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# Composition of Essential Oils in Needles and Barks of Turkish Red Pine (*Pinus brutia* Ten.) Infested by *Marchalina hellenica* Genn.

Sastav eteričnih ulja u iglicama i kori turskoga  
crvenog bora (*Pinus brutia* Ten.) zaraženoga  
insektom *Marchalina hellenica* Genn.

## ORIGINAL SCIENTIFIC PAPER

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**ABSTRACT** • The scale insect *Marchalina hellenica* Genn. plays a key role in pine honey production and is hosted mainly by Turkish red pine (*Pinus brutia* Ten.). The needles of Turkish red pine are rich in essential oils. Moreover, essential oils can affect the host selection of insects. The essential oils in the needles and barks of *Marchalina hellenica*-infested and non-infested Turkish red pine were obtained via water distillation and their composition was determined by GC-MS analyses. The composition of the essential oils was found to differ in the needles of non-infested Turkish red pine and in those of pine infested by *Marchalina hellenica*. The Mann-Whitney U test results showed that the  $\beta$ -caryophyllene level was higher in the needles of Turkish red pine infested by *Marchalina hellenica*, whereas the junipen level was higher in the essential oil of non-infested Turkish red pine bark. Pimaral and  $\alpha$ -guaiene were detected only in the needles of infested trees, but were absent in the needles of non-infested trees. These components may be markers that can act on their own or as part of the whole in the host tree selection of *Marchalina hellenica*. In addition, the cis-verbenone component found in Turkish red pine bark might play a role in attracting *Marchalina hellenica*. This phenomenon should be further investigated through additional studies.

**KEYWORDS:** *Marchalina hellenica*; *Pinus brutia*; essential oils; GC-MS; needles; bark

**SAŽETAK** • Ljuskavica *Marchalina hellenica* Genn. ima ključnu ulogu u proizvodnji meda od borovine, a uglavnom živi na turskome crvenom boru (*Pinus brutia* Ten.). Igljice tog bora bogate su eteričnim uljima. Štoviše, eterična ulja za insekte mogu biti presudna pri odabiru domaćina na kojemu će se nastaniti. Eterična ulja iz iglica turskoga crvenog bora zaraženoga i nezaraženoga ljuskavicom *Marchalina hellenica* dobivena su vodenom destilacijom, a njihov je sastav određen GC-MS analizama. Utvrđeno je da je sastav eteričnih ulja u iglicama ne-

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*zaraženoga turskoga crvenog bora i u iglicama bora zaraženoga s Marchalina hellenica različit. Rezultati Mann-Whitneyjeva U-testa pokazali su da je razina  $\beta$ -kariofilena veća u iglicama crvenoga turskog bora zaraženoga s Marchalina hellenica, dok je razina kleke viša u eteričnom ulju nezaražene kore turskoga crvenog bora. Pimaral i  $\alpha$ -guaiene otkriveni su samo u zaraženim iglicama stabala, ali ih nije bilo u iglicama nezaraženih borova. Te komponente mogu biti markeri koji pri selekciji stabla domaćina Marchaline hellenice mogu djelovati sami ili kao dio cjeline. Osim toga, komponenta cis-verbenon, pronađena u kori turskoga crvenog bora, mogla bi imati važnu ulogu u privlačenju Marchaline hellenice. Tu je pojavu potrebno detaljnije istražiti u dodatnim studijama.*

**KLJUČNE RIJEČI:** *Marchalina hellenica; Pinus brutia; eterična ulja; GC-MS; iglice; kora*

## 1 INTRODUCTION

### 1. UVOD

The majority of the world's pine honey production takes place in Turkey. However, the essence of pine honey is created by *Marchalina hellenica* Genn (*M. hellenica*), which is mainly hosted by Turkish red pine (*Pinus brutia* Ten.). The scale insect *M. hellenica* lives in the cracks under the scales of bark. After sucking the sap of the host tree and receiving the nutrients it requires, the insect secretes the remainder rectally. This secretion, called *honeydew*, is then collected by bees and converted into pine honey, the worldwide production of which is only carried out in Turkey and Greece (Beşçeli and Ekici, 1968; Santas, 1983; Gürkan and Boşgelmez, 1989; Thrasylvoulou and Manikis, 1996; Gösterit and Gürel, 2011).

*M. hellenica* is distributed on the mainland and throughout some islands of Turkey and Greece as well as on the Italian island of Ischia. Although the widest distribution area of *M. hellenica* in Turkey is found in the Muğla region, it is also spread along the Aegean coast. *M. hellenica* selects *Pinus brutia* in particular in Turkey, and both *Pinus halapensis* and *Pinus brutia* in Greece. However, it has previously been reported that it can infest *Pinus pinea*, *Pinus nigra*, *Pinus sylvestris*, and *Pinus pinaster*. Moreover, suggestions that *M. hellenica* is also hosted by *Cedrus libani* in Turkey and by *Abies cephalonica* in Greece have been proposed (Beşçeli and Ekici, 1968; Selmi, 1983; Margaritopoulos *et al.*, 2003; Bacandritsos, 2004; Gounari, 2006; Ülgentürk *et al.*, 2012). According to the Food and Agriculture Organization (FAO) report AUS-69/1 dated 13.07.2015, *M. hellenica* has begun to appear in *Pinus radiata* and *Pinus halapensis* in Australia as well (Anonymous, 2017).

Furthermore, there is disagreement over the instar periods of *M. hellenica* in Greece. Although Erlinghagen (2001) and Bacandritsos *et al.* (2004) reported two instar periods, Gounari (2006; 2008) suggested three instar periods. Nevertheless, it is generally agreed that *M. hellenica* exhibits three instar periods in Turkey (Gürkan and Boşgelmez, 1989; Ülgentürk *et al.* 2012).

Coniferous trees and their parts contain rich extractives. Şahin and Yalcin (2017) determined the most common essential oil components in coniferous tree needles and a wide range of studies have been carried

out on the determination of essential oil components in various parts of *Pinus brutia*, including the needles (Roussis *et al.*, 1995; Sezik *et al.*, 2008; Mateus, 2008; Koutsaviti *et al.*, 2014; Yener *et al.*, 2014), bark (Salman, 2009; Bağcı *et al.*, 2011), cones (Loizzo *et al.*, 2008; Tumen *et al.*, 2010), and twigs (Ghosn *et al.*, 2006; Ustun *et al.*, 2012).

In coniferous trees, essential oils and some other extractives may be effective in the selection of host trees by insects. It was reported that some of the extractive components in coniferous trees might have attractive or deterrent effects on insects (Metcalf and Kogan, 1987; Jactel *et al.*, 1996; Franceschi *et al.*, 2005; Erbilgin *et al.*, 2006; Keeling and Bohlmann, 2006).

However, very limited research has been conducted on the interaction of *M. hellenica* with the extractive content of host trees. The terpene composition of the oleoresin (Mita *et al.*, 2002) and needles (Gallis *et al.*, 2011) of Aleppo pine infested by *M. hellenica* and the needles (Topcan, 2017) of Turkish red pine hosting *M. hellenica* have been investigated.

Although a few studies have been conducted to determine the interaction of *M. hellenica* with some tree species, a better understanding of specific tree chemical properties under controlled conditions was clearly needed. Consequently, the essential oil composition of Turkish red pine needles and bark from selected regions was evaluated against *M. hellenica* infections. After collecting samples, the level of essential oil compounds was determined based on the degree of changes. By performing the sample collection process in different areas and seasons and using the bark as a material as well as the needle, this study provides an additional new dimension compared to previous studies. The essential oil compounds of the needles and bark of *M. hellenica*-infested and uninfested Turkish red pine were examined. The results found in this study were evaluated in terms of the use of these components as a mark of host tree selection for *M. hellenica*.

## 2 MATERIALS AND METHODS

### 2. MATERIJALI I METODE

The Turkish red pine needle and bark samples from trees infested by *M. hellenica* and from non-infested trees were collected in three different seasons from

**Table 1** Needle sample collection areas and periods  
**Tablica 1.** Područja i razdoblja prikupljanja uzoraka iglica

Area/Season <i>Područje/Sezona</i>	First (Season 1) <i>1. sezona</i>	Second (Season 2) <i>2. sezona</i>	Third (Season 3) <i>3. sezona</i>
Area 1 (A1): Ula – Muğla	July 2016	October 2016	February 2017
Area 2 (A2): Yerkesik – Muğla	July 2016	October 2016	February 2017
Area 3 (A3): Gökova – Muğla	July 2016	November 2016	January 2017

three different trial areas consisting of natural stands in Muğla Province. Table 1 presents the collection areas and seasons. The coexistence of Turkish red pine trees hosting and not hosting *M. hellenica* was considered as the criterion in the selection of the study area. In total, twenty trees were selected in each trial area, ten of which were infested with *M. hellenica* and ten were not infested. Needle and bark samples were taken from the same trees in all three seasons. Samples from *M. hellenica*-infested trees were mixed together homogeneously. The same procedure was applied to samples from non-infested trees.

To obtain the essential oils of the needles and barks, 350 g samples were subjected to hydro distillation for 3 h using a Clevenger-type apparatus. The essential oils were kept in the refrigerator at -18 °C until gas chromatography-mass spectrometry (GC-MS) analysis. The Varian 2100 GC-MS system was used to characterize the essential oil components of the samples. The NIST 2008 library data and the “Eight Peak Index of Mass Spectra”, “Monoterpenes”, and “Identification of Essential Oils by Ion Trap Mass Spectroscopy” spectrophotometer atlases were used to identify the components. In addition, characterization was supported by taking into account the retention times of the components and calculating the Kovats index values. Standard substances were also characterized in the column under the same conditions and their retention times and essential oil components were compared. The GC-MS analysis conditions for the experimental procedure were as follows:

- Carrier gas: Helium
- Injection temperature: 250 °C
- Column temperature: The oven temperature was held at 60 °C for 5 min, and then increased up to 280 °C in increments of 4 °C/min and held at this temperature for 15 min.
- Split ratio: 1:50
- Ion source temperature: 150 °C
- Ionization energy: 70 eV
- Mass range: 28 - 650 m/z
- Injection volume: 0.2 µL
- Column properties: DB-1 nonpolar capillary column (Agilent), length: 30 m, inner diameter: 0.25 mm, film thickness: 0.25 µm

The evaluation of the normal distribution status of the dataset was performed using the Shapiro-Wilk test. The non-parametric Mann-Whitney U test was used to determine whether the two groups (infested by *M. hellenica* and non-infested) differed from each other.

### 3 RESULTS AND DISCUSSION

#### 3. REZULTATI I RASPRAVA

##### 3.1 Chemical composition of essential oils in pine needles

###### 3.1. Kemijski sastav eteričnih ulja u borovim iglicama

As shown in Table 4 (Supplement), the presence of 51 and 63 components, respectively, were revealed in the needles of *M. hellenica*-infested and non-infested Turkish red pine in Area 1. The main components of the essential oils of infested and non-infested tree needles were detected as  $\alpha$ -pinen,  $\beta$ -pinen,  $\beta$ -caryophyllene, germacrene D, cembrene, and thunbergol. In all three seasons, the  $\beta$ -caryophyllene ratio of the infested tree needles (12.84, 13.15, and 15.55 %) was higher than in the non-infested tree needles (11.29, 8.24, and 12.89 %); however, the  $\alpha$ -pinen and  $\beta$ -pinen ratios differed. Pimaral and  $\alpha$ -guaiene were identified only in the infested tree needles and these were in low amounts. The existence of 34 and 37 components, respectively, appeared in the infested and non-infested Turkish red pine needles in Area 2 (Table 5, Supplement). The main components of the essential oil composition of the infested and non-infested tree needles in A2 were similar to those in A1. Cembrene and thunbergol were not determined as major components in A2. A contrasting situation was seen in the ratios of  $\alpha$ -pinen and  $\beta$ -pinen. Pimaral and  $\alpha$ -guaiene were found only in the needles of infested trees, as was the case in A1.

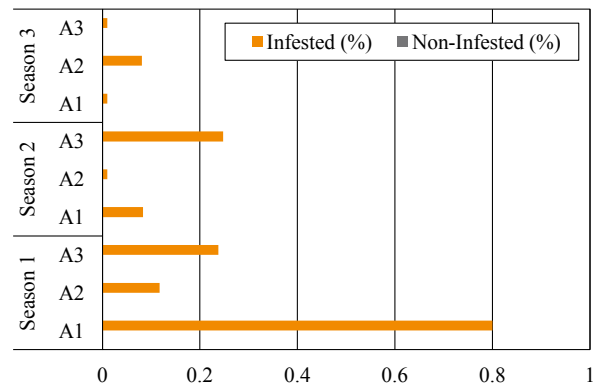
The 32 and 30 components, respectively, identified in infested and non-infested Turkish red pine needles in Area 3, are shown in Table 6 (Supplement). The major components of the essential oils of infested and non-infested tree needles were detected as  $\alpha$ -pinene,  $\beta$ -pinene,  $\beta$ -caryophyllene, and germacrene D, as was the case in A2. A comparison of the infested and non-infested tree needles revealed that the rate of  $\beta$ -caryophyllene was higher in the infested tree needles, whereas the ratios of  $\alpha$ -pinene and  $\beta$ -pinene were higher in the non-infested tree needles. The seasonal  $\beta$ -caryophyllene ratios were determined to be 16.22, 20.61, and 14.54 % in the infested tree needles and 7.68, 9.98, and 11.05 % in the non-infested tree needles, respectively. Pimaral appeared only in the needles of infested trees, as observed in A1 and A2.

The rate of  $\beta$ -caryophyllene was higher in infested tree needles in all three seasons and all three trial

areas, except for the second season of A2 (Figure 1). However, no such relation was observed between the  $\alpha$ -pinene and  $\beta$ -pinene ratios or the infested and non-infested tree needles. Therefore, according to the results of the Mann-Whitney U test (Table 2), only the  $\beta$ -caryophyllene ratio was found higher in infested trees at  $\alpha = 0.05$  ( $p = 0.038$ ). Although the germacrene D ratio showed a tendency similar to that of  $\beta$ -caryophyllene, there was statistically no correlation between it and the infested tree needles. Moreover, no significant difference was found among the other components. In all trial areas, pimaral was identified only in the needles of infested trees (Figure 2), whereas  $\alpha$ -guaiene was detected in the needles of infested trees only in A1 and A2, but was not found in either infested or non-infested tree needles in the A3 trial region (Figure 3). The findings of the pimaral and  $\alpha$ -guaiene were seen as remarkable.

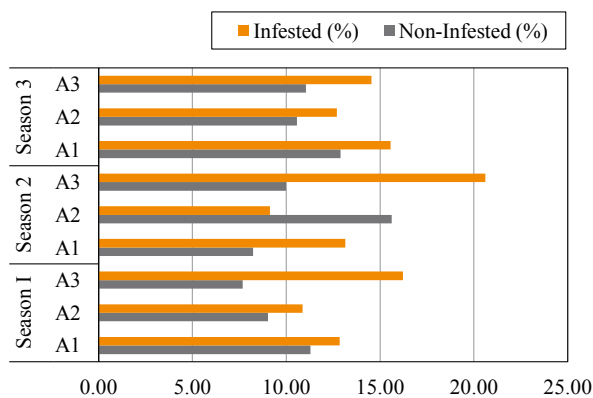
Gallis *et al.* (2011) reported that, when  $\beta$ -caryophyllene,  $\alpha$ -humulene, and neoabietol ratios were higher in the needles of Aleppo pine infested with *M.*

*hellenica*, the ratio of the cembrene was higher in non-infested trees. Kleinhentz *et al.* (1999) indicated that the  $\beta$ -caryophyllene ratio was higher in the needles of maritime pine infested by *Dioryctria sylvestrella* Ratz.



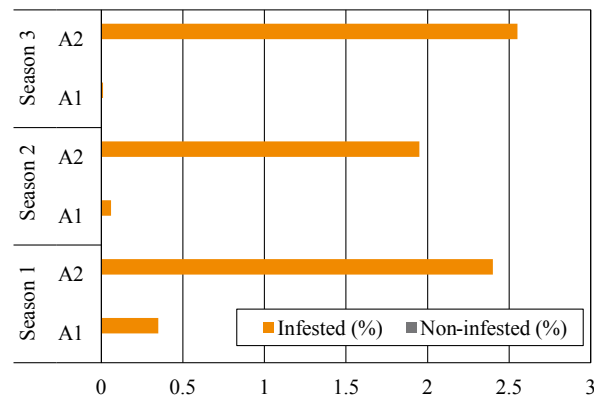
**Figure 2** Pimaral levels in needles of *M. hellenica*-infested and non-infested Turkish red pine

**Slika 2.** Razine pimarala u iglicama turskoga crvenog bora zaraženoga s *M. hellenica* i u iglicama nezaraženoga turskog crvenog bora



**Figure 1**  $\beta$ -caryophyllene levels in needles of *M. hellenica*-infested and non-infested Turkish red pine

**Slika 1.** Razine  $\beta$ -kariofilena u iglicama turskoga crvenog bora zaraženoga insektom *M. hellenica* i u iglicama nezaraženoga turskog crvenog bora



**Figure 3**  $\alpha$ -guaiene levels in needles of *M. hellenica*-infested and non-infested Turkish red pine

**Slika 3.** Razine  $\alpha$ -guaiene u iglicama turskoga crvenog bora zaraženoga s *M. hellenica* i u iglicama nezaraženoga turskog crvenog bora

**Table 2** Results of Mann-Whitney U test of essential oil components of needles from *M. hellenica*-infested and non-infested trees

**Tablica 2.** Rezultati Mann-Whitneyjeva U-testa komponenata eteričnog ulja iglica s borova zaraženih i nezaraženih insektom *M. hellenica*

Component Komponenta	Mann-Whitney U	Z	Asymp. Sig. (2-tailed)	Mean ranks Prosječni rangovi		Sample number Broj uzorka	
				Non-Infested Nezaražen	Infested Zaražen	Non-Infested Nezaražen	Infested Zaražen
$\alpha$ -Pinene	26.000	-1.281	.200	11.11	7.89	9	9
$\beta$ -Pinene	29.000	-1.015	.310	10.78	8.22	9	9
$\alpha$ -Terpineol	23.000	-1.545	.122	11.44	7.56	9	9
<b><math>\beta</math>-Caryophyllene</b>	<b>17.000</b>	<b>-2.075</b>	<b>.038*</b>	<b>6.89</b>	<b>12.11</b>	<b>9</b>	<b>9</b>
Germacrene D	22.000	-1.634	.102	7.44	11.56	9	9
$\delta$ -Cadinene	40.000	-.044	.965	9.44	9.56	9	9
Cembrene	36.500	-.353	.724	9.06	9.94	9	9
Thunbergol	36.000	-.397	.691	9.00	10.00	9	9

\*significant for  $\alpha = 0.05$  / značajno za  $\alpha = 0,05$



In our study, the  $\beta$ -caryophyllene level was found to be statistically higher in the needles of Turkish red pine infested by *M. hellenica*. However, no correlation was found between *M. hellenica* and cembren.

Mita *et al.* (2002) reported that the sensitivity of Aleppo pine to attack by *M. hellenica* was significantly correlated with a high ratio of  $\alpha$ -pinene and low ratios of limonene and  $\alpha$ -terpinyl acetate. Topcan (2017) demonstrated that the amount of  $\alpha$ -pinene decreased in the needles of Turkish red pine infested by *M. hellenica* during times of their intense feeding. In our research, although  $\alpha$ -pinene was a major component in the needles of infested and non-infested trees, statistically, no correlation was found between the  $\alpha$ -pinene level in the needles of Turkish red pine and *M. hellenica*.

Sakai and Yamasaki (1990) demonstrated that the pimaral isolated from *Pinus densiflora* and *Pinus tunbergii* exhibited an attractive effect for *Monochamus alternatus* females. In our work, pimaral was detected only in the needles of Turkish red pine infested by *M. hellenica*, despite being quantitatively low. Likewise,  $\alpha$ -guaiene was determined only in the needles of Turkish red pine infested by *M. hellenica*. No previous studies were found correlating  $\alpha$ -guaiene with *M. hellenica* or other insects. Therefore, this finding is of great importance as it is being presented to the literature for the first time. The similarities and differences appeared when our results on the needles were compared to the literature findings.

### 3.2 Chemical composition of essential oils in pine bark

#### 3.2. Kemijski sastav eteričnih ulja u borovoj kori

The presence of 40 and 44 components, respectively, were determined in the bark of infested and non-infested Turkish red pine trees in the Area 1 trial region

(Table 7, Supplement). The major components of the infested and non-infested tree bark were  $\alpha$ -terpineol,  $\beta$ -caryophyllene, borneol, myrtenol, junipen, *cis*-myrtenol, and caryophyllene oxide. The junipen and caryophyllene oxide ratios in the bark of infested trees were revealed as 9.85, 15.11, 16.22, 4.01, 4.64, and 8.17 %, respectively, whereas the ratio of these components in the bark of non-infested trees were found as 6.44, 7.42, 7.13, 1.92, 2.66, and 2.22 %, respectively. For all three seasons, *cis*-verbenone was detected only in the bark of infested trees (25.65, 4.65, and 3.79 %), but was absent in the bark of non-infested trees.

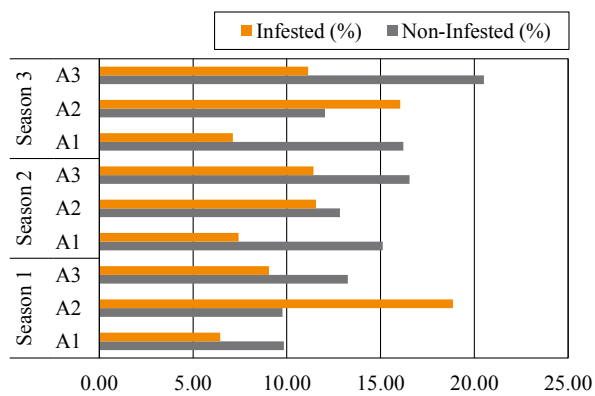
A total of 39 components appeared in both infested and non-infested Turkish red pine bark in the Area 2 trial region. These components are shown in Table 8 (Supplement). As with the A1 trial region, for all three seasons, although *cis*-verbenone was only seen in the bark of infested trees (2.75, 3.13, and 1.24 %), it was not found in the bark of non-infested trees. Like in the A1 trial region, in the A2 trial region, the main components of infested and non-infested tree bark were  $\alpha$ -terpineol, junipen,  $\beta$ -caryophyllene, myrtenol, caryophyllene oxide, borneol, and *cis*-myrtenol.

The existence of 34 and 37 components, as listed in Table 9 (Supplement), were revealed in the bark of infested and non-infested Turkish red pine in the A3 trial region. The main components in the essential oils of the infested and non-infested tree bark were identified as  $\alpha$ -terpineol, junipen,  $\beta$ -caryophyllene, caryophyllene oxide, and borneol, as in the A1 and A2 trial regions. Unlike the other two trial areas, although *cis*-verbenone was determined in the bark of both infested and non-infested trees, it was found to be higher in the bark of infested trees for all three seasons. According to the seasons, *cis*-verbenone ratios were determined as 1.51, 3.44, and 2.64 % in the bark of infested trees and

**Table 3** Results of Mann-Whitney U test of essential oil components of *M. hellenica*-infested and non-infested barks  
**Tablica 3.** Rezultati Mann-Whitneyjeva U-testa komponentata eteričnog ulja kore zaražene s *M. hellenica* i nezaražene borove kore

Component Komponenta	Mann-Whitney U	Z	Asymp. Sig. (2-tailed)	Mean ranks Prosječni rankovi		Sample number Broj uzorka	
				Non-Infested Nezaražen	Infested Zaražen	Non-Infested Nezaražen	Infested Zaražen
Fenchol	26.000	-1.280	.200	11.11	7.89	9	9
<i>trans</i> -pinocarveol	34.000	-.574	.566	8.78	10.22	9	9
Borneol	32.000	-.751	.200	10.44	8.56	9	9
Myrtenol	26.000	-1.280	.200	7.89	11.11	9	9
<i>cis</i> -myrtenol	34.000	-.574	.566	10.22	8.78	9	9
<b>Junipen</b>	<b>20.000</b>	<b>-1.810</b>	<b>.070*</b>	<b>11.78</b>	<b>7.22</b>	<b>9</b>	<b>9</b>
$\beta$ -Caryophyllene	28.000	-1.104	.270	10.89	8.11	9	9
$\alpha$ -Caryophyllene	36.000	-.397	.691	10.00	9.00	9	9
Caryophyllene oxide	39.000	-.132	.895	9.67	9.33	9	9
Isoaromadendrene oxide	36.000	-.397	.691	9.00	10.00	9	9
Aaromadendrene oxide	27.000	-1.192	.233	8.00	11.00	9	9

\*significant for  $\alpha = 0.05$  / značajno za  $\alpha = 0,05$



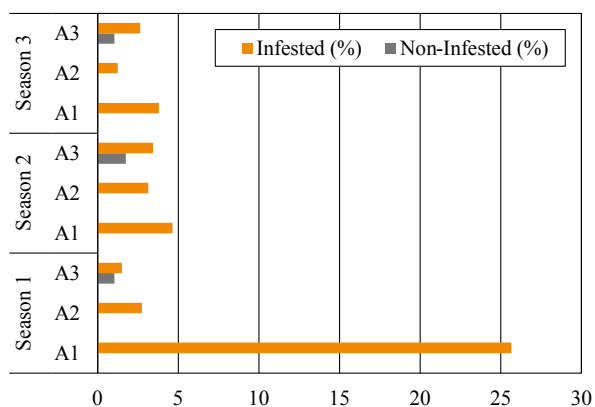
**Figure 4** Junipen levels in barks of *M. hellenica*-infested and non-infested Turkish red pine

**Slika 4.** Razine kleke u iglicama turskoga crvenog bora zaraženoga s *M. hellenica* i u iglicama nezaraženoga turskog crvenog bora

1.04, 1.75, and 1.05 % in the bark of non-infested trees, respectively.

According to the results of the Mann-Whitney U test (Table 3), the junipen ratio was higher in the bark of non-infested trees at  $\alpha = 0.10$  ( $p = 0.07$ ). No significant difference was observed in the other components. Figure 4 presents the junipen levels of the infested and non-infested tree bark. Although the *cis*-verbenone was identified only in the bark of infested trees in the A1 and A2 trial regions, it was detected in the bark of both infested and non-infested trees in the A3 trial region, but was quantitatively higher in the bark of infested trees. The *cis*-verbenone ratios of the infested and non-infested tree bark are presented in Figure 5.

Verbenon is one of the components used in creating pheromone traps (Rudinsky, 1973; Bedard *et al.*, 1980). In our study, although *cis*-verbenone was determined only in the bark of Turkish red pine infested by *M. hellenica* in the A1 and A2 trial regions, it was found in the bark of both infested and non-infested trees in the A3 trial region, but at a higher level in the



**Figure 5** *Cis*-verbenon levels in barks of *M. hellenica*-infested and non-infested Turkish red pine

**Slika 5.** Razine *Cis*-verbenona u iglicama turskoga crvenog bora zaraženoga s *M. hellenica* i u iglicama nezaraženoga turskog crvenog bora

infested bark. When the data obtained from our research and the findings in the related literature were evaluated together, it was concluded that the *cis*-verbenone component in Turkish red pine bark might have an effect that attracts *M. hellenica*.

Kleinhentz *et al.* (1999) reported that the junipen ratio was higher in the liber and resin of maritime pine not infested by *Dioryctria sylvestrella* Ratz. In addition, Kovalcuk *et al.* (2015) stated that the junipen rate was higher in the bark of Scots pine not infested by *Hylobius abietis* L. In our study, the junipen level was determined to be statistically higher in the bark of Turkish red pine not infested by *M. hellenica*. The results for junipen obtained in our study were compatible with the literature findings.

## 4 CONCLUSIONS

### 4. ZAKLJUČAK

Essential oils can play a key role in the selection of host trees by insects. Therefore, the GC-MS analyses of the essential oils are of great importance. The GC-MS results of this study indicated that the essential oil composition of non-infested Turkish red pine trees and of those infested by *M. hellenica* were different. This could suggest that the *cis*-verbenone in Turkish red pine bark might exhibit an attractive effect for *M. hellenica*. The  $\beta$ -caryophyllene, junipen, *cis*-verbenone, pimaral, and  $\alpha$ -guaiene determined via GC-MS analyses may be markers that can act on their own or as a part of the whole in the host tree selection of *M. hellenica*. However, in order to verify our claim, additional studies are needed, both in *M. hellenica*-infested forests and under laboratory conditions. Our findings have created an important basis for future studies focusing on the selection of host trees of *M. hellenica* and including olfactometer and orientation tests.

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## SUPPLEMENT – DODATAK

**Table 4** A1 trial region: Essential oil composition in needles of *M. hellenica*-infested and non-infested Turkish red pine  
**Tablica 4.** Probno područje A1: sastav eteričnog ulja u iglicama turskoga crvenog bora nezaraženoga i zaraženoga s *M. hellenica*

Component / Komponenta	RI	SEASON 1 (Area, %)		SEASON 2 (Area, %)		SEASON 3 (Area, %)	
		NI	I	NI	I	NI	I
<b><math>\alpha</math>-pinen</b>	940	0.07	tr	11.77	8.55	13.79	21.51
Camphene	948	tr	tr	0.30	0.26	0.27	0.41
$\alpha$ -fenchene	955	tr	-	tr	-	35.10	-
<b><math>\beta</math>-pinen</b>	961	0.32	0.09	31.40	29.99	1.92	3.98
3-carene	1004	0.09	tr	0.91	0.66	1.01	0.72
Pseudolimonen	1012	0.04	tr	1.98	2.38	tr	42.68
$\alpha$ -Phellandrene	1015	-	tr	-	0.06	-	tr
D-limonen	1026	0.12	tr	2.04	0.27	0.45	tr
Terpineolen	1080	0.19	tr	0.53	0.76	0.34	0.33
Linalol	1106	0.28	tr	0.21	0.14	0.32	tr
Fenchol	1108	0.22	tr	tr	0.11	tr	tr
<i>trans</i> -Pinocarveol	1121	0.02	-	tr	-	tr	-
<i>cis</i> - $\beta$ -Terpineol	1131	0.10	tr	tr	0.05	tr	tr
Isoborneol	1138	0.26	tr	0.11	0.11	0.08	tr
Myrtenol	1154	0.04	-	tr	-	tr	-
Terpinen-4-ol	1159	0.26	tr	0.11	0.16	0.08	tr
$\alpha$ -Terpineol	1175	7.04	0.53	1.98	2.79	1.30	0.59
Estragole	1221	0.03	tr	tr	0.04	tr	tr
<i>cis</i> -Geraniol	1225	0.03	-	tr	-	tr	-
Thymol Methyl ether	1228	0.03	tr	tr	0.02	tr	tr
Linalyl acetate (Bergamiol)	1239	0.72	tr	0.50	0.09	1.51	0.17
$\alpha$ -Terpineol acetate (Terpinyl acetate)	1357	1.98	0.18	1.37	0.75	2.56	0.93
Aromadendrene	1363	0.04	tr	tr	0.29	tr	tr
Isobornyl acetate	1268	0.44	tr	0.28	0.50	0.29	0.35
T-Gurjunene	1374	0.04	-	tr	-	tr	-
$\beta$ -Elemen	1382	0.96	0.11	0.49	0.65	0.66	0.13



Table 4 continuation / Tablica 4. nastavak

Component / Komponenta	RI	SEASON 1 (Area, %) 1. sezona (površina, %)		SEASON 2 (Area, %) 2. sezona (površina, %)		SEASON 3 (Area, %) 3. sezona (površina, %)	
		NI	I	NI	I	NI	I
Eugenol methyl ether	1387	0.79	0.05	0.25	0.41	tr	0.26
$\beta$ -Cubebene	1412	0.52	tr	0.24	0.24	0.27	0.18
<b><math>\beta</math>-Caryophyllene</b>	1415	11.29	12.84	8.24	13.15	12.89	15.55
$\alpha$ -Caryophyllene	1443	31.49	-	18.15	-	2.63	-
$\alpha$ -Amorphene	1452	0.16	-	0.08	-	tr	-
$\alpha$ -Selinene	1465	2.63	-	1.79	-	tr	-
$\alpha$ -Guaiene	1470	-	0.35	-	0.06	-	tr
<b>Germacrene D</b>	1472	1.26	3.13	0.67	19.72	tr	3.11
Phenethyl pivalate	1477	-	0.20	-	0.72	-	0.56
$\alpha$ -Muurolole	1487	0.58	-	0.28	-	0.59	-
Isoeugenol methyl ether	1491	0.75	-	0.10	-	0.59	-
T-Muurolole	1493	1.06	0.16	0.39	0.41	tr	0.43
$\alpha$ -Himachalene	1495	1.21	-	0.79	-	1.65	-
<i>cis</i> - $\alpha$ -Bisabolene	1499	-	0.25	-	1.22	-	1.51
Nerolidol	1512	-	0.10	-	0.09	-	tr
$\delta$ -Cadinene	1513	2.97	0.44	1.26	1.25	1.63	1.20
Caryophyllene oxide	1561	0.41	0.23	0.08	0.22	0.16	0.17
Aromadendrene oxide	1576	0.25	0.19	0.05	0.06	tr	tr
Germacrene-D-4-ol	1578	tr	0.07	tr	0.16	0.16	0.15
Lauric acid ethyl ester (Dodecanoic acid ethyl ester)	1582	-	tr	-	tr	-	0.11
<i>trans</i> - $\alpha$ -longipinocarveol	1596	0.09	-	tr	-	tr	-
$\alpha$ -Cubanol	1601	0.64	-	0.18	-	tr	-
tau-Cadinol	1616	1.27	0.85	0.34	0.37	0.35	0.16
$\alpha$ -Cadinol	1622	2.13	1.60	0.56	0.57	0.54	tr
$\delta$ -Cadinol	1631	0.39	0.29	0.10	0.12	tr	tr
tau-Muurolole	1639	0.12	-	tr	-	tr	-
<i>trans</i> -farnesol	1677	-	tr	-	tr	-	0.23
Benzylbenzoate	1719	0.21	0.10	0.04	0.07	tr	tr
Sclaren	1801	0.08	-	tr	-	0.12	-
<i>trans</i> farnesyl acetate	1814	0.31	0.84	0.17	0.31	0.12	0.12
Benzoic acid phenethyl ester	1830	0.07	-	tr	-	tr	-
<i>Cis</i> -3-Hexenyl cinnamate	1846	0.04	0.15	tr	0.02	tr	tr
Kaur-16-ene	1908	0.38	-	0.22	-	tr	-
Undefined	-	-	1.76	-	0.45	-	tr
Verticillol	2226	3.09	5.84	1.08	1.40	1.41	0.27
<b>Thunbergol</b>	2235	14.14	46.40	3.97	5.31	9.71	3.14
Pimaral	2241	-	0.80	-	0.08	-	tr
<b>Cembrene</b>	2253	6.69	15.28	3.10	3.97	3.93	0.74
Undefined	-	-	2.21	-	0.66	-	0.31
Scloreol	2174	-	3.05	-	0.14	-	tr
Ethyl Linoleate	2191	0.22	-	3.54	-	tr	-
Pimara-7,15-dien-3-one	2257	0.63	-	0.11	-	0.14	-
Methyl abietate	2342	0.24	-	0.16	-	tr	-
Androst-4-ene-3,17 dione	2392	-	1.09	-	0.12	-	tr
Methyl neoabietate	2411	0.39	0.82	0.09	0.08	tr	tr
Methyl 7,13,15-abietatrienoate	2421	0.15	-	tr	-	tr	-
Undefined	-	tr	-	tr	-	0.25	-

RI – Retention Index, I – Infested, NI – Non-Infested, I. Season – July 2016, II. Season – October 2016, III. Season – February 2017, tr – traces, tr <0.01

RI – indeks retencije, I – zaražen bor, NI – nezaražen bor, 1. sezona – srpanj 2016., 2. sezona – listopad 2016., 3. sezona – veljača 2017., tr – u tragovima, tr <0,01

**Table 5** A2 trial – region: Essential oil composition in needles of *M. hellenica*-infested and non-infested Turkish red pine  
**Tablica 5.** Probno područje A2: sastav eteričnog ulja u iglicama turskoga crvenog bora nezaraženoga i zaraženoga s *M. hellenica*

Component / Komponenta	RI	SEASON 1 (Area, %)		SEASON 2 (Area, %)		SEASON 3 (Area, %)	
		NI	I	NI	I	NI	I
<b><math>\alpha</math>-pinen</b>	940	12.09	9.92	1.92	14.10	14.35	13.31
Camphene	948	0.41	0.31	0.06	0.35	0.30	0.27
<b><math>\beta</math>-pinen</b>	961	32.07	28.89	7.56	36.94	30.51	27.33
3-Caren	1004	1.01	0.69	0.55	0.71	0.87	0.46
Pseudolimonen	1012	2.43	1.81	0.79	2.03	2.66	2.08
D-limonen	1026	2.71	2.32	1.25	2.28	2.37	2.04
Terpineolen	1080	1.52	0.89	0.51	0.51	0.39	0.32
Linalol	1106	0.24	0.20	0.23	0.11	0.22	0.14
Fenchol	1108	0.27	0.16	0.09	tr	tr	tr
<i>cis</i> - $\beta$ -Terpineol	1131	0.09	0.07	0.05	tr	tr	tr
Isoborneol	1138	0.22	0.15	0.13	tr	tr	tr
Terpinen-4-ol	1159	0.24	0.17	0.18	0.12	0.09	0.08
$\alpha$ -Terpineol	1175	5.14	3.18	2.68	1.90	1.16	0.92
Linalyl acetate (Bergamiol)	1239	0.21	0.25	1.04	0.43	1.00	0.76
Isobornyl acetate	1268	0.35	0.59	0.74	0.64	0.46	0.65
$\alpha$ -Terpineol acetate (Terpinyl acetate)	1357	0.81	1.53	1.61	1.64	1.28	3.18
$\beta$ -Bourbonene	1379	0.13	0.13	0.16	0.10	0.15	Tr
$\beta$ -Elemen	1382	0.56	0.75	1.22	0.69	0.44	0.36
Eugenol methyl ether	1387	0.47	0.04	0.84	0.40	0.46	0.42
$\beta$ -Cubebene	1412	0.27	0.33	0.39	0.23	0.18	0.20
<b><math>\beta</math>-Caryophyllene</b>	1415	9.03	10.86	15.61	9.14	10.56	12.70
$\alpha$ -Caryophyllene	1443	2.02	-	3.40	-	2.17	-
$\alpha$ -Guaiene	1470	-	2.40	-	1.95	-	2.55
<b>Germacrene D</b>	1472	16.45	23.89	34.50	20.04	22.43	25.69
T-Murolene	1493	0.40	0.58	0.65	0.41	0.36	0.51
$\alpha$ -Himachalene	1495	0.91	-	1.97	-	0.96	-
$\delta$ -Cadinene	1513	1.30	1.60	2.05	1.24	0.98	1.34
Caryophyllene oxide	1561	0.19	0.32	0.36	0.17	0.12	tr
$\alpha$ -Cubanol	1601	0.40	-	0.26	-	0.12	-
tau-Cadinol	1616	0.63	0.67	0.83	0.30	0.24	0.29
$\delta$ -Cadinol	1631	1.10	1.16	1.49	0.49	0.40	0.42
Benzylbenzoate	1719	0.07	-	0.08	-	tr	-
<i>trans</i> farnesyl acetate	1814	0.14	0.18	0.28	0.18	0.11	0.06
Verticillol	2226	0.68	0.74	2.01	0.33	0.37	0.33
Thunbergol	2235	2.81	2.45	7.93	1.42	2.93	2.30
Pimaral	2241	-	0.12	-	tr	-	0.08
Cembrene	2253	2.56	2.57	6.45	1.15	1.35	1.11
Methyl neobietate	2411	0.11	0.09	0.14	tr	tr	0.11

RI – Retention Index, I – Infested, NI – Non-Infested, I. Season – July 2016, II. Season – October 2016, III. Season – February 2017, tr – traces, tr <0.01

RI – indeks retencije, I – zaražen bor, NI – nezaražen bor, 1. sezona – srpanj 2016., 2. sezona – listopad 2016., 3. sezona – veljača 2017., tr – u tragovima, tr <0,01

**Table 6** A3 trial region: Essential oil composition in needles of *M. hellenica*-infested and non-infested Turkish red pine  
**Tablica 6.** Probno područje A3: sastav eteričnog ulja u iglicama turskoga crvenog bora nezaraženoga i zaraženoga s *M. hellenica*

Component / Komponenta	RI	SEASON 1 (Area, %)		SEASON 2 (Area, %)		SEASON 3 (Area, %)	
		NI	I	NI	I	NI	I
<b><math>\alpha</math>-pinene</b>	940	19.74	2.29	17.07	tr	17.59	11.54
Camphene	948	0.52	0.08	0.39	tr	0.43	0.22
<b><math>\beta</math>-pinene</b>	961	34.39	6.04	35.97	0.41	26.25	20.98
3-Caren	1004	0.94	0.56	1.73	0.43	1.33	1.27
Pseudolimonen	1012	2.11	0.69	2.05	0.17	2.32	2.16

Table 6 continuation / Tablica 6. nastavak

Component / Komponenta	RI	SEASON 1 (Area, %)		SEASON 2 (Area, %)		SEASON 3 (Area, %)	
		1. sezona (površina, %)	I	2. sezona (površina, %)	I	3. sezona (površina, %)	I
D-limonen	1026	2.34	0.94	3.43	0.27	2.12	1.59
Terpineolen	1080	0.86	0.44	0.60	0.18	0.39	0.22
Linalol	1106	0.26	0.08	0.30	0.21	0.11	tr
Isoborneol	1138	0.16	0.10	tr	0.08	0.04	tr
Terpinen-4-ol	1159	0.16	0.10	0.13	0.10	0.07	tr
$\alpha$ -Terpineol	1175	3.49	2.36	2.67	2.47	1.07	0.71
Linalyl acetate (Bergamiol)	1239	0.45	-	0.51	-	0.41	-
Isobornyl acetate	1268	0.45	0.43	0.41	0.53	0.43	0.33
$\alpha$ -Terpineol acetate (Terpinyl acetate)	1357	-	1.37	-	2.71	-	1.77
$\beta$ -Bourbonene	1379	0.10	0.30	0.15	0.29	0.23	0.28
$\beta$ -Elemen	1382	0.42	0.89	0.37	0.89	0.35	0.39
Eugenol methyl ether	1387	0.36	0.44	0.21	0.26	0.31	0.15
$\beta$ -Cubebene	1412	0.21	0.52	0.13	0.51	0.23	0.29
<b><math>\beta</math>-Caryophyllene</b>	1415	7.68	16.22	9.98	20.61	11.05	14.54
$\alpha$ -Caryophyllene	1443	1.67	3.55	1.98	4.46	2.34	3.05
<b>Germacrene D</b>	1472	16.22	41.85	15.49	39.29	25.56	29.10
T-Muurolene	1493	0.52	1.13	0.34	1.09	0.37	0.72
$\alpha$ -Himachalene	1495	0.90	1.79	1.20	2.34	1.65	1.52
$\delta$ -Cadinene	1513	1.55	3.28	1.17	3.20	1.91	2.10
$\alpha$ -Cubanol	1601	0.22	0.23	tr	0.16	tr	0.19
tau-Cadinol	1616	0.39	0.94	0.17	0.74	0.44	0.28
$\delta$ -Cadinol	1631	0.57	1.47	0.32	0.91	0.86	0.40
Benzylbenzoate	1719	0.06	0.12	tr	tr	0.01	tr
trans farnesyl acetate	1814	-	0.59	-	0.56	-	0.20
Verticillol	2226	0.38	-	0.32	-	1.59	-
Thunbergol	2235	1.63	5.48	1.75	8.27	0.49	3.87
Pimaral	2241	-	0.24	-	0.25	-	tr
Cembrene	2253	1.25	5.35	1.15	8.48	0.07	2.03
Methyl neobietate	2411	-	0.14	-	0.14	-	0.06

RI – Retention Index, I – Infested, NI – Non-Infested, I. Season – July 2016, II. Season – October 2016, III. Season – February 2017, tr – traces, tr <0.01

RI – indeks retencije, I – zaražen bor, NI – nezaražen bor, 1. sezona – srpanj 2016., 2. sezona – listopad 2016., 3. sezona – veljača 2017., tr – u tragovima, tr <0,01

Table 7 A1 trial region: Essential oil composition in bark of *M. hellenica*-infested and non-infested Turkish red pineTablica 7. Probno područje A1: sastav eteričnog ulja u kori turskoga crvenog bora nezaraženoga i zaraženoga s *M. hellenica*

Component / Komponenta	RI	SEASON 1 (Area, %)		SEASON 2 (Area, %)		SEASON 3 (Area, %)	
		1. sezona (površina, %)	I	2. sezona (površina, %)	I	3. sezona (površina, %)	I
<b><math>\alpha</math>-pinen</b>	940	0.09	1.97	0.34	tr	0.70	2.51
Camphane	948	-	0.20	-	tr	-	0.24
<b><math>\beta</math>-pinen</b>	961	0.26	0.54	0.16	tr	0.46	0.78
3-Caren	1004	0.25	1.38	0.60	tr	1.83	1.82
p-Simen	1013	tr	0.41	0.18	tr	0.24	0.60
D-Limonen	1026	0.11	0.86	0.39	tr	0.57	1.03
Dehidro p-simen	1058	tr	0.19	0.19	0.04	0.21	0.29
Fenchone	1075	-	0.26	-	0.09	-	0.64
Terpineolen	1080	0.51	1.01	1.02	0.15	1.42	1.49
Fenchol	1108	1.80	2.45	2.91	1.44	1.18	2.83
$\alpha$ -Campholenal	1116	0.42	0.19	0.84	0.09	0.41	0.27
Trans-Pinocarveol	1121	2.03	1.15	4.23	0.17	1.43	2.40
Camphor	1128	0.99	1.03	2.50	1.02	0.93	2.59
Cis- $\beta$ -Terpineol	1131	0.79	0.86	1.27	0.90	0.62	1.42
Isoborneol	1138	1.07	1.38	1.66	1.16	0.71	2.03
Borneol	1147	5.20	5.04	7.99	6.44	2.88	7.43
Myrtenol	1154	5.37	2.74	7.31	4.14	3.50	5.53

Table 7 continuation / Tablica 7. nastavak

Component / Komponenta	RI	SEASON 1 (Area, %)		SEASON 2 (Area, %)		SEASON 3 (Area, %)	
		1. sezona (površina, %)		2. sezona (površina, %)		3. sezona (površina, %)	
		NI	I	NI	I	NI	I
Terpinen-4-ol	1159	3.03	1.60	3.35	2.12	1.66	2.41
Para-Simen-8-ol	1168	1.18	-	1.68	-	0.53	-
<b><math>\alpha</math>-Terpineol</b>	1175	34.69	27.42	3.91	40.21	19.66	33.04
Berbenone	1191	1.50	-	3.62	-	1.09	-
<b>Cis-Verbenon</b>	1196	-	25.65	-	4.65	-	3.79
Cis-Carveol	1200	0.73	0.43	0.87	1.01	0.49	1.12
<b>Cis-Myrtanol</b>	1208	3.80	2.21	5.23	5.36	1.55	3.39
Perilla aldehyde (Perillal)	1217	0.47	-	0.38	-	0.21	-
Estragole	1221	0.68	0.62	0.79	0.51	0.41	0.38
Perilla alcohol (Perillol)	1290	0.41	0.38	0.24	0.18	tr	
Carvacrol	1295	0.17	0.12	0.22	0.39	0.15	0.29
$\alpha$ -Longipinen	1338	0.74	0.42	1.19	0.34	1.09	0.38
$\alpha$ -Gurjunene	1387	0.53	0.76	0.90	0.78	0.84	0.49
<b>Junipen</b>	1401	9.85	6.44	15.11	7.42	16.22	7.13
<b><math>\beta</math>-Caryophyllene</b>	1415	9.69	3.79	12.87	4.48	11.26	4.90
$\alpha$ -Caryophyllene	1443	2.16	0.89	2.88	1.11	2.77	1.07
$\beta$ -Farnesene	1450	0.20	-	0.23	-	0.20	-
Methyl eugenol	1456	0.23	0.19	0.27	0.34	0.60	0.23
Germacrene-D	1472	-	0.38	-	0.53	-	Tr
<b>Caryophyllene oxide</b>	1561	4.01	1.92	4.64	2.66	8.17	2.22
Isoaromadendrene oxide	1572	1.33	0.76	2.12	2.30	2.77	0.74
Aromadendrene oxide	1576	1.49	1.00	2.58	3.97	4.65	1.34
Benzylbenzoate	1719	0.12	0.11	0.21	0.24	0.06	Tr
Isopimaric acid methyl ester	2217	0.13	-	0.24	-	1.06	-
Verticillol	2226	0.99	0.62	1.35	1.43	2.57	1.48
Pimaral	2241	0.58	0.83	0.91	2.50	2.93	0.75
Cembrene	2253	0.35	-	0.43	-	0.69	-
Dehydroabietic aldehyde	2263	0.60	-	0.66	-	0.56	-
Undefined	-	0.44	-	0.36	-	0.32	-
Methyl dehydroabietate	2301	0.81	1.20	0.94	1.34	0.33	0.41
Methyl abietate	2342	0.20	0.58	0.20	0.49	0.07	0.08

RI – Retention Index, I – Infested, NI – Non-Infested, I. Season – July 2016, II. Season – October 2016, III. Season – February 2017, tr – traces, tr <0.01

RI – indeks retencije, I – zaražen bor, NI – nezaražen bor, 1. sezona – srpanj 2016., 2. sezona – listopad 2016., 3. sezona – veljača 2017., tr – u tragovima, tr <0,01

Table 8 A2 trial region: Essential oil composition in bark of *M. hellenica*-infested and non-infested Turkish red pineTablica 8. Probno područje A2: sastav eteričnog ulja u kori turskoga crvenog bora nezaraženoga i zaraženoga s *M. hellenica*

Component / Komponenta	RI	SEASON 1 (Area, %)		SEASON 2 (Area, %)		SEASON 3 (Area, %)	
		1. sezona (površina, %)		2. sezona (površina, %)		3. sezona (površina, %)	
		NI	I	NI	I	NI	I
$\alpha$ -pinen	940	3.40	0.20	0.03	tr	2.77	4.05
Camphene	948	0.36	-	tr	-	0.18	-
$\beta$ -pinen	961	1.16	0.16	0.02	tr	1.33	0.08
3-Caren	1004	2.44	0.69	0.10	0.05	3.25	1.70
p-Simen	1013	0.47	0.15	0.02	tr	0.35	0.05
D-limonen	1026	0.85	0.38	0.04	0.03	1.06	3.72
$\gamma$ -Terpinen	1048	0.17	-	0.02	-	0.20	-
Dehidro para-simen	1058	0.24	-	0.05	-	0.18	-
Terpineolen	1080	1.77	1.07	0.28	0.28	1.71	1.82
Fenchol	1108	1.62	1.43	1.24	0.72	1.41	1.62
$\alpha$ -Campholenal	1116	0.71	0.61	0.35	0.12	0.51	0.85
trans-Pinocarveol	1121	2.53	1.23	1.60	0.67	2.08	2.48
Camphor	1128	1.51	0.78	0.91	0.42	1.39	1.13
cis- $\beta$ -Terpineol	1131	0.64	0.65	0.77	0.44	0.87	0.70
Isoborneol	1138	0.67	0.58	0.76	0.37	3.37	3.44



Table 8 continuation / Tablica 8. nastavak

Component / Komponenta	RI	SEASON 1 (Area, %) 1. sezona (površina, %)		SEASON 2 (Area, %) 2. sezona (površina, %)		SEASON 3 (Area, %) 3. sezona (površina, %)	
		NI	I	NI	I	NI	I
Borneol	1147	4.01	2.91	3.90	2.24	2.68	2.41
<b>Myrtenol</b>	1154	20.32	3.11	4.11	2.80	6.24	5.31
Terpinen-4-ol	1159	2.31	1.73	1.89	1.08	2.06	2.04
<b><math>\alpha</math>-Terpineol</b>	1175	21.86	23.66	33.76	22.72	24.30	20.74
<b>Cis-Verbenon</b>	1191	-	2.75	-	3.15	-	1.24
Cis-Carveol	1200	0.88	0.59	0.96	0.69	0.79	0.47
<b>Cis-Myrtanol</b>	1208	4.86	1.95	3.91	2.97	3.77	1.81
Perilla aldehyde (Perillal)	1217	0.66	-	0.56	-	0.32	-
Estragole	1221	1.07	1.16	0.77	0.56	0.46	0.83
Perilla alcohol (Perillol)	1290	0.30	0.44	0.48	0.51	Tr	tr
Carvacrol	1295	0.27	0.25	0.42	0.35	0.26	0.12
$\alpha$ -Longipinen	1338	0.87	1.96	1.09	1.04	1.05	1.57
$\alpha$ -Gurjunene	1387	0.70	1.57	0.90	0.84	0.64	0.91
<b>Junipen</b>	1401	9.77	18.86	12.83	11.55	12.04	16.05
<b><math>\beta</math>-Caryophyllene</b>	1415	6.11	9.12	9.25	8.05	8.96	11.16
$\alpha$ -Caryophyllene	1443	1.23	2.18	2.38	2.36	1.97	2.42
Methyl eugenol	1456	0.50	0.89	0.41	1.19	0.53	0.71
<b>Caryophyllene oxide</b>	1561	1.64	5.96	5.26	4.61	5.45	4.87
Isoaromadendrene oxide	1572	1.29	2.67	1.92	4.78	2.79	1.24
Aromadendrene oxide	1576	1.39	3.36	1.92	6.30	2.79	1.75
Benzylbenzoate	1719	0.14	0.30	0.24	0.59	0.09	0.08
Verticillol	2226	-	1.83	-	4.19	-	0.78
Pimaral	2241	0.64	1.35	2.21	3.84	1.44	0.64
Cembrene	2253	-	0.53	-	0.97	-	0.30
Pimara-7.15-dien-3-one	2257	-	1.18	-	2.01	-	0.26
Dehydroabietic aldehyde	2263	0.32	0.94	1.90	2.89	0.34	0.40
Methyl dehydroabietate	2301	0.31	0.64	1.77	3.23	0.24	0.21
Methyl abietate	2342	tr	0.16	0.98	1.36	0.10	0.06

RI – Retention Index, I – Infested, NI – Non-Infested, I. Season – July 2016, II. Season – October 2016, III. Season – February 2017, tr – traces, tr <0.01

RI – indeks retencije, I – zaražen bor, NI – nezaražen bor, 1. sezona – srpanj 2016., 2. sezona – listopad 2016., 3. sezona – veljača 2017., tr – u tragovima, tr <0,01

Table 9 A3 trial region: Essential oil composition in bark of *M. hellenica*-infested and non-infested Turkish red pineTablica 9. Probno područje A3: sastav eteričnog ulja u kori turskoga crvenog bora nezaraženoga i zaraženoga s *M. hellenica*

Component / Komponenta	RI	SEASON 1 (Area, %) 1. sezona (površina, %)		SEASON 2 (Area, %) 2. sezona (površina, %)		SEASON 3 (Area, %) 3. sezona (površina, %)	
		NI	I	NI	I	NI	I
$\alpha$ -pinen	940	tr	tr	10.93	1.26	3.79	0.28
Camphene	948	tr	-	0.82	-	0.16	-
$\beta$ -pinen	961	tr	tr	3.72	0.46	2.26	0.24
3-Caren	1004	tr	tr	6.11	0.70	1.80	0.44
D-limonen	1026	tr	tr	2.00	0.94	0.64	0.39
$\gamma$ -Terpinen	1048	tr	-	0.26	-	0.10	-
Dehidro para-simen	1058	tr	tr	0.38	1.57	0.11	1.49
Terpineolen	1080	tr	-	2.69	-	1.04	-
Fenchol	1108	1.37	0.43	1.71	1.75	0.90	1.89
$\alpha$ -Campholenal	1116	0.13	tr	0.78	0.59	0.72	0.73
trans-Pinocarveol	1121	0.89	0.32	2.00	1.66	1.90	-
Camphor	1128	0.41	0.05	1.59	2.09	0.60	1.44
cis- $\beta$ -Terpineol	1131	0.76	0.60	0.39	1.36	0.63	1.43
Isoborneol	1138	1.36	0.85	1.09	1.40	2.32	1.43
Borneol	1147	6.03	5.06	4.36	5.88	1.77	5.27
Myrtenol	1154	2.59	3.54	1.98	1.92	2.79	3.55
Terpinen-4-ol	1159	1.49	0.71	1.59	1.87	1.06	1.60

Table 9 continuation / Tablica 9. nastavak

Component / Komponenta	RI	SEASON 1 (Area, %) 1. sezona (površina, %)		SEASON 2 (Area, %) 2. sezona (površina, %)		SEASON 3 (Area, %) 3. sezona (površina, %)	
		NI	I	NI	I	NI	I
<b><math>\alpha</math>-Terpineol</b>	1175	30.68	30.17	18.90	31.73	8.28	26.22
<b>Cis-Verbenon</b>	1191	1.04	1.51	1.75	3.44	1.05	2.64
Cis-Carveol	1200	0.62	0.66	0.42	0.52	0.53	0.97
Cis-Myrtanol	1208	2.95	3.83	1.33	1.04	0.61	1.24
Estragole	1221	0.85	0.50	0.78	0.64	0.28	0.21
Carvacrol	1295	0.21	0.65	tr	0.30	0.07	0.13
$\alpha$ -Longipinen	1338	0.73	0.28	1.64	0.79	1.99	0.88
$\alpha$ -Gurjunene	1387	1.11	0.71	1.16	0.68	1.20	1.06
<b>Junipen</b>	1401	13.26	9.05	16.55	11.42	20.51	11.14
<b><math>\beta</math>-Caryophyllene</b>	1415	6.41	7.03	9.76	11.32	21.49	14.91
$\alpha$ -Caryophyllene	1443	1.60	2.20	2.00	2.78	5.04	3.47
Methyl eugenol	1456	0.34	0.57	0.43	1.74	Tr	0.42
<b>Caryophyllene oxide</b>	1561	2.68	6.55	1.41	2.87	9.15	5.88
Isoaromadendrene oxide	1572	1.84	4.86	0.52	2.10	1.64	1.66
Aromadendrene oxide	1576	2.32	6.02	0.68	2.80	2.33	2.78
Benzylbenzoate	1719	0.17	2.00	tr	tr	0.02	0.03
Pimaral	2241	0.89	3.15	0.31	1.80	1.48	2.35
Cembrene	2253	-	3.66	-	tr	-	0.68
Dehydroabietic aldehyde	2263	14.67	-	tr	-	0.74	-
Methyl dehydroabiatate	2301	1.60	3.63	tr	0.61	0.85	0.47
Methyl abiatate	2342	1.02	1.42	tr	tr	0.17	0.39

RI – Retention Index, I – Infested, NI – Non-Infested, I. Season – July 2016, II. Season – October 2016, III. Season – February 2017, tr – traces, tr <0.01

RI – indeks retencije, I – zaražen bor, NI – nezaražen bor, 1. sezona – srpanj 2016., 2. sezona – listopad 2016., 3. sezona – veljača 2017., tr – u tragovima, tr <0,01

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