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Chemical Characterization of Lebonan Cedar Tar

Analiza kemijskog sastava katrana od libanonskog cedra

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ABSTRACT • *Chemical characterization of Cedrus libani A. Rich tar, obtained by the traditional method and Jenkner Retort, was studied. Roots and fallen branches were used and analyzed separately. FID-GC and GC-MS were used for the characterization. The tar yield of Jenkner Retort was 60 %, while it is 30-40 % in the traditional method. 41 compounds were identified. β-himachalane (22-28 %) was found to be the main compound in all tars. α-himachalane (6-10%) and longifolene (7-9 %) were the other important compounds. Some compounds, which have antifungal and insecticides effects like deodarone and E-(α)-atlantone, were determined only in tars of roots. Phenolic compounds were not determined by the traditional method.*

Key words: *Cedrus libani, himachalane, pyrolysis, GC-MS, Jenkner Retort*

SAŽETAK • *U radu je prikazana analiza kemijskog sastava katrana od libanonskog cedra (Cedrus libani A. Rich) dobivenoga tradicionalnim postupkom i uz pomoć Jenknerove retorte. Napravljena je zasebna analiza za staro korijenje i za otpale grane libanonskog cedra. Za kemijsku karakterizaciju primijenjene su metode FID-GC i GC-MS. Prinos katrana uz pomoć Jenknerove retorte bio je 60 %, a prinos tradicionalnom metodom iznosio je 30 – 40 %. Pritom je identificiran 41 kemijski spoj. Ustanovljeno je da je β-himakalan (22 – 28 %) glavni spoj svih analiziranih katrana. Drugi po važnosti bili su A-himakalan (6 – 10 %) i longifolen (7 – 9 %). Neki spojevi koji imaju antifungalne i insekticidne učinke kao što su deodaron i E-(α)-atlanton pronađeni su samo u korijenju. U katranu dobivenom tradicionalnim postupkom nisu pronađeni fenolni spojevi.*

Ključne riječi: *libanonski cedar, himakalan, piroliza, GC-MS, Jenknerova retorta*

1 INTRODUCTION
1. UVOD

Pyrolysis is a thermal method converting biomass into gas, liquid and char product in the absence of oxygen. Sort of biomass and thermal conditions affect the yield of these products. Slow pyrolysis (low temperature, long residence time) enhances the charcoal yield, where in fast pyrolysis (moderate temperature, short residence time) liquid-tar yield is high (Bridgwater, 2003; Li *et al.*, 2008; Wang *et al.*, 2009)

Tar is a complex mixture which has a distinctive smoky smell and mainly dark brown color. Generally, *Pinus* sp., *Juniperus* sp., *Fagus* sp., *Betula* sp., *Picea* sp., and *Cedrus* sp. tree species are used for tar production. This liquid product has been used in wood preser-

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vation since ancient times specially in the impregnation of ships and boats (Reunanen *et al.*, 1989; Reunanen *et al.*, 1990; Reunanen *et al.*, 1993).

It was also widely used in veterinary as an antiseptic that is applied to wounds and scratches, treatment for intestinal parasites by adding some drops to drinking water and as repellent against insects and snakes. There are also some applications for the human health care eg. ulcers, dandruff, eczema (Kurt *et al.*, 2008). Despite its distinctive smell and some toxic effect, inhaling of tar helps asthma and, especially in Marrakesh, it is believed to keep away Satan from small babies (Julin, 2008). Tars can be burned as fuel, too (Bridgwater, 2003).

Lebanon Cedar (*Cedrus libani* A. Rich) is a native Mediterranean species with a pleasant odor and valuable wood. In the ancient times, it had a religious meaning and was used as a medicine (Hafızoglu, 1987a). Today there is a legal restriction to cut these trees. Nonetheless, villagers use the fallen branches and old roots to produce cedar tar.

In the last decade, pyrolysis of woody plants has been studied by modern methods from different aspects because of its advantage in storage and variable use (Chiodo *et al.*, 2015; Das and Sarmah, 2015; Hagner *et al.*, 2015; Niu and Liu, 2015; Lij *et al.*, 2015; St Pierre *et al.*, 2015). However, traditional tar production has been used for several millennia. From this point of view, the aim was to compare the tar composition obtained by traditional and modern pyrolysis methods and also to determine the difference between root and branch wood of a tree.

2 MATERIALS AND METHODS
2. MATERIJALI I METODE

2.1 Materials
2.1. Materijali

In this study, Lebanon Cedar (*Cedrus libani* A. Rich), a native species in Turkey, was used for tar production. Due to the legal restrictions of Cedar wood production, fallen branches and old roots were used. Samples were taken at the altitude of 1400 m from Büyüknacar-Pazarcık, Kahramanmaraş, Turkey. The moisture content of branches and roots were 5.83 % and 7.5 %, respectively. Traditionally obtained tar was taken from the same location and parts of tree.

2.2 Method
2.2. Metoda

2.2.1 Sample preparation
2.2.1. Priprema uzoraka

Two different methods, Traditional and Jenkner Retort, were used for tar production in this study. Traditional method, which is in principle a kind of distillation, was applied by the local people. 400-500 kg sample was used in this method. Two holes with different dimensions, depending on the amount of wood material used, were dug nearby and generally the smaller, where tar is collected, was located down-slope from the larger. A pipe was placed between two holes for the tar flow. Inner and upper part of the hole, where ignition occurs, were plastered with mud and clay. If required, small holes were opened on the top to keep the burning process active. For a better tar yield, wood samples were inserted vertically in the hole (Hjulstrom *et al.*, 2006; Kurt *et al.*, 2008). Jenkner Retort was used for the pyrolysis of cedar samples. A cylindrical stainless steel reactor with 12 cm internal diameter and 26 cm height was used. The temperature of retort was measured by Pt-Rh-Pt thermocouple. Branches and roots were applied separately. For each sample, approximately 600 g was placed in the reactor and heated by electric furnace. With the rate of 4 °C/min, temperature raised to max. 400 °C. Bio-char, end-product, was weighted after the experiment and gas yield was calculated by taking the difference (Sutcu *et al.*, 2004). Three repetitions were done for each experiment.

2.2.2 Characterization
2.2.2. Karakterizacija

For the chemical analysis, three different solvents, n-hexane, diethyl ether and dichloromethane, were used for the liquid-liquid extraction. 5 ml tar was extacted with 30 ml solvent and diluted to 100 ml. 1 ml aliquot was taken from the diluted sample and evaporated under the nitrogen before silylation (Ekman and Holmbom, 1989). Silylated samples were analyzed by FID-GC (Flame Ionization Detector – Gas Chromatography) (Shimadzu GC-2010) and GC-MS (Gas Chromatography - Mass Spectrometry) (Shimadzu GCMS-2010 Plus) equipped with TRB-5 type column 30 m x 0.25 mm (0.25 μm film thickness). Helium was used as carrier gas with 1.03 ml/min flow. Temperature program was 60 °C (1 min.) raised to 120 °C with the rate of 4°C/min and held for 10 min. with the same rate raised to 200 °C (held 10 min.) and then to 300 °C (held 10 min.). Detector temperature was 300 °C. Split ratio was 1:10. Three injections were done for each sample.

3 RESULTS AND DISCUSSION
3. REZUTATI I RASPRAVA

3.1 Product yields
3.1. Prinos proizvoda

In the traditional method, the yield was 30-40 % (150 – 200 kg tar/400 – 500 kg wood material), while with Jenkner Retort system it was 60 %. When modern closed systems are used, it is possible to control and apply high temperatures, which affect the yield. The temperature is an important parameter affecting the yield of pyrolysis products (Duman *et al.*, 2011; Ozciftci and Özbay, 2013). Table 1 presents the yield of Jenkner Retort products. As seen, tar is the main product. Tar was obtained at 230 °C from branches and at 300 °C from roots. No significant difference was observed between the yield of roots and branches.

According to Koch P. (1972), with destructive distillation, the yield of char was found as 15-20 % and 6-12 % of tar in southern pine stump wood.

During the process, two different colors of tar were observed. Dark black color was from resinous roots and

Table 1 Yield of Jenkner Retort products, %
Tablica 1. Prinos proizvoda Jenknerovom retortom, %

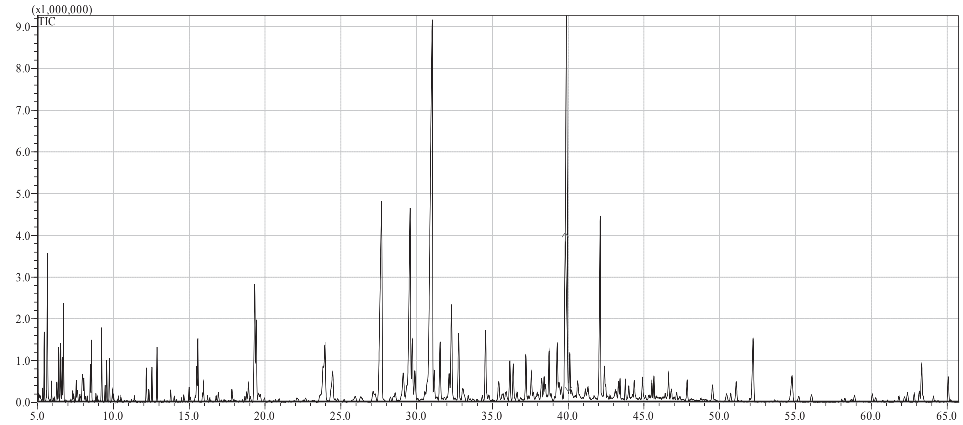
	Gas *Plin*	**Liquid (Tar)** *Tekućina (katran)*	**Char** *Ugljen*
Root / *Korijen*	12.2	62.1	25.6
Branch / *Grane*	11	64.5	24.4

yellow color from branches. Unfortunately, the gas product cannot be used as in the closed Jenkner Retort system.

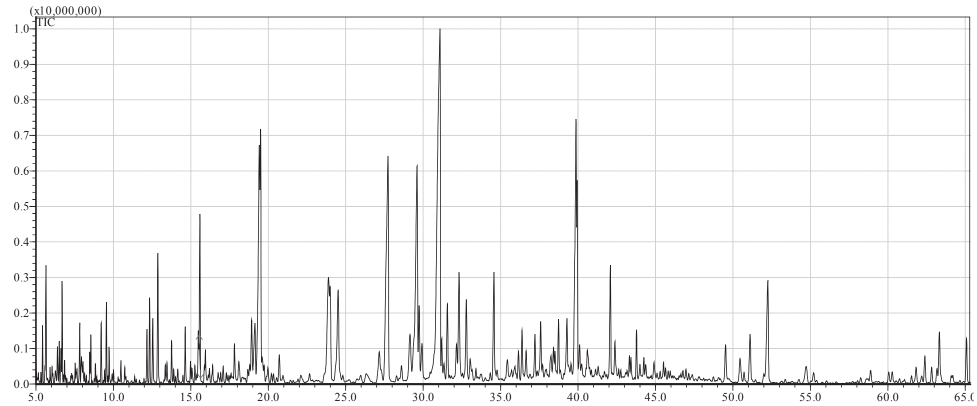
3.2 Analysis of tars
3.2. Analiza katrana

Cedar tar, obtained with different methods and from different parts of wood, was analyzed by GC-MS and FID-GC. Chemical composition of tars is shown in Table 2. Phenols, alcohols, acids, sesquiterpenoids and fatty-resin acids are the constituents of tars with different ratios. Almost 70 % of tar produced by Jenkner Retort was identified; however, this ratio was 50 % in the traditional method.

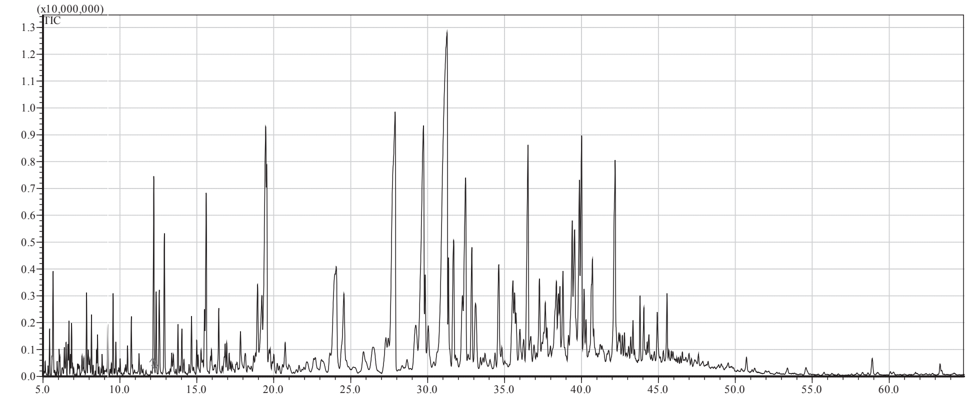
β-himachalane (22-28 %), a sesquiterpene, is the most abundant compound in all tars. It is found to be higher in the branches. α-himachalane (6-10 %) and longifolene (7-9 %) are the other important compounds. As known, β-himachalane and its derivates obtained from cedrus have antifungal and insecticidal effects (Daoubi *et al.*, 2005; Derwich *et al.*, 2010; Singh and Agarwal, 1988). Although the results showed similarities with literature, himachalol, which was found in the essential oil of cedar wood obtained by



A: Traditional / *tradicionalni postupak*



B: Jenkner Retort - branch / *u Jenknerovoj retorti – grane*



C: Jenkner Retort - root / *u Jenknerovoj retorti – grane*

Figure 1 GC-MS Chromatograms of cedrus tar diluted in dichloromethane
Slika 1. GC-MS kromatogrami katrana od cedra razrijeđenoga u diklormetanu

Table 2 Chemical composition of *Cedrus libani* tar obtained by different methods, %
Tablica 2. Kemijski sastav katrana od libanonskog cedra proizvedenoga različitim metodama, %

Compound *Kemijski spoj*	**RT**	**Root (Jenkner Retort)** *Korijen (u Jenknerovoj retorti)*			**Branch (Jenkner Retort)** *Grane (u Jenknerovoj retorti)*			**Traditional** *Tradicionalni postupak*		
		A	B	C	A	B	C	A	B	C
3-methyl-3-butanoic acid / *3-metil-3-maslačna kiselina*	10.32	0.31	0.13	0.34	0.47	0.80	0.54	-	0.84	1.28
Phenol / *fenol*	13.67	0.31	0.25	0.11	-	0.16	0.44	0.13	-	-
Propanoic acid / *propanoična kiselina*	14.02	-	-	0.11	-	-	-	-	-	-
Acetic acid / *octena kiselina*	14.56	-	0.05	0.37	-	0.36	-	-	-	-
2,6-Dimethyl styrene / *2,6-dimetil stiren*	15.07	0.31	0.24	-	-	-	-	-	-	-
p-Cresol-1 / p-krezol-1	17.02	0.23	0.31	-	0.21	0.29	0.34	-	-	-
p-Cresol-2 / *p-krezol-2*	17.44	0.47	0.75	0.64	0.34	0.38	0.65	-	-	-
1,3-methyl phenyl ethanone / *1,3-metil fenil etanon*	19.06	0.27	0.32	-	-	-	-	-	-	-
2,4-dimethyl phenol / *2,4-dimetil fenol*	21.06	0.25	0.29	0.28	0.28	0.30	0.28	-	-	-
Guaiacol / *gvajakol*	21.23	0.99	1.23	0.44	0.68	0.19	1.61	-	-	-
2,6-dimethylphenoxy / *2,6-dimetilfenoksi*	23.46	0.05	-	-	-	-	-	-	-	-
4-methyl-guaiacol / *4-metil-gvajakol*	27.70	-	-	2.97	2.89	2.95	5.79	0.29	2.63	1.11
4-methyl-guaiacol+catechol* *4-metil-gvajakol+katehol**	27.75	3.04	5.13	-	-	-	-	-	-	-
Catechol / *katehol*	27.79	-	-	1.57	-	4.96	2.22	0.36	0.94	1.34
4-methyl-catechol / *4-metil-katehol*	32.43	0.38	1.41	1.78	-	6.70	3.76	0.84	1.87	2.09
3-methyl-catechol / *4-metil-katehol*	33.20	-	-	-	0.48	-	-	-	-	-
α-Himachalane / *α-himakalan*	35.70	9.41	8.78	7.48	10.1	6.01	9.22	6.57	10.1	7.29
Longifolene / *longifolen*	37.07	8.75	7.73	7.22	9.70	7.11	8.75	7.69	9.40	7.80
Cedren / *cedren*	37.16	-	0.63	0.64	1.24	1.02	1.24	1.25	1.39	1.16
β-Himachalane / *β-himakalan*	38.30	25.1	22.5	21.9	28.1	23.1	25.8	23.7	25.8	23.0
(+)-Cuparane / *(+)-kuparan*	38.59	2.26	1.99	2.33	1.61	1.11	1.49	-	-	-
iso-longifolene / *izo-longifolen*	39.10	1.05	1.05	1.13	0.64	-	-	0.59	0.53	0.59
iso-Eugenol / *izo-eugenol*	40.79	1.26	1.23	1.57	2.05	2.44	1.84	1.56	1.39	1.50
Dihydro-Ar-Tumerone / *dihidro-ar-tumeron*	42.12	0.87	1.55	1.52	-	-	-	-	-	-
β-Himachalenoxide / *β-himakalenoksid*	42.55	-	-	-	-	-	-	1.22	1.00	1.11
Ar-tumerone / *Ar-tumeron*	44.39	1.98	1.74	2.05	0.61	0.99	0.36	-	-	-
β-Atlantone / *β-atlanton*	44.56	0.85	1.43	1.05	-	-	-	-	-	-
Tumerone / *tumeron*	45.35	1.82	1.53	1.82	0.98	0.99	0.86	1.73	1.23	1.50
Deodarone / *deodaron*	45.67	2.40	2.06	2.31	-	-	-	-	-	-
E-(α)-Atlantone / *E-(α)-atlanton*	48.29	3.55	3.46	4.21	-	-	-	-	-	-
16:0 acid / *16:0 kiselina*	60.23	tr	tr	tr	tr	tr	tr	tr	tr	tr
Dehydroabietan *dehidroabietan*	61.33	-	-	-	1.87	2.86	1.84	-	-	-
9-18:1 acid / *9-18:1 kiselina*	66.94	tr	tr	tr	tr	tr	tr	tr	-	tr
Pimaric acid / *pimarinska kiselina*	70.74	-	-	-	0.58	0.35	-	-	-	-
Sandrocopimaric acid / *sandrokopimarna kiselina*	71.11	tr	tr	tr	-	0.28	0.16	tr	tr	-
iso-Pimaric acid / *izo-pimarinska kiselina*	71.22	tr	tr	tr	1.62	1.50	1.36	1.70	1.27	1.58
Dehydroabietic acid / *dehidroabietična kiselina*	72.53	tr	tr	tr	1.09	1.28	1.04	1.01	0.74	0.90
Abietic acid / *abietična kiselina*	73.36	-	-	-	-	-	-	0.70	0.52	0.64
22:0 acid / *22:0 kiselina*	78.33	tr	tr	tr	tr	tr	tr	tr	tr	tr
24:0 alcohol / *24:0 alkohol*	80.67	tr	tr	tr	tr	tr	tr	tr	tr	tr
24: acid / *24:0 kiselina*	81.53	tr	tr	tr	tr	tr	tr	tr	tr	tr
Total non-identified compounds *Ukupno neidentificirani spojevi*		33.1	32.4	35.3	33.5	28.1	33.1	50.6	40.3	47.1

tr: trace (< % 0.1) A: n-Hexane, B:Diethylether, C:Dichloromethane, RT: TRB-5 type column,*:were not seperated. *tr: u tragovima (<0,1 %) A: n-heksan, B: dietileter, C: diklormetan, RT: TRB-5 kolona, *: nisu izdvojeni.*

Clevenger apparatus at 100 °C (Hafizoglu, 1987b; Fleisher and Fleisher, 2000; Saab *et al.*, 2005), was not detected in our samples. β-himachalenoxide was only determined by the traditional method.

Likewise the yield, the composition of tar also differs with thermal conditions (Wang *et al.*, 2009). Phenols, guaicol, creosol and 2.4-dimethyl phenol, were clearly observed only in the Jenkner Retort system. These compounds indicated that temperature applied in the traditional method was not sufficient for lignin degradation. Another compound iso-eugenol, precursors of lignin biosynthesis, was determined almost more than 2% in the branches. As known, in the softwood branches, the lignin content was higher (Fengel and Wegener, 2003). Phenols, obtained from biomass pyrolysis oils are valuable chemicals. They have disinfectant and antidiarrheal characteristics. They are also used as intermediates in synthesis of pharmaceutical products, adhesives and special polymers (Amen-Chen *et al.*, 1997; Ozbay, 2015).

Resin and fatty acids were found in all samples, while in roots they were less than 0.01 % (trace). Catechol, used in the production of pesticides, was found in higher amounts (2.2-6.7 %) in the tars obtained from branches.

Deodarone, E-(α)-atlantone, dihydro-Ar-tumore, which have an antifungal and insecticides effect, were found only in roots (Satrani *et al.*, 2006; Chaudhary *et al.*, 2012). Another compound, tumorene, used as a repellent against mosquito larvae, was identified in all tars (Champakaew *et al.*, 2007).

Three solvents with different polarities were used to dissolve tars for the analysis (Fig. 1). Although no significant difference was observed, dichloromethane gave the highest yield (59.6-71.8 %) of solubility among the identified compounds. However, β-himachalane, the main compound, was found to be higher in the hexane extract.

4 CONCLUSION
4. ZAKLJUČAK

In this study, chemical characterization of *Cedrus libani* A. Rich tar obtained by pyrolysis with Jenkner Retort system and traditional method was studied. Roots and fallen branches were used and analyzed separately. The tar yield of Jenkner Retort was 60 %, while it is 30-40 % in the traditional method, which revealed the effect of temperature. 41 compounds were identified. Phenols, alcohols, acids, sesquiterpenoids and fatty-resin acids are the main constituents. Phenols and methoxy groups were seen only in Jenkner Retort system. However, sesquiterpenoids (β-himachalane, α- himachalane), used for veterinary purposes, were determined in all samples. Traditional method can be used by the local veterinary, if compounds that have antifungal and insecticides are determined in these samples.

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