# Physical, Mechanical and Bonding Performance of Calabrian Pine (*Pinus brutia* Ten.) as Influenced by Heat Treatment

## Utjecaj toplinske obrade na fizikalna i mehanička svojstva te na svojstva lijepljenih spojeva drva kalabrijskog bora (*Pinus brutia* Ten.)

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**ABSTRACT** • *In this study, the effects of heat treatment on some physical, mechanical, wettability and bonding properties of Calabrian pine (Pinus brutia Ten.) were investigated. Specimens were exposed to heat under atmospheric pressure at four different temperatures (120, 150, 180, 210 °C) and three different time levels (2, 5, 8 hours). Weight loss, bending strength (MOR), modulus of elasticity (MOE) in bending, Young's modulus in compression, compression strength parallel to grain, contact angle, and bonding performance using shear tests were evaluated. All of the properties of the specimens tested were affected by heat treatment of different intensity. As a result, softer treatments yielded some increase in mechanical properties, but increase of time and temperature resulted in significant decrease in mechanical properties with decreasing mass loss. Contact angle measurements before and after treatment indicated a significant increase in wood hydrophobicity. Shear strength of the specimens were diminished when time and temperature of heat treatment were increased.*

***Key words:*** *Calabrian pine, heat treatment, mechanical properties, bonding*

**SAŽETAK** • *U radu su prikazani rezultati istraživanja učinaka toplinske obrade na neka fizikalna i mehanička svojstva, sposobnost kvašenja i svojstva lijepljenih spojeva drva kalabrijskog bora (Pinus brutia Ten.). Uzorci su izloženi utjecaju topline pod atmosferskim tlakom pri četiri različite temperature (120, 150, 180 i 210 °C) i tri različita vremena izlaganja (2, 5 i 8 sati). Primjenom smičnih testova određeni su gubitak mase, čvrstoća na savijanje (MOR), modul elastičnosti (MOE) pri savijanju, Youngov modul pri tlačnom opterećenju, tlačna čvrstoća paralelno s vlakancima, kontaktni kut i svojstva lijepljenog spoja. Toplinska obrada drva utjecala je na sva istraživana svojstva uzoraka kalabrijskog bora, ali je taj utjecaj bio različitog intenziteta. Rezultati su pokazali da blaži uvjeti toplinske obrade neznatno pridonose povećanju vrijednosti mehaničkih svojstava, a porast vremena i temperature toplinske obrade*

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*rezultirali su značajnim smanjenjem vrijednosti mehaničkih svojstava i smanjenjem gubitka mase. Mjerenje kontaktnog kuta na površini uzoraka prije i nakon toplinske obrade pokazalo je značajan porast hidrofobnosti drva. Pri povećanju vremena i temperature toplinske obrade smanjila se smična čvrstoća uzoraka.*

***Ključne riječi:*** *kalabrijski bor, toplinska obrada, mehanička svojstva, lijepljenje*

## 1 INTRODUCTION
## 1. UVOD

Preservation of wood using heat-treatment process is one of the modification processes that alter the physical, chemical and mechanical properties of wood. Studies on heat treatment of wood are not new. They started in the 1920s by Tiemann and gained interest in the last two decades leading to commercialization (Esteves and Pereira, 2009). According to Boonstra (2008), the reasons for this interest may include decreasing of durable timber, increasing demand of sustainable building materials, deforestation of sub-tropical forests and restrictive regulations. In the heat-treatment process, wood is exposed to temperatures up to 160-250 °C, usually above 200 °C depending on the species used and the desired material properties (Kocaefe *et al*., 2008).

While thermal treatment of wood results in darkened color, decreased moisture performance, improved biological durability and improved dimensional stability, most of its mechanical properties will decrease depending upon species tested, temperature and duration of exposure (Esteves and Pereira, 2009). During the process, hemicelluloses are the first to decompose, then lignin softens and finally cellulose and hydrophilic groups are modified (Bekhta and Niemz, 2003). Since hardwoods contain higher proportion of hemicelluloses than softwoods, the degradation is more severe (Esteves *et al.*, 2007).

Different commercial treatments appeared due to the increase of demand for heat treated wood in different countries during the last two decades. Increase in demand also resulted in the number of investigations conducted on different wood species. Some selected properties of heat treated wood species can be found in Esteves and Pereira (2009), but in general, color change, equilibrium moisture content (EMC), dimensional stability, bending properties and compression strength were studied in most investigations.

The purpose of this study was to investigate the influence of the heat treatment on some physical, mechanical, wettability as well as bonding performance of Calabrian pine wood. The effect of heat treatment on physical and mechanical properties of wood is well-understood, but investigations on wettability and bonding behavior are scarce. Calabrian pine covers the largest area (3 096 064 ha) among conifers grown in Turkey, which corresponds to approximately 15 % of the total forest area. Calabrian pine is a fast-growing tree; its wood is an important raw material for various fields, including construction (Bektas *et al*. 2003).

## 2 MATERIALS AND METHODS
## 2. MATERIJALI I METODE

Small clear wood samples were prepared from Calabrian pine (*Pinus brutia* Ten.) logs harvested from Bucak Forest District in Turkey. They were approximately 50 cm in diameter. The logs were transferred and sawn to lumber. Only sapwood lumbers were used in order to prepare small clear specimens.

Three pieces (2, 5, 8 hours) of lumber, free of visible defects, were used in preparation of each individual test. Five matched heat treatment samples (0, 120, 150, 180, 210 °C), with the dimensions 20 x 80 x 480 mm, were cut from each lumber. Specimens were exposed to heat under atmospheric pressure. For shear tests, PVA adhesive was used. After heat treatment, small samples were cut for testing. At least 6 replicates were prepared for each test group.

Before testing and after heat treatment process, specimens were conditioned in climatic chambers at 65 % relative humidity (RH) at a temperature of 20 °C. To minimize the influence of the MC change, specimens were tested immediately after removal from the climatic chamber. Wood MC was determined by the oven-drying method. Apparent densities of the samples were calculated according to TS 2472 (2005) using stereometric method, which is based on measurement of the sample volume and mass. The properties of wood samples investigated include mass loss (ML, %), bending modulus of elasticity (MOE), bending strength (MOR), compression strength (CS), Young's modulus in compression (YM), contact angle using water (CAW), contact angle using hexane (CAH) and shear strength (SS).

Weight loss was measured by the difference of dry weight before and after thermal treatments. Mechanical tests were performed using UTM with the capacity of 50 kN. Static bending tests were performed in a three-point bending apparatus with a span length of 360 mm and a loading speed of 6 mm/min, according to TS 2474 (1976) and 2478 (1976). Compression tests were conducted using an extensometer with a loading speed of 2 mm/minute according to TS 2595 (1997). Shear tests were performed according to TS 3459 (2012). After the mechanical tests, the load-deflection curves were analyzed in order to calculate the mechanical properties. The contact angles of water and hexane droplets on untreated and heat-treated samples were measured by the sessile drop method in the grain of sapwood cut in tangential surfaces using KSV-CAM 101 tensiometer. Hexane was chosen as non-polar test liquid to avoid problems due to polarity of wood. All measurements were performed at 20 °C and 65 % relative humidity.

Analysis of variance (ANOVA) general linear model procedure was run for data with SAS statistical analysis software to interpret effects of temperature and duration of exposure on the properties of clear wood samples.

## 3 RESULTS AND DISCUSSION
## 3. REZULTATI I RASPRAVA

Average values for ML, MOE, MOR, CS, YM, CAW, CAH and SS of the specimens tested are presented in Table 1. The average density of the control samples were 0.54 g/cm$^3$ with a correlation coefficient of 11 %. There was a good match among the density values in the various treatment groups. With respect to available literature references for similar MC, the measured density values were comparable.

ML values (%) of the specimens tested are presented in Figure 1 and ranged between 0.80 % and 15.97 %, depending on the temperature and duration of exposure. The values of ML (%) are accumulated with increasing temperature and duration of exposure. The values found in this study are similar to those reported by Esteves and Pereira (2009). ML is mainly due to the degradation of the hemicelluloses, which are the most heat sensitive polymers of the wood (Bourgois and Guyonnet, 1988). Mass loss is also one of the most important aspects of heat treatment and is accepted as indication of quality (Esteves and Pereira, 2009).

In general, the change of mechanical properties depends on the tree species and on conditions of the heat treatment. Bending properties of the samples tested in this study were significantly affected by heat treatment. MOE seemed to increase for softer treatments (%) and decrease for more severe treatments as shown in Figure 2. Results indicate that 2-hour heat treatment of samples increased the MOE values of the samples by 6 %, while 8-hour treatment decreased the MOE values by 7.8 %. There is no significant difference between the MOE values of control samples and 5-hour treatments.

There is no significant difference between the MOR values of the control samples and 2-hour treatments. For the 5 hour treatments, the MOR values of the samples were increased by 5 % and 3 % when the temperature was raised to 120 and 150 °C, while 180 and 210 °C exposures decreased the MOR values by 5 % and 7 %, respectively. After 8-hour heat treatment, the MOR values of the samples were increased by 5 % and 8 % when the temperature was raised to 120 and 150 °C, while 180 and 210 °C exposures decreased the MOR values by 7 % and 55 %, respectively. Influence of heat treatments on the MOR values is shown in Figure 3.

The effect of heat treatment on the MOR values of the wood is greater than on the MOE values (Esteves and Pereira, 2009; and Ross, 2010). According to Esteves and Pereira (2009), softer heat treatments yield

**Table 1** Average values of ML, MOE, MOR, YM, CS, SS, CAW and CAH
**Tablica 1.** Prosječne vrijednosti veličina ML, MOE, MOR, YM, CS, SS, CAW i CAH

| Temp °C | Hour | ML % | MOE N/mm$^2$ | MOR MPa | YM MPa | CS MPa | SS MPa | CAW ° | CAH ° |
|---|---|---|---|---|---|---|---|---|---|
| Control | 2* | 0 | 8181 (9.93)** | 102 (7.41) | 10111 (14.46) | 52 (5.39) | 8.45 (10.48) | 60.49 (23.14) | 13.87 (48.22) |
| | 5* | 0 | 12365 (9.16) | 136 (7.37) | 17093 (9.03) | 74 (2.51) | 8.45 (10.48) | 45.66 (20.52) | 13.65 (40.24) |
| | 8* | 0 | 10519 (13.54) | 114 (9.99) | 13022 (13.07) | 53 (10.31) | 8.45 (10.48) | 31.20 (35.11) | 13.48 (50.63) |
| 120 | 2 | 0.8 (4.75) | 9012 (7.79) | 105 (10.17) | 13595 (23.78) | 57 (10.09) | 6.87 (8.81) | 68.96 (35.11) | 13.16 (35.07) |
| 150 | | 1.5 (4.2) | 8537 (10.53) | 101 (5.57) | 11242 (13.74) | 54 (7.81) | 9.96 (6.96) | 66.54 (7.06) | 11.62 (39.44) |
| 180 | | 1.81 (7.1) | 8034 (9.48) | 103 (11.02) | 11114 (8.86) | 57 (3.69) | 6.99 (8.59) | 79.13 (20.42) | 12.46 (51.58) |
| 210 | | 9.42 (2.12) | 9296 (9.71) | 103 (9.10) | 16659 (12.15) | 67 (8.79) | 2.75 (13.00) | 100 (5.12) | 8.89 (30.24) |
| 120 | 5 | 2.05 (5.85) | 13584 (7.39) | 165 (7.37) | 20104 (10.39) | 75 (14.33) | 5.96 (44.22) | 71 (33.53) | 12.19 (52.43) |
| 150 | | 2.13 (3.4) | 11779 (10.96) | 160 (6.26) | 16233 (12.53) | 77 (8.21) | 8.07 (14.80) | 89.29 (11.19) | 11.18 (57.32) |
| 180 | | 3.04 (4.5) | 11786 (10.70) | 122 (4.10) | 15819 (14.54) | 74 (6.30) | 6.57 (19.06) | 93.26 (5.59) | 9.84 (48.43) |
| 210 | | 11.53 (3.5) | 10939 (11.59) | 105 (6.88) | 12630 (14.18) | 65 (12.57) | 3.15 (18.60) | 111.15 (5.57) | 9.82 (27.29) |
| 120 | 8 | 2.85 (6.5) | 10433 (9.81) | 115 (6.41) | 14156 (10.47) | 59 (5.22) | 12.28 (16.13) | 83.88 (18.51) | 11.39 (37.13) |
| 150 | | 3.49 (5.15) | 10182 (12.09) | 113 (14.31) | 13337 (12.74) | 58 (11.24) | 7.68 (17.90) | 114.91 (11.76) | 10.23 (47.18) |
| 180 | | 7.56 (3.0) | 9964 (9.25) | 104 (7.57) | 12414 (9.32) | 61 (6.47) | 4.92 (19.28) | 104.28 (3.58) | 9.50 (39.62) |
| 210 | | 15.97 (15) | 9099 (5.79) | 51 (10.12) | 11145 (11.70) | 52 (9.88) | 2.38 (10.43) | 87.50 (5.12) | 9.79 (20.08) |

ML - mass loss, % / *gubitak mase,* %; MOE - bending modulus of elasticity / *modul elastičnosti pri savijanju*; MOR - bending strength / čvrstoća na savijanje; YM - Young's modulus in compression / *Youngov modul pri tlačnom opterećenju*; CS - compression strength / *tlačna čvrstoća*; SS - shear strength / *smična čvrstoća*; CAW - contact angle using water / *kontaktni kut primjenom vode*; CAH - contact angle using hexane / *kontaktni kut primjenom heksana*; * number indicates piece of lumber from which samples were made, not hours of treatment duration / *broj označava uzorak piljenice od koje su izrađeni uzorci, a ne vrijeme trajanja toplinske obrade*; **values in parenthesis are coefficient of variations / *vrijednosti u zagradama označuju koeficijent varijacije*

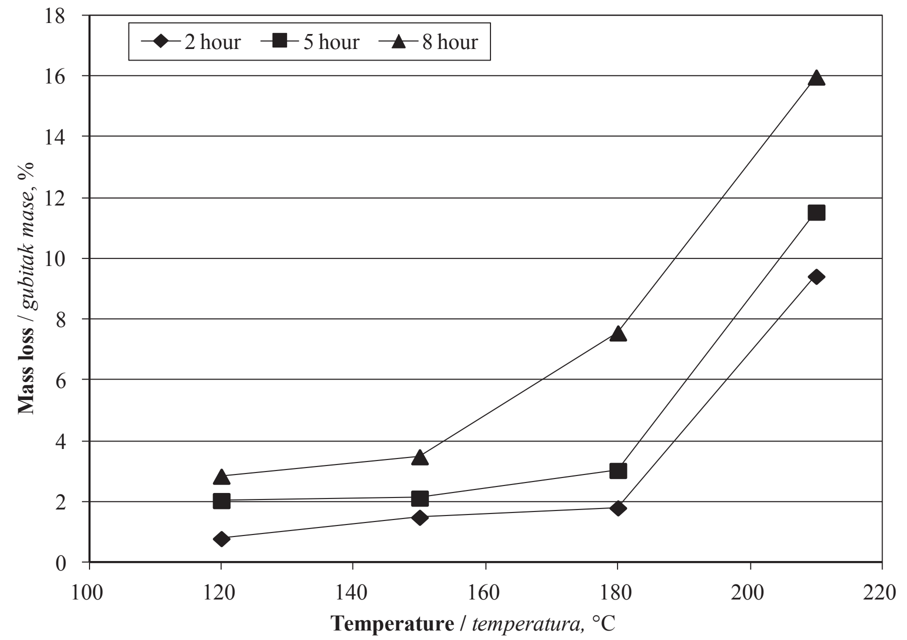


**Figure 1** Effect of heat treatment on ML (%) of Calabrian pine
**Slika 1.** Utjecaj toplinske obrade drva kalabrijskog bora na gubitak mase

an increase in the MOE values, while severe treatments result in a decrease in the MOE values. The findings presented by Altınok *et al.* (2010) support this idea. Kubojima *et al.* (2000) state that the effects of heat treatment on the MOR are less in nitrogen than in air.

Figure 4 presents the effects of heat treatment on the YM values. Test results show that 2 hour-treatments increase the YM values by 20 %, while 5 hour-treatments decrease the YM values by 23 %. There is no significant difference between the values of control samples and 8-hour treatments. There is no significant difference between the values of control samples and 150-180 °C exposures. Kubojima *et al.* (1998) reported similar results for Sıtka spruce exposed to 120, 160, and 200 °C. According to Kubojima *et al*. (1998), the increase in the YM can be explained by the increase in the crystallinity index of cellulose, while the decrease is due to the degradation.

Figure 5 illustrates the effects of heat treatment on the CS values. Results indicate that 2 and 8-hour treatments increase the CS values by 10 % and 14 %, respectively, while 5-hour treatments decrease the CS values by 14 % in comparison to control samples.

There is a contradiction in the literature on whether the CS is decreased by heat treatment or not.

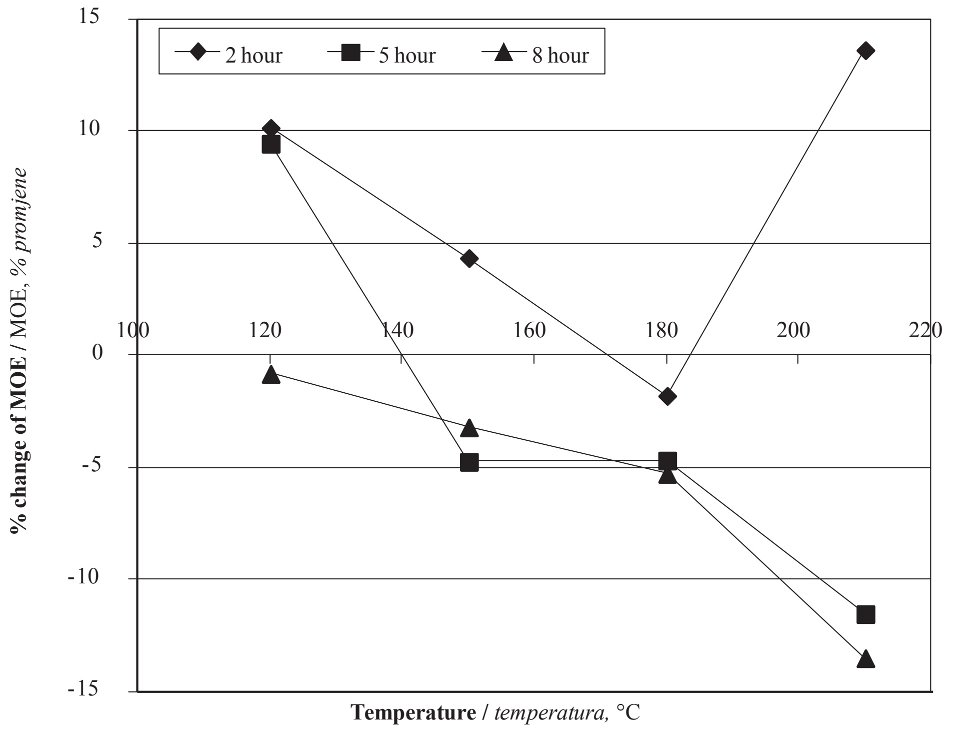


**Figure 2** Effect of heat treatment on MOE values of Calabrian pine
**Slika 2.** Utjecaj toplinske obrade drva kalabrijskog bora na modul elastičnosti pri savijanju

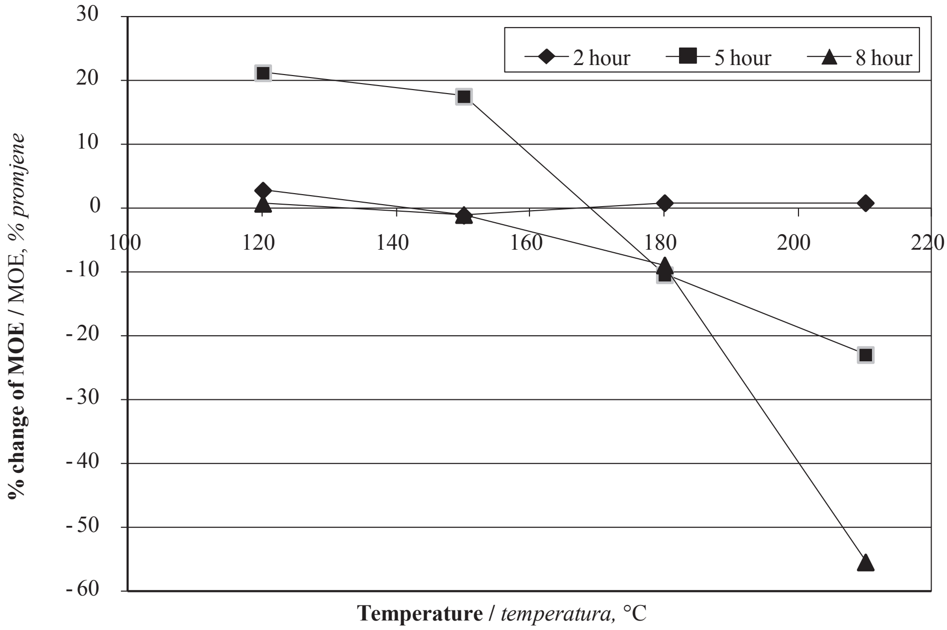


**Figure 3** Effect of heat treatment on MOR values of Calabrian pine
**Slika 3.** Utjecaj toplinske obrade drva kalabrijskog bora na čvrstoću na savijanje

Some authors (Ünsal and Ayrılmış, 2005; Korkut *et al.*, 2008a; Korkut *et al.*, 2008b) found that the CS is decreased by heat treatment, while others (Boonstra and Blomberg, 2007; Altınok *et al.*, 2010) reported an increase with heat treatment.

The reduction of mechanical properties due to heat treatment can be explained by the degradation of hemicelluloses, as the main reason, followed by crystallization of amorphous cellulose. Cross-linking of lignin may contribute to the mechanical properties in the longitudinal direction. The lower equilibrium moisture content may have positive effects on strength properties, but degradation of chemical compounds ousts this effect (Esteves and Pereira, 2009).

While the influence of heat treatment on the physical and mechanical behavior of wood is relatively well known (Esteves and Pereira, 2009), investigations of the wettability and bonding performance are limited. So far, only a few studies have investigated the wettability and bonding performance of wood (Kocaefe *et al.,* 2008; Sernek *et al.*, 2008; Altınok *et al.,* 2010; Perçin and Uzun, 2014).

Heat-treated wood shows lower affinity to water and a strongly modified wettability leading to important changes of its behavior with most coating or gluing processes (Petrissans *et al.*, 2003). According to Hakkou *et al.* (2005), wood is totally hydrophilic with a contact angle value near zero for heat treatments below 120 °C; after this threshold value, contact angle suddenly changes to reach 90° for treatment temperature between 120 and 160 °C; for higher temperatures, it remains constant near 90°. Oliveira *et al.* (2010) state that contact angle also varies for sapwood and heartwood.

Results of this study show that the contact angle using both water and hexane was significantly changed for all treatments, but the change was drastic when hig-

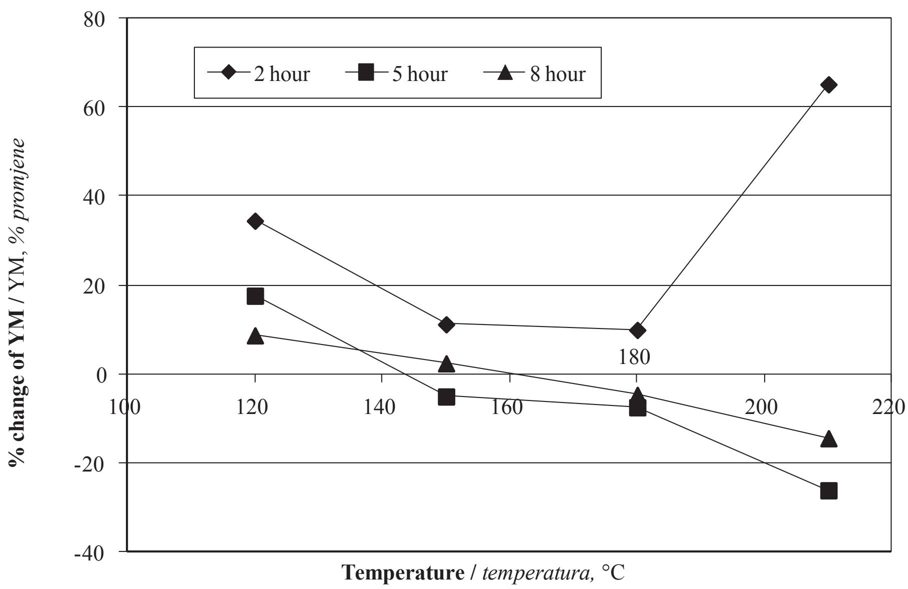


**Figure 4** Effect of heat treatment on YM values of Calabrian pine
**Slika 4.** Utjecaj toplinske obrade drva kalabrijskog bora na Youngov modul pri tlačnom opterećenju

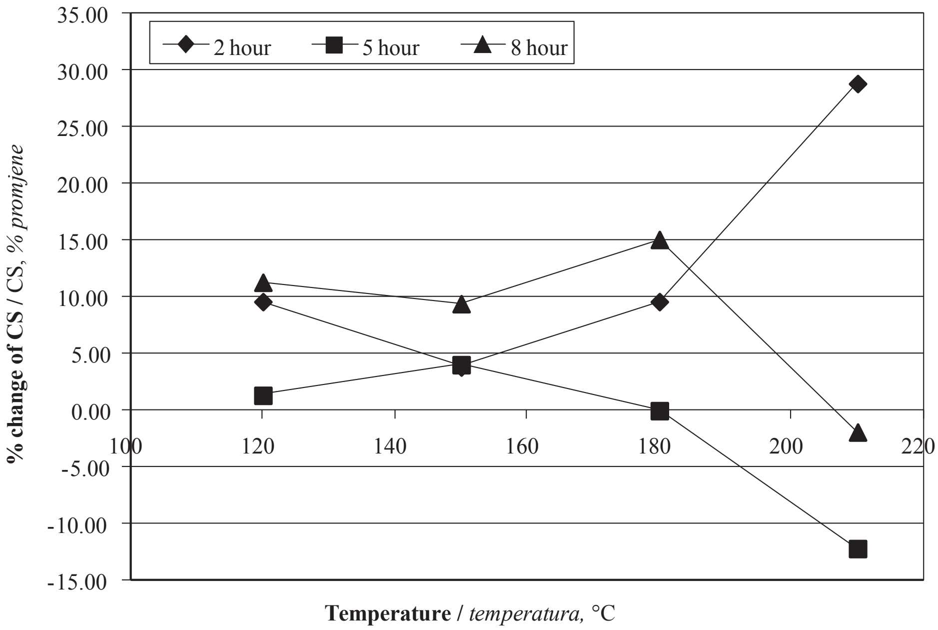


**Figure 5** Effect of heat treatments on CS of Calabrian pine
**Slika 5.** Utjecaj toplinske obrade drva kalabrijskog bora na tlačnu čvrstoću

her temperatures were applied for a longer time (Figure 6 and 7). Generally, the contact angle using water drops is increasing from 120 to 210 °C due to a higher hydrophobic condition of the wood sample, when higher temperatures are applied for a longer time. The contact angle decreases after 150 °C, thus evidencing that the wood recovers its hydrophilic nature. Treatments between 200 and 260°C can cause significant degradation in hemicelluloses content of wood, which release a great content of acetic acid (Weiland *et al.*, 1998). The phenolic carboxylic acid and 4-O-methyl-glucuronic and galacturonic acids, produced as a result of the hydrolysis of wood, also contribute to wood acidity (Windeisen *et al.*, 2007). Chen *et al.* (2012) state that heat treatment in oxygen leads to lower pH values than in nitrogen. Production of acid groups in heat treated wood for longer durations and high temperatures may cause a decrease in the contact angle when polar water drops are used. Polar water may form new linkages with acidic functional groups on the surface of heat treated wood. Drying defects occurring on the surface of the samples may enhance water–surface interaction, resulting in a decrease of the contact angle (Oliveira *et al.* 2010).

Thermal decomposition of hemicelluloses and cellulose of wood with the thermal treatment may lead to reduce the wettability of wood. Decreasing effect of heat treatment on the wettability was also observed by Hakkou *et al.* (2005) and Kocaefe *et al.* (2008). Petrissans *et al.* (2003) suggested that one of the possible reasons for the decrease of wettability could be the increase of cellulose crystallinity.

Results of this study reveal that bonding performance of 2-5-8 hour treated samples were decreased by 20-34 % comparing to control samples. When 120 °C exposure was applied, the bonding strength of the samples did not change significantly. 150 °C exposure seems to increase the bonding strength, but 180 °C and 210 °C exposures drastically reduced the bonding

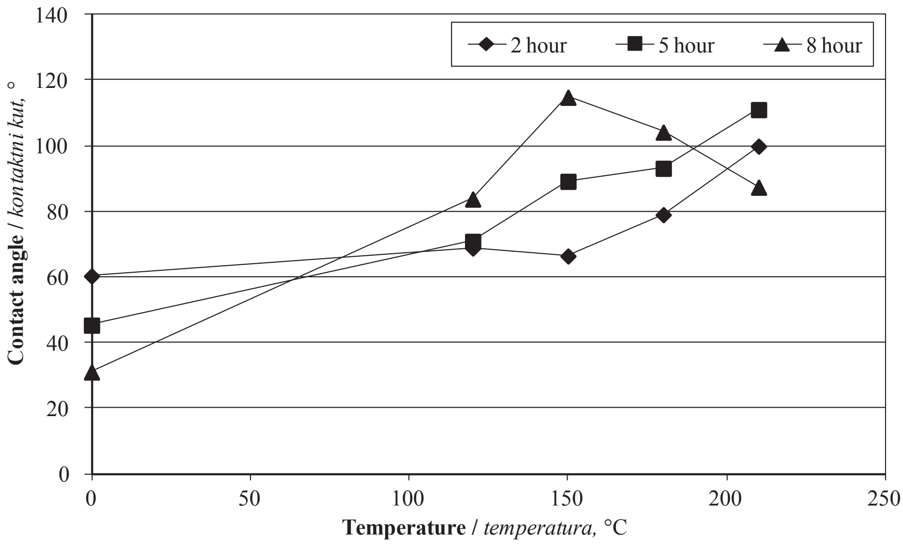


**Figure 6** Effects of heat treatment on contact angle measured by water drops
**Slika 6.** Utjecaj toplinske obrade drva kalabrijskog bora na kontaktni kut izmjeren kapljicama vode

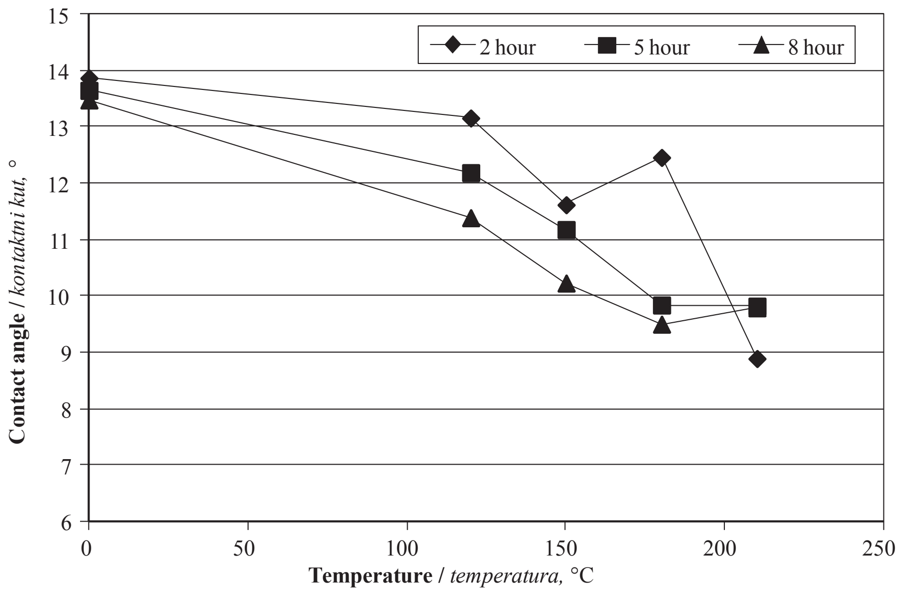


**Figure 7** Effects of heat treatment on contact angle measured by hexane drops
**Slika 7.** Utjecaj toplinske obrade drva kalabrijskog bora na kontaktni kut izmjeren kapljicama heksana

strength (30 % and 67 %). Figure 8 shows the change in bonding strength depending upon heat treatment duration and temperatures.

Decrease in shear strength was reported by several studies including Šernek *et al*. (2007), who reported a decline of 13 % for spruce bonded with PF and UF, each by heat treatment at 210 °C for 2 hours. Another study by Šernek *et al.* (2008) reported that shear strength was decreased by 23 % for heat treated spruce. Kol *et al.* (2009) also presented lower shear strength for heat treated Tali and Iroko woods at 180 °C for 2 hours. Perçin and Uzun (2014) reported similar reduction of bonding strength after heat treatment for wood species of pine, beech, scots pine and poplar using PVAc-D4 type adhesives. Esen and Özcan (2012) presented similar reduction of bonding strength after heat treatment for wood species of oak using several types of adhesives.

The only study that revealed an increase was reported by Altınok *et al.* (2010), who applied heat treatments at 100 and 150 °C for 4 hours on PVA and PU bonded samples.

## 4 CONCLUSIONS
## 4. ZAKLJUČAK

Increase of duration and temperature during heat treatment results in a significant weight loss, which is one of the quality parameters of the process. Softer treatments resulted in some improvement in mechanical properties, probably due to lower equilibrium moisture content. MOE and Young's modulus decreased significantly under treatment conditions of longer exposure and higher temperature. The effect of heat treatment on MOR is more severe. CS seems to be increasing with treatment severity. Heat treated samples become hydrophobic at the temperature ranging between 120 and 210 °C. Wettability was significantly reduced by heat treatment resulting in lower shear

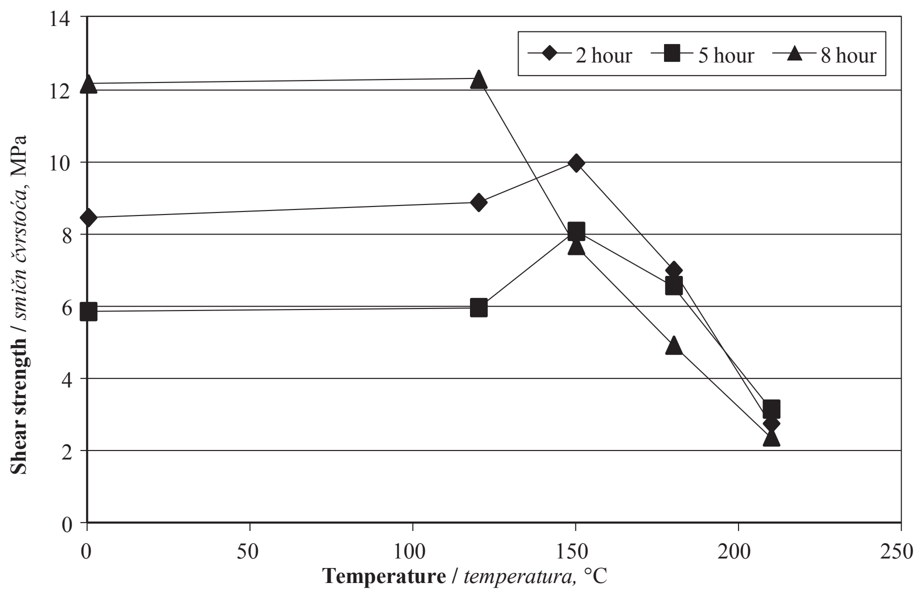


**Figure 8** Effect of temperature and duration of exposure on shear strength of Calabrian pine
**Slika 8.** Utjecaj toplinske obrade drva kalabrijskog bora na smičnu čvrstoću

strength. Since mechanical properties were significantly reduced, heat treated wood of Calabrian pine may not be suitable for load carrying members.

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