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An Evaluation of Modulus of Elasticity, Dimensional Stability and Bonding Strength of Bonded Heat-Treated Wood

Procjena modula elastičnosti, dimenzijske stabilnosti i čvrstoće lijepljenja toplinski obrađenog drva

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ABSTRACT • In this study, the effects of heating on the mechanical and sorption properties of pine and spruce wood were investigated. The study was carried out using wood heated in laboratory. Specimens were divided into the following three groups: specimens of one group were not exposed to heating, whereas specimens of two other groups were subjected to heating at the temperature of 190 °C and 215 °C, in the air under atmospheric pressure. Specimens were then bonded together using a cold press and four different systems of not heated and heated (at different temperatures) samples were formed: two not heated samples and two samples heated at different temperatures. After that, newly formed systems were moistened and dried in a climatic chamber and measurements of dimensional changes were carried out. Then the mechanical properties were observed again using two different methods - method of transverse vibrations and method of three point static bending. A positive and strong correlation coefficient was determined between static and dynamic MOE values, however, dynamically determined MOE was up to 16 % higher. None of the bonded specimens became loosened during soaking and drying, which means that wood, heated at the different temperatures, could be bonded together thereby obtaining a system of heat-treated wood that may have better mechanical or sorption properties than solid wood.

Key words: heat-treated wood, pine, spruce, mechanical properties, nondestructive evaluation method, three point static bending, bonding

SAŽETAK • U radu je prikazano istraživanje utjecaja toplinske obrade drva na mehanička i sorpcijska svojstva borovine i smrekovine. Istraživanje je provedeno na drvu koje je toplinski obrađeno u laboratoriju. Uzorci su podijeljeni u sljedeće tri skupine: uzorci koji nisu bili izloženi toplinskoj obradi, uzorci toplinski obrađeni u zraku – pri atmosferskom tlaku i temperaturi 190 °C, te na uzorke obrađene na temperaturi 215 °C. Zatim su uzorci slijepjeni u hladnoj preši tako da su izradena četiri slijepjena spoja: od uzorka toplinski neobrađenog drva i top-

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linski obradenog drva pri 190 °C, od uzorka toplinski neobradenog drva i toplinski obradenog drva pri 215 °C, od dva uzorka toplinski neobradenog drva i od uzorka drva toplinski obrađenoga pri dvije različite temperature. Nakon toga novonastali su spojevi navlaženi i osušeni u klimatskoj komori te su provedena mjerena dimenzijskih promjena. Zatim su dvjema različitim metodama – metodom transverzalnih vibracija i metodom trotočkastoga statičkog savijanja – ispitana njihova mehanička svojstva. Utvrđen je pozitivan i visok koeficijent korelacije između vrijednosti statičkoga i dinamičkog modula elastičnosti, pri čemu je dinamički modul elastičnosti bio do 16 % veći od statičkoga. Nijedan od slijepljenih spojeva pri navlaživanju i sušenju nije izgubio čvrstoću, što znači da se drvo toplinski obrađeno pri različitim temperaturama može lijepiti. Tako dobiven slijepjeni spoj toplinski obradenog drva može imati bolja mehanička ili sorpcijska svojstva od masivnog drva.

Ključne riječi: toplinski obrađena drvo, borovina, smrekovina, mehanička svojstva, nerazorne metode ispitivanja, trotočkasto statičko savijanje, lijepljenje

1 INTRODUCTION

1. UVOD

Wood is a natural, renewable and environmentally friendly material, which consists mainly of cellulose, hemicellulose, lignin and extractive substances. It is one of the strongest and most widely used organic materials in furniture and construction industry. Although wood is an excellent renewable building material, it has some disadvantages, such as its degradation under outdoor conditions and hygroscopicity. Wood based materials absorb and desorb moisture from the surrounding environment resulting in dimensional instability. Numerous methods have been tested in order to improve the durability and stability of wood dimensions avoiding the use of chemicals. One of the most effective methods used to enhance wood dimensional stability is thermal treatment. (Rowell *et al.*, 2009; Priadi and Hiziroglu, 2013; Akyildiz and Ates, 2008; Bekhta and Niemz, 2003)

Thermal wood processing involves the use of only three components - water, steam and high temperature, and, therefore, makes heated wood an eco-friendly alternative to chemically impregnated wood. During heat treatment, a large number of chemical changes occur, including the esterification of hydroxyl groups and reduction of hemicellulose and the number of accessible OH groups within wood. Consequently, heat treatment can improve the natural quality and properties of wood, such as resistance to biological deterioration. Nevertheless, the level of modification depends on temperature and time of treatment, atmosphere, wood species and its properties, wood initial humidity and dimension of the samples. It is also indicated that heat treated wood is more resistant to natural weathering. Surface damages such as cracking and mould growing on coated heat treated wood were found to be less than on the painted control wood. Although resulting in improved dimensional stability and biological durability, the main drawback of this method is the poor mechanical performance of the final wood product (Korkut and Hiziroglu, 2014; Li *et al.*, 2011; Missio *et al.*, 2015; Tomak *et al.*, 2014).

Heat treatment generally results in an apparent reduction of mechanical properties of the members due to material losses in the cell wall, hemicellulose degradation and modification of long chain molecules. The strength properties begin to deteriorate at temperatures

over 150 °C. The wood becomes more brittle, and bending and tensile strength decrease by 10 – 30 %. Therefore, in general heat treated wood would not be an ideal product where high strength properties for constructional applications are desired. However, heat treated wood can be used for aesthetic places such as garden, kitchen, and sauna furniture, cladding on wooden buildings, bathroom cabinets, floor material, musical instruments, ceilings, inner and outer bricks, doors and window joinery, and a variety of other outdoor and indoor wood applications (Korkut *et al.*, 2008; Ozcan, *et al.*, 2012; Korkut 2008).

Some earlier experiments demonstrated that heat treatment did not cause any significant changes in the values of modulus of elasticity. In addition, it was observed that the modulus of elasticity of heat treated specimens was slightly higher than the one of untreated wood and it was determined that the transverse tensile strength of the specimens decreased by 26 % (Santos, 2000; Bal, 2014).

Wood is a biological material with a heterogeneous structure. Therefore, during the analysis of its mechanical properties, the extensive spread of data is obtained. One of the possible solutions is to use a large quantity of specimens and to subject data to statistical processing. Another solution is to apply non-destructive testing methods for the evaluation of mechanical properties. The main advantage provided by this method is that specimens remain intact and there is no need to cut out specimens with certain dimensions. In addition, the use of dynamic methods for the analysis of specimens allows quite accurate determination of their modulus of elasticity (Baltrušaitis, *et al.*, 2010; Santos, 2000).

Currently, there is a lack of information on strength properties of bonded heat treated wood. Therefore, the objective of this study is to develop different systems by bonding natural and thermally modified wood and to evaluate their mechanical and sorption properties.

2 MATERIAL AND METHODS

2. MATERIJAL I METODE

Studies were carried out using specimens of spruce and pine wood cut out of planed beams with the following measurements: 250x20x6 mm. Each group contained 20 pieces of specimens. The average density of pine specimens was 595 kg/m³, and of spruce - 417

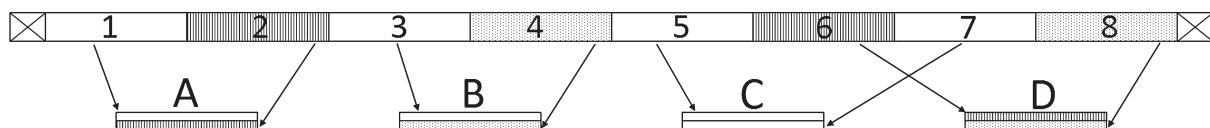


Figure 1 Specimens cutting and bonding scheme. 1, 3, 5, 7 – non heat-treated; 2, 6 – for treatment at the temperature of 190 °C; 4, 8 - for treatment at the temperature of 215 °C; x – cutout edges; A, B, C, D – systems of bonded specimens

Slika 1. Shema rezanja i lijepljenja uzoraka: 1, 3, 5, 7 – toplinski neobradeni uzorci; 2, 6 – uzorci za toplinsku obradu pri temperaturi 190 °C; 4, 8 – uzorci za toplinsku obradu pri temperaturi 215 °C; x – rubovi za izrezivanje; A, B, C, D – sustavi slijepljjenih uzoraka

kg/m³. Specimens were heated in the air under atmospheric pressure for 2 hours. When specimens have an excessive moisture content (>10 %), wood can develop cracks during the heating process and uneven color after the heating (Akyildiz *et al.*, 2009). In order to prevent this before the beginning of the heating process specimens underwent conditioning at the temperature of 25 ± 2 °C and 35 ± relative humidity of 5 % for 10 days and after that their moisture content was about 8 %. Before the determination of mechanical properties, conditioning was carried out at the temperature of 20 ± 2 °C and relative humidity of 65 ± 5 %.

Subsequently, using a cold press, specimens were bonded with a polyurethane adhesive and the following four different systems, with the following dimensions 250x20x12 mm, were formed: A – unheated specimens and specimens heated at 190 °C, B – unheated specimens and specimens heated at 215 °C, C – unheated specimens and unheated specimens, and D – specimens heated at 190 °C and 215 °C (Figure 1). Bonding conditions were applied according to the specifications of the adhesive manufacturer: the minimum pressure of 0.6 N/mm² and bonding time of 60 minutes at the temperature of 20 °C. Specimens moisture content before bonding was as follows: unheated specimens – from 9 % to 9.5 %, heated at 190 °C – about 6 %, heated at 215 °C – about 4 %.

After completing the adhesive bonding process, specimens were moistened at the temperature of 23 °C ± 1 °C and relative humidity of 80 % ± 5 %, and dried at the temperature of 23 °C ± 1 °C and relative humidity of 40 % ± 5 % in a climatic chamber. It took 10 days to complete each cycle, and after then measurements of dimensional changes and weight were carried out. The width of different bonded parts of specimens were measured in order to estimate dimensional stability, to determine how un-heated and heated, at different temperatures, parts of specimens affected each other to establish their mutual dimensional changes.

Then the mechanical properties of newly formed heated wood systems were established, using two different methods: non-destructive method of transverse vibrations and three point static bending test, according to standard EN310. The special test stand (Vobolis and Albrektas, 2007) was used to determine the modulus of elasticity (MOE) on the basis of the non-destructive testing method, which also allowed assessing the mechanical properties of specimens (Figure 2).

For three point static bending test, a universal tensile/bending testing machine was used. During the deter-

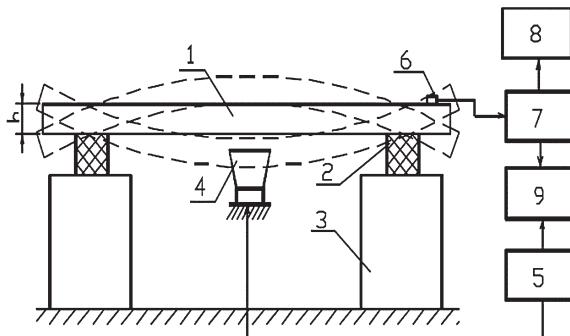


Figure 2 The scheme of the non-destructive MOE test stand: 1 – specimen; 2 – vibration damping material (foam rubber); 3 – massive supports; 4 – loudspeaker; 5 – vibration generator; 6 – sensor; 7 – measuring instrument; 8 – oscilloscope; 9 – phase meter

Slika 2. Shema uređaja za nerazorno ispitivanje modula elastičnosti: 1 – uzorak; 2 – materijal za prigušenje vibracija (pjenasta guma); 3 – masivni nosači; 4 – zvučnik; 5 – generator vibracija; 6 – senzor; 7 – mjerni instrument; 8 – osciloskop; 9 – fazni mjerač

mination of three point static bending and non-destructive MOE, the load was applied and the sensor was fastened on the unheated side of the specimen.

Other specimens were prepared to establish the bonding strength: fracture zone was removed and from the remaining two parts, samples of 100 mm length were formed (Figure 3).

One part of the specimen was placed in a dry place, while other underwent moistening and drying cycles. Moistening was carried out in a water bath and drying was performed in a kiln at the temperature of 45 °C ± 2 °C and relative humidity of 10 % ± 2 %. It took 7

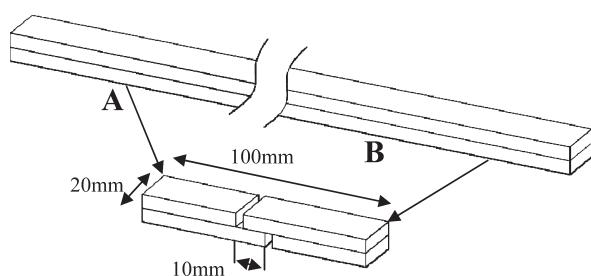


Figure 3 The scheme of preparation of specimens for bonding strength. A – group for moistening and drying, B – control group

Slika 3. Shema pripreme uzoraka za ispitivanje čvrstoće slijepljjenog spoja. A – skupina za navlaživanje i sušenje, B – kontrolna skupina

days for each cycle. The shear test of the bond line was performed on the basis of the standard EN205 using a universal tensile/bending testing machine.

Finally, statistical analysis of data (Pekarskas, 2007) was performed.

3 RESULTS AND DISCUSSION

3. REZULTATI I RASPRAVA

The obtained results demonstrate that thermal modification influenced the sorption properties and dimensional stability of specimens. It was also established that thermal modification had a greater impact on pine specimens. This is related to wood density - higher density wood is known to be less resistant to the effect of heat. The obtained data are in line with the literature data (Chaouch *et al.*, 2010). Figure 2 shows a change in the weight of specimens during the moistening and drying cycles.

It was determined that, during the moistening and drying cycle, the greatest shift in weight occurred in the case of spruce specimens of group C. There was an almost identical change in the weight of the spruce specimens of groups A, B and D. Such result may have been influenced by the density of specimens: higher density wood contains more cellulose, which is a hydrophilic wood component. This means that higher density wood contains more free OH groups to which water molecules are connected when wood undergoes moistening (Jakimavičius, 2008). The density of the section of group D specimens, which was heated at temperatures of 190 °C and 215 °C, was only 0.3 % and 0.7 % lower, respectively, than the density of the unheated section of group B specimens. The density of the section of group B specimens, which was heated at a temperature of 215 °C, was 5.4 % lower than the density of the section of group D specimens, which was heated at the same temperature.

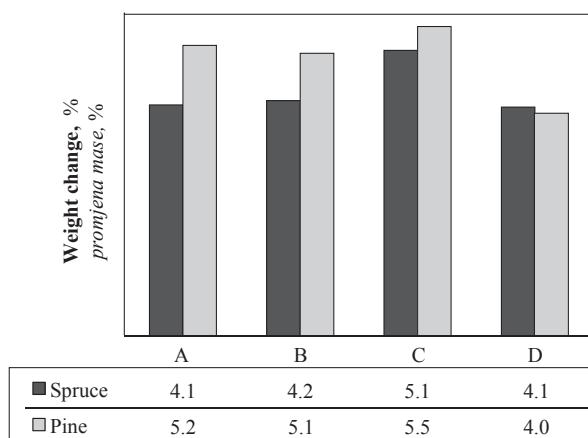


Figure 4 The average change in the weight of specimens, %:
A – unheated and heated at 190 °C, B – unheated and heated at 215 °C, C – unheated and unheated, D – heated at 190 °C and 215 °C

Slika 4. Prosječna promjena mase uzoraka, %: A – toplinski neobrađeni i toplinski obrađeni uzorak drva pri 190 °C, B – toplinski neobrađeni i toplinski obrađeni uzorak drva pri 215 °C, C – dva toplinski neobrađena uzorka drva, D – toplinski obrađeni uzorci drva pri 190 °C i 215 °C

When analyzing a change in the weight of pine specimens, one can observe that the largest and smallest shift in weight occurred in the case of the specimens of groups C and D, respectively (Figure 4). Meanwhile, the changes were almost identical in the weight of the specimens of groups A and B. It can be stated that the section of the specimen, which was heated at a temperature of 215 °C and included in system B, did not have any significant impact on sorption properties in comparison to system A. Tables 1 and 2 provide data about a shift in the width measurement of the different sections of bonded specimens.

When comparing the change in the measurements of the unheated section, it was found that the smallest shift in width occurred in the case of specimens of unheated group A. The change in the width of the unheated sections of group A of specimens of spruce and pine was ~1.3 and 1.06 - 1.18 times smaller, respectively, than that of groups B and C. The smallest shift in the width of the section heated at a temperature of 190 °C was also recorded in the case of system A. The change in the width of the section of this system, heated at a temperature of 190 °C, was 1.43 and 1.16 times smaller, respectively, in the case of spruce and pine specimens. In the case of specimens of spruce and pine, the most stable section of the specimen heated at a temperature of 215 °C was in systems B and D, respectively. However, in terms of width change, the difference in the sections of spruce specimens heated at a temperature of 215 °C (1.4-fold) was greater than the one of pine specimens (1.1-fold). In summary, it can be concluded that the lowest and highest dimensional stability was recorded in the case of groups C and A of both types of wood specimens, respectively

Having established the MOE of specimens, it was determined that values obtained by means of the

Table 1 The average shift in the measurement of spruce specimens, %

Tablica 1. Prosječna promjena dimenzija smrekovih uzoraka, %

System Spoj	Non-heated part <i>Toplinski neobrađeni dio</i>	190 °C part <i>Toplinski obrađeni dio pri 190 °C</i>	215 °C part <i>Toplinski obrađeni dio pri 215 °C</i>
A	0.82	0.67	
B	1.09		0.61
C	1.10		
D		0.96	0.85

Table 2 The average shift in the measurement of pine specimens, %

Tablica 2. Prosječna promjena dimenzija borovih uzoraka, %

System Spoj	Non-heated part <i>Toplinski neobrađeni dio</i>	190 °C part <i>Toplinski obrađeni dio pri 190 °C</i>	215 °C part <i>Toplinski obrađeni dio pri 215 °C</i>
A	1.06	0.86	
B	1.12		1.05
C	1.25		
D		1.00	0.94

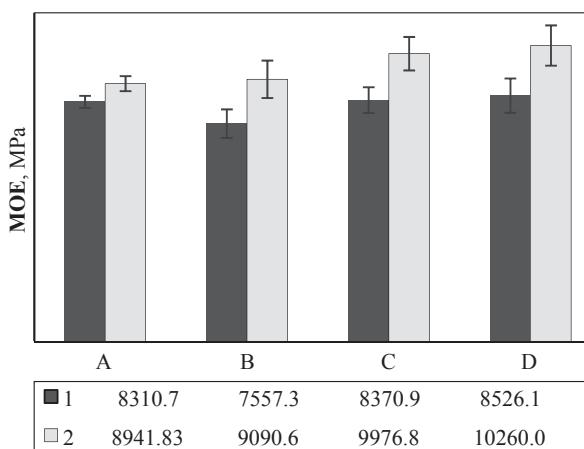


Figure 5 MOE of spruce specimens, MPa: 1 – static MOE, 2 – dynamic MOE. A – unheated and heated at 190 °C, B – unheated and heated at 215 °C, C – unheated and unheated, D – heated at 190 °C and 215 °C

Slika 5. Modul elastičnosti smrekovine, MPa: 1 – statički modul elastičnosti, 2 – dinamički modul elastičnosti; A – toplinski neobrađen i toplinski obrađen uzorak drva pri 190 °C, B – toplinski neobrađen i toplinski obrađen uzorak drva pri 215 °C, C – dva toplinski neobrađena uzorka drva, D – toplinski obrađeni uzorci drva pri 190 °C i 215 °C

dynamic method are 7 – 16 % higher than the values obtained by means of the static method. This was expected, because it is well known that the dynamic MOE values are higher than the static MOE values. Other researches (Shan-qing and Feng, 2007; Divos and Tanaka, 2005; Nzokou *et al.*, 2006) also obtained similar results. Figures 5 and 6 present the values of the MOE of spruce and pine specimens, which were obtained using the static and dynamic methods.

The obtained data reveal that, when spruce specimens were researched by means of both the static and dynamic methods, the highest values of the MOE were obtained in the case of specimens of group D. The

lowest dynamic and static values of the MOE were established in the case of specimens of group A and B, respectively. However, the dynamic values of the MOE of specimens of group A and B differ insignificantly – only by 1.7 %. In addition, when the statistical analysis of data was performed, two values were excluded as statistically unreliable from the calculations of the MOE of specimens of group A and such value was removed from specimens of group B. This also may have influenced such result. The highest value of the MOE, obtained in the case of specimens of group D, can be explained by the fact that prior to heating, the MOE of the unbonded specimens of this group, which was measured using the dynamic method, was 1 % higher in comparison to specimens of group C. When the MOE is measured by means of the dynamic method after heating and bonding, a similar result is obtained – the MOE of group D specimens is 2.8 % higher in comparison to group C specimens. Denser and dryer wood is known to have better mechanical properties in most cases (Spatz *et al.*, 2013). The equilibrium moisture content of heated wood is lower in comparison to unheated wood, thus this also may have had an impact on such results.

When pine specimens were analyzed, it was found that there was a coincidence between the highest and lowest values of the MOE, which was determined using both the static and dynamic method. The highest value of the MOE was recorded in the case of specimens of group C and the lowest value of the MOE was registered in the case of specimens of group A. The values of the MOE of these groups, which were established by means of the static and dynamic method, differed by 8.7 % and 11.4 %, respectively. The lowest value of the MOE of specimens of group A can be explained by the fact that, before the heating and bonding, this group of specimens had the lowest value of the MOE, which was determined by the dynamic method, in all specimens of pine: in comparison to specimens of group C, the MOE of specimens of group A was ~8.1 % lower.

In order to evaluate the reliability of the MOE data, the coefficient of variation was calculated. The obtained data demonstrate that the results are reliable and that their dissemination is insignificant. In the case of the values of the MOE of spruce and pine specimens, which were established by means of the static method, the coefficient of variation was 6.5 - 12.5 % and 3.9 - 7.9 %, respectively. In the case of the values of the MOE of spruce and pine specimens, which were established using the dynamic method, the coefficient of variation was 4.5 - 10.9 % and 5.0 - 9.1 %, respectively.

In order to compare the statically and dynamically obtained values of the MOE of specimens, a correlation coefficient was determined (Table 3). Although, there is a 7 – 16 % difference between the statically and dynamically obtained values of the MOE of specimens, the correlation coefficients obtained between them display a strong relationship. Only the correlation coefficient of specimens of group D spruce was ~12 % lower in comparison to the correlation coefficients of other specimens. However, it is still included in the concept of a

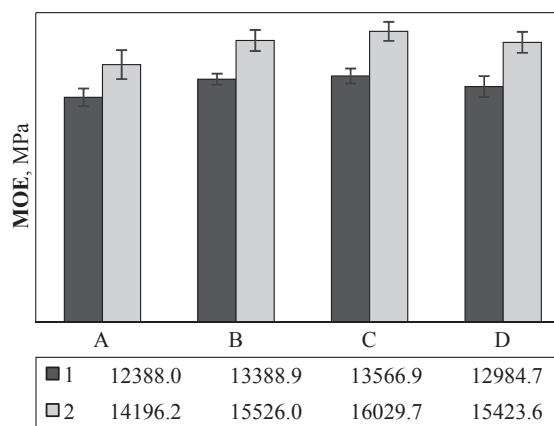


Figure 6 MOE of pine specimens, MPa: 1 – static MOE, 2 – dynamic MOE. A – unheated and heated at 190 °C, B – unheated and heated at 215 °C, C – unheated and unheated, D – heated at 190 °C and 215 °C

Slika 6. Modul elastičnosti borovine, MPa: 1 – statički modul elastičnosti, 2 – dinamički modul elastičnosti; A – toplinski neobrađen i toplinski obrađen uzorak drva pri 190 °C, B – toplinski neobrađen i toplinski obrađen uzorak drva pri 215 °C, C – dva toplinski neobrađena uzorka drva, D – toplinski obrađeni uzorci drva pri 190 °C i 215 °C

Table 3 Correlation coefficient between static and dynamic MOE values

Tablica 3. Koeficijent korelacije između vrijednosti statičkoga i dinamičkog modula elastičnosti

Correlation coefficient / Koeficijent korelacije				
Group / Skupina	A	B	C	D
Spruce / Smrekovina	0.85	0.86	0.88	0.76
Pine / Borovina	0.84	0.88	0.88	0.83

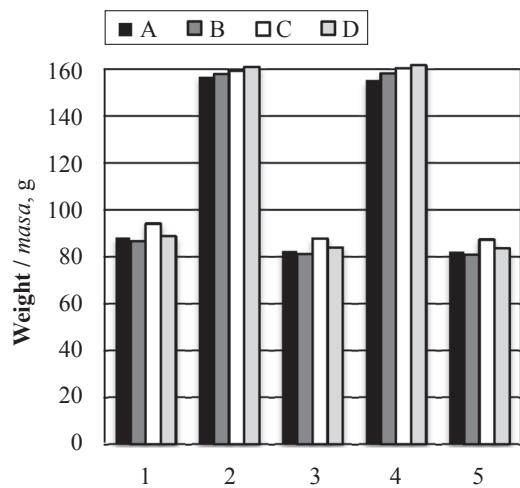


Figure 7 Spruce weight after moistening–drying cycles, g: 1 – primary, 2, 4 – after moistening, 3, 5 – after drying. A – unheated and heated at 190 °C, B – unheated and heated at 215 °C, C – unheated and unheated, D – heated at 190 °C and 215 °C

Slika 7. Masa smrekovih uzoraka nakon ciklusa navlaživanja i sušenja, g: 1 – prije navlaživanja; 2, 4 – nakon navlaživanja; 3, 5 – nakon sušenja; A – toplinski neobrađeni i toplinski neobrađeni i toplinski obrađen uzorak drva pri 190 °C, B – toplinski neobrađeni i toplinski obrađen uzorak drva pri 215 °C, C – dva toplinski neobrađena uzorka drva, D – toplinski obrađeni uzorci drva pri 190 °C i 215 °C

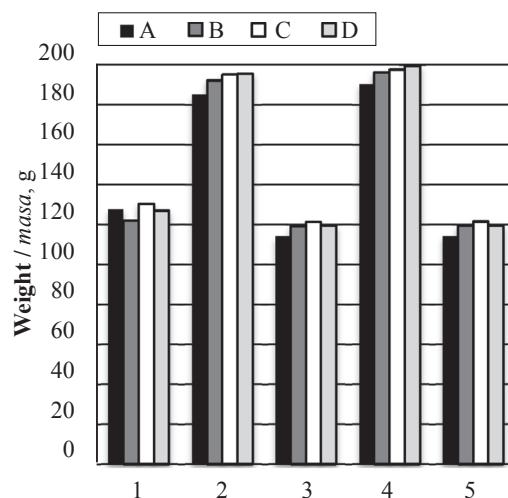


Figure 8 Pine weight after moistening – drying cycles, g: 1 – primary, 2, 4 – after moistening, 3, 5 – after drying. A – unheated and heated at 190 °C, B – unheated and heated at 215 °C, C – unheated and unheated, D – heated at 190 °C and 215 °C

Slika 8. Masa borovih uzoraka nakon ciklusa navlaživanja i sušenja, g: 1 – prije navlaživanja; 2, 4 – nakon navlaživanja; 3, 5 – nakon sušenja; A – toplinski neobrađeni i toplinski neobrađeni i toplinski obrađen uzorak drva pri 190 °C, B – toplinski neobrađeni i toplinski obrađen uzorak drva pri 215 °C, C – dva toplinski neobrađena uzorka drva, D – toplinski obrađeni uzorci drva pri 190 °C i 215 °C

strong correlation relationship. The correlation between static and dynamic MOE values were compared by several authors and strong correlation was also established between these two MOE values (Perstorper 1994; Jugo and Ozarska 1996).

After the cycle of soaking and drying, none of the bonded specimens became loosened, though the weights of some groups of specimens changed nearly twice (Figure 7 and 8). The obtained data reveal that the weights of spruce specimens shifted 1.3 – 1.5 times

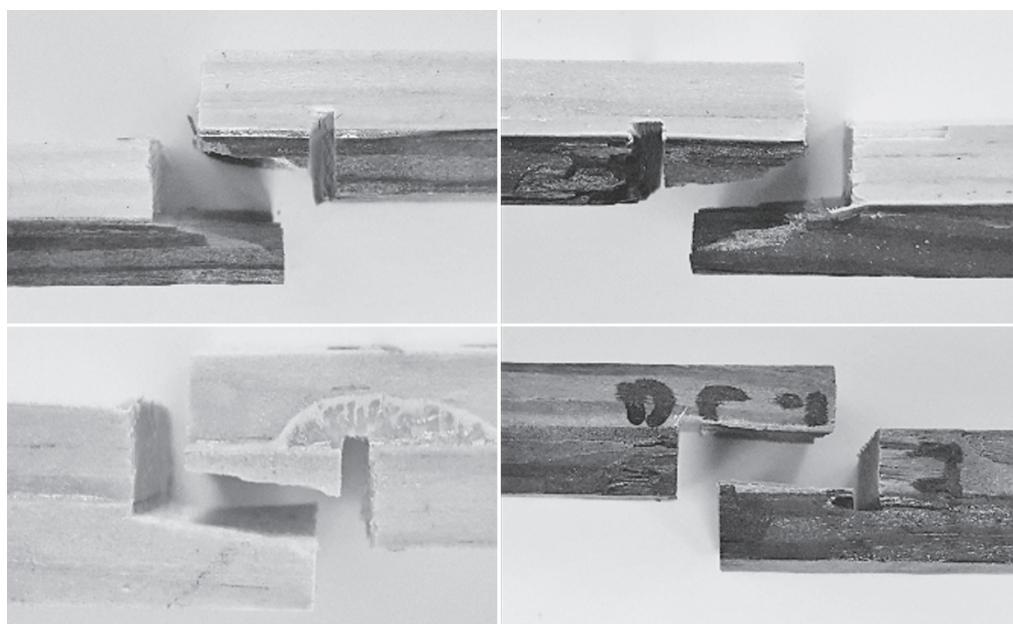


Figure 9 Specimens after bond line test
Slika 9. Uzorci nakon ispitivanja čvrstoće slijepljjenog spoja

Table 4 Part which failed, %: A – unheated and heated at 190 °C, B – unheated and heated at 215 °C, C – unheated and unheated, D – heated at 190 °C and 215 °C**Tablica 4.** Udjel uzoraka na kojima je nastao lom, %: A – toplinski neobrađen i toplinski obrađen uzorak drva pri 190 °C, B – toplinski neobrađen i toplinski obrađen uzorak drva pri 215 °C, C – dva toplinski neobrađena uzorka drva, D – toplinski obrađeni uzorci drva pri 190 °C i 215 °C

	Spruce / Smrekovina				Pine / Borovina			
	A	B	C	D	A	B	C	D
Unheated / <i>Toplinski neobrađeno</i>	7.5 %	0 %	100 %		5 %	0 %	100 %	
Heated at 190 °C / <i>Toplinski obrađeno pri 190 °C</i>	92.5 %			40 %	95 %			22.5 %
Heated at 215 °C / <i>Toplinski obrađeno pri 215 °C</i>		100 %		60 %		100 %		77.5 %

more than the ones of pine. This is related to wood density. Similar results were obtained in previous research (Navickas and Albrektas, 2013).

Having performed the testing of the strength of the bond line of specimens, it was established that the specimens of all groups cracked through the wood (figure 9). None of the specimens cracked through the bond line, therefore it can be claimed that a polyurethane adhesive is suitable for the bonding of both natural and thermally modified wood.

Spruce and pine specimens of group A and B mostly failed in the heat treated part, while specimens of group D mostly failed in the part heated at 215 °C (Table 4).

4 CONCLUSIONS

4. ZAKLJUČAK

1. During the cycle of moistening and drying, the highest and lowest dimensional stability was established in the case of groups A and C of both types of wood, respectively.
2. The dynamically determined MOE was up to 16 % higher than the statically determined MOE. However, the correlation coefficient between these values was 0.76 - 0.88.
3. The highest value of the MOE was statically and dynamically established in the case of spruce specimens of group D and pine specimens of group C.
4. During the cycle of soaking and drying, the weights of spruce and pine specimens changed by 69 – 96 % and 45 – 67 %, respectively. However, none of the bonded specimens became loosened.
5. When performing the testing of the strength of the bond line of specimens, 100 % of specimens cracked through the wood, which implies that the bond line was stronger.
6. PUR adhesives are suitable for ensuring reliable bonding of wood subjected to thermal modification at different temperatures.

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