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Wood Waste Turned Into Value Added Products: Thermal Plasticization by Benzylation Process

Pretvorba drvnog otpada u proizvod dodane vrijednosti: toplinsko plastificiranje primjenom procesa benzilacije

ABSTRACT • *Sawdust is usually considered as waste in wood-using industries. These materials can be converted into value added thermoplastics by means of benzylation. Products can be utilized in different applications where plastics are used. Thermoplasticization process was carried out with benzyl chloride under different alkaline conditions, 15, 25 and 35 %, respectively. Alkali ions and concentration affect the substitution reaction. Tailored material structure was remarkably changed. In order to detect changes, crystallinity index of the materials and thermal properties were analyzed. In FTIR spectra, the peaks appeared at 698 cm^{-1} and 740 cm^{-1}, which indicates the aromatic C-C angular deformation. Multiple peak appears at 3030 cm^{-1}, which indicates benzylation of the materials. The peak increase can be observed at 1596 cm^{-1} due to the aromatic deformation. Benzyl groups attached to hydroxyl to form ether groups increase the peak intensity. As a result of that, hydrogen bond energy changes and crystallinity of the materials is reduced. This substitution of functional groups changes the decomposition temperature of modified materials. It reduces the decomposition temperature to between 330 and 350 ºC. Thermogravimetric analysis revealed that modified products were characterized by poorer thermal stability compared to raw materials.*

Keywords: thermoplastic resin, hydrogen bond energy, crystallinity, thermal properties

SAŽETAK • *Piljevina se u drvoprerađivačkoj industriji često smatra otpadom. Međutim, ona se postupkom benzilacije može pretvoriti u termoplastični materijal kao proizvod dodane vrijednosti. Tako dobiveni materijal moguće je upotrijebiti za različite namjene za koje se obično rabe plastične mase. Toplinski postupak plastificiranja provodi se benzil kloridom pri različitim lužnatim uvjetima (15, 25 ili 35 %). Koncentracija lužnatih iona utječe na reakciju supstitucije. Struktura materijala značajno se mijenja. Radi otkrivanja promjene, analizirani su indeks kristaliničnosti materijala i toplinska svojstva. U infracrvenom dijelu spektra pikovi su se pojavili na 698 cm^{-1}*

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i 740 cm⁻¹, što upućuje na aromatske C-C kutne deformacije. Višestruki maksimum pojavljuje se pri 3030 cm⁻¹, što označava benzilaciju materijala. Zbog aromatske deformacije porast pika može se primijetiti na 1596 cm⁻¹. Benzilne skupine vezane na hidroksilne skupine tvore eterske skupine i povećavaju intenzitet pika, zbog čega se mijenja energija vodikove veze i kristaliničnost materijala se smanjuje. Taj postupak supstitucije na funkcionalnim skupinama mijenja temperaturu razgradnje modificiranih materijala. Smanjuje se temperatura dekompozicije na 330 i 350 °C. Termogravimetrijska analiza pokazala je da modificirani proizvodi imaju lošiju toplinsku stabilnost nego sirovina od koje su proizvedeni.

Ključne riječi: *termoplastične smole, energija vodikove veze, kristaliničnost, toplinska svojstva*

1 INTRODUCTİON
1. UVOD

Increased utilization of wood results in more waste due to debarking, cutting, shaving, sanding, etc. How to deal with this waste is an important issue. Environmental concern and deficiency in natural resources impose solutions that involve reducing waste and recovering and recycling this material to conserve natural resources. Progress has been made in efforts to reduce waste, but additional works need to be done to solve this problem.

Sawdust and wood shavings are produced by a number of sectors and this raw material is part of the municipal waste stream. A tremendous range of products can be obtained from these lignocellulosic waste materials due to complexity of cell wall structure. Lignocellulosic materials refer to woody and nonwoody plants that are composed of cellulose, hemicellulose, and lignin. Chemical properties of these materials make them suitable for a large number of products. The utilization of lignocellulosic materials in the production of plastic and composites is becoming more and more attractive (Biswas *et al.,* 2006; Bodirlau *et al.,* 2008; Çöpür *et al.,* 2007; Ebringerova and Heinze, 2000; Hassan *et al.,* 2001; Hon and Luis, 1989; Hon and Ou, 1989; Pereira *et al.,*1997; Rowell, 1990; Su *et al.,* 2015).

Plastics are being favored for many applications in place of other materials due to formability, light weight and strength properties (Hon and Shiraishi, 2001). In order to plasticize, lignocelullosic materials are chemically modified. Chemical modification of cell wall polymers is usually carried out either in acidic or alkali conditions (Chen *et al.,* 2012; Huang *et al.,* 2014; Qu *et al.,* 2014). Chemical alteration of hydroxyl groups can reduce the interaction among the chain segments, creates free volume and changes the thermal properties of the material (Nakano, 1994). New modified lignocellulosic materials have thermoplasticity, when large or many side-chains are introduced by chemical modification.

Thermoplastic materials are usually produced from petrochemicals, which is dependent on limited source of fossil fuels whose price is fluctuating and may be running out. In addition to that, fossil fuels release gases which cause the greenhouse effect. One solution is to use tailored renewable resources. Therefore, the objectives of this paper were to explore the chemical modification of Turkish red pine sawdust *via* etherification to impart thermoplasticity. Thermoplasticity was achieved by benzylation process, and characterization of the material was accomplished with crystallinity index and thermogravimetric analysis.

2 MATERIAL AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

Turkish red pine (TRP) sawdust were obtained from the Isparta region in Turkey. Materials were dried under room temperature. Meal samples were prepared using a Wiley mill and ground to pass various mesh screens (60-80 mesh).

2.2 Methods
2.2. Metode

2.2.1 Alkali treatment
2.2.1. Lužnata obrada

Sawdust was treated with different concentrations of sodium, lithium, potassium hydroxide and guanidine for 24 hrs at room temperature. After treatment, materials were rinsed and dried at 102±3 ºC and tested with Fourier transform infrared spectroscopy (FTIR).

2.2.2 Benzylation process
2.2.2. Proces benzilacije

Benzylation process was used similarly to Hon and Ou process. Dried and ground samples (3 grams) were pretreated with various alkali concentrations (15, 25, 35 %) for preswelling. The slurry was transferred to a 250 ml round bottom flask containing benzyl chloride (BC) (27.6 ml). The reaction was conducted at 110 ºC for 6 hours with continuous stirring. The crude benzylated material was collected by filtration and exhaustively washed with water to remove any residuals and finally washed with ethanol to remove any residual benzyl alcohol. The final product was dried overnight at 40 ºC.

2.2.3 Fourier transform infrared spectroscopy (FTIR)
2.2.3. Fourierova transformacija infracrvene spektroskopije

FTIR spectra were obtained on untreated and benzylated substrates from KBr pellet using Perkin Elmer spectrum one model FTIR spectrometer. Each sample was scanned 10 times between 4000 and 400 cm⁻¹ and changes in the chemical structure were recorded.

2.2.4 Thermal properties
2.2.4. Toplinska svojstva

Perkin Elmer Diamond model thermogravimetric analyzer (TGA) was used to study the thermal properties of etherified samples. The heating rate was set at 10 ºC/min and the temperature ranged from 25 to 600 ºC. Measurements on 4 mg samples were carried out under nitrogen atmosphere (100 ml/min).

2 RESULTS AND DISCUSSION
2. REZULTATI I RASPRAVA

The degree of crystallinity is an important parameter for plasticization of lignocellulosic materials. Cell wall polymers contain significant amounts of hydroxyl groups. Chemical modification of lignocellulosic materials usually takes place between these hydroxyl groups of the cell wall polymers and chemical reagent with or without catalyst (Rowell, 1990). Tailored hydroxyl groups can help lignocellulosic materials to exhibit thermoplastic properties due to change in hydrogen bonding ability.

Introducing new chemicals to cell wall polymers causes breaking up of some hydrogen bonds and changes crystallinity. This change indicates the possibility of plasticization and can be detected with FTIR spectroscopy. In order to detect the change in crystallinity, different peak ratios and hydrogen bond energy can be used (Poletto *et al.*, 2013; Akgul *et al.,* 2007). The ratio between the heights of the bands at 1370 cm^{-1} and 2900 cm^{-1} was used to determine the total crystalline index (TCI), and the ratio between the areas of the bands at 1430 cm^{-1} and 896 cm^{-1} was used to determine lateral order index (LOI) of the materials (Akerholm, *et al.*, 2004; Ma, 2007). The energy of the hydrogen bonds *EH* for the OH stretching band was calculated using equation 1:

$$EH = \frac{1}{K} \cdot \left[\frac{(v_o - v)}{v_o} \right] \qquad (1)$$

Where v_o is the standard frequency corresponding to free OH groups (3650 cm^{-1}), v is the frequency
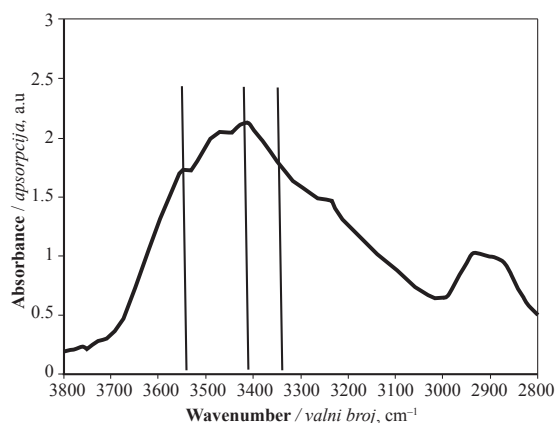

Figure 1 Assignment of hydroxyl bands in FTIR spectra for untreated Turkish red pine
Slika 1. Raspored hidroksilnih skupina na infracrvenom dijelu spektra za netretiranu piljevinu od drva turskoga crvenog bora

of the bonded OH groups, and *K* is a constant (1/ *K* = 2.625 · 10^2 kJ).

In order to increase reactivity and swell lignocellulosic materials before modification, the material was treated with alkaline solution. Alkaline treatments contracts microfibrils in the cell wall and changes the dimension (Nakano, 2010). The mechanism of microfibril contraction and anisotropic dimensional changes of cells in wood are treated with aqueous NaOH solution. As a result, chemical reagent can reach and react with hydroxyl groups on cell wall polymers. In untreated materials, there are free, intra and inter hydrogen bonded hydroxyl groups available (Figure 1). The intramolecular hydrogen bond in lignin can be observed at 3560-3580 cm^{-1} (Kondo, 1997; Poletto *et al.,* 2012). Intramolecular hydrogen bond in cellulose appears around 3430 cm^{-1} and 3340 cm^{-1} (Kondo, 1997; Poletto *et al.,* 2014). After alkaline treatment, these hydroxyl groups affected crystalline structure changes. Table 1 shows the crystallinity and hydrogen bond energy change after alkaline treatment (Table 1).

There is a small difference in crystallinity index and hydrogen bond energy (EH) of the materials. TCI

Table 1 Crystallinity of alkaline treated materials of Turkish red pine (TRP)
Tablica 1. Kristaliničnost materijala piljevine turskog crvenog bora (TPR) tretiranog lužinama

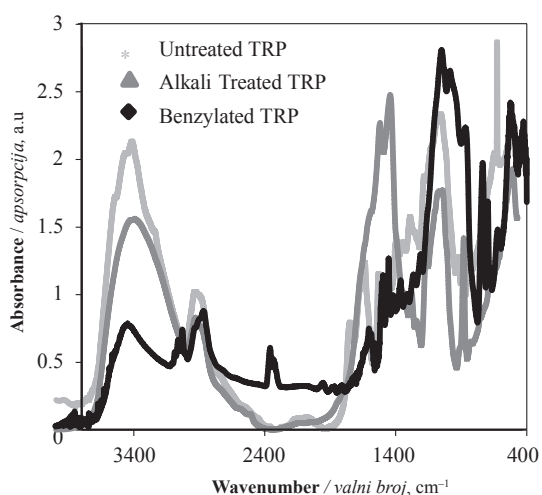|	**Crystallinity of raw materials** / *Kristaliničnost sirovine*					
	TCI	LOI	EH (kJ)	δυ (cm^{-1}) 2	δυ (cm^{-1}) 3	δυ (cm^{-1}) 6
Untreated Turkish red pine	0.43	0.36	15.75	2.80	2.79	2.77
15 % NaOH TRP sawdust	7.99	2.18	13.59	2.80	2.78	2.77
25 % NaOH TRP sawdust	5.56	2.81	12.87	2.80	2.78	2.77
35 % NaOH TRP sawdust	5.04	21.63	14.38	2.80	2.78	2.77
15 % KOH TRP sawdust	1.50	9.43	16.54	2.80	2.78	2.77
25 % KOH TRP sawdust	2.07	2.73	17.69	2.80	2.78	2.77
35 % KOH TRP sawdust	2.24	10.74	17.04	2.80	2.78	2.77
15 % LiOH TRP sawdust	3.61	1.17	16.54	2.80	2.78	2.77
25 % LiOH TRP sawdust	1.96	1.37	17.98	2.80	2.78	2.77
35 % LiOH TRP sawdust	2.14	1.44	20.35	2.80	2.78	2.77
15 % Guanidine TRP sawdust	3.60	20.20	16.54	2.80	2.78	2.77
25 % Guanidine TRP sawdust	4.70	0.25	17.40	2.80	2.78	2.77
35 % Guanidine TRP sawdust	0.89	4.05	16.54	2.80	2.78	2.77

Figure 2 FTIR spectra of untreated and chemically treated TRP sawdust of Turkish red pine (TRP)
Slika 2. FTIR spektar netretirane i kemijski tretirane piljevine od drva turskoga crvenog bora

and LOI of the untreated sample were lower than those of alkaline treated materials. In contrast, hydrogen bond energy was high considering the alkaline treated materials. Alkaline treated samples change the crystalline structure and hydrogen bond energy and the degree of this change increases as NaOH < KOH ≤ Guanidine < LiOH at the same concentration. Wood powder mercerization under alkaline conditions and the polymorphic

transition take place under the alkaline solution. As the concentration of alkali, in particular NaOH, increases, TCI decreases. In contrast to that, LOI increased with the NaOH, KOH and LiOH concentration. It is probably due to the transition between cellulose polymorph and the effect of alkali ion (Nakano, 2010; Borysiak and Doczekalska, 2005). The lowest TCI and LOI values were observed on untreated materials, which may indicate that the amorphous region is higher in untreated materials than after alkali treatment when mercerization takes place. Hydrogen bond energy indicates the interaction between the adjacent cellulose chains (Poletto *et al.,* 2012). It increases when cellulose chains approach each other, and the packing density also increases. There was a slight difference among bond distances in alkali treated materials. Hydrogen bond energy increased with alkali concentration.

After alkali treatment, the slurry was transferred to a round bottom flask containing benzyl chloride. The benzylation process proceeded with continuous stirring. Untreated and chemically treated samples show different FTIR spectra (Figure 2). Benzylation of the materials caused a reduction of hydroxyl peaks at 3400 cm^{-1} and an increase of the aromatic C-C axial deformations around 1595 cm^{-1} (Figure 3). In addition to that, aromatic multiple peak appeared at 3030-3100 cm^{-1}. This could be due to monosubstituted benzyl rings. Guanidine showed spectra similar to those of untreated materials.

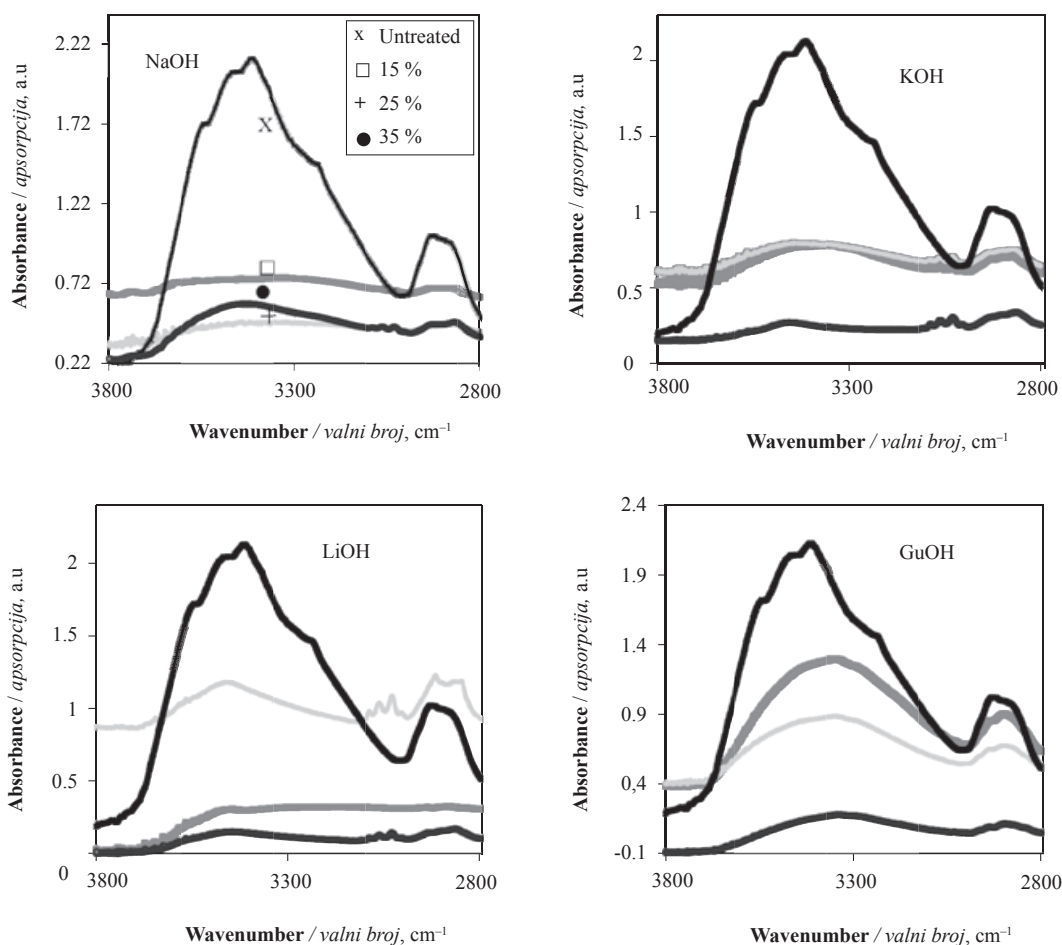**Figure 3** Aromatic stretch at 3030 - 3050 cm^{-1} due to monosubstituted benzyl rings
Slika 3. Aromatski dio na 3030 – 3050 cm^{-1} zbog monosupstitucije benzilskih prstenova
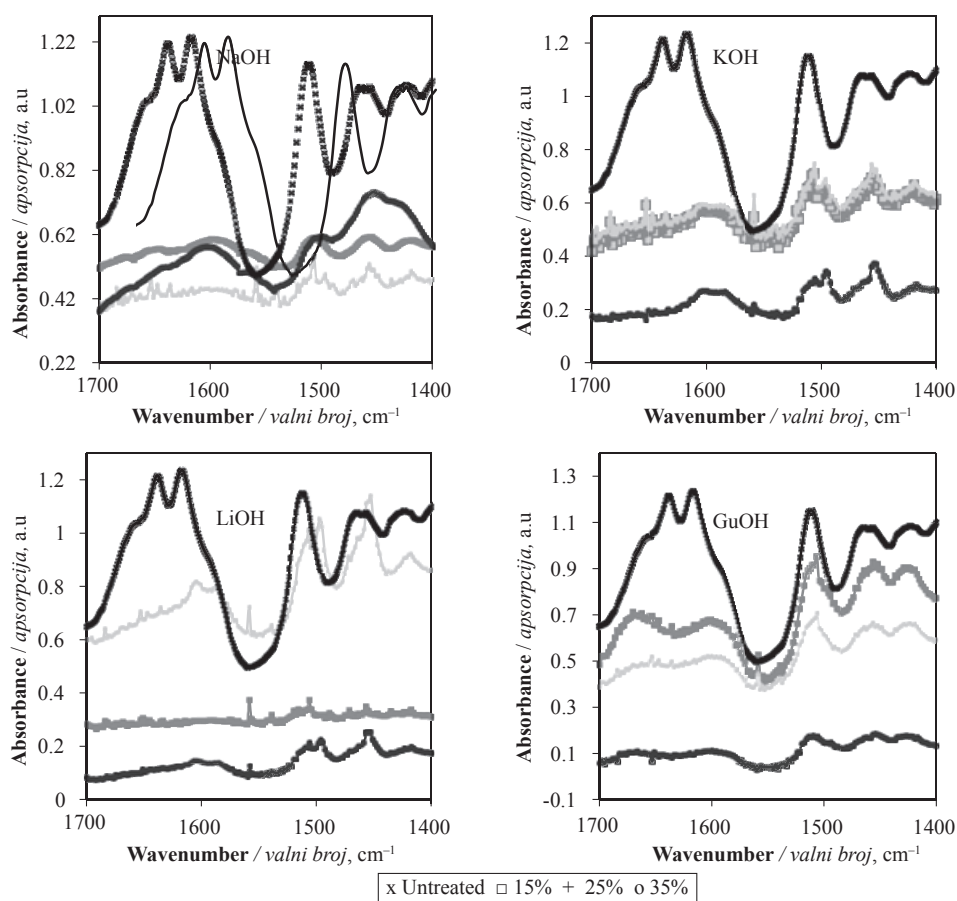
x Untreated □ 15% + 25% o 35%

Figure 4 Increase of aromatic C-C axial deformations around 1595 cm^{-1} due to benyzlation
Slika 4. Porast aromatske C-C aksijalne deformacije oko 1595 cm^{-1} zbog benzilacije
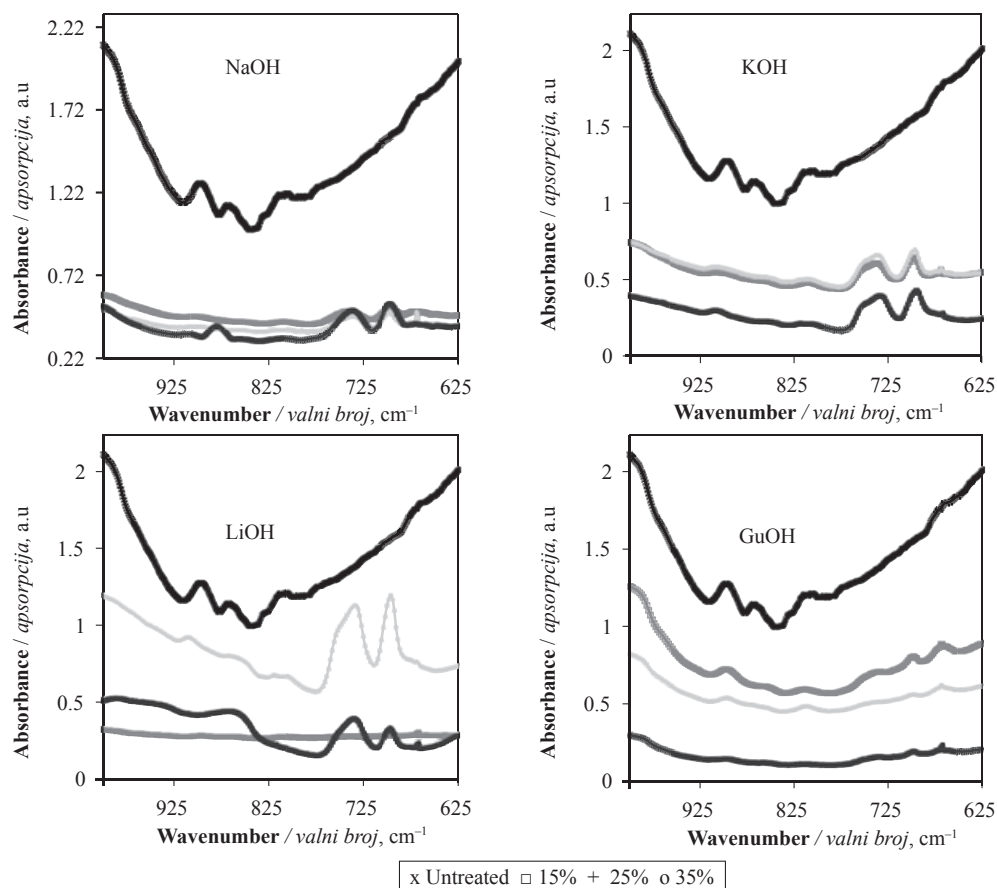

x Untreated □ 15% + 25% o 35%

Figure 5 Appearance of bands at 698 cm^{-1}, 740 cm^{-1}
Slika 5. Pojava skupina na 698 cm^{-1}, 740 cm^{-1}
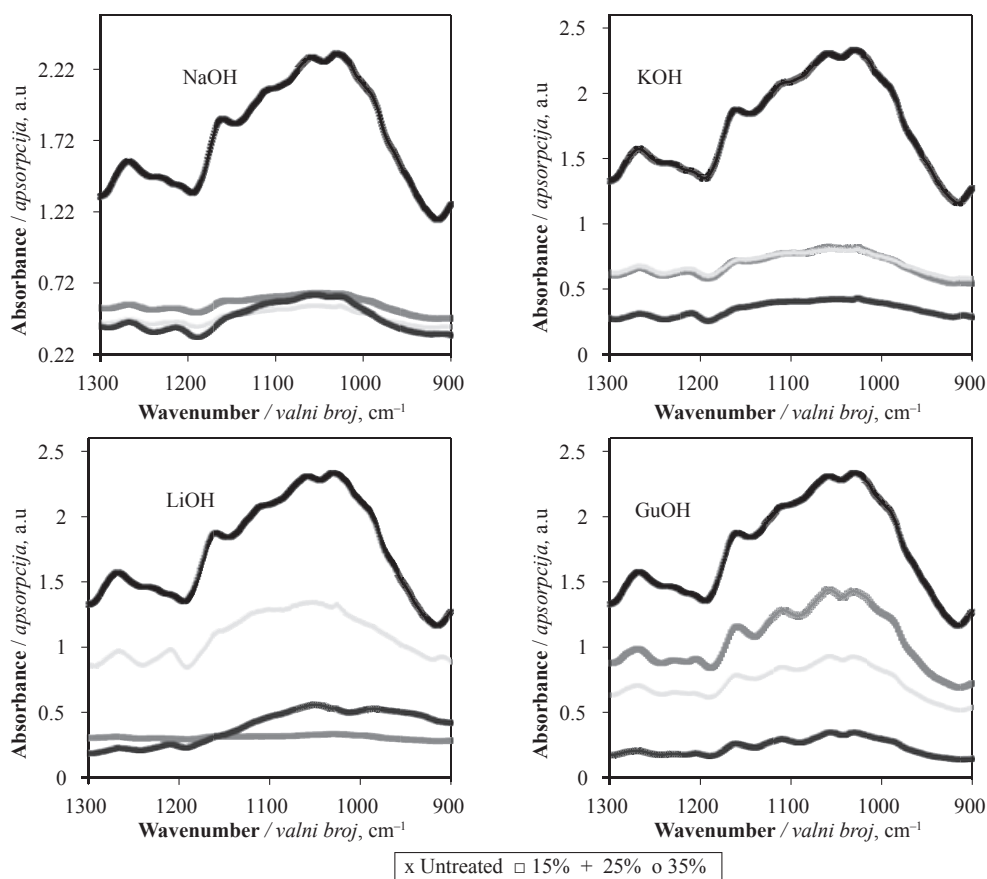
x Untreated □ 15% + 25% o 35%

Figure 6 C-O stretch of the substituted benzyl ether
Slika 6. C-O dio etera koji je supstituiran benzilom

The peak at 670 cm^{-1} gives information on C–OH out of plane bending (Ma, 2007). Spectra of the samples showed a reduction of peak intensity at 670 cm^{-1}. In contrast, peak appears at 698 cm^{-1} due to aromatic C-C angular deformation in treated materials (Figure 5). In addition to that, benzyl group can be detected at 740 cm^{-1}. The guanidine peak is lower compared to others. Even though it is strong alkali, the size of the molecule may restrict the entrance into the cell wall. After the reaction, an increase of aryl ether band at 1265 cm^{-1} and of alkyl ether band at 1155-1060 cm^{-1} (due to C-O stretch from the substituted benzyl ether) was observed. (Figure 5). This absorbance band implied the substitution of hydroxyl groups in materials with benzyl groups.

Cellulose is the main component in wooden materials and it controls the thermal properties of wood (Hon and Shiraishi, 2001). Hydrogen bonds between cellulose chain and in the chain shift to melting temperature over decomposition temperature. Modification of wood helped to disrupt any hydrogen bonds between adjacent chains and in the chain. Therefore, it helps wood to become thermoformable.

Figure 7 shows differential thermogravimetric analysis (DTGA) curves of alkali treated with different alkali ions, concentration and untreated materials. The first step of weight loss, from room temperature to 120 °C, was related to the evaporation of absorbed water. The second step ends near 410 °C, and can be described by an abrupt mass loss stage that was related to the main thermal decomposition process. A derivative

weight loss curve can be used to tell the point at which weight loss is most apparent (Figure 8). According to the results obtained, raw materials begin to decompose at a higher temperature (T_i = 380 °C) when compared to modified materials (T_i = 330-340 °C).

It was seen that there was a significant difference between modified and untreated materials in relation to their respective thermal decomposition temperatures, which may be related to the difference in crystallinity between untreated and modified materials. Tailored hydroxyl groups change the crystalline structure of the materials, when the intra and intermolecular hydrogen bonds between chains are broken. Rearrangement of the bonding ability of the treated materials reduces the decomposition temperature. As a result, the crystalline domains in the untreated materials show a higher thermal stability, when compared to modified materials.

4 CONCLUSION
4. ZAKLJUČAK

Sawdust is a very promising source of raw materials for commercial products such as cellulose, plastics, etc. for many countries due to its availability and abundance. However, it is considered as waste. Thermoplasticization can be achieved by benzylation. Alkaline species and alkali concentration affect the substitution reaction and change the material properties. These changes are significant with NaOH, and it can be detected with crystallinity, hydrogen bond energy change and thermal analysis. Hydroxyl groups were substituted
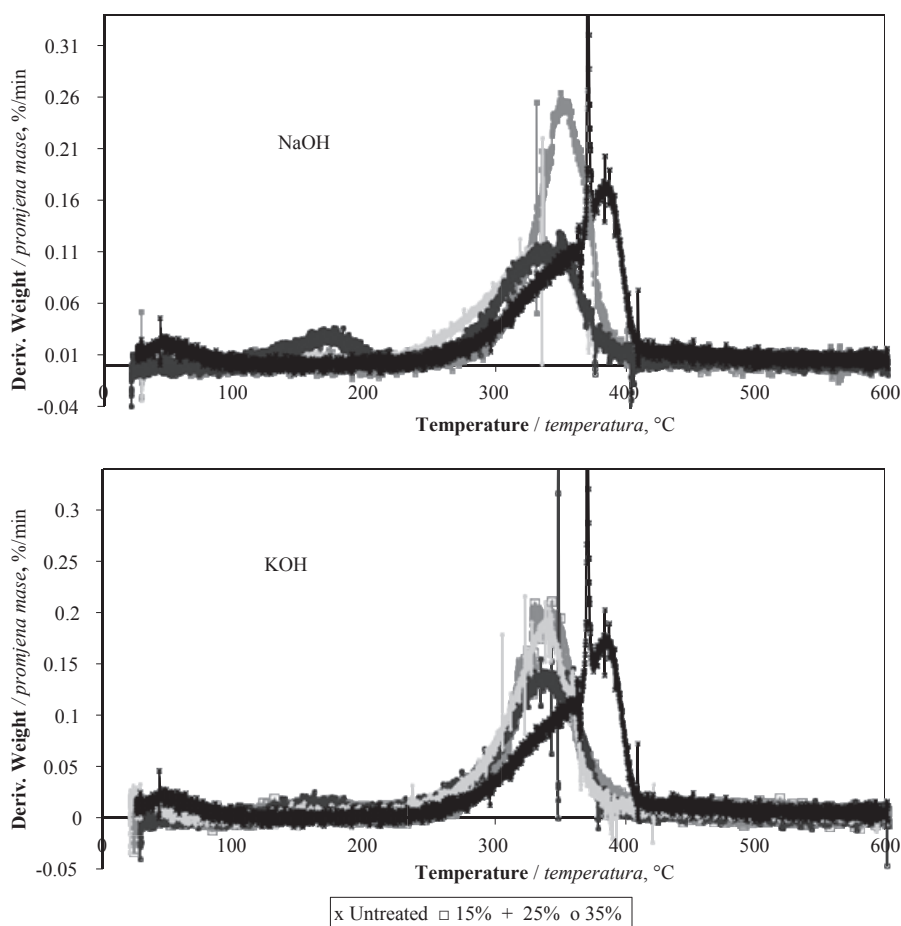
Figure 7 DTGA thermogram of untreated and benzylated wood with different alkali
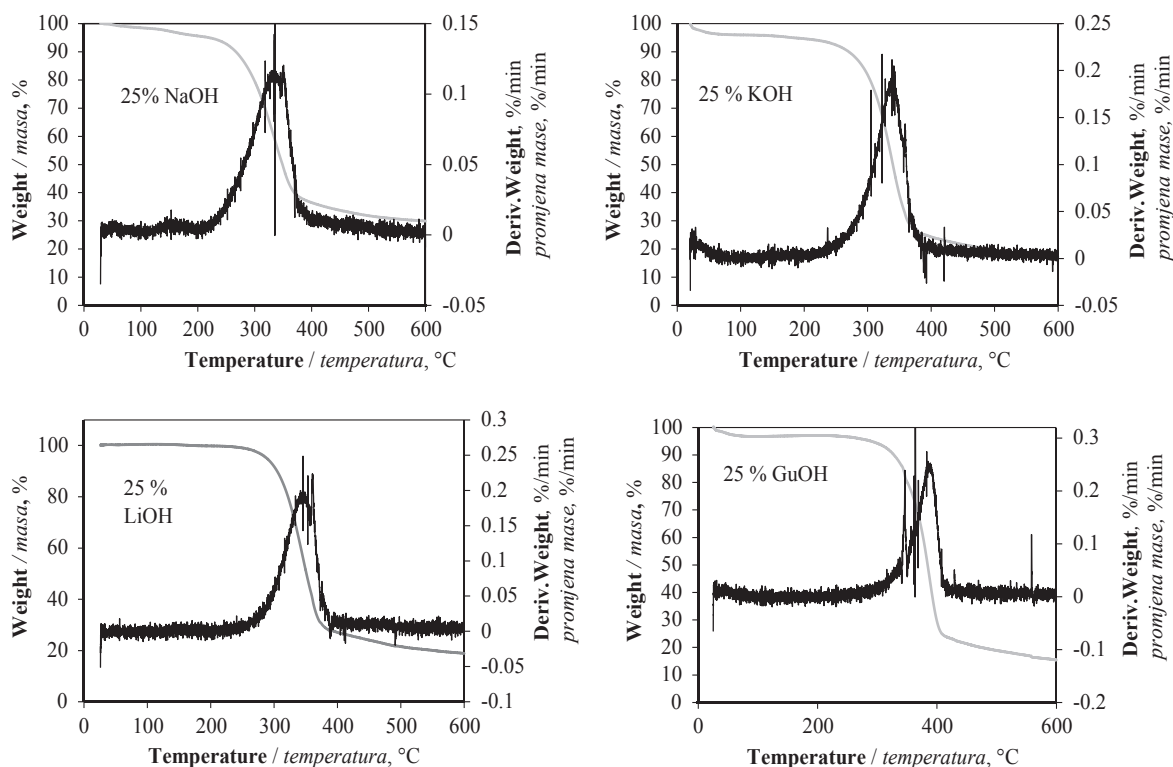**Slika 7**. DTGA termogrami netretiranog drva i drva benziliranoga različitim lužinama

Figure 8 DTG thermograms of benzylated materials under different alkali concentrations
Slika 8. DTG termogrami benziliranog materijala pri različitim koncentracijama lužine

with benzyl groups and this caused the change of FTIR spectra. Guanidine shows different trends. This could be due to the molecule size. It cannot penetrate the pore on the cell wall, because the pore size could be smaller than the molecule size. Na Li and K are effective on thermoplasticization. Multiple peaks appear at around 3030 cm^{-1} indicating benzylation of the materials. The appearance of the peak at 698 cm^{-1} and 740 cm^{-1} indicates the aromatic C-C angular deformation. Thermogravimetric analysis revealed that modified products were characterized by poorer thermal stability compared to raw materials. Deformed crystallinity of the materials reduced significantly the decomposition temperature, and the reduction of the decomposition temperature ranged between 40 to 60 ºC.

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