wall, but can form complexes with hydroxyl groups. A significant potential site for the absorption of boron is the hydroxyls of the carboxylic acids and phenolic groups. In recent years, a lot of research has been done on delaying or preventing leaching of boron compounds from wood and increasing the potential use of boron compounds. Nevertheless, the leaching resistant results reported so far appear generally poor (Townsend and Solo-Gabriele, 2006; Yamaguchi, 2003; Baysal *et al.,* 2003; Obanda *et al.,* 2008; Yalinkilic *et al.,* 1999). In addition, in our previous study, in which the retention and leaching properties of boron and copper were determined, it was observed that the leaching of boron and copper was significantly reduced, especially with the use of octanoic acid (Yildiz *et al.,* 2019).

The objective of this study was to investigate decay and fire resistance of the wood samples impregnated with boron-copper based solutions. Boric acid, borax, copper hydroxy carbonate, ethanolamine, octanoic acid and benzalkonium chloride were used as chemical substances of 4 different preservative solutions. Scots Pine (*Pinus sylvestris*) wood was treated with the preservatives by vacuum-pressure impregnation system according to Bethell method. Decay resistance performances against white and brown rot fungi were determined and combustion tests were performed for the impregnated wood samples as well as for one unimpregnated control sample. Consequently, the effects of the substances in decay resistance and combustion tests were investigated. The aim of impregnation with developed chemicals was to protect the wood against fungi, to increase its resistance to flame and, accordingly, to extend its life.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

The sapwood samples used in the study were obtained from the Scots pine (*Pinus sylvestris*) trees of Bolu Dörtdivan region. The logs of wood were selected as healthy, smooth fibrous, knotless, exhibiting normal growth, undamaged by fungus and insect pest. Sapwood blocks were cut in dimensions of 5 mm × 15 mm × 30 mm for fungal decay-resistance tests, and 13 mm × 13 mm × 76 mm for combustion tests. Before treatment, all wood blocks were conditioned at (20±2) °C and (65±5) % relative humidity for two weeks.

The impregnation chemicals used in the study were water based and the contents were boric acid (Eti Mining Corp.), borax decahydrate (Eti Mining Corp.), copper hydroxy carbonate (Tekkim), ethanolamine (Merck), benzalkonium chloride (Tekkim) and octanoic acid (Merck). Four different impregnation chemicals coded as BC1-BC4 were prepared at the molar ratios shown in Table 1 for 1000 mL of concentrated solution.

Figure 1 Vacuum-pressure wood impregnation system
Slika 1. Vakuumsko-tlačni sustav za impregnaciju drva

Concentrated impregnation chemicals obtained as a result of all processes were mixed in concentrations of 3 % in water and impregnated into the wood placed in the vacuum-pressure tank by Bethell Method according to ASTM D1413-07 (ASTM 2007). To accomplish this, specimens were vacuumed under pressure of 60 mmHg for 30 min and then placed in a solution under 10 bar pressure for 60 min. The impregnation processes of the solutions into the wood were carried out in a vacuum-pressure wood impregnation system within the Boron Research Institute (Figure 1). The samples were left to dry at room temperature after impregnation.

Table 1 Molar concentration (mol/L) of concentrated solution
Tablica 1. Molarna koncentracija (mol/L) koncentrirane otopine

Component / *Komponenta*	**BC1, mol/L**	**BC2, mol/L**	**BC3, mol/L**	**BC4, mol/L**
Boric acid / *borna kiselina*	0.60	0.60	0.68	0.60
Borax / *boraks*	0.06	0.06	0.08	0.06
Copper hydroxy carbonate *bakrov(II) karbonatni hidroksid*	0.47	0.47	0.34	0.47
Ethanolamine / *etanolamin*	3.76	3.76	2.82	3.76
Octanoic acid / *oktanska kiselina*	-	0.34	0.34	0.34
Benzalkonium chloride *benzalkonijev klorid*	-	-	0.08	0.12

2.1 Retention and Leaching Tests
2.1. Ispitivanje retencije i ispiranja

Sapwood blocks were cut in dimensions of 20 mm × 20 mm × 20 mm from Scots pine logs. Before treatment, all wood blocks were conditioned at (20±2) °C and (65±5) % relative humidity for two weeks. Each sample group was subjected to leaching procedure separately. Five cubes of treated wood specimens were submerged in 400 mL of distilled water. Afterwards, the leachate was removed and replaced with fresh distilled water after 6, 24, 48, 72, 96, and 120 hours in a sequence. Each leachate sample was collected and stored for copper and boron analyses. Vibratory Disc Mill was used to grind the impregnated wood specimens for 1.5 minutes, a sample of each variation was taken in a beaker and nitric acid (65 % Merck) was added. The solutions were filtered using filter paper and then diluted with distilled water after acid treatment. Inductively Coupled Plasma-Mass Spectrometer (ICP-MS Perkin Elmer) was used to determine the amount of boron and copper in impregnated and leachate samples.

2.2 Decay resistance tests
2.2. Ispitivanje otpornosti prema propadanju

Decay resistance performances against white and brown rot fungi were determined according to modified EN 113 (EN 1996) standard. For the Scots pine, experiments were carried out on two different fungus species and four different solution variations. The sample sizes of wood were modified to 5 mm x 15 mm x 30 mm dimensions specified in the standard and then oven dry weights of the prepared samples were measured. After the impregnation process, the wood samples were dried in the air-conditioning chamber at (20±2) °C and (65±5) % relative humidity (Figure 2a) and then placed in each petri dish as one test and one control sample, with 20 replicates for each variation. 48 % malt-agar mixture was used for the growth medium of the fungi. In order to sterilize the prepared solution, the flasks were covered with aluminum foil and kept in an autoclave at 121 °C for 20 minutes and allowed to cool in the inoculation cabinet. After cooling well, approximately 23 mL was poured into each petri dish. After the white (*Trametes versicolor* L.) and brown (*Coniophora puteana* L.) rot fungi were inoculated into the nutrient media, the petri dishes were kept in the air conditioning chamber until the fungal growth was completed. At the end of the period, the test and control samples were placed in the petri dishes before the decay and were placed in the incubator at (22±1) °C and (70±5) % relative humidity and out of light for 8 weeks (Figure 2b). Then, the samples were taken from petri dishes and kept in the oven at (103±2) °C until they reached constant weight and their weights were recorded as full dry weight after fungi attack. Mass loss (*ML*) was calculated according to the following Eq.

$$ML(\%) = \frac{M_0 - M_1}{M_0} \cdot 100 \qquad (1)$$

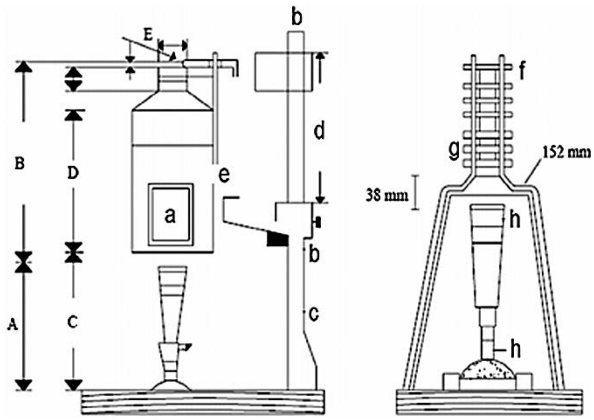
Where, M_1 (g) is the final dry weight of specimens after fungal exposure and M_0 (g) is the initial dry weight of samples.

Also, in this study, the strengths of Scots pine samples impregnated with preservatives according to EN 350 (EN 2016) standard were classified. It accepts a maximum of 3 % mass loss in standard test specimens and five strength classes are formed based on X value (Test *ML* (%) / Control *ML* (%)). These strength classes are: very durable $X \leq 0.15$; $0.30 \geq$ durable > 0.15; $0.60 \geq$ moderately durable > 0.30; $0.90 \geq$ light resistant > 0.60; undurable > 0.90.

2.3 Combustion tests
2.3. Ispitivanje gorenja

Combustion tests were performed according to ASTM E160-50 standard (ASTM 1975). The impregnated and control specimens were adjusted to (27±2) °C temperature and 30 % - 35 % relative humidity in the air-conditioning cabinet prior to the combustion process. Each stand, with 24 samples, was placed verti-

Figure 2 Test specimens (a) in air-conditioning chamber, (b) in petri dishes
Slika 2. Ispitni uzorci: a) u klimatizacijskoj komori, b) u Petrijevim zdjelicama



a. Mica glass
b. End of slide
c. Flame burner guide
d. Slide
e. Millivolt meter inlet
f. Wood specimens
g. Wire cage
h. Flame burner

Figure 3 Combustion test device
Slika 3. Uređaj za ispitivanje gorenja

cally, 2 samples on each floor. In each experiment, one control and impregnated wood samples with four different impregnation chemicals were combusted. Tests for each parameter were performed in triplicate. Samples were exposed to a (25±2) cm flame, under a gas pressure of 0.5 kg/cm^2 for 180 seconds. Following the combustion with fire, the flame was extinguished and the samples were allowed to combust autogenously until they collapsed. Temperature changes during combustion were determined using a thermometer. During the combustion process, the gas pressure was kept constant at the level specified in the standard, and the combustion test parameters were measured for three combustion stages as flame source combustion, non-flame source combustion and glowing combustion. Combustion test device is shown in Figure 3.

In order to determine the effect of flame source combustion, non-flame source combustion and glowing combustion on the impregnated wood samples, the variance analysis was applied to the groups. According to the analysis of variance, the importance levels of the significant factors were determined by Duncan test.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

The amount of boron-copper retention and leaching rates are shown in Table 2. BC1 samples showed lower retention of boron and copper. Also, almost all boron was leached from the impregnated wood in BC1. As can be seen from the results of BC2, the relative increase in boron and copper fixation is considered to be due to the addition of octanoic acid. However, when BC3 and BC4 samples were examined, it was observed that the addition of benzalkonium chloride to the solution slightly reduced the retention and leaching properties of boron. As seen from all the results in Table 2, octanoic acid and especially benzalkonium chloride enhanced copper retention, and BC4 solution had the maximum copper retention level.

Decay resistance performances against white *(Trametes versicolor)* and brown *(Coniophora puteana)* rot fungi and combustion test results have been analyzed. Decay resistance tests ended after malt-agar mixture had been completely dry in 4 months. Mass loss of the untreated control samples was between 8 - 10 %. Mass loss of control samples and impregnated wood materials and chemical mass loss prevention ratios were calculated and it was determined that all impregnation chemicals protect wood against fungi. The results of the decay test are given in Figure 4.

The highest mass loss of 1.01 % was observed in BC1 sample for *Trametes versicolor* fungi. The whole mass loss of other samples was below this value. According to the test results, mass loss of BC2, BC3 and BC4 samples was 0.75 %, 0.50 % and 0.58 %, respectively. The success of the solutions used in the experiments in preventing mass loss was found to be statisti-

Table 2 Boron copper retention and percentage released from wood samples treated with various solutions
Tablica 2. Retencija bora i bakra te postotak otpuštanja bora i bakra iz uzoraka drva zaštićenog različitim otopinama

Solution types *Vrsta otopine*	**Avg. boron retention** *Srednja vrijednost retencije bora,* kg/m^3	**Avg. copper retention** *Srednja vrijednost retencije bakra,* kg/m^3	**Avg. boron released** *Srednja vrijednost otpuštanja bora,* %	**Avg. copper released** Srednja vrijednost otpuštanja bakra, %
BC1	0.203±0.04	0.834±0.03	99.52±0.4	10.49±0.3
BC2	0.279±0.02	0.909±0.04	74.34±0.6	5.98±0.2
BC3	0.258±0.01	0.905±0.03	75.88±0.5	4.79±0.2
BC4	0.233± 0.02	1.290 ± 0.05	81.44±0.7	4.43±0.2

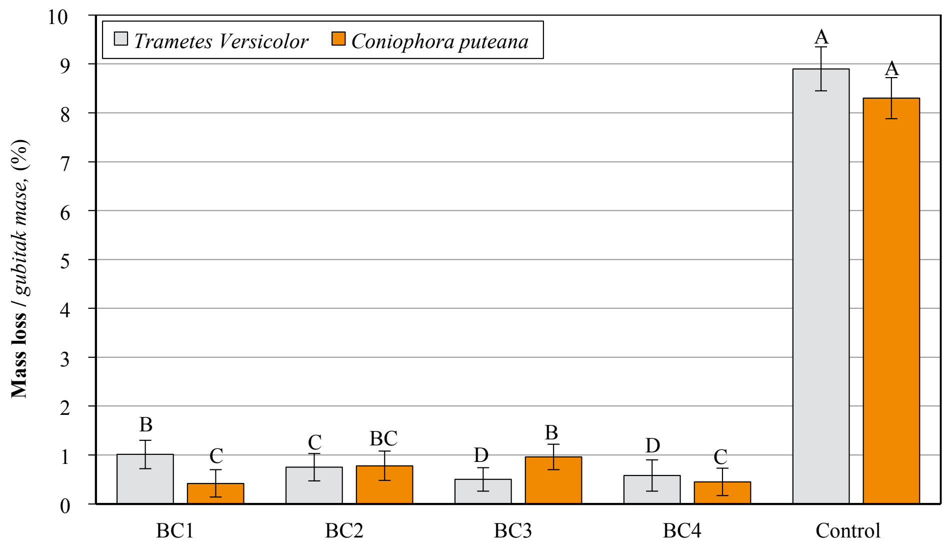


Figure 4 Mass loss (%) of samples exposed to *Trametes versicolor* and *Coniophora puteana* (Number followed by the same letter indicates no statistically significant differences according to the Least-Significant-Difference Test with 0.95 confidence)
Slika 4. Gubitak mase (u %) uzoraka izloženih gljivama *Trametes versicolor* i *Coniophora puteana* (broj iza kojeg slijedi isto slovo znači da nema statistički značajnih razlika prema testu najmanje značajne razlike s pouzdanošću od 0,95)

cally significant; however, the difference between the BC3 and BC4 solutions was found to be statistically insignificant. Also, *Coniophora puteana* fungi caused the mass loss of 0.96 % on BC3 as the highest value. Mass loss of BC1, BC2 and BC4 samples was 0.42 %, 0.78 % and 0.45 %, respectively. Considering the mass losses, in terms of *Trametes versicolor* fungus, its success in preventing mass loss was statistically significant, but the difference between the BC1 and BC4 solutions was found to be statistically insignificant.

Test results have shown that all prepared chemicals are highly resistant to both *Trametes versicolor* and *Coniophora puteana* fungi. The effects of copper and boron compounds mentioned previously in various studies on fungi were found to be compatible with the test results of this study (Kartal *et al.,* 2019; Humar *et al.,* 2007; Cao and Kamdem, 2004; Mourant *et al.,* 2009; Thevenon *et al.,* 2009; Kartal *et al.,* 2004).

According to the test results, it was determined that octanoic acid increased the resistance of impregnated wood against *Trametes versicolor*, while the resistance of wood against *Coniophora puteana* fungi was slightly decreased. The efficacy of octanoic acid against fungi has also been demonstrated in different studies (Humar *et al*., 2007; Schmidt, 1985). By addition of benzalkonium chloride, fungal effect has been more significant against *Trametes versicolor* and *Coniophora puteana*. Benzalkonium chloride is a salt of quaternary ammonia compounds and known as disinfectant and fungicidal activity in the literature (Terzi *et al*., 2011; Pernak *et al*., 2004; Preston and Nicholas, 1982) which corresponds to the results of this study.

The results of the combustion tests are given in Table 3. It was observed that the temperatures formed in the combustion with flame source, non-flame combustion and glowing combustion were in the range of 150-185 °C, 460-530 °C and 200-235 °C, respectively. These temperatures have delayed the ignition. For the Scots pine control samples, temperatures of 227.3 °C in the combustion with flame source, 532.7 °C in the non-flame combustion and 237.0 °C in the glowing combustion were measured. These results show that impregnated wood samples have better flame resistance than control samples.

BC1 samples (150.1 °C) gave the most positive results in terms of temperature during the combustion with flame source, while BC4 samples (184.7 °C) gave the most negative results except for the control samples. However, depending on the temperature of the control samples, this value can still be considered positive.

The lowest temperature values were obtained in the samples impregnated with BC3 (461.3 °C) and BC4 (495.7 °C) chemicals during combustion in non-flame source stage. The temperature of wood impregnated with chemical BC2 reached the highest value (530.3 °C) and a result similar to the control group was encountered. The lowest temperature value of the core during the glowing combustion stage was observed in BC1 (201.0 °C) and BC3 (202.3 °C) samples. The temperature of wood impregnated with chemical BC2 reached the highest value (232.0 °C) and a result close to the control group was obtained.

When taking into consideration the results of the combustion tests, it is seen that the wood samples impregnated with boron compounds known to increase the flame resistance give better results than the control samples. The fire-retardant effect of boron compounds

Table 3 Results of Duncan tests for combustion properties
Tablica 3. Rezultati Duncanova testa za svojstva gorivosti

Solution types *Vrsta otopine*	**Combustion properties** / *Svojstva gorivosti*		
	Combustion with flame source *Gorenje s izvorom plamena,* °C	**Non-flame source combustion** *Gorenje bez izvora plamena,* °C	**Glowing combustion** *Gorenje žarenjem,* °C
BC1	150.1 ± 6.2^{A}	509.7 ± 14.6^{BC}	201.0 ± 4.4^{A}
BC2	154.3 ± 7.1^{AB}	530.3 ± 16.4^{C}	232.0 ± 13.6^{C}
BC3	168.1 ± 7.8^{ABC}	461.3 ± 18.7^{A}	202.3 ± 4.7^{A}
BC4	184.7 ± 3.5^{C}	495.7 ± 19.1^{B}	219.3 ± 9.9^{B}
Control / *kontrolni uzorak*	227.3 ± 10.6^{D}	532.7 ± 13.9^{CD}	237.0 ± 16.1^{C}

Number followed by the same letter indicates no statistically significant differences according to the Least-Significant-Difference Test with 0.95 confidence. / *Broj iza kojeg slijedi isto slovo znači da nema statistički značajnih razlika prema testu najmanje značajne razlike s pouzdanošću od 0,95.*

has already been proven by different studies (Örs *et al.,* 1999; Baysal *et al.,* 2003; Temiz *et al.,* 2008). However, it can be seen that the increase in the amount of organic matter in the impregnation chemical is an important factor in the results obtained near the temperature of the control samples.

The addition of octanoic acid to the impregnation chemical did not cause a significant change in combustion with flame temperatures, but caused a significant increase in temperature compared to the results of non-flame combustion and glowing combustion. This result showed that octanoic acid in general decreased the flame resistance partially. Benzalkonium chloride, on the other hand, caused a notable increase in the combustion with flame source temperature and caused lower combustion temperatures in case of non-flame combustion and glowing combustion.

4 CONCLUSIONS
4. ZAKLJUČAK

This study investigates the combustion and decay resistance properties of boron-copper based solutions which were developed by our group. Scots pine (*Pinus sylvestris*) wood was treated with the preservatives by vacuum-pressure impregnation system. According to the decay tests, all prepared chemicals are highly resistant to both *Trametes Versicolor* and *Coniophora puteana* fungi. In addition to boron and copper, octanoic acid and benzalkonium chloride to some degree also provide fungicidal efficacy in the wood. The combustion test results demonstrate that, while boron compounds have a significant fire-retardant impact on the wood, octanoic acid decreases the flame resistance feebly. Furthermore, benzalkonium chloride causes a slight increase in the combustion with flame source temperature and lower combustion temperatures in case of non-flame combustion and glowing combustion.

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