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Thermal Degradation of Bonding Strength of Aspen Plywood

Toplinska degradacija čvrstoće lijepljenja ploče od uslojenog drva jasike

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ABSTRACT • *The objective of this research was to study the effect of exposure time on the bonding strength of aspen plywood at elevated temperatures. The plywood samples were manufactured under laboratory conditions using two types of adhesive: urea-formaldehyde (UF) and phenol-formaldehyde (PF). The plywood samples were tested after exposure to three different temperatures (150 °C, 200 °C and 250 °C) and three exposure time levels (1, 2 and 3 hours) at each temperature. Additionally, a set of control samples was tested at room temperature. The quality of bonding was assessed by shear strength test in compliance with the requirements of the standard EN 314-1. The mass and density losses as well as colour changes of the plywood samples were also determined. The findings of this study indicated that exposure of plywood panels to elevated temperature caused significant degradation of their bonding strength. PF plywood samples lost 63.2 % of their initial strength after 3 h of exposure at 250 °C, while UF samples lost 65.9 % of their initial strength already after 3 h of exposure at the temperature of 200 °C. Statistical regression-based models were also developed for predicting the loss of plywood bonding strength as functions of mass and density losses and total colour difference. As the mass/density losses or total colour difference of panels increased, the losses in bonding strength increased too.*

Keywords: *plywood; heat treatment; bonding strength; mass loss; density loss; colour change*

SAŽETAK • *Cilj ovog istraživanja bio je ispitati utjecaj vremena izlaganja toplini na čvrstoću lijepljenja ploča od uslojenog drva jasike. Uzorci ploča od uslojenog drva proizvedeni su u laboratorijskim uvjetima uporabom dvije vrste ljepila: urea-formaldehidnog (UF) i fenol-formaldehidnog (PF) ljepila. Uzorci ploča od uslojenog drva ispitani su nakon izlaganja različitim temperaturama (150 °C, 200 °C i 250 °C) tijekom različitog vremena izlaganja (1, 2 i 3 sata). Usto, skupina kontrolnih uzoraka ispitana je pri sobnoj temperaturi. Kvaliteta lijepljenja ocijenjena je testom smične čvrstoće u skladu sa zahtjevima norme EN 314-1. Također, određeni su gubitci mase i smanjenje gustoće, kao i promjena boje uzoraka ploča od uslojenog drva. Rezultati ove studije pokazali su da izlaganje ploča od uslojenog drva povišenoj temperaturi uzrokuje znatno smanjenje njihove čvrstoće lijepljenja. Uzorci ploča od uslojenog drva zalijepljeni PF ljepilom izgubili su 63,2 % svoje početne čvrstoće nakon tri sata izlaganja na 250 °C, dok su uzorci ploča od uslojenog drva zalijepljeni UF ljepilom izgubili 65,9 % početne čvrstoće već nakon tri sata izloženosti temperaturi 200 °C. Nadalje, razvijeni su statistički regresijski modeli za predviđanje gubitka čvrstoće lijepljenja ploča od uslojenog drva kao funkcije gubitka mase, smanjenja gustoće i ukupne razlike u boji. Kako se povećavao gubitak mase, smanjivala gustoća ili povećavala ukupna razlika u boji ploča, tako su se povećavali i gubitci čvrstoće lijepljenja.*

Ključne riječi: *ploča od uslojenog drva; toplinsko tretiranje; čvrstoća lijepljenja; gubitak mase; smanjenje gustoće; promjena boje*

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1 INTRODUCTION
1. UVOD

Global plywood production and consumption are increasing (FAO 2018). Plywood is widely used in various industries, in particular in construction. Due to its high physical and mechanical properties, plywood is used in construction side by side with solid wood. In Ukraine, birch and alder are used mainly for plywood production while aspen is much less used. One of the reasons for the lack of use of aspen in the production of plywood is the lack of information on the behaviour of such plywood when exposed to high temperatures. The increasing use of plywood in construction requires knowledge of the behaviour of plywood in different environmental conditions. In particular, structural members made using plywood can be subjected to high temperatures. Therefore, knowledge of the properties of plywood under high temperature is extremely important.

The performance of solid wood or wood-based composites, such as particleboard, flakeboard, waferboard, and oriented strandboard (OSB) at or after exposure to elevated temperatures has been well studied (Suchsland and Enlow 1968; Hsu *et al*., 1989; Zhang *et al*., 1997; Bekhta *et al*., 2003; Ohlmeyer and Lukowsky 2004; Del Menezzi and Tomaselli 2006, Bekhta and Marutzky 2007; Okino *et al*., 2007). These studies generally reported that the post heat treatment improved dimensional stability and enhanced durability and fungal resistance of materials, while decreasing the mechanical properties of the composites. The performance of plywood panels at or after exposure to elevated temperatures is less studied. Some authors (Sinha *et al*., 2011b; Zhou *et al*., 2012; Lunguleasa *et al*., 2018) found that the bending strength of plywood panels decreased with the increase of the heat treatment temperature and exposure time. Sinha *et al*., (2011a) also proposed regression models to predict the loss of plywood strength as a function of temperature and duration of heat treatment. Candan *et al*., (2012) showed that (a) increasing the severity of thermal treatment of plywood panels resulted in smoother surfaces; (b) the wettability properties of the treated panels were lower than those of the untreated panels; (c) the treated panels exhibited more hydrophobic characteristics for outdoor application.

Shear strength is one of the main parameters of plywood panels by which their quality of bonding is evaluated. An extensive literature search did not reveal any information about the effects of elevated temperatures on adhesive bonding performance of interior aspen plywood. The objective of this research was to investigate the effects of heat-treatment on the adhesive bonding strength of the plywood panels made using different types of adhesive.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

In this study, an aspen (*Populus tremula* L.) rotaty cut wood veneer was used. The thickness of the veneer was 1.5 mm, and the moisture content of veneer sheets was 6-8 %.

The plywood panels had been bonded using commercial urea-formaldehyde (UF) KFC-0.1-MYY and phenol formaldehyde (PF) Vatex-244 resins. We chose UF and PF resins because these adhesives are most commonly used for the manufacture of plywood (UF is interior-type resin, while PF is a more heat-resistant, exterior-type resin). UF and PF adhesives had the following parameters, respectively: solid content 66 % and 46 %, dynamic viscosity 379.3 and 213.7 MPa·s, gel time 55 s (at 100 °C) and 120 s (at 150 °C), free formaldehyde content not more than 0.15 and 0.1 %, hydrogen ion concentration (pH) 8.6 and 12. UF adhesive solution used in the manufacturing was composed of 100 parts of UF resin by weight, 15 parts of wheat flour by weight, and 4 parts of 15 % concentrated NH_4Cl by weight. The PF resin was used for plywood panel manufacturing without any filler or additive.

Plywood panels of 300 mm × 300 mm were made in an electrically heated hydraulic laboratory press. The specific pressing pressure of 1.8 MPa and temperature of 130 °C were used, and 6 min pressing time (during the last 30 s of the press cycle the pressure was continuously reduced to 0 MPa). In this study, we applied the same pressing temperature of 130 °C, as well as the same pressing modes to both adhesives, only to be able to compare the properties of UF and PF plywood panels made under the same conditions, knowing that the same pressing modes for these two adhesives may affect, to some extent, the final properties of the panels. The adhesive spreads were 120 and 130 g/m^2 based on wet mass for UF and PF adhesive, respectively. The adhesive was applied onto one side of every uneven ply. The plies were assembled perpendicularly to each other (veneer sheets were laid up tight/loose) to form plywood of five plies. Adhesive was applied on the veneer surface with a hand roller spreader. After pressing, the plywood panels were subjected to conditioning for 5 days, after which all panels were cut to extract test samples according to the standard requirements. Three plywood panels were made for each experimental condition and control.

The heat treatment of the test samples was performed in a laboratory temperature controlled ventilated oven SNOL 67/350 (AB "Utenos Elektrotechnika", Lithuania) with ±1 °C sensitivity under atmospheric pressure. All samples were heat treated at a temperature of 150 °C, 200 °C and 250 °C during 1, 2 and 3 hours at each temperature in the presence of air. Once the samples were taken out of the oven, they were cooled to room temperature before testing. Thereafter, the dimensions and weights of the plywood samples were measured to calculate the density of the plywood panels according to the standard EN 323:1993. Moreover, after heat treatment and cooling, the mass and density losses were determined and colour parameters were estimated. Then, bonding strength test was carried out according to the relevant standard (EN 314-1:2003). The bonding strength was determined by adhesive layer near the middle of the sample, i.e. at the second layer of glue. The quality of bonding was assessed in compliance with the requirements of this standard, pre-treatment according to point 5.1.1 (interior conditions): samples immersion for 24-h in water at a temperature of 20±3 °C. Fifteen samples were used for each variant of bonding strength mechanical testing. Additionally, a set of control samples was tested at room temperature.

The colour measurements of all specimens were recorded on the surface of plywood samples before and after thermal treatment with a colorimeter Minolta Croma-Meter CR-300. The *CIEL*a*b** colour system was used, where L^* describes the lightness, a^* and b^* describe the chromatic coordinates on the green-red and blue-yellow axis, respectively. From the $L^*a^*b^*$ values, the colour uniformity was calculated as a difference in the lightness (ΔL^*) and chromaticity parameters (Δa^* and Δb^*) between heat-treated and un-treated plywood samples. In addition, hue angle (h), saturation (C^*) and total colour difference (ΔE) were calculated too. On the hue circle, $h = 0°$ denotes redness and $h = 90°$ denotes yellowness. Saturation C^*, corresponding to the distance between the colour and the centre of the chromaticity plane, is a measure of colour intensity. For each specimen, 10 random measurements of surface colour were taken.

The analysis of variance (ANOVA) was conducted to study the effect of the heat treatment temperature and exposure time on the properties of plywood samples at the 0.95 confidence interval.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Mass and density losses of heat-treated plywood samples
3.1. Gubitak mase i smanjenje gustoće toplinski tretiranih uzoraka ploča od uslojenog drva

Table 1 shows the mass and density losses of the plywood samples bonded with UF and PF adhesives under different conditions of heat treatment. The mass and density losses of the UF-bonded samples are slightly greater than the mass and density losses of the PF-bonded samples. As can be seen from Table 1, the increase in mass and density losses of the samples with increasing temperature and duration of heat treatment is typical (characteristic) of heated plywood samples. Particularly dramatic loss of mass and density of the samples is observed under heat treatment at 250 °C for 3 hours. At the temperature of 250 °C, the mass and density losses for both UF- and PF-bonded plywood samples exceeded 20 %. At the temperature of 150 °C for treatment time of 1-3 hours, the mass and density losses averaged 7.6-8.1 % and 4.0-7.8 % for UF- and PF-bonded plywood samples, respectively. These losses are obviously connected with the evaporation of moisture from the plywood samples. With the increase of the heat treatment temperature up to 200 °C, the mass and density losses continued to increase. The highest mass losses of 33.1 % and 36.6 % were observed for PF and UF samples at 250 °C/3 h heat treatment. At high treatment temperature, the significant mass loss is caused by the release of various by-products during degradation of wood hemicellulose (Bekhta and Niemz, 2003; Poncsak *et al.*, 2006).

The highest density losses of 25.0 % and 27.7 % were observed for PF and UF samples at 250 °C/3 h heat treatment. Besides, the density of plywood samples decreases more with increasing temperature at the same treatment time than with increasing the duration of treatment at a certain temperature. With increasing processing time, the plywood samples lost more mass and consequently their density decreased.

Similarly, applying heat treatment to OSB panels, some researchers (Del Menezzi and Tomaselli, 2006; Mendes *et al.,* 2013) have established that, with the increase in treatment temperature and duration, the panels lost more mass, and finally, their density reduced. Lunguleasa *et al.* (2018) also observed that the mass loss of beech plywood panels increased whereas bending strength decreased with the increase of heat treatment duration and temperature.

3.2 Colour changes of plywood samples
3.2. Promjene boje uzoraka ploča od uslojenog drva

It is possible to use colour to evaluate the strength of heated plywood panels. Therefore, as a degradation indicator, colour changes were measured. The colour pa-

Table 1 Mass and density losses of aspen plywood samples depending on the type of adhesive and treatment temperature and time
Tablica 1. Gubitak mase i smanjenje gustoće uzoraka ploča od uslojenog drva jasike ovisno o vrsti ljepila, temperaturi i vremenu izlaganja

Temperature of treatment, °C *Temperatura tretiranja, °C*	**Time of treatment, h** *Vrijeme izlaganja,* h	**Mass losses, %** *Gubitak mase, %*		**Density losses, %** *Smanjenje gustoće, %*	
		PF	**UF**	**PF**	**UF**
150	1	7.64	7.90	4.37	7.33
	2	7.96	8.07	6.66	7.61
	3	7.67	8.12	4.04	7.76
200	1	9.71	8.77	10.09	5.93
	2	11.58	9.94	13.45	6.09
	3	13.03	10.26	13.06	11.32
250	1	22.50	27.64	15.23	21.98
	2	30.17	29.69	22.76	23.81
	3	33.09	36.60	25.04	27.69

rameters L^*, a^*, b^*, h, C^* and the differences of ΔL, Δa, Δb, ΔC, and ΔE are presented in Tables 2 and 3. The heat treatment provides a darkening of the surface of the plywood panels. The degree of colour change of the samples depends on the temperature to which the samples were subjected. At 150 °C, the changes in colour were slight. This is because the major change in the material was caused by the loss of water and volatile organic compounds that lead to physical changes. The darkening of the surfaces of the samples increased gradually, depending on the change in the treatment temperature ranging from 150 to 250 °C. Colour changes are significant at higher temperatures, mainly due to the transformation of carbohydrates, phenols and other extracts from the middle to the outside of the sample during the evaporation of moisture (Zhang *et al.*, 2013). Other authors (Hsu *et al.*, 1989; Del Menezzi and Tomaselli, 2006) have also observed such panel darkening when working with panel heat treatment.

Heat treatment of plywood samples reduces lightness of colour L^* and chromatic coordinates a^* and b^*. These parameters could be used as a factor for determining the level of the treatment of samples. The values of total colour difference are very high and proportional to the temperature applied (Table 3). The highest values of total colour difference were observed at the temperature of 250 °C, namely 56.4 % and 61.0 % for PF and UF bonded samples, respectively. Total colour difference is slightly higher for UF-bonded samples than for PF-bonded samples. At the highest treatment temperature of 250 °C, the colour of the samples changed from light to black, namely the lightness L^* of the samples decreased by 65.2 % and 67.6 % for the PF- and UF-bonded samples, respectively. At the same treatment conditions, the saturation C^* of the samples decreased by 73.0 % and 80.3 %, respectively, for the samples glued with PF and UF.

Colour changes, as well as mass loss of the plywood samples during heat treatment, are mainly caused by the chemical changes in the wood components, in particular by the degradation of hemicelluloses. This is confirmed by the close relationship between these parameters. We found a good correlation between mass loss and total colour difference (Fig. 1). The greater the mass loss, the stronger the colour changes and, conversely, the greater the colour changes, the greater the mass loss (Fig. 1). The finding of previous researches (Yildiz *et al.*, 2006; Nazerian *et al.*, 2011) also showed that heat treatment causes important degradations of the material and significant mass loss, resulting in a decrease of the hemicellulose content.

Table 2 Colour parameters of plywood samples before and after heat treatment (*T* – temperature of treatment; τ - time of treatment)
Tablica 2. Parametri boje uzoraka ploča od uslojenog drva prije i nakon toplinske obrade (*T* – temperatura tretiranja; τ – vrijeme izlaganja)

T, °C	*τ*, h	PF					UF				
		*L**	*a**	*b**	*h*	*C**	*L**	*a**	*b**	*h*	*C**
Untreated / *Netretirani uzorci*		83.72	6.44	18.25	1.23	19.35	87.40	6.64	17.02	1.20	18.27
150	1	82.42	7.39	21.69	1.24	22.91	83.64	7.38	20.45	1.22	21.74
	2	82.71	7.12	20.17	1.23	21.39	82.78	7.64	23.83	1.26	25.02
	3	83.13	7.31	20.90	1.23	22.14	82.20	8.21	21.91	1.21	23.40
200	1	62.91	11.4	24.13	1.13	26.67	59.96	12.10	24.74	1.12	27.54
	2	58.72	11.7	23.72	1.11	26.43	50.58	11.80	21.47	1.07	24.50
	3	55.11	11.3	23.40	1.12	25.98	42.41	11.71	18.47	1.01	21.87
250	1	30.00	3.56	5.73	1.01	6.75	28.29	2.36	2.70	0.85	3.59
	2	29.14	2.85	4.38	0.99	5.23	28.64	3.11	3.73	0.88	4.86
	3	29.87	3.59	5.37	0.98	6.46	28.78	2.68	2.81	0.81	3.88

Table 3 Colour differences of plywood samples before and after heat treatment (T – temperature of treatment; τ - time of treatment)
Tablica 3. Razlike u boji uzoraka ploča od uslojenog drva prije i nakon toplinske obrade (*T* – temperatura tretiranja; τ – vrijeme izlaganja)

T, °C	*τ*, h	PF					UF				
		Δ*L**	Δ*a**	Δ*b**	Δ*C**	Δ*E*	Δ*L**	Δ*a**	Δ*b**	Δ*C**	Δ*E*
150	1	-1.30	0.95	3.44	3.56	3.80	-3.76	0.74	3.43	3.47	5.14
	2	-1.01	0.68	1.92	2.04	2.27	-4.62	1.00	6.81	6.76	8.29
	3	-0.59	0.87	2.65	2.79	2.85	-5.20	1.57	4.89	5.13	7.31
200	1	-20.81	4.92	5.88	7.32	22.18	-27.44	5.46	7.72	9.27	29.02
	2	-25.00	5.21	5.47	7.07	26.12	-36.82	5.16	4.45	6.23	37.45
	3	-28.61	4.85	5.15	6.63	29.47	-44.99	5.07	1.45	3.60	45.30
250	1	-53.72	-2.88	-12.52	-12.61	55.23	-59.11	-4.28	-14.32	-14.68	60.97
	2	-54.58	-3.59	-13.87	-14.13	56.43	-58.76	-3.53	-13.29	-13.41	60.35
	3	-53.85	-2.85	-12.88	-12.89	55.44	-58.62	-3.96	-14.21	-14.39	60.45

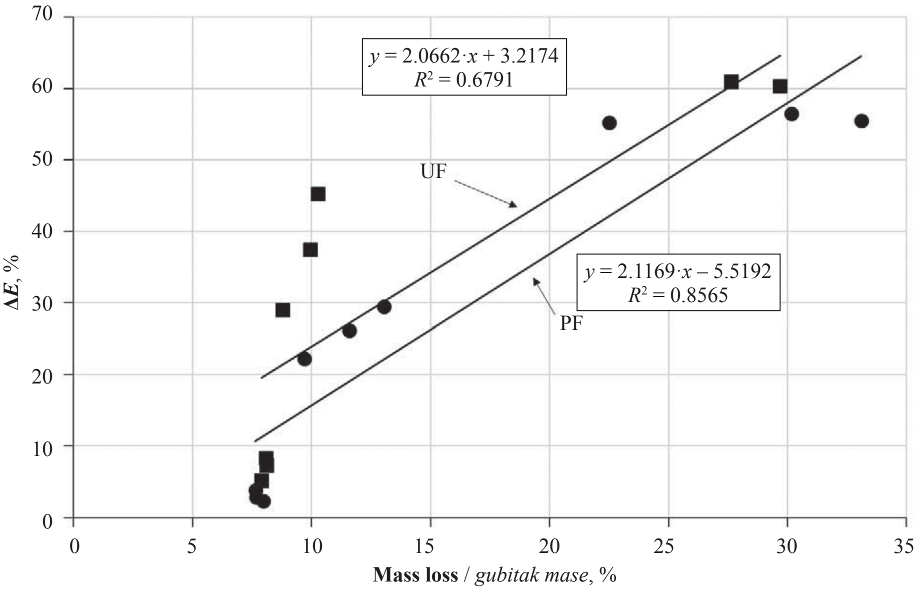

Figure 1 Correlation between total colour difference and mass loss of samples
Slika 1. Korelacija između ukupne razlike u boji i gubitka mase uzoraka

3.3 Shear strength of the plywood samples
3.3. Smična čvrstoća uzoraka ploča od uslojenog drva

Figures 2 and 3 show the dependence of the bonding strength on the temperature and duration of heat treatment. The highest decrease in bonding strength was observed for UF and PF plywood samples treated at 250 °C/3h, and a smaller one for the samples treated at 150 °C/1h.

For the PF plywood samples heat treated at the temperatures of 150 °C, 200 °C and 250 °C for 1-3 hours, the bonding strength is lower than that for the control untreated samples (Figures. 2, 3), but the average values are higher than 1 MPa (except the treatment at 250 °C /3h, 0.98 MPa with cohesive wood failure of 12 %). Therefore, the heat treated PF-bonded samples meet the requirements for plywood for use in interior conditions according to EN 314-2:1993, although shear strength is lower than that for untreated plywood samples. For the UF plywood samples treated at high temperatures of 150 °C and 200 °C for 1-3 h, the average values of bonding strength are also higher than 1 MPa. Further increasing temperature of heat treatment up to 250 °C at these durations leads to the reduction of bonding strength, and the average values are lower than 1 MPa, namely 0.71 MPa (cohesive wood failure of 25 %) and 0.78 MPa (cohesive wood failure of 30 %) for treatments at 250 °C /1h and 250 °C /2h, respectively. For UF-bonded samples heated at 250 °C for 3 h, it was not possible to determine the bonding strength due to the delamination of the samples.

Plywood, is a layered composite that contains wood and resin as adhesive. As plywood has a layered structure, its strength is highly dependent on adhesive between the layers. The degradation of the PF adhesive used in the manufacturing of plywood panels occurs at a temperature above 175 °C, while the degradation of

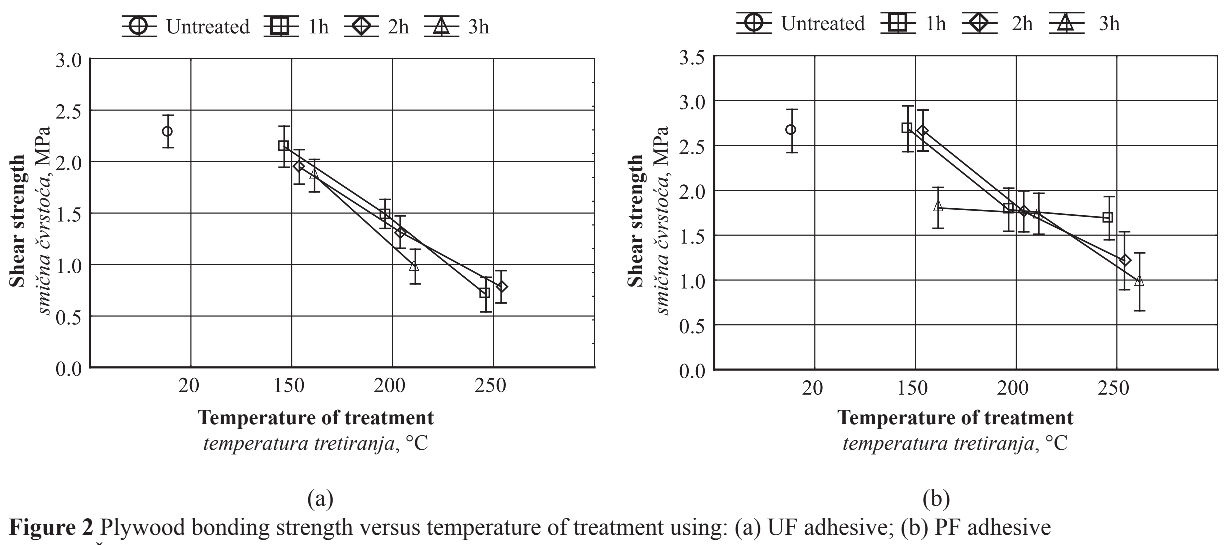

Figure 2 Plywood bonding strength versus temperature of treatment using: (a) UF adhesive; (b) PF adhesive
Slika 2. Čvrstoća lijepljenja ploča od uslojenog drva u ovisnosti o temperaturi tretiranja: (a) ploče s UF ljepilom, (b) ploče s PF ljepilom

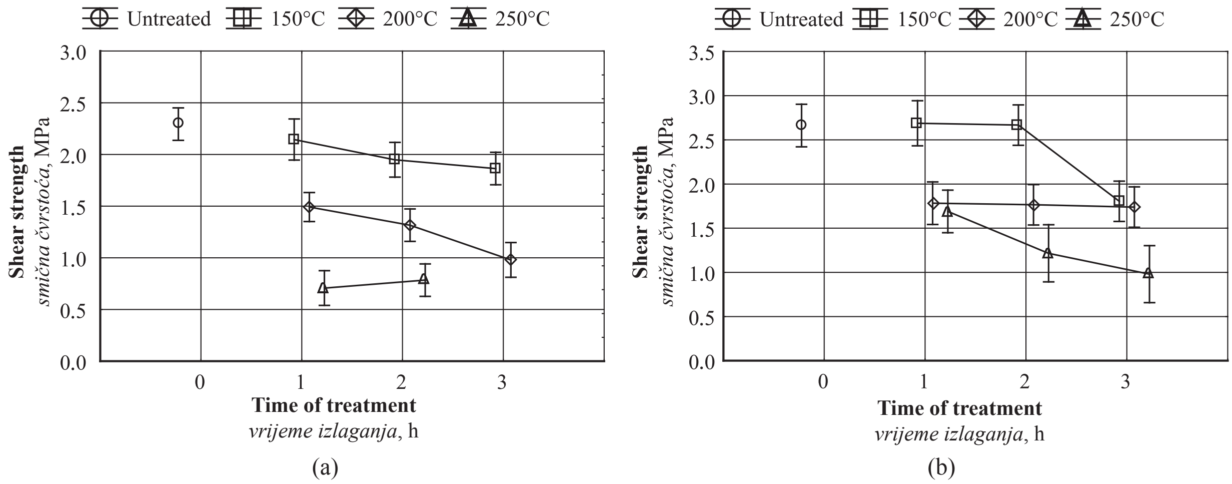

Figure 3 Plywood bonding strength versus time of treatment using: (a) UF adhesive; (b) PF adhesive
Slika 3. Čvrstoća lijepljenja ploča od uslojenog drva u ovisnosti o vremenu izlaganja toplini: (a) ploče s UF ljepilom, (b) ploče s PF ljepilom

UF adhesive occurs at a lower temperature. Therefore, when plywood samples are exposed to an elevated temperature, the degradation of the adhesive lines causes more rapid degradation in strength, especially for UF-bonded samples. The temperatures of 200 °C and 250 °C are high enough to cause both wood and resin to degrade. Therefore, during the heat treatment process, the panels degrade generally not only by degradation of wood veneers, but also by degradation of the adhesive and consequently also the veneer adhesion (Lunguleasa *et al.*, 2018).

As stated above, heat treatment of the plywood samples leads to loss of moisture, evaporation of extractives, degradation of hemicellulose and degradation of the resin. Together, all these lead to mass and density losses as well as to colour changes of the samples. As a result, there is a loss in bonding strength of the samples. Therefore, it was important to find out whether there are correlations between bonding strength and mass/density losses and total colour difference of the samples.

It was established that a good correlation exists between bonding strength loss and mass loss, between bonding strength loss and density loss, and between bonding strength loss and total colour difference of the samples (Figure 4). Linear regression models were

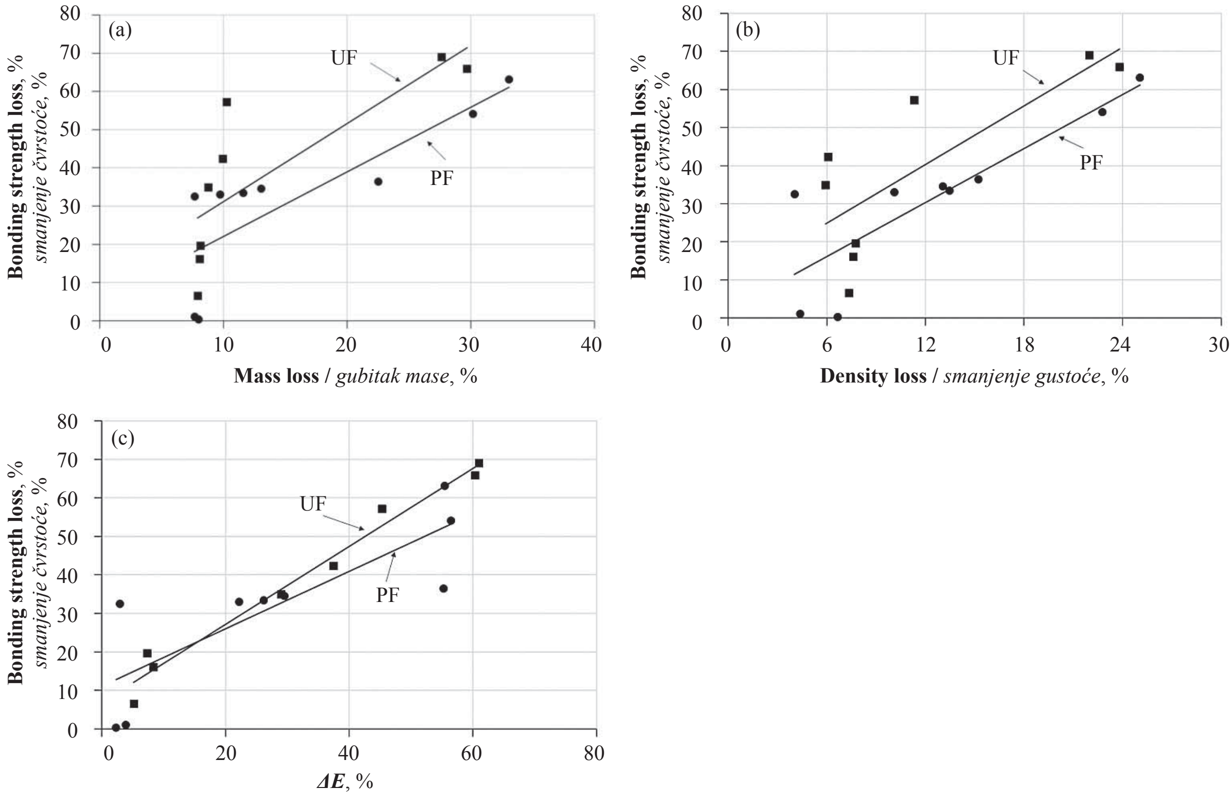

Figure 4 Correlation between bonding strength loss and: (a) mass loss; (b) density loss; (c) total colour difference
Slika 4. Korelacija između smanjenja čvrstoće lijepljenja ploča od uslojenog drva i (a) gubitka mase, (b) smanjenja gustoće, (c) ukupne razlike u boji

proposed to predict bonding strength loss (in %) depending on the mass loss (*ML*), density loss (*DL*) and total colour difference (ΔE) of the samples:

for UF-bonded samples:

Bonding strength loss = 2.0421 × *ML* + 10.795 ($R^2 = 0.63$)

Bonding strength loss = 2.5634 × *DL* + 9.5515 ($R^2 = 0.62$)

Bonding strength loss = 1.012 × ΔE + 6.8641 ($R^2 = 0.98$)

for PF-bonded samples:

Bonding strength loss = 1.6877 × *ML* + 5.2274 ($R^2 = 0.67$)

Bonding strength loss = 2.3664 × *DL* + 1.9497 ($R^2 = 0.73$)

Bonding strength loss = 0.7453 × ΔE + 11.093 ($R^2 = 0.68$).

In our previous study, we also found that the colour parameters can be estimated quantitatively and used as a prediction of wood strength (Bekhta and Niemz, 2003), although some authors (Unsal *et al.*, 2009) consider that darkening is a weak indicator in estimating the static flexural strength of the post heat-treated MDF panels.

4 CONCLUSIONS
4. ZAKLJUČAK

The findings of this study indicated that the exposure of aspen plywood panels to elevated temperature, especially above 200 °C, caused significant degradation of their bonding strength. The effect of temperature on the loss of bonding strength is more significant than the duration of heat treatment. The higher the heat treatment temperature and the longer the time, the more significant are the changes in plywood surface colour and in bonding strength. PF plywood samples lost 63.2 % of their initial strength after 3 h of exposure at 250 °C, while UF samples lost 65.9 % of their initial strength already after 3 h of exposure at the temperature of 200 °C; at 250 °C for 3 h the UF-bonded samples collapsed (delaminated). Statistical regression-based models were also developed for predicting the bonding strength loss of aspen plywood panels as functions of mass and density losses and total colour difference. As the mass/density losses or total colour difference of panels increased, the losses in bonding strength increased too.

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