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Effects of Catalysts on Modulus of Rupture and Chemical Structure of Heat–Treated Wood

Učinci katalizatora na modul loma i kemijsku strukturu toplinski obrađenog drva

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ABSTRACT • *Heat treatment process, which is widely used in the wood industry, has shown some negative effects on the mechanical strength of wood. The objective of this study was to investigate the effects of catalysts on the modulus of rupture (MOR), mass loss and chemical structure of heat–treated Scotch pine (Pinus sylvestris L.) samples. For this purpose, some catalysts (50 % NaOH and 47 % KOH solutions, solid KOH) were added to the heat treatment process. Heat treatment experiments were performed under the nitrogen atmosphere at the temperature of 212 °C for 2 h. The MOR and chemical changes monitored by FT-IR spectra were then examined for the test groups. According to the results of this study, the use of commercial solid potassium hydroxide (KOH) in heat treatment decreased the degree of strength loss and mass loss of heat-treated wood. The strength (MOR) loss of samples heat-treated in the presence of potassium hydroxide was found to be only 5.4 %, while the strength loss in non-catalytic treatment was found to be 12.5 %.*

Keywords: *heat treatment, Scotch pine, mechanical strength, catalyst, chemical properties*

SAŽETAK • *Proces toplinske obrade, koji se često primjenjuje u drvnoj industriji, pokazao je neke negativne učinke na mehaničku čvrstoću drva. Cilj ove studije bio je ispitati učinke katalizatora na modul loma (MOR), gubitak mase i kemijsku strukturu uzoraka toplinski obrađenog drva običnog bora (Pinus sylvestris L.). S tom namjerom u procesu toplinske obrade drva primijenjeni su katalizatori (50-postotna otopina NaOH i 47-postotna otopina KOH te kruti KOH). Eksperimentalna toplinska obrada drva provedena je u atmosferi dušika, pri temperaturi 212 °C tijekom 2 h. Modul loma i kemijske promjene praćene na spektrima FT-IR za odabrane su skupine uzoraka analizirani. Rezultati studije pokazali su da se primjenom komercijalnoga krutog kalijeva hidroksida (KOH) kao katalizatora pri toplinskoj obradi drva gubitak čvrstoće i gubitak mase toplinski obrađenog drva smanjuju. Utvrđeno je da je gubitak čvrstoće (MOR) uzoraka obrađivanih toplinom uz prisutnost kalijeva hidroksida samo 5,4 %, a gubitak čvrstoće u nekatalitičkom je tretmanu bio 12,5 %.*

Ključne riječi: *toplinska obrada, obični bor, mehanička čvrstoća, katalizator, kemijska svojstva*

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1 INTRODUCTION
1. UVOD

Wood heat treatment by pre-pyrolysis is used to improve wood properties such as its decay durability and dimensional stability without chemical treatment (Rowell *et al.,* 2009; Kamdem *et al.*, 2002).

Wood heat treatment has become widespread in forestry and wood industry. According to statistics, the production volume of ThermoWood increased from 18799 m^3 in 2001 to 159333 m^3 in 2015. Also, almost half of the production volume implemented in 2015 was produced from pine wood (ThermoWood Production Statistics, 2015).

Thermal treatment at high temperature causes considerable changes in the chemical composition of wood. During heat treatment, hemicelluloses are degraded, ramification of lignin takes place, the relative amount of crystalline cellulose increases due to degradation of the amorphous components, extractive content decreases and wood acidity increases (Weiland and Guyonnet, 2003; Tumen *et al.,* 2010; Hakkou *et al.*, 2005; Boonstra and Tjeerdsma, 2006; Kocaefe *et al.*, 2008; Brito *et al.,* 2008; Kamdem *et al.,* 2002, Chen *et al.*, 2012).

Heat treatment process has shown some negative effects on the mechanical strength of wood. The reduction of mechanical strength may limit the use of heat-treated wood in fields requiring high strength (Shi *et al.,* 2007; Mburu *et al.,* 2008; Ates *et al.,* 2009; Şahin Kol, 2010; Guofu and Shengxia, 2012; Cao *et al.,* 2012; Kartal *et al.,* 2008).

During the heat treatment process, degradation of hemicelluloses releases acetic acid, which acts as a catalyst for decomposition of carbohydrate. Decomposition of carbohydrate results in strength and mass loss. Acid concentration, leading to strength loss in heat-treated wood increases with increasing treatment temperature and duration (Tjeerdsma *et al.*, 1998; McDonald *et al.,* 1999; Sivonen *et al.,* 2005; Sundqvist *et al.*, 2006).

In recent years, numerous studies have been performed by using various methods in order to improve the mechanical properties of heat-treated wood. Kartal *et al.* (2008) studied the effects of boron impregnation and heat treatment on chemical and mechanical properties of wood. Thus, wood samples treated with either boric acid (BA) or di-sodium octoborate tetrahydrate (DOT) solutions were exposed to heat treatment. They reported that BA and DOT treatments decreased the pH value of wood. Additionally, *MOR* losses after heat treatment in DOT-treated samples were found to be higher than in the untreated samples. Awoyemi and Westermark (2005) studied the effects of borate impregnation that were applied to wood before heat treatment on the strength properties of thermally modified wood. It was found that borate impregnation reduced the severity of the strength loss during heat treatment and this was attributed to the buffering effect of the alkali on the acidity of wood, which could have mitigated the degree of degradation. LeVan and Winandy (1990) stated that acidic fire retardant chemicals and high temperature increased the degree of acid hydrolysis in the wood, thereby causing a loss in strength. Wang *et al.* (2012) studied the effect of pH on chemical and mechanical properties of thermally modified wood. They found that disodium octoborate tetrahydrate (*pH* = 8.3) and buffering solutions reduced the mass loss of thermally treated wood and improved the modulus of rupture and modulus of elasticity. Chemical analyses also showed that degradation of hemicelluloses was inhibited by disodium octoborate tetrahydrate and boric acid/sodium hydroxide (buffering solutions) pretreatments within the temperature range of 180-200 °C, which may explain the mechanical property improvement. Winandy (1997) stated that using boron-based buffers before thermal treatment reduced the severity of thermal degradation. Percin *et al.* (2015) determined that the mechanical strength losses of samples impregnated with borax were generally lower than those of non-impregnated controls.

In previous studies, the strength loss in heat-treated wood was reported to be related to acid concentration. In this study, some basic catalysts (50 % NaOH and 47 % KOH solutions, solid KOH) were used in order to investigate the effects of the basic catalysts added to the heat treatment process on the modulus of rupture (*MOR*) and chemical changes revealed by FT-IR ATR spectroscopy.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Wood material
2.1. Drvo

Scotch pine (*Pinus sylvestris* L.) sapwood samples (non-deficient, without decay and insect damages) were selected as test materials. At least 28 samples with dimensions of 20 mm x 20 mm x 300 mm per treatment were oven dried at 103 ± 2 °C until a constant oven-dry weight was obtained prior to heat treatment experiments.

2.2 Catalysts
2.2. Katalizatori

The commercial catalysts used in the experiments were 50 % sodium hydroxide (NaOH) solution, 47 % potassium hydroxide (KOH) solution and solid potassium hydroxide (KOH). NaOH and KOH solutions were purchased from Merck, and solid KOH was purchased from Sigma-Aldrich.

The technical specifications of the catalysts are given in Table 1. (Sigma Aldrich Product Specification Sheet, 2013; MERCK Use Information Sheet, 2016a; MERCK Use Information Sheet, 2016b).

2.3 Heat treatment experiments
2.3. Provedba toplinske obrade

Heat treatment experiments were performed in a vacuum oven under the nitrogen atmosphere at the temperature of 212 °C for 2 h. During the experiments, the heating rate and temperature were controlled with a PID (Proportional–Integral–Derivative) controller. In a non-catalytic heat treatment experiment, the oven-

Table 1 Technical specifications of catalysts
Tablica 1. Tehnička obilježja katalizatora

Specification / *Obilježje*	**NaOH solution** / *Otopina NaOH*	**KOH solution** / *Otopina KOH*	**Solid KOH** / *Kruti KOH*
Melting point / *talište*	-	-	361 °C
Density / *gustoća*	1.53 g/cm³ (20 °C)	1.47 g/cm³ (20 °C)	2.04 g/cm³ (20 °C)
pH	>14 (20 °C)	>13.5 (20 °C)	>14 (20 °C)
Boiling point / *vrelište*	143 °C	135 °C	719 °C
Viscosity / *viskoznost*	79 mPa·s (20 °C)	8.7 mPa·s (20 °C)	-

Table 2 Test groups
Tablica 2. Ispitivane skupine uzoraka

Group code *Oznaka skupine*	**Catalyst** *Katalizator*	**Amount of catalyst** *Količina katalizatora* %	**Temperature** *Temperatura* °C	**Time** *Vrijeme* h
UT	NO / *bez katalizatora*	-	Untreated	-
HT	NO / *bez katalizatora*	-	212	2
N1	NaOH solution / *otopina NaOH*	15	212	2
N2	NaOH solution / *otopina NaOH*	30	212	2
K1	KOH solution / *otopina KOH*	15	212	2
K2	KOH solution / *otopina KOH*	30	212	2
SK1	Solid KOH / *kruti KOH*	15	212	2
SK2	Solid KOH / *kruti KOH*	30	212	2

*UT – Untreated / *netretirano drvo*, HT – Heat-treated / *toplinski obrađeno drvo*

dried samples were weighed and placed into the oven. In the catalytic heat treatment experiments, the oven was loaded with the samples and various amount of catalyst (15 %wt and 30 %wt). The samples were heated until a final temperature of 212 °C and maintained for 2 h at this temperature. The final, treatment system was stopped and allowed to cool down to a drying temperature of 103 °C under nitrogen atmosphere. The labeled test groups are given in Table 2.

2.4 Determination of mass loss and MOR
2.4. Određivanje gubitka mase i modula loma

The mass loss (*ML*) of the samples was determined according to equation (1).

$$ML = \frac{M_{ut} - M_t}{M_{ut}} \cdot 100 \qquad (1)$$

Where:
ML – mass loss (%),
M_{ut} – initial oven-dry mass of the sample before heat treatment (g),
M_t – oven-dry mass of the same sample after heat treatment (g).

The samples with dimensions of 20 mm x 20 mm x 300 mm were conditioned at 20 ± 2 ºC and 65 ± 5 % relative humidity for about 15 days to reach the equilibrium moisture content prior to MOR tests. MOR of the samples was tested according to Turkish standard (TS 2474, 1976). At least 28 samples were used for each treatment group to determine MOR.

2.5 Fourier transform infrared spectroscopy (FTIR)
2.5. Infracrvena spektroskopija s Fourierovom transformacijom (FTIR)

FT-IR spectra of untreated, heat-treated and catalytic heat-treated wood samples were determined by using an Alpha FTIR-ATR instrument (Bruker Alpha FTIR-ATR instrument).

Spectra were determined directly using ATR technique in the range from 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1}.

2.6 Statistical analysis
2.6. Statistička analiza

An analysis of variance ($p \leq 0.05$) was conducted to evaluate the importance of differences between experimental groups. In order to measure specific differences between pairs of means, post hoc test was performed. Significant differences between the groups and homogeneity groups were determined by Duncan's multiple range test (DMRT).

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1. Mass loss and modulus of rupture
3.1. Gubitak mase i modul loma

The mass loss and *MOR* test results of heat treatment experiments are given in Figures 1 and 2.

The analysis of variance ($p \leq 0.05$) was conducted to evaluate the effects of heat treatment performed in the presence of different catalysts on mass loss and *MOR*. The test results showed that the parameters that affect mass loss and *MOR* were statistically significant. Subsequently, the Duncan's multiple range test was applied in order to determine homogenous groups (Table 3).

According to Table 3 and Figure 1, the use of solid KOH resulted in the highest mass loss of heat-treated wood samples. Due to the acid–reducing effects of the catalysts used on wood, the mass loss of the samples generally reduced as compared to non-catalytic heat treatment.

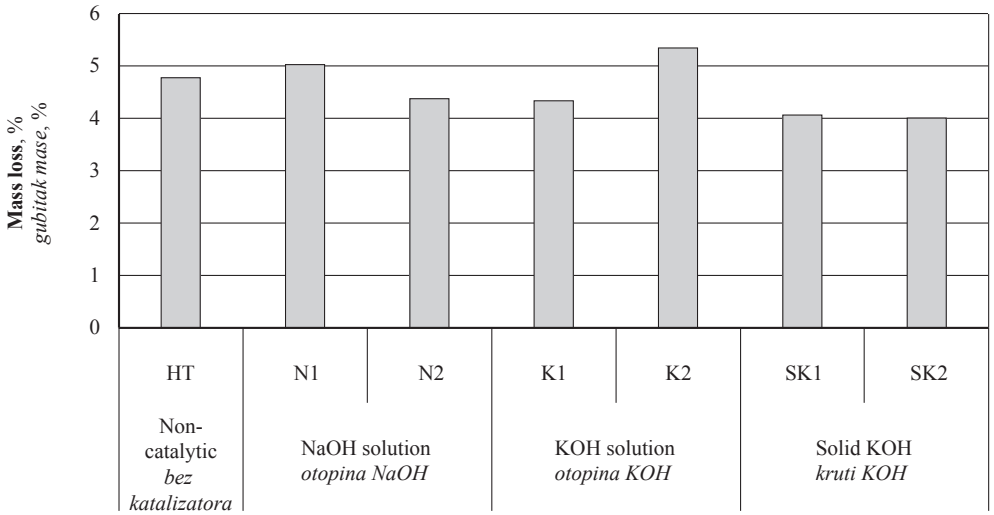

Figure 1 Mass loss of samples after heat treatment at temperature of 212 °C for 2 hours
Slika 1. Gubitak mase uzoraka nakon toplinske obrade pri temperaturi 212 °C tijekom 2 h

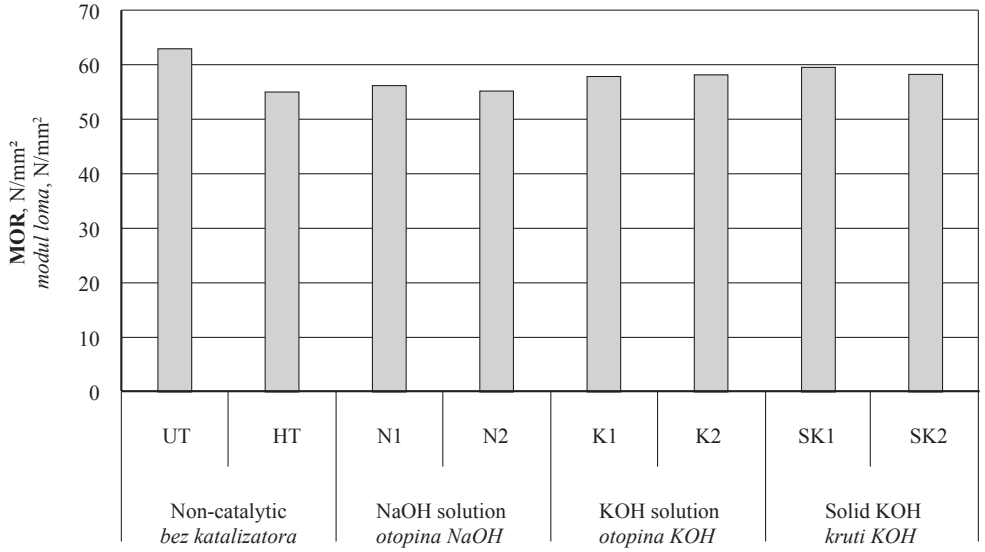

Figure 2 MOR test results of samples after heat treatment at temperature of 212 °C for 2 hours
Slika 2. Modul loma uzoraka nakon toplinske obrade pri temperaturi 212 °C tijekom 2 h

There were significant reductions in MOR of samples after heat treatment both in the catalytic and non-catalytic treatments (Table 3). As seen in Figure 2, heat treatment experiments performed in the presence of catalyst decreased the degree of strength loss of wood as compared to non-catalytic heat treatment. The strength (MOR) loss of samples heat-treated in the presence of solid KOH was found to be only 5.4 %, while the strength loss in non-catalytic treatment was found to be 12.5 %. However, the samples MOR was not significantly affected by the amount of catalyst, except for solid KOH, which was a bit different.

After heat treatment, the lowest mass loss (4 %) was found in samples heat-treated in the presence of solid KOH. Correspondingly, the highest MOR (59.56 N/mm^2) was found in the same samples and the lowest (55.04 N/mm^2) in non-catalytic (heat-treated) samples. The results showed that the use of catalyst (NaOH and KOH solutions, solid KOH) in heat treatment, especially solid KOH decreased the degree of strength loss. The most likely reason is that the use of catalyst reduces the release of acetic acid leading to strength loss in heat-treated wood. Awoyemi (2008) stated that the use of buffer reduces the release of acetic acid. Consequently, the degree of strength loss of heat-treated wood decreases significantly with increasing borate concentration from 0.1 to 0.3 M. This was undoubtedly due to the buffering effect of alkali on the strength properties of heat-treated wood.

3.2 Fourier transform IR analysis
3.2. FTIR analiza

Figure 3 presents the FTIR spectra of untreated, heat-treated and catalytic heat-treated pine wood samples. The peaks located at 805 cm^{-1}, 896 cm^{-1} and 1372 cm^{-1} corresponding to C-H deformation in cellulose and hemicelluloses decreased in the heat-treated samples when compared to the untreated samples (Tjeerdsma and Militz, 2005; Naumann *et al.,* 2005; Pandey and Pitman, 2003). However, the decreases of heat-treated samples with catalyst were less than those of heat-treated samples without the catalyst. This indi-

Table 3 Results of the Duncan's multiple range tests
Tablica 3. Rezultati Duncanovih testova

Group code *Oznaka skupine*	**Catalyst** *Katalizator*	**Amount of catalyst** *Količina katalizatora* %	**Temperature** *Temperatura* ºC	**Time** *Vrijeme* h	**SV**	**Mass loss** *Gubitak mase* %	**MOR** N/mm²
UT	NO / *bez katalizatora*	-	Untreated	-	Mean SD HG	- - -	62.97 8.70 B
HT	NO / *bez katalizatora*	-	212	2	Mean SD HG	4.77 0.77 BC	55.04 7.37 A
N1	NaOH solution / *otopina NaOH*	15	212	2	Mean SD HG	5.02 0.85 BC	56.22 11.01 A
N2	NaOH solution / *otopina NaOH*	30	212	2	Mean SD HG	4.37 1.56 AB	55.22 12.28 A
K1	KOH solution / *otopina KOH*	15	212	2	Mean SD HG	4.33 0.66 AB	57.88 8.50 A
K2	KOH solution / *otopina KOH*	30	212	2	Mean SD HG	5.34 1.05 C	58.17 7.57 A
SK1	Solid KOH / *kruti KOH*	15	212	2	Mean SD HG	4.06 0.84 A	59.56 9.64 AB
SK2	Solid KOH / *kruti KOH*	30	212	2	Mean SD HG	4.00 0.63 A	58.26 6.72 A

HG – Homogeneity group (groups with the same letters in the column indicate that there is no statistical difference (p<0.05) between the samples) / *grupa homogenosti (skupine s istim slovima u stupcu upućuju na to da među uzorcima ne postoji statistička razlika, p <0,05);* SD – Standard deviation / *standardna devijacija;* SV – Statistical values / *statističke vrijednosti*

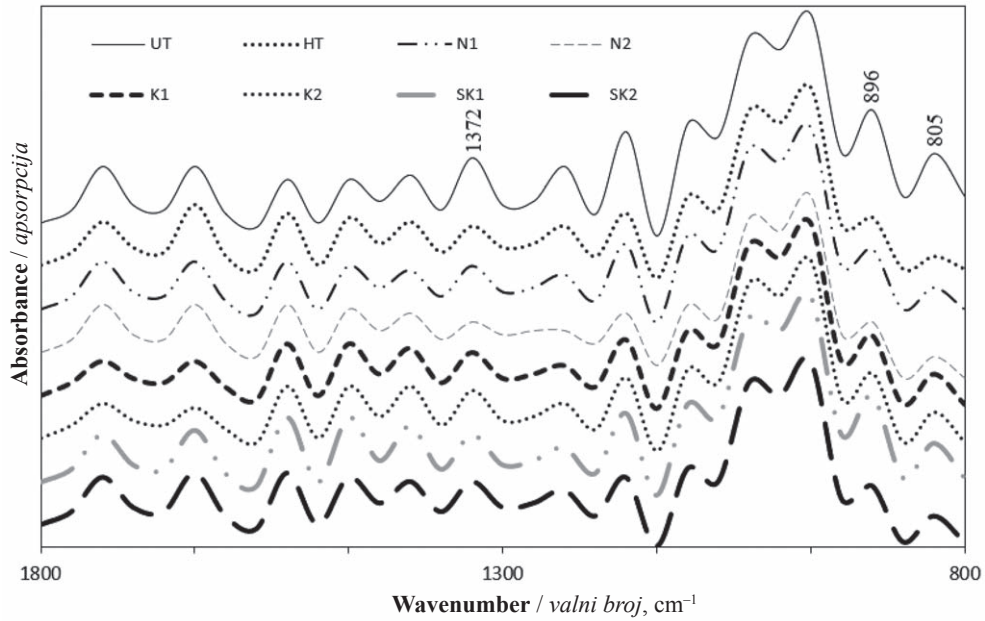

Figure 3 FTIR spectra of wood samples: respectively from top to bottom, UT, HT, N1, N2, K1, K2, SK1, SK2
Slika 3. FTIR spektri uzoraka drva; odozgo prema dolje: UT, HT, N1, N2, K1, K2, SK1, SK2

cates that the use of the catalyst in heat treatment reduced the hemicellulose degradation.

4 CONCLUSIONS
4. ZAKLJUČAK

The effects of heat treatment in the presence of catalyst on the chemical structure and *MOR* of scotch pine wood were investigated. Heat treatment in the presence of catalyst (NaOH and KOH solutions, solid KOH), especially commercial solid potassium hydroxide (KOH) could decrease the degree of strength loss and mass loss by reducing the release of acid leading to thermal degradation during heat treatment. According to FTIR analysis results, these differences could be attributed to the buffering effect of the catalysts on the strength properties of heat-treated wood.

The *MOR* losses of samples heat-treated in the presence of solid KOH (5.4 %) were lower than those in the presence of KOH solution (7.6 %), NaOH solution (10.7 %) and non-catalytic (12.5 %). However, significant differences between homogeneity groups were only found in treatments in the presence of solid KOH.

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