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The Effect of Chip Size and Alkaline Pre-Hydrolysis Conditions on Following Soda Pulping of Hornbeam Wood

Utjecaj veličine sječke i uvjeta alkalne predhidrolize na natronski postupak proizvodnje celuloze od grabovine

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ABSTRACT • *The impact of chip size and hydrolysis temperature on changing chemical composition of hornbeam wood and following soda pulping is investigated. Three chip sizes, two temperatures (60 and 90 ˚C) and 120 minutes retention time in pre-hydrolysis step were selected. After pre-hydrolysis treatment, the sample was divided into two portions; one third was used for chemical analysis and the other two thirds for soda pulping. The reference soda pulping conditions were used on either treated or untreated chips. The influence of pre-hydrolysis was determined measuring cellulose and lignin content, residual alkali and the hemicelluloses removal. The lignin and cellulose content were marginally increased and the hemicellulose removal was higher at larger chip size. Total yield and rejects, kappa number and strength properties of the unbleached pulp were measured using corresponding Tappi standard test methods. The pulping total yield and rejects of the treated chips varied between 30.31 % and 48.14 % and 0.83 % to 7.31 %, respectively. The reject from soda pulping of untreated chips was 24.16. Pre-hydrolysis treatment reduced the tensile index, but the tear index was only marginally improved.*

Keywords: *hemicelluloses, pre-hydrolysis, yield, strength, residual alkali, hornbeam, soda pulping*

SAŽETAK • *U radu je istražen utjecaj veličine sječke i temperature hidrolize na promjene kemijskog sastava grabovine te naknadni natronski proces proizvodnje celuloze. Za predhidrolizu su odabrane tri veličine sječke, dvije temperature (60 i 90 °C) i retencijsko vrijeme od 120 minuta. Uzorak je nakon predhidrolize podijeljen na dva dijela, pri čemu je jedna trećina iskorištena za kemijsku analizu, a ostale su dvije trećine upotrijebljene za natronski postupak proizvodnje celuloze. Referentni uvjeti natronskog postupka primijenjeni su na predtretiranoj i netretiranoj sječki. Utjecaj predhidrolize određen je mjerenjem sadržaja celuloze i lignina, količine zaostale lužine i izdvajanja hemiceluloza. Sadržaj celuloze i lignina samo je neznatno porastao, dok je izdvajanje hemiceluloza bilo veće za sječku većih dimenzija. Ukupan prinos vlakana i udio neadekvatnih vlakana, kappa broj i čvrstoća nebijeljene natronske celuloze određeni su u skladu s odgovarajućim Tappi standardima. Ukupan prinos vlakana i*

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udio neadekvatnih vlakana za netretiranu su drvnu sječku iznosili 30,31 i 48,14 %, odnosno 0,83 do 7,31 %. Udio neadekvatnih vlakana dobivenih natronskim postupkom netretirane sječke iznosio je 24,16 %. Predhidrolizom je smanjen vlačni indeks, uz neznatno poboljšanje indeksa kidanja.

Ključne riječi: *hemiceluloze, predhidroliza, prinos, čvrstoća, zaostala lužina, grabovina, natronski postupak proizvodnje celuloze*

1 INTRODUCTION
1. UVOD

Chemical industry and energy sector of the world are suffering from increasing consumption and limited supply, because they are almost fully relying on non-renewable oil and natural gas resources. Therefore, this sector is continuously searching for alternative sources of feedstock, and among them lignocellulosic material as renewable sources with low cost and potential volume has been a viable alternative introducing biorefinery concept (Zhang *et al*., 2015).

Biorefinery concept is considered as a path to sustainability for partial fulfillment of the future demand for chemical feedstock and energy (Hatti-Kaul, 2010). This concept integrates biomass conversion processes to produce fuel, power, and value added chemical from biomass. It is analogous to today's petroleum refining, which produces multiple fuels and products from petroleum (Vachalova *et al*., 2014).

Pulping residual utilization as chemical production feedstock has been followed by two paths: 1- conversion of residual lignin and hydrolyzed carbohydrates from spent liquor (black liquor) prior to burning the spent liquor, and 2- pre-hydrolysis and extraction of hydrolysate before pulping. The first path has been used for a long period in the production of sugars, vanillin and other derivatives from soft wood pulping liquors. However, the second path is new and has been pursued very extensively during last two decades following the biorefinery concept or forest biorefinery (Ormshy *et al*., 2012).

Available literature presents information on acid and alkaline per-hydrolysis of different lignocellulosic material to extract carbohydrate hydrolysate to be used as chemical feedstock and also facilitate following pulping reactions. Some of the research works are directed to extract hydrolysate for enzymatic bio-conversion in biofuel and bio-chemical production (Ormsby *et al*., 2012; Carvalherio *et al*., 2008; Shen *et al*., 2018; Liu *et al*., 2018; Lu *et al*., 2017; Travaini *et al*., 2016; Dawan *et al*., 2008; Song *et al*., 2011). Kurian *et al.* (2010) enzymatically fermented the xylose and other hemicellulose products from sweet corn in the production of bio-ethanol. Teherzadeh and Karimi (2007) reviewed the acid pre-hydrolysis of various lignocellulosic material. Yan *et al.* (2017) used acidic hot water treatment to improve and characterize the value added non-carbohydrate compounds from poplar wood. Mirahmadi *et al*. (2010) applied mild alkaline pre-hydrolysis on spruce and birch wood to extract sugar compounds for the production of both bio-ethanol and bio-gas, while Jiang *et al*. (2017) applied acid and alkali pretreatment on pine wood to enhance the bio-oil yield. Sugarcane bagasse (Boopathy *et al*., 2008) and softwood (Gulbrandsin *et al*., 2017) have been studied for ethanol production.

In another direction, the research work has been concentrated to investigate the effect of alkaline pre-hydrolysis of depithed corn stover on the hemicellulose extraction and the performance of pre-hydrolyzed corn stover in Soda/AQ pulping (Cheng *et al*., 2010). Kautto *et al*. (2010) investigated the pre-hydrolysis of softwood chips prior to kraft pulping and stated that the kappa number, yield and strength, except tear strength of the pulp, were reduced. Giant bamboo was pre-hydrolyzed before kraft and Soda/AQ pulping to produce value-added byproducts and facilitate pulping reactions (Vena *et al*., 2010). Further research on the application of alkaline and acid pre-hydrolysis on wood chips and non-wood resources are credited to Li *et al*. (2010), Helmerius *et al*. (2010), Jahan *et al*. (2014), Cheng *et al*. (2010), Garcia *et al*. (2011) and Liu *et al*. (2011). Ayrilmis *et al*. (2017) investigated the effect of chip size on hemicelluloses extraction and the effect on flakeboard properties.

The importance of chemical feedstock production from lignocellulosic material in conjunction with pulping directed this research to investigate the effect of chip size and alkaline pre-hydrolysis temperature on hornbeam wood to extract the carbohydrate hydrolysate and to determine its effect on soda pulping.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Raw material
2.1. Sirovina

Hornbeam (*Carpinus betulus*) bolt (length 100 cm and diameter 40 cm) was cut from a longer log at a forest harvesting operation yard in Northern Iran and was transferred to Pulp and Paper Research Laboratory, Islamic Azad University, Karaj Branch. First, the bolt was debarked and then converted to narrower long boards using a band saw. The boards were then chipped using a drum chipper (Pallmann Drum Chipper PHT 430X120). The chips were air dried at ambient temperature to reach equilibrium moisture content and were visually classified in three different sizes (Table 1). The selected chips were stored in plastic bags until used.

2.2 Chemical analysis
2.2. Kemijska analiza

Chemical components of untreated and treated chips including cellulose and lignin were measured using the following Tappi test procedures: Powder preparation; T257 cm-20, extractive free powder; T264 cm-07, Lignin; T222 0m-06 (Tappi, 2008), Cellulose content was measured by Kurschner-Hoffer method.

Table 1 Average dimensions of classified chips
Tablica 1. Prosječne dimenzije klasificirane sječke

Dimension *Dimenzija* / **Chip class** *Klasa sječke*	**Length**, **mm** *Duljina*, mm	**Width**, **mm** *Širina*, mm	**Thickness**, **mm** *Debljina*, mm
Small / *mala*	13.74	7.50	2.10
Medium / *srednja*	20.40	9.05	3.14
Large / *velika*	30.86	17.74	4.98

2.3 Alkaline pre-hydrolysis
2.3. Alkalna predhidroliza

Alkaline pre-hydrolysis of chips was performed using 7.5 % (w/w) reagent grade sodium hydroxide (Merck, Germany), 120 minutes time and two temperatures (60 and 90 ˚C). A sample weighing 150 grams (bone dry) was mixed with 1500 milliliter distilled water and 7.5 % (w/w) sodium hydroxide (based on the dry weight of the chips) was added to the mixture in a plastic bag. The bag was sealed and heated in hot water bath at constant temperature with occasional manual shaking to ensure uniformity. At the end of treatment time, the content of the plastic bag was discharged on a screen (200 mesh) and the liquor (filtrate) was collected. This liquor was used to determine residual sodium hydroxide and dissolved hemicelluloses. The per-hydrolyzed chips were divided into two portions: one-third for the determination of chemical composition and the other two-thirds were used for soda pulping experiments.

2.4 Hemicelluloses precipitation
2.4. Količina hemiceluloza

The dissolved hemicelluloses were determined as follow. First, the pH of the filtrate was adjusted to 5 using 4N H_2SO_4 and the solution was kept refrigerated for 24 hours to precipitate the dissolved lignin in the filtrate. Then, after the separation of lignin, 100 milliliters of the hydrolysate was transferred to a 1000 milliliters beaker and 400 milliliters 96 % ethanol was added. The solution was refrigerated for another 24 hours. Thereafter, the solution was centrifuged for 5 minutes at 7500 rpm. The hemicelluloses were separated and dried in an oven set at 40 ˚C for 24 hours. The weight of the hemicelluloses was determined and the percentage based on original wood was calculated.

2.5 Pulping
2.5. Priprema pulpe

Soda pulping was used on per-hydrolyzed chips with and without alkaline pre-hydrolysis. Pulping conditions were kept constant as follow: sodium hydroxide; 20 % based on bone dry weight of chips, cooking temperature and time; 175 °C and 90 minutes after reaching the cooking temperature, wood to liquor ratio: 1:8. Untreated and treated chips were pulped using a 4-liter rotating digester (Ghomes Wood and Paper Equipment Manufacturing Co.). At the end of each pulping time, the content of the cylinder was discharged on a 200 mesh screen, and the spent liquor was collected. The cooked material was defibrated using a 25 cm laboratory single disc refiner (Ghomes Wood and Paper Equipment Manufacturing Co) in three passes and then the pulp was screened using a set of two screens, a 14-mesh screen on top of 200 mesh screens. Material retained on the 14-mesh screen (R14) was considered as reject (shives), and the fibers passed through the 14-mesh screen and retained on the 200-mesh screen (P14-R200) were considered as accept (screened yield), which was added to reject and the total yield was calculated.

The following TAPPI standard test methods were used for pulp and hand sheets evaluation: Kappa number, T236 om-06; Drainage, T227 om-04; Hand sheet preparation, T205 om-06; Tear strength, T414 om-04; and Tensile strength, T494 om-06 (Tappi, 2008).

2.6 Statistical analysis
2.6. Statistička analiza

Analysis of variance (ANOVA) was used for statistical analysis of the data and in case the significant difference between the averages was observed, then Duncan Multiple Range Test was used for grouping the averages.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Alkaline pre-hydrolysis
3.1. Alkalna predhidroliza

The results of the cellulose and lignin measurements of treated and untreated hornbeam chips are summarized in Table 2. Each value in Table 2 is the average of three measurements.

Both cellulose and lignin content of treated hornbeam chips is higher than that of the untreated chips, which is due to the removal of hemicelluloses by alkaline treatment.

The results of the statistical analysis on the effect of pre-hydrolysis temperature and chip size on the measured properties are shown in Table 3. The influence of treatment temperature on residual alkali, cellulose content of treated chips and hemicelluloses removal were not statistically significant but the effect of temperature on lignin content of the pre-hydrolyzed chips was statistically significant at 99 % confidence level. However, the effect of chip size on residual alkali was statistically significant at 95 % confidence level and the effect of chip size on cellulose, lignin and hemicelluloses removal was significant at 99 %confidence level. The interactive effect of both variables on residual alkali was not

Table 2 The average cellulose and lignin content of treated and untreated hornbeam chips
Tablica 2. Prosječni sadržaj celuloze i lignina u tretiranoj i netretiranoj sječki grabovine

Variables / *Varijable* \ **Properties** / *Svojstva*		**Cellulose** *Celuloza*	**Lignin** *Lignin*
Temperature, °C *Temperatura*, °C	**Chip size** *Veličina sječke*	%	%
60	Small / *mala*	52.5	19.0
	Medium / *srednja*	55.5	19.5
	Large / *velika*	48.0	15.0
90	Small / *mala*	53.5	18.5
	Medium / *srednja*	53.0	19.0
	Large / *velika*	52.5	20.5

significant and the effect on cellulose, lignin and hemicelluloses removal was statistically significant at 99 % and 95% significance level, respectively (Table 3).

The interactive effect of chip size and pre-hydrolysis temperature on hemicelluloses removal is illustrated in Figure 1. The amount of hemicelluloses removed varied between the lowest value of 2.98 % and the highest value of 7.14 % of the original weight of the wood. Higher amount of hemicelluloses were removed from larger chips, which indicates the penetration of the alkali deep into the chips and removal of more hemicelluloses. The effectiveness of the sodium hydroxide in dissolution and hydrolysis of hemicelluloses is higher at higher treatment temperature, which relates to more severe condition.

The results of hornbeam wood alkaline pre-hydrolysis indicate the potential of this treatment to dissolve and remove the hemicelluloses (Garcia *et al.*, 2011; Helmerius *et al*., 2010; Li *et al*., 2011; Ormsby *et al*., 2012; Carvalherio *et al*., 2008; Liu *et al*., 2018; Lu *et al*., 2017; Song *et al*., 2011, Vena *et al*., 2010). Alkaline pretreatment breaks the lignin-carbohydrate bonds and dissolves the hemicelluloses. Figure 2 schematically shows the location of hemicelluloses in cell wall structure and possible lignin-carbohydrate bonding. These hemicelluloses can be a source of carbohydrate as feedstock for bio-ethanol production in biorefinary concept. As shown in Figure 3, the sodium hydroxide charged on the chips is not totally absorbed by the wood and between 2 to 3.8 % remained indicating the sufficiency of the alkali charge for the treatment condition applied. The absorption of the sodium hydroxide at lower tempera-

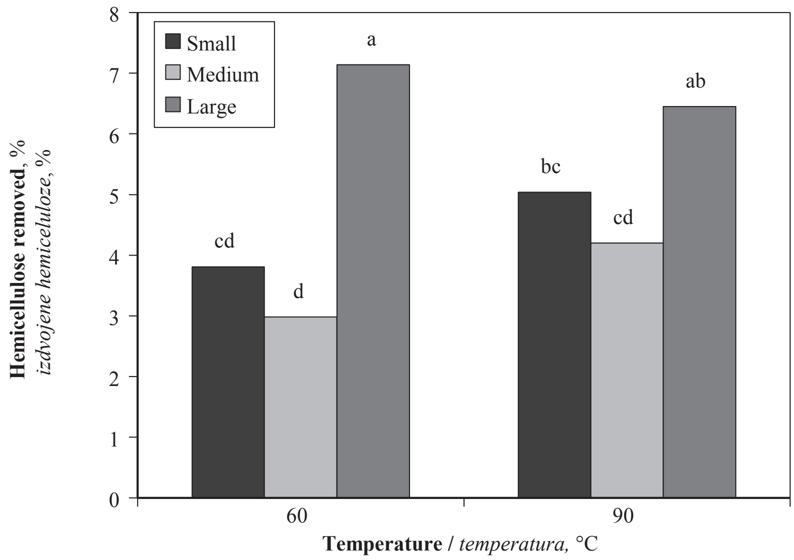

Figure 1 The influence of hornbeam chips alkaline pre-hydrolysis variables on hemicelluloses removal (Lower case letters on the bars show the Duncan grouping of the averages)
Slika 1. Utjecaj varijabli alkalne predhidrolize sječke grabovine na izdvojene hemiceluloze (mala slova iznad stupaca prikazuju Duncanovo grupiranje prosjeka)

Table 3 Analysis of variance of the effect of experimental variables on chemical components of pre-hydrolyzed hornbeam chips, hemicelluloses removal (*F* value and significance level)
Tablica 3. Analiza varijance utjecaja istraživanih varijabli na kemijski sastav predhidrolizirane sječke grabovine i izdvojene hemiceluloze (*F*-vrijednost i razina značajnosti)

Properties / *Svojstva* \ **Variables** / *Varijable*	**Temperature** *Temperatura*	**Chip size** *Veličina sječke*	**Chip size × Temperature** *Veličina sječke × temperatura*
Residual alkali / *alkalni ostatak*	3.612^{ns}	4.769*	0.2525^{ns}
Cellulose / *celuloza*	1.956^{ns}	61.839**	16.653**
Lignin / *lignin*	10.560**	3.956*	17.869**
Hemicelluloses removal / *izdvojene hemiceluloze*	1.163^{ns}	19.879**	4.067*

Significance level: **,99 %; *,95 %; ns, not significant / *Razina značajnosti: **,99 %; *,95 %; ns, nije značajno*

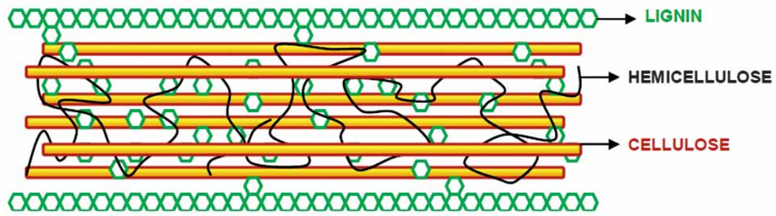

Figure 2 Schematic presentation of cell wall structure showing the bonding between lignin and carbohydrates (Mussatto and Teixeira, 2010)
Slika 2. Shematski prikaz strukture stanične stijenke koji prikazuje vezu između lignina i ugljikohidrata (Mussatto i Teixeira, 2010.)

ture was better than at higher temperature. Even though less sodium hydroxide is absorbed at higher temperature, more hemicelluloses is removed, which shows the effectiveness of chip size on penetration of alkali.

The cellulose content of the treated chips was higher than that of untreated chips, because of the hemicelluloses removal. Otherwise the charged sodium hydroxide is not strong enough to degrade and dissolve the cellulose (Travaini *et al*., 2016). The lignin content of the treated chips is marginally (about 2.5 % of the original content of 18 %) increased as well, which is also due to hemicelluloses removal and partial lignin hydrolysis (Travaini *et al*., 2016).

3.1 Pulp properties
3.1. Svojstva pulpe

The usual pulping process for hardwoods is kraft pulping, which provides fast reactions between pulping chemicals and lignin. Soda pulping reactions compared to kraft pulping is slow and pulping time is longer. However, in this study, soda pulping was selected to demonstrate the impact of pre-hydrolysis on pulping performance. The results of pulp properties measurements showed that chip prehydrolysis of chips facilitated the penetration of pulping chemicals and caused faster pulping reactions and lignin removal (Kautto *et al*., 2010).

The results of the statistical analysis of the effect of prehyloysis temperature and chip size on soda pulp properties are summarized in Table 4. The effect of both variables on pulping total yield and the reject are statistically significant at 99 % coincidence level. However, the influence of the temperature on pulp drainage and kappa number is not significant, but the impact of chip size and the interactive effect of two variables were determined to be significant at either 99 % or 95 % confidence level (Table 4).

The total yield of the pulps produced from treated chips varied between the lowest value of 30.41 % using smallest chips and the highest value of 48.14 %. The total yield of pulp produced from untreated chips was 41.93 % (Figure 4). At smaller chip sizes, the total yield is lower, which indicates the effectiveness of the alkaline hydrolysis of the wood components (Vena *et al*., 2010; Jahan *et al*., 2012). Even though the total yield of the pulps does not vary too extensively, the pulping rejects measurement showed interesting results as the consequence of chip treatment. The amount of rejects of the pulps produced from treated chips is very low (0.83 % to 7.31 %) compared to 24.16 % for pulps from untreated chips. There is no statistically significant difference between the averages of the treated chips pulp rejects. The results of pulping yield and rejects measurement show that the chips alkaline prehydrolysis opens the structure of wood, undergoes hydrolysis and dissolves the hemicellulose (Figure 1), which facilitates the penetration of alkali into the wood structure and the removal of lignin. This phenomenon reduces the pulping yield, and the amount of reject (Jahan *et al*., 2012; Vena *et al*., 2010).

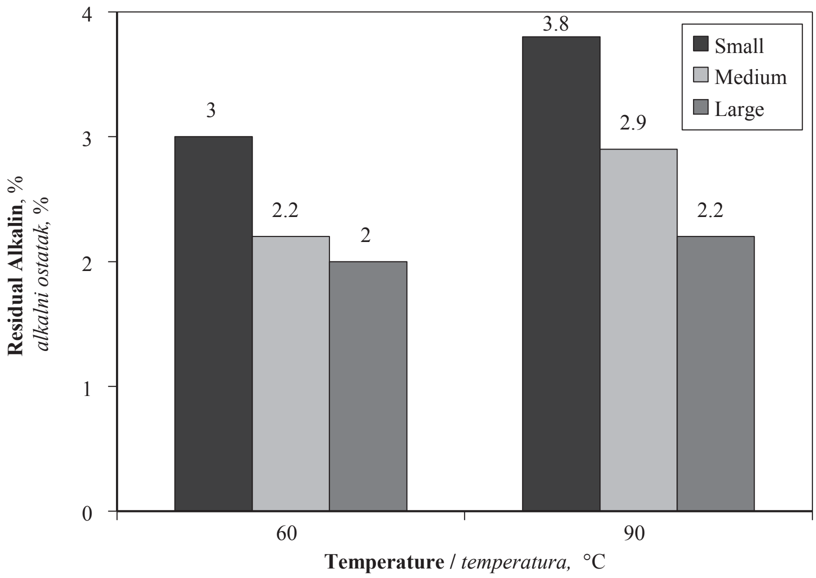

Figure 3 The influence of hornbeam chips alkaline pre-hydrolysis variables on residual sodium hydroxide after treatment
Slika 3. Utjecaj varijabli alkalne predhidrolize sječke grabovine na preostali natrijev hidroksid nakon tretmana

Table 4 Analysis of variance of the effect of experimental variables on properties of soda pulp produced from pre-hydrolyzed hornbeam chips (*F* value and significance level)
Tablica 4. Analiza varijance utjecaja istraživanih varijabli na natronski postupak proizvodnje celuloze iz predhidrolizirane sječke grabovine (*F*-vrijednost i razina značajnosti)

Variables / *Varijable* — **Properties** / *Svojstva*	**Temperature** *Temperatura*	**Chip size** *Veličina sječke*	**Chip size × Temperature** *Veličina sječke × temperatura*
Total Yield / *ukupan prinos*	15.449**	77.519**	85.872**
Reject / *odbačeno*	0.502 ns	51.839**	2.640 ns
Drainage / *drenaža*	0.640 ns	11.360**	0.320 ns
Kappa No. / *Kappa broj*	0.732 ns	3.339*	4.852**
Tensile strength / *vlačna čvrstoća*	0.180 ns	65.498**	11.571**
Tear strength / *čvrstoća na kidanje*	0.001**	0.144 ns	0.217 ns

Significance level: **,99 %; *,95 %; ns, not significant / *Razina značajnosti: **,99 %; *,95 %; ns, nije značajno*

The kappa number of soda pulps prepared from treated chips was lower than that from un-treated chips (control), which indicates faster removal of lignin from treated chips (Jahan *et al*., 2012; Vena *et al*., 2010). The kappa number of pulps from treated chips ranges between the highest value of 34.73 and the lowest value of 28.42 compared to kappa number of control pulp (34.33) (Garcia *et al*.,2011; Vena *et al*., 2010). Of course, the kappa number of the pulp without reject (accept pulp) was measured. However, the results reveal that pre-hydrolysis breaks the lignin-carbohydrate bond and facilitates the lignin dissolution and removal (Travaini *et al*., 2016; Li *et al*., 2010). The measured drainage of the soda pulps was 17 ˚SR compared to 20 ˚SR for pulp from untreated chips. This also shows the potential of hydrophilic hemicelluloses to hold water and their removal causes easy water drainage from the pulp.

The strength properties of soda pulps produced from alkaline pre-hydrolyzed hornbeam chips are measured and the statistical analysis of the data is provided in Table 4. The alkaline pre-treatment of hornbeam chips reduced the tensile index of the soda pulps produced from the treated chips (Figure 5), but the tear index of the pulp was only marginally improved (Figure 6) (Kautto *et al*., 2010; Vena *et al*., 2010; Jahan *et al*., 2012).The effect of chip size and the combined effect of two variables on tensile index was statistically significant at 99 % confidence level. However, the effect of the variable on tear index was not statistically significant.

The presence of low molecular weight hemicelluloses in pulp fibers enhances the bonding potential of fibers. Hemicelluloses absorb more water compared to cellulose and develop more hydrogen bonds between the carbohydrates. The tensile index of paper produced from pulp fibers strongly depends on the intensity of hydrogen bonds. Elimination of hemicelluloses will reduce the number of hydrogen bonds and consequently lower tensile strength of the paper. The removal of hemicelluloses deteriorates the fiber to fiber bonding and the consequence of this phenomenon is also the reduction of paper tensile index (Kautto *et al*., 2010). However, the removal of hemicelluloses from the wood cell wall initiates the fiber contraction followed by the so-called hornification phenomenon after paper drying, which improves the fiber inherent strength and tear index (Jahan *et al*., 2010).

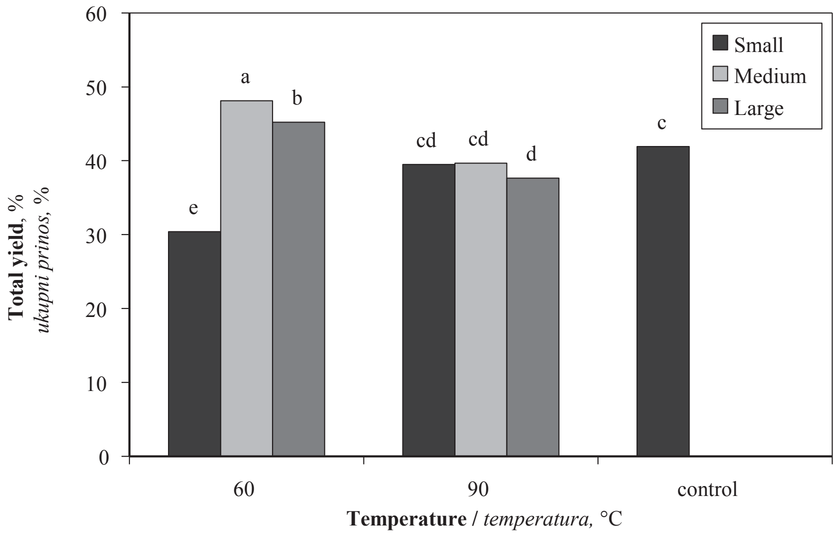

Figure 4 The influence of hornbeam chips alkaline pre-hydrolysis variables on total yield of soda pulps (Lower case letters on the bars show the Duncan grouping of the averages)
Slika 4. Utjecaj varijabli alkalne predhidrolize sječke grabovine na ukupan prinos natronski proizvedene celuloze (mala slova iznad stupaca prikazuju Duncanovo grupiranje prosjeka)

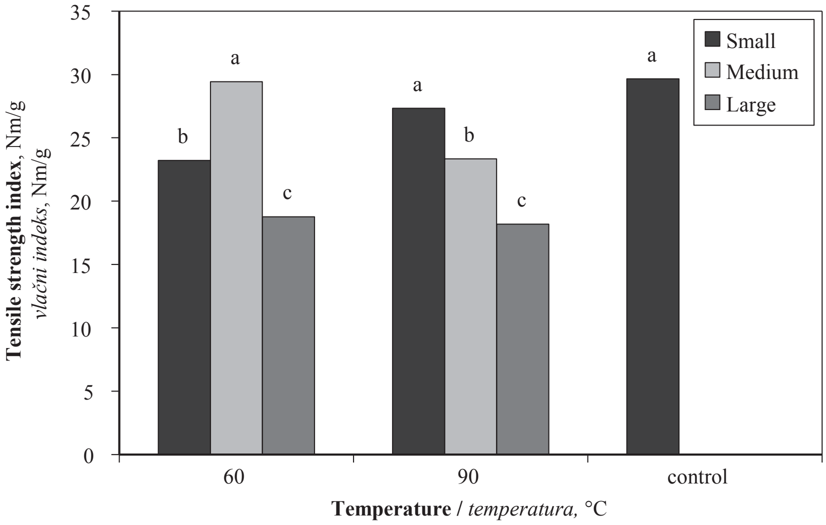

Figure 5 The influence of hornbeam chips alkaline pre-hydrolysis variables on tensile index of soda pulps (Lower case letters on the bars show the Duncan grouping of the averages)
Slika 5. Utjecaj varijabli alkalne predhidrolize sječke grabovinena vlačni indeks natronski proizvedene celuloze (mala slova iznad stupaca prikazuju Duncanovo grupiranje prosjeka)

4 CONCLUSIONS
4. ZAKLJUČAK

Alkaline pre-hydrolysis of hornbeam wood chips can be used to remove up to 7.14 % of the hemicelluloses from hornbeam wood and breaks the lignin carbohydrate bonds. The disruption of the bond opens the structure of the cell wall and facilitates the penetration of pulping liquor. The soda cooking performance of the treated chips is faster than that of untreated chips and the pulping rejects of untreated chips are higher than those of treated chips.

Alkaline pre-hydrolysis and subsequent partial removal of hemicelluloses and disruption of the ligninficarbohydrate linkages facilitates the lignin removal. Therefore, the kappa number of the produced pulp was reduced. The treatment deteriorates the fiber to fiber bonding and reduces the tensile strength index of the soda pulps. However, the tear strength index is preserved and marginally improved, which can be attributed to better delignification as well as sound and longer fibers in the produced pulp.

Further research should be focused on temperature fields of the samples of other wood-based materials (such as chipboard, plywood), samples with wood joints, samples with treated surface, samples with flame retardant treatment, the use of radiant heat, etc.

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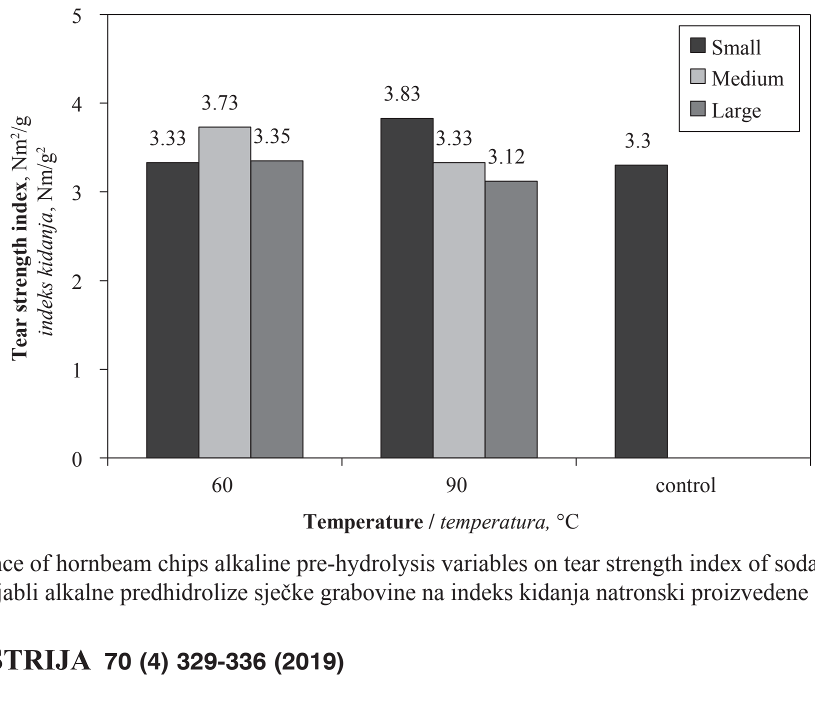

Figure 6 The influence of hornbeam chips alkaline pre-hydrolysis variables on tear strength index of soda pulping
Slika 6. Utjecaj varijabli alkalne predhidrolize sječke grabovine na indeks kidanja natronski proizvedene celuloze

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