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Calorific Value and Chemical Properties in Juvenile and Mature Wood of Thermally-Modified *Eucalyptus Grandis*

Kalorijska vrijednost i kemijska svojstva toplinski modificiranoga juvenilnog i zrelog drva eukaliptusa (*Eucalyptus grandis*)

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ABSTRACT • *During thermal modification, timber is exposed to temperatures approaching 200 ºC for several hours and wood properties change. This study was aimed at evaluating the calorific value and chemical properties of juvenile and mature wood of thermally modified Eucalyptus grandis. Boards were taken from 30-year-old E. grandis trees and thermally modified at 180ºC. Samples of the untreated and thermally modified wood were transformed into chips and then processed into sawdust for the energy and chemical characterization of juvenile and mature wood. The results show that the thermal modification causes: (1) significant increases of 3.7 % and 6.8 %, respectively, in the net calorific value of juvenile and mature wood; (2) significant increases of 24.8 % and 47.6 %, respectively, in the extractive content of juvenile and mature wood; (3) significant decreases of 4.2 % and 8.3 % in the respective holocellulose content; and (4) significant decreases in the galactose, xylose and glucose contents. It was demonstrated that the influence of thermal treatment was lower in juvenile wood than in mature wood.*

Key words: *biomass, energy, chemical properties, Eucalypts, thermally-modified wood*

SAŽETAK • *Tijekom postupaka toplinske modifikacije drvo se nekoliko sati izlaže temperaturama do 200 ºC, zbog čega se mijenjaju njegova svojstva. Cilj ove studije bio je vrednovati kalorijsku vrijednost i kemijska svojstva toplinski modificiranoga juvenilnog i zrelog drva eukaliptusa (Eucalyptus grandis). Piljenice su izrađene od tridesetogodišnjih stabala eukaliptusa i toplinski modificirane pri 180 ºC. Za istraživanje energijskih i kemijskih svojstava juvenilnoga i zrelog drva uzorci nemodificiranoga i toplinski modificiranog drva prerađeni su u iverje, a zatim u piljevinu. Rezultati su pokazali da toplinska modifikacija uzrokuje (1) značajno povećanje neto kalorijske vrijednosti juvenilnoga drva od 3,7 % i 6,8 % zrelog drva; (2) značajno povećanje ekstraktivnih tvari od 24,8 % u*

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juvenilnom drvu i 47,6 % u zrelom drvu; (3) značajno smanjenje sadržaja holoceluloze od 4,2 % u juvenilnom drvu i 8,3 % u zrelom drvu te (4) značajno smanjenje sadržaja galaktoze, ksiloze i glukoze. Dokazano je da je utjecaj toplinske modifikacije na energijska i kemijska svojstva juvenilnoga drva manji nego na svojstva zrelog drva.

Ključne riječi: *biomasa, energija, kemijska svojstva, eukaliptus, toplinski modificirano drvo*

1 INTRODUCTION
1. UVOD

During thermal modification, timber is exposed to temperatures approaching 200 ºC for several hours. Several heat treatments are used commercially in Europe. They differ in the temperature applied, the use of nitrogen or steam or oils as an inert fluid, and their application to wet or dry wood (Brito *et al.*, 2008; Calonego *et al.*, 2010, 2012; Esteves *et al.*, 2007; Wikberg and Maunu, 2004).

Studies showed an improvement of equilibrium moisture content and dimensional stability of 61.0 % and 90.0 % in *Eucalyptus globulus* wood after thermal modification at 190-210 ºC (Esteves *et al.*, 2007), and reductions of 21.5 % and 23.2 % in equilibrium moisture content and volumetric swelling in *E. grandis* wood when thermally-modified at 180 ºC (Calonego *et al.*, 2012). The same wood showed significant reductions of 15.7 % in the weight loss after exposure to *Pycnoporus sanguineus* fungi when thermally-modified at 180 ºC (Calonego *et al.*, 2010).

When the thermal treatment temperature is increased from 180 ºC to 220 ºC, an increase occurs in the number of specimens of *E. grandis* with fragile failures of 10.5 % to 100.0 % in compression to parallel to grain, and of 15.8 % to 100.0 % in static bending (Calonego *et al.*, 2012). Temperatures greater than 180 °C caused internal cracks in wood of thermally-modified *E. grandis* (Calonego and Severo, 2010). The thermal treatment of *Eucalyptus saligna* at 180 °C caused a decrease of up to 53.2 %, 62.8 %, 33.8 %, and 35.1 % in the arabinose, galactose, mannose, and xylose contents, respectively, whereas there was a proportional increase of up to 32.5 % in the lignin content of wood (Brito *et al.*, 2008).

Untreated *Pinus pinaster* wood and thermally modified wood at 260 ºC for 0.5 h, 1.0 h, 2.0 h and 4.0 h showed lignin contents of 28 %, 41 %, 51 %, 54 % and 84 %, respectively. The respective gross calorific values were 17932.1, 21076.4, 22164.9, 22181.7 and 25845.1 kJ/kg (Bourgois and Guyonnet, 1988).

Felfti *et al*. (2005) studied briquette of thermally modified wood between 220 ºC and 270 ºC during 0.5 to 1.5 h and concluded that the increase in the temperature and time of the thermal treatment caused degradation of hemicelluloses and improved the calorific value. The gross calorific value was 20020.0 kJ/kg for untreated wood and 21065.1 kJ/kg and 21209.1 kJ/kg, respectively, for thermally modified wood at 220 ºC for 1.5 h and 250 ºC for 0.5 h.

However, the wood is a heterogeneous material and its chemical and physical variations occur due to several factors, e.g. tree species, silviculture, and especially the wood anatomy. Juvenile wood can be defined as being close to the pith and technologically, it differs from mature wood on account of several properties such as: fiber length, density, stability of wood and chemical properties (Bao *et al*., 2001; Calonego *et al*., 2005a, 2014; Severo *et al*., 2012; Zobel and Van Buijtenen 1989).

Moreover, fine residues (wood shavings, sawdust and powder) of wood manufacturing and processing industries have a great potential for generating energy by combustion. According to Quirino *et al*. (2004), the gross calorific value of two hundred fifty-eight species of wood varies from 16039.6 to 22290.5 kJ/kg. Zanuncio *et al*. (2013) concluded that the gross calorific value of *Eucalyptus urophylla* wood varies between 19770.1 and 20017.1 kJ/kg. Calonego *et al*. (2005b) showed that the gross calorific value of *E. grandis* wood was 19897.8 kJ/kg.

Since there is no information about the effects of thermal treatment on calorific value of *E. grandis* wood, the aim of this study was to evaluate the calorific value and chemical properties of juvenile and mature wood of this species after thermal modification.

2 MATERIALS AND METHODS
2. MATERIJAL I METODE

This study utilized wood from 30-year-old *E. grandis* trees from the Forestry Institute of São Paulo located in Manduri, São Paulo, Brazil. Four trees were felled and sectioned into 2.9 m logs. The first log from each tree with diameters between 30 cm and 35 cm (measured at half height) were cut into flat saw boards. The boards that contained the pith were cut into 28-mm thick pieces. Subsequently, all the boards were dried up to 10.0 % moisture content in a dry kiln.

2.1 Thermal treatments of boards
2.1. Toplinska obrada piljenica

Four dried boards were planed to 24-mm thickness and cut into smaller pieces measuring 0.60 m in length. Regions with cracks and knots were discarded. One of these smaller pieces was kept in its original condition (untreated wood), and the other pieces were reserved for the thermal treatment (thermally modified wood).

The material was placed in an electric oven with a programmable controller and thermally modified in the Laboratory of Wood Drying and Preservation of UNESP, Botucatu, SP, in Brazil. The treatment started at an initial temperature of 100 over a period of 14 h and then was increased (1.34 ºC/minutes) up to 180 ºC and maintained over a period of 2.5 h according to the application of the patent developed by Severo and

Calonego (2011). After the end of the thermal treatment, wood pieces were allowed to cool naturally until they reached 30 ºC.

Subsequently, samples of the untreated and thermally modified woods were transformed into chips and then processed into sawdust in a slicer type Willey with 20 mesh sieve size (0.85mm) for the energy and chemical characterization of juvenile and mature wood. The juvenile and mature wood regions were defined according to Oliveira *et al*. (1997). The anatomical characterization of the wood used in this study showed that the juvenile wood is confined up to 80 mm from the pith.

2.2 Calorific value of wood
2.2. Kalorijska vrijednost drva

The untreated and thermally modified wood sawdust were transformed into pellets and placed in an oven at 103±2 ºC, and maintained in this condition until they reached 0 % moisture content.

The pellet was placed in a calorimeter PARR 1201 in the Laboratory of Applied Physics in the Department of Physics and Biophysics, IB - UNESP, Botucatu, SP, in Brazil. The gross calorific value was determined by bomb calorimeter method according to the standards presented in ABNT NBR-8633 (1984). After the combustion, the Equation 1 was used for determining the gross calorific value of the pellets.

$$GCV = \frac{\left(K \cdot W_{\mathrm{W}}\right)}{W_{\mathrm{P}}} \cdot \Delta t \qquad (1)$$

Where:
GCV - gross calorific value, kcal/kg;
W_W - weight of water used in the calorimeter, 2.5 kg;
W_P - oven-dry weight of pellet, kg;
Δt - temperature gradient before and after combustion, ºC;
K - calorimeter constant, 489.

The net calorific value was determined by gross calorific value and equilibrium moisture content of the wood according to the Equation 2 presented in Zanuncio *et al*. (2013).

$$NCV = GCV - (0.0114 \cdot EMC \cdot GCV) \qquad (2)$$

Where:
NCV - net calorific value, kcal/kg;
GCV - gross calorific value, kcal/kg;
EMC - equilibrium moisture content, %.

Subsequently, the gross and net calorific values determined in kcal/kg were mathematically transformed into kJ/kg.

2.3 Chemical properties of wood
2.3. Kemijska svojstva drva

The material used for chemical analysis was classified between 40 and 60 mesh (0.42 and 0.25 mm). The extractives content was determined by extraction sequences with ethanol/toluene 1/2 (v/v), ethanol and hot water (TAPPI T 264 cm-97, 1999). The acid-insoluble Klason lignin (TAPPI T 222 om-98, 1999), and holocellulose contents [holocellulose = 100 − (% Lignin + % Extractives Totals)] were determined in extractive-free wood.

The arabinose, galactose, xylose, mannose and glucose were analysed by High-Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection (HPAE -PAD) in the filtrate obtained of the acid-insoluble Klason lignin. The analysis was carried out by using a chromatograph Thermo-Dionex, ICS-5000 with Pulsed Amperometric Detection, CarboPac PA 1 column and NaOH 0,5M as eluent at a flow rate of 1 mL/min (Sullivan, 1994).

For the evaluation of calorific value and chemical properties, a Kolmogorov-Smirnov's normality test was performed at 5 % significance. All variables had normal distribution. Subsequently, a parametric test (two-way ANOVA) was performed at 5 % significance taking into account the type of wood and the thermal treatment for the comparison of means of calorific value, extractive, lignin, holocellulose and sugar contents of wood. The Jandel SigmaStat version 2.0 was used for statistical analysis.

3 RESULTS AND DISCUSSION
3. REZULTATI I RASPRAVA

3.1 Calorific value of thermally modified wood
3.1. Kalorijska vrijednost toplinski modificiranog drva

The gross calorific values of juvenile and mature wood from untreated *E. grandis* were 19998.3 and 19355.6 kJ/kg (Table 1). These results are similar to those cited by Calonego *et al*. (2005b), Zanuncio *et al*. (2013), and Quirino *et al*. (2004).

Table 1 shows that the gross calorific value of *E. grandis* wood has not been significantly changed by thermal modification at 180 ºC, although the respective treatment promoted a decrease of up to 3.2 % in terms of physical property studied. Similar results were showed by Felfti *et al*. (2005), who concluded that the minimum condition that promotes significant improvements in a wood calorific value was 250 ºC.

Juvenile wood of untreated and thermally modified *E. grandis* wood, at air conditions (20 ºC and 74 % RH), presents equilibrium moisture content of 10.9 % and 9.1%, respectively. In mature wood, the respective moisture contents were 11.1 % and 8.4 %.

Table 1 shows that the thermal treatment at 180 ºC promoted significant reductions of 16.5 % and 24.3 % in the equilibrium moisture content of juvenile and mature woods. The influence of thermal treatment was lower on juvenile wood than on mature wood both in terms of calorific value and moisture content. Similar behavior was reported by Severo *et al*. (2012) and Calonego *et al*. (2014), who studied the changes of some properties of *E. grandis* and *P. elliotti* var. *elliottii* woods during thermal modification. According to the authors, the juvenile wood has an adverse effect on modification.

Due to smaller equilibrium moisture content in the thermally-modified wood of *E. Grandis,* more useful energy is generated through combustion, since the moisture present in certain materials uses part of the gross energy released. Thus the net calorific value is the better way to quantify the energy potential of wood. Table 1

Table 1 Equilibrium moisture content and calorific value of juvenile and mature wood from thermally modified *E. grandis*
Tablica 1. Ravnotežni sadržaj vode i kalorijska vrijednost toplinski modificiranoga juvenilnog i zrelog drva eukaliptusa

Wood *Drvo*	**E.M.C. at air – 20 ºC and 74 % RH** *EMC pri temperaturi zraka 20 ºC i vlažnosti od 74 %* %					**Gross calorific value** *Bruto kalorijska vrijednost* kJ/kg			**Net calorific value** *Neto kalorijska vrijednost* kJ/kg		
	N	U	N	TM	Red. or (Inc.) %	U	TM	(Inc.) %	U	TM	(Inc.) %
Juvenile *juvenilno*	4	10.9 [0.3]	4	9.1 [0.4]	16.5*	19998.3 [802.2]	20278.8 [803.5]	(1.4) NS	17520.5 [753.6]	18169.0 [720.6]	(3.7)*
Mature *zrelo*	4	11.1 [0.5]	4	8.4 [0.4]	24.3*	19355.6 [892.2]	19975.2 [732.3]	(3.2) NS	16911.3 [772.9]	18057.3 [686.6]	(6.8)*
Red. or (Inc.) %		(1.8) NS		7.7 *		3.2 NS	1.5 NS		3.5 NS	0.6 NS	

E.M.C. – Equilibrium moisture content / *ravnotežni sadržaj vode*, Red. – Reduction / *smanjenje*, Inc. – Increase / *povećanje*, N – Repeated number of samples / *broj uzoraka*, U – Untreated wood / *netretirano drvo*, TM – Thermally-modified wood / *toplinski modificirano drvo*, []– standard deviation / *standardna devijacija*, * – Significant difference by F test at 95 % probability / *signifikantna razlika prema F-testu i vjerojatnosti 95 %*, NS – Non-significant difference / *nesignifikantna razlika*.

shows that the thermal treatment at 180 ºC promoted significant increase of 3.7 % and 6.8 %, respectively, in the net calorific value of juvenile and mature woods.

3.2 Chemical properties of thermally modified wood
3.2. Kemijska svojstva toplinski modificiranog drva

The lignin, holocellulose, and extractive contents of juvenile wood from untreated *E. grandis* were 26.12 %, 73.69 %, and 2.06 %, respectively. In mature wood, these components were 28.27 %, 72.77 %, and 2.46 %. These results are presented in Table 2, and are similar to those cited by Bao *et al*. (2001), Brito *et al*. (2008), and Zobel and Van Buijtenen (1989).

Table 3 verifies that the galactose, xylose, and glucose contents in juvenile wood from untreated *E. grandis* were 0.32%, 16.35%, and 57.02%, respectively. In mature wood from untreated *E. grandis*, the respective contents were 0.55%, 14.18%, and 58.02%. These results are similar to those reported by Brito *et al*. (2008) for *E. grandis* wood.

The results of qualitative determination of sugars are also presented in Figure 1 and 2. The peak of

Table 2 Chemical compounds of juvenile and mature woods from thermally modified *E. grandis*
Tablica 2. Kemijska svojstva toplinski modificiranoga juvenilnog i zrelog drva eukaliptusa

Wood *Drvo*	**Extractives content** *Sadržaj ekstrativnih tvari* %					**Insoluble lignin content** *Sadržaj netopljivog lignina* %			**Holoceluloses content** *Sadržaj holoceluloze* %		
	N	U	N	TM	(Inc.) %	U	TM	(Inc.) %	U	TM	Red. %
Juvenile *juvenilno*	4	2.06 [0.21]	4	2.57 [0.59]	(24.8)*	26.12 [0.69]	26.12 [1.15]	0.0NS	73.69 [0.79]	70.61 [1.52]	4.2*
Mature *zrelo*	4	2.46 [0.48]	4	3.63 [0.74]	(47.6)*	28.27 [1.12]	30.41 [1.34]	(7.6) NS	72.77 [0.71]	66.71 [1.43]	8.3*
Red. or (Inc.) %		(19.4)*		(41.3) *		(8.2) *	(16.4)*		1.3 NS	5.5*	

Red. – Reduction / *smanjenje*, Inc. – Increase / *povećanje*, N - Repeated number of samples / *broj uzoraka*, U – Untreated wood / *netretirano drvo*, TM – Thermally-modified wood / *toplinski modificirano drvo*, []– standard deviation / *standardna devijacija*, * - Significant difference by F test at 95 % probability / *signifikantna razlika prema F-testu i vjerojatnosti 95 %*, NS – Non-significant difference / *nesignifikantna razlika*.

Table 3 Sugar content of juvenile and mature wood from thermally modified *E. grandis*
Tablica 3. Sadržaj šećera toplinski modificiranoga juvenilnog i zrelog drva eukaliptusa

Wood *Drvo*	**Galactose content** *Sadržaj galaktoze* %					**Xylose content** *Sadržaj ksiloze* %			**Glucose content** *Sadržaj glukoze* %		
	N	U	N	TM	Red. %	U	TM	Red. %	U	TM	(Inc.) %
Juvenile *juvenilno*	4	0.32 [0.10]	4	0.33 [0.07]	(3.1)NS	16.35 [0.71]	15.49 [0.51]	5.3*	57.02 [1.35]	54.80 [1.15]	3.9*
Mature *zrelo*	4	0.55 [0.06]	4	0.34 [0.11]	38.2*	14.18 [0.62]	12.21 [1.62]	13.9*	58.02 [0.79]	54.16 [1.11]	6.7*
Red. or (Inc.) %		(71.9)*		(3.0) NS		13.3 *	21.2 *		(1.8) NS	1.2 NS	

Red. – Reduction / *smanjenje*, Inc. – Increase / *povećanje*, N – Repeated number of samples / *broj uzoraka*, U – Untreated wood / *netretirano drvo*, TM – Thermally-modified wood / *toplinski modificirano drvo*, []– standard deviation / *standardna devijacija*, * – Significant difference by F test at 95 % probability / *signifikantna razlika prema F-testu i vjerojatnosti 95 %*, NS – Non-significant difference / *nesignifikantna razlika*.

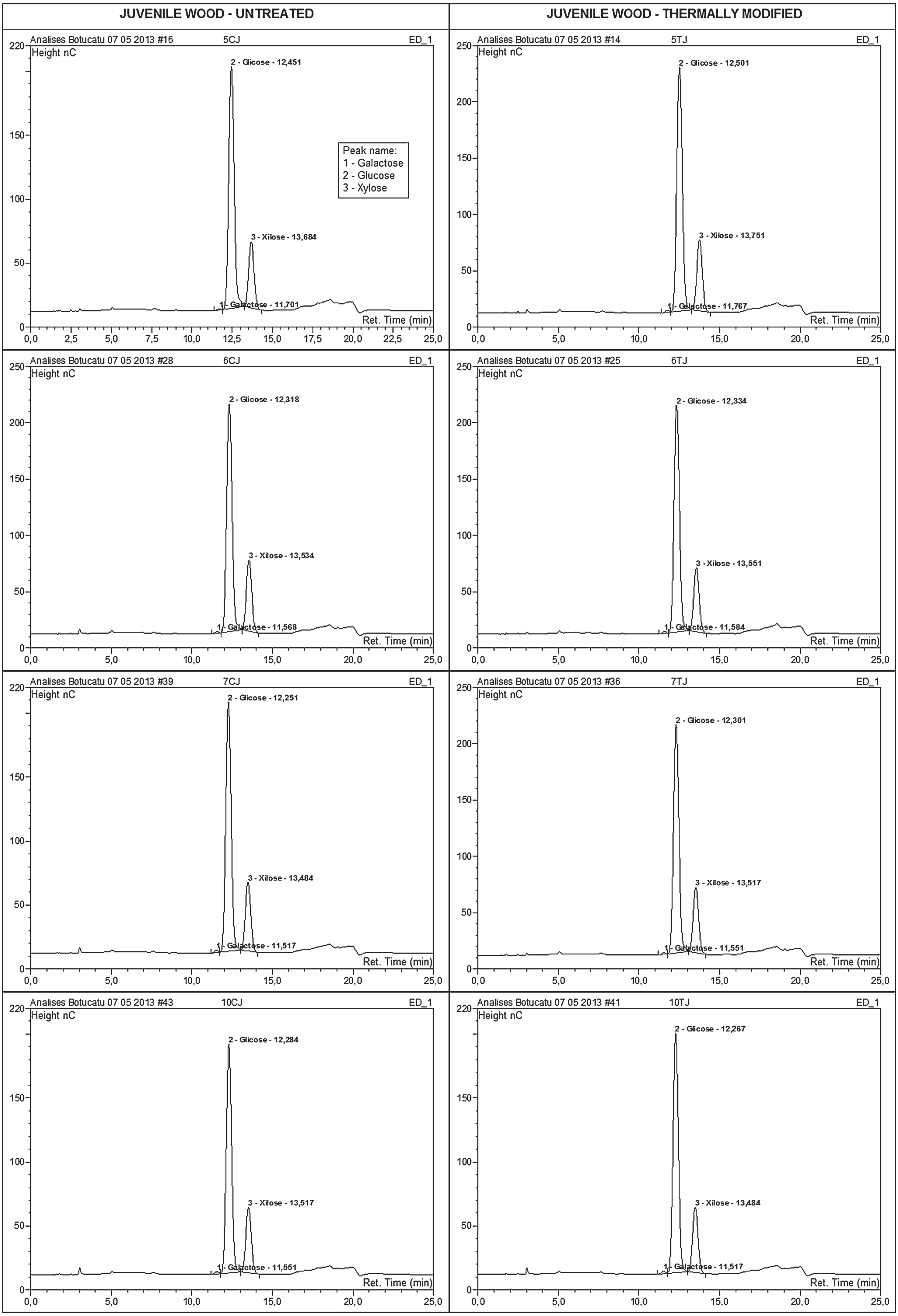

Figure 1 Chromatograms of extracts of juvenile wood from thermally modified *E. grandis*
Slika 1. Kromatogrami ekstraktivnih tvari u toplinski modificiranome juvenilnom drvu eukaliptusa

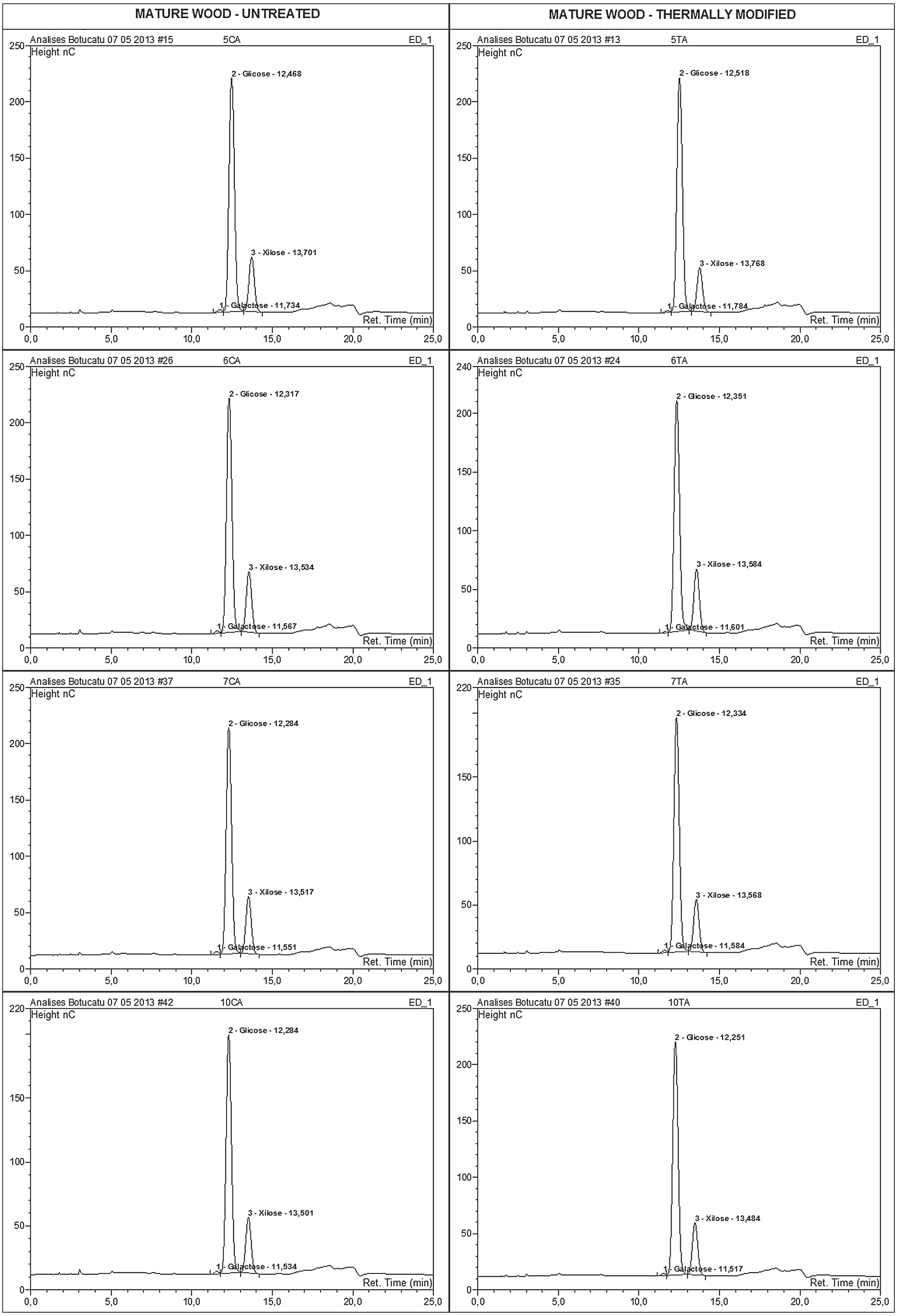

Figure 2 Chromatograms of extracts of mature wood from thermally modified *E. grandis*
Slika 2. Kromatogrami ekstraktivnih tvari u toplinski modificiranome zrelom drvu eukaliptusa

galactose, xylose and glucose by the chromatograms of extracts of untreated and thermally modified wood can be seen.

The effect of thermal treatment on the chemical properties of *E. grandis* wood is shown in detail in Table 2 and 3.

The current study demonstrates that the juvenile and mature wood from thermally modified *E. grandis* presented 2.57 % and 3.63 % in the respective extractive contents when compared with the values found for untreated wood, which were 2.06 % and 2.46 %. Mature wood, when thermally modified, showed a proportional increase of 7.6 % in the insoluble lignin content, whereas juvenile wood showed no change by thermal treatment. The mature wood, when thermally modified, showed greater significant reduction (8.3 %) in holocellulose content than juvenile wood (4.2 %).

The thermal treatment of mature wood of *E. grandis* at 180 °C caused a decrease of 38.2 %, 13.9 %, and 6.7 % in the galactose, xylose, and glucose contents. The influence of thermal treatment in juvenile wood was lower than in mature wood. Similar changes were reported by Bourgois and Guyonnet (1988), Brito *et al*. (2008), Severo *et al*. (2012), Bächle *et al*. (2010) and Wikberg and Maunu (2004), who studied the variations in the chemical properties of other kinds of wood during the thermal modification.

The changes in chemical properties of thermally modified *E. grandis* wood can explain the increase in the calorific value. Similar behavior was reported by Bourgois and Guyonnet (1988), who concluded that the thermal treatment in *Pinus pinaster* wood at 260 ºC during 0.5 to 4 h caused an increase between 46.4 % and 200 % in the lignin contents, and between 17.5 % and 44.1 % in the gross calorific value. These results are similar to those reported by Felfti *et al*. (2005) who concluded that the increase in the temperature and the time of the thermal treatment causes degradation of hemicelluloses and increase the calorific value of wood.

4 CONCLUSIONS
4. ZAKLJUČAK

This study shows that thermal modification of *Eucalyptus grandis* wood increases its net calorific value up to 6.8 %. Regarding chemical properties, significant increase was detected in the extractive content of juvenile and mature wood and reduction in the holoceluloses and sugar contents, when it was submitted to the thermal modification at 180 ºC. Finally, the influence of thermal treatment in juvenile wood was lower than in mature wood.

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