

Mustafa Burak Arslan¹, Selçuk Küçükaydın², Meltem Taş-Küçükaydın³,
Mehmet Emin Duru³, Halil Turgut Şahin⁴

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.

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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 β -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 α -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-

¹ Author is researcher at General Directorate of Forestry, Ege Forestry Research Institute, İzmir, Turkey

² Author is researcher at Department of Medical Services and Techniques, Köyceğiz Vocational School of Health Services, Muğla Sıtkı Koçman University, Muğla, Turkey.

³ Author is researcher at Department of Chemistry, Faculty of Sciences, Muğla Sıtkı Koçman University, Muğla, Turkey.

⁴ Author is researcher at Department of Forest Products Engineering, Faculty of Forestry, Isparta University of Applied Sciences, Isparta, Turkey.

zaraženoga turskoga crvenog bora i u iglicama bora zaraženoga s Marchalina hellenica različit. Rezultati Mann-Whitneyjeva U-testa pokazali su da je razina β -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 α -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* infestations. 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 α -pinen, β -pinen, β -caryophyllene, germacrene D, cembrene, and thunbergol. In all three seasons, the β -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 α -pinen and β -pinen ratios differed. Pimaral and α -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 α -pinen and β -pinen. Pimaral and α -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 α -pinene, β -pinene, β -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 β -caryophyllene was higher in the infested tree needles, whereas the ratios of α -pinene and β -pinene were higher in the non-infested tree needles. The seasonal β -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 β -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 α -pinene and β -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 β -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 β -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 α -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 α -guaiene were seen as remarkable.

Gallis *et al.* (2011) reported that, when β -caryophyllene, α -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 β -caryophyllene ratio was higher in the needles of maritime pine infested by *Dioryctria sylvestrella* Ratz.

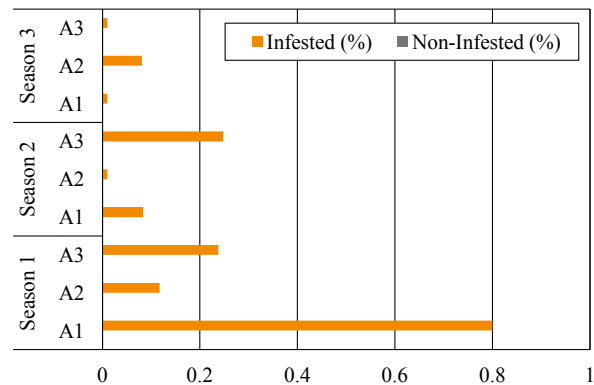


Figure 2 Pimaral levels in needles of *M. hellenica*-infested and non-infested Turkish red pine

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

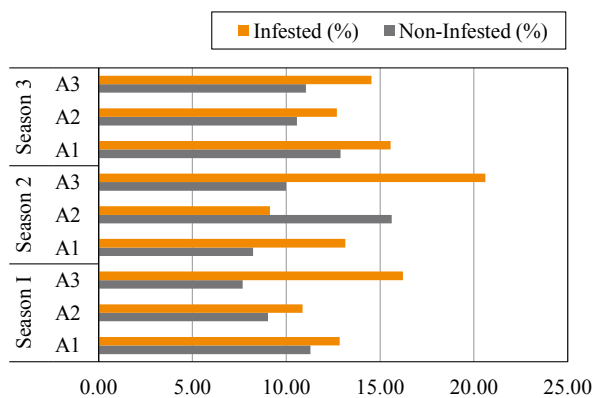


Figure 1 β -caryophyllene levels in needles of *M. hellenica*-infested and non-infested Turkish red pine

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

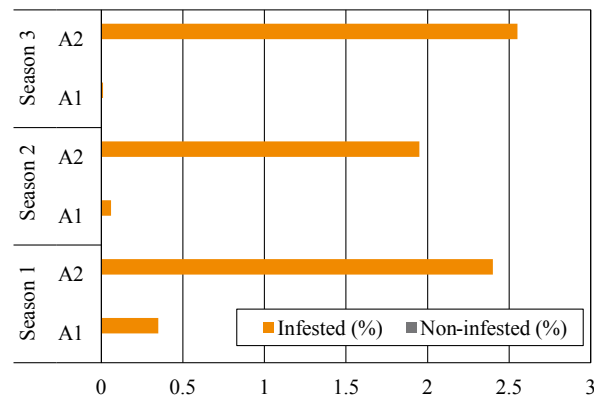


Figure 3 α -guaiene levels in needles of *M. hellenica*-infested and non-infested Turkish red pine

Slika 3. Razine α -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 |
| α -Pinene | 26.000 | -1.281 | .200 | 11.11 | 7.89 | 9 | 9 |
| β -Pinene | 29.000 | -1.015 | .310 | 10.78 | 8.22 | 9 | 9 |
| α -Terpineol | 23.000 | -1.545 | .122 | 11.44 | 7.56 | 9 | 9 |
| β-Caryophyllene | 17.000 | -2.075 | .038* | 6.89 | 12.11 | 9 | 9 |
| Germacrene D | 22.000 | -1.634 | .102 | 7.44 | 11.56 | 9 | 9 |
| δ -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 β -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 α -pinene and low ratios of limonene and α -terpinyl acetate. Topcan (2017) demonstrated that the amount of α -pinene decreased in the needles of Turkish red pine infested by *M. hellenica* during times of their intense feeding. In our research, although α -pinene was a major component in the needles of infested and non-infested trees, statistically, no correlation was found between the α -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 thunbergii* 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, α -guaiene was determined only in the needles of Turkish red pine infested by *M. hellenica*. No previous studies were found correlating α -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

3.2.1. 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 α -terpineol, β -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 α -terpineol, junipen, β -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 α -terpineol, junipen, β -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 |
| Junipen | 20.000 | -1.810 | .070* | 11.78 | 7.22 | 9 | 9 |
| β -Caryophyllene | 28.000 | -1.104 | .270 | 10.89 | 8.11 | 9 | 9 |
| α -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 $\alpha = 0,05$

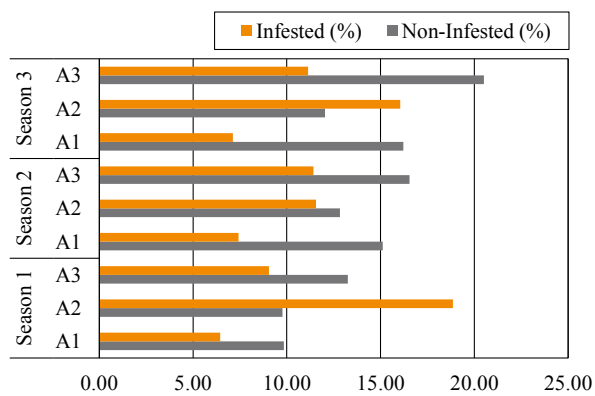


Figure 4 Junipen levels in barks of *M. hellenica*-infested and non-infested Turkish red pine

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

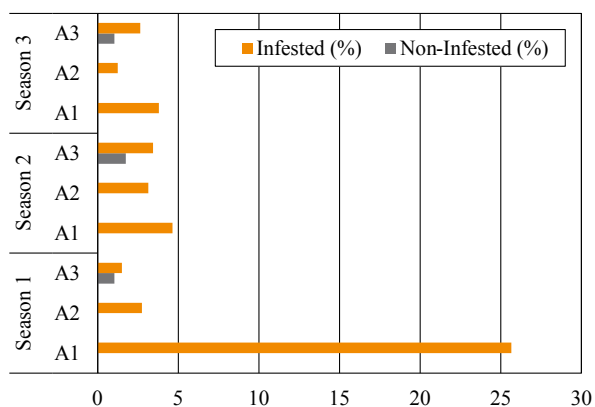


Figure 5 *Cis*-verbenon levels in barks of *M. hellenica*-infested and non-infested Turkish red pine

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 β -caryophyllene, junipen, *cis*-verbenone, pimaral, and α -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, %) 1. sezona (površina, %) | | SEASON 2 (Area, %) 2. sezona (površina, %) | | SEASON 3 (Area, %) 3. sezona (površina, %) | |
|--|------|---|------|---|-------|---|-------|
| | | NI | I | NI | I | NI | I |
| α-pinen | 940 | 0.07 | tr | 11.77 | 8.55 | 13.79 | 21.51 |
| Camphene | 948 | tr | tr | 0.30 | 0.26 | 0.27 | 0.41 |
| α -fenchene | 955 | tr | - | tr | - | 35.10 | - |
| β-pinen | 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 |
| α -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> - β -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 |
| α -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 |
| α -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 | - |
| β -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 |
| β -Cubebene | 1412 | 0.52 | tr | 0.24 | 0.24 | 0.27 | 0.18 |
| β-Caryophyllene | 1415 | 11.29 | 12.84 | 8.24 | 13.15 | 12.89 | 15.55 |
| α -Caryophyllene | 1443 | 31.49 | - | 18.15 | - | 2.63 | - |
| α -Amorphene | 1452 | 0.16 | - | 0.08 | - | tr | - |
| α -Selinene | 1465 | 2.63 | - | 1.79 | - | tr | - |
| α -Guaiene | 1470 | - | 0.35 | - | 0.06 | - | tr |
| Germacrene D | 1472 | 1.26 | 3.13 | 0.67 | 19.72 | tr | 3.11 |
| Phenethyl pivalate | 1477 | - | 0.20 | - | 0.72 | - | 0.56 |
| α -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 |
| α -Himachalene | 1495 | 1.21 | - | 0.79 | - | 1.65 | - |
| <i>cis</i> - α -Bisabolene | 1499 | - | 0.25 | - | 1.22 | - | 1.51 |
| Nerolidol | 1512 | - | 0.10 | - | 0.09 | - | tr |
| δ -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> - α -longipinocarveol | 1596 | 0.09 | - | tr | - | tr | - |
| α -Cubanol | 1601 | 0.64 | - | 0.18 | - | tr | - |
| tau-Cadinol | 1616 | 1.27 | 0.85 | 0.34 | 0.37 | 0.35 | 0.16 |
| α -Cadinol | 1622 | 2.13 | 1.60 | 0.56 | 0.57 | 0.54 | tr |
| δ -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 |
| Thunbergol | 2235 | 14.14 | 46.40 | 3.97 | 5.31 | 9.71 | 3.14 |
| Pimaral | 2241 | - | 0.80 | - | 0.08 | - | tr |
| Cembrene | 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 |
| α-pinen | 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 |
| β-pinen | 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> - β -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 |
| α -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 |
| α -Terpineol acetate (Terpinyl acetate) | 1357 | 0.81 | 1.53 | 1.61 | 1.64 | 1.28 | 3.18 |
| β -Bourbonene | 1379 | 0.13 | 0.13 | 0.16 | 0.10 | 0.15 | Tr |
| β -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 |
| β -Cubebene | 1412 | 0.27 | 0.33 | 0.39 | 0.23 | 0.18 | 0.20 |
| β-Caryophyllene | 1415 | 9.03 | 10.86 | 15.61 | 9.14 | 10.56 | 12.70 |
| α -Caryophyllene | 1443 | 2.02 | - | 3.40 | - | 2.17 | - |
| α -Guaiene | 1470 | - | 2.40 | - | 1.95 | - | 2.55 |
| Germacrene D | 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 |
| α -Himachalene | 1495 | 0.91 | - | 1.97 | - | 0.96 | - |
| δ -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 |
| α -Cubanol | 1601 | 0.40 | - | 0.26 | - | 0.12 | - |
| tau-Cadinol | 1616 | 0.63 | 0.67 | 0.83 | 0.30 | 0.24 | 0.29 |
| δ -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 |
| α-pinene | 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 |
| β-pinene | 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 |
| α -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 |
| α -Terpineol acetate (Terpinyl acetate) | 1357 | - | 1.37 | - | 2.71 | - | 1.77 |
| β -Bourbonene | 1379 | 0.10 | 0.30 | 0.15 | 0.29 | 0.23 | 0.28 |
| β -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 |
| β -Cubebene | 1412 | 0.21 | 0.52 | 0.13 | 0.51 | 0.23 | 0.29 |
| β-Caryophyllene | 1415 | 7.68 | 16.22 | 9.98 | 20.61 | 11.05 | 14.54 |
| α -Caryophyllene | 1443 | 1.67 | 3.55 | 1.98 | 4.46 | 2.34 | 3.05 |
| Germacrene D | 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 |
| α -Himachalene | 1495 | 0.90 | 1.79 | 1.20 | 2.34 | 1.65 | 1.52 |
| δ -Cadinene | 1513 | 1.55 | 3.28 | 1.17 | 3.20 | 1.91 | 2.10 |
| α -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 |
| δ -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 |
| α-pinen | 940 | 0.09 | 1.97 | 0.34 | tr | 0.70 | 2.51 |
| Camphane | 948 | - | 0.20 | - | tr | - | 0.24 |
| β-pinen | 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 |
| α -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- β -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 | - |
| α-Terpineol | 1175 | 34.69 | 27.42 | 3.91 | 40.21 | 19.66 | 33.04 |
| Berbenone | 1191 | 1.50 | - | 3.62 | - | 1.09 | - |
| Cis-Verbenon | 1196 | - | 25.65 | - | 4.65 | - | 3.79 |
| Cis-Carveol | 1200 | 0.73 | 0.43 | 0.87 | 1.01 | 0.49 | 1.12 |
| Cis-Myrtanol | 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 |
| α -Longipinen | 1338 | 0.74 | 0.42 | 1.19 | 0.34 | 1.09 | 0.38 |
| α -Gurjunene | 1387 | 0.53 | 0.76 | 0.90 | 0.78 | 0.84 | 0.49 |
| Junipen | 1401 | 9.85 | 6.44 | 15.11 | 7.42 | 16.22 | 7.13 |
| β-Caryophyllene | 1415 | 9.69 | 3.79 | 12.87 | 4.48 | 11.26 | 4.90 |
| α -Caryophyllene | 1443 | 2.16 | 0.89 | 2.88 | 1.11 | 2.77 | 1.07 |
| β -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 |
| Caryophyllene oxide | 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 |
| α -pinen | 940 | 3.40 | 0.20 | 0.03 | tr | 2.77 | 4.05 |
| Camphene | 948 | 0.36 | - | tr | - | 0.18 | - |
| β -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 |
| γ -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 |
| α -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- β -Terpineol | 1131 | 0.64 | 0.65 | 0.77 | 0.44 | 0.87 | 0.70 |
| Isborneol | 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 |
| Myrtenol | 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 |
| α-Terpineol | 1175 | 21.86 | 23.66 | 33.76 | 22.72 | 24.30 | 20.74 |
| Cis-Verbenon | 1191 | - | 2.75 | - | 3.15 | - | 1.24 |
| Cis-Carveol | 1200 | 0.88 | 0.59 | 0.96 | 0.69 | 0.79 | 0.47 |
| Cis-Myrtanol | 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 |
| α -Longipinen | 1338 | 0.87 | 1.96 | 1.09 | 1.04 | 1.05 | 1.57 |
| α -Gurjunene | 1387 | 0.70 | 1.57 | 0.90 | 0.84 | 0.64 | 0.91 |
| Junipen | 1401 | 9.77 | 18.86 | 12.83 | 11.55 | 12.04 | 16.05 |
| β-Caryophyllene | 1415 | 6.11 | 9.12 | 9.25 | 8.05 | 8.96 | 11.16 |
| α -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 |
| Caryophyllene oxide | 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 |
| α -pinen | 940 | tr | tr | 10.93 | 1.26 | 3.79 | 0.28 |
| Camphene | 948 | tr | - | 0.82 | - | 0.16 | - |
| β -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 |
| γ -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 |
| α -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- β -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 |
| α-Terpineol | 1175 | 30.68 | 30.17 | 18.90 | 31.73 | 8.28 | 26.22 |
| Cis-Verbenon | 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 |
| α -Longipinen | 1338 | 0.73 | 0.28 | 1.64 | 0.79 | 1.99 | 0.88 |
| α -Gurjunene | 1387 | 1.11 | 0.71 | 1.16 | 0.68 | 1.20 | 1.06 |
| Junipen | 1401 | 13.26 | 9.05 | 16.55 | 11.42 | 20.51 | 11.14 |
| β-Caryophyllene | 1415 | 6.41 | 7.03 | 9.76 | 11.32 | 21.49 | 14.91 |
| α -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 |
| Caryophyllene oxide | 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

Corresponding address:

MUSTAFA BURAK ARSLAN

General Directorate of Forestry, Ege Forestry Research Institute, İzmir, TURKEY,

e-mail: mustafaburakarslan@ogm.gov.tr