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Original article (Orijinal araştırma)

Chemical components and insecticidal effects of essential oils from three lavender cultivars against adult *Sitophilus granarius* (L., 1758) (Coleoptera: Curculionidae)[1]

Farklı lavanta çeşitlerinin kimyasal bileşenleri ve *Sitophilus granarius* (L., 1758) (Coleoptera: Curculionidae) erginlerine karşı insektisidal etkisi

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Abstract

The study was conducted to determine the insecticidal and behavioral effect of essential oils (EOs) extracted from three lavender cultivars, Hemus, Raya and Yubileina, against adult stage of *Sitophilus granarius* (L., 1758) (Coleoptera: Curculionidae) under laboratory conditions. The contact, fumigant and repellent effects of plant EOs were investigated and possible germination inhibitory effects of the EOs on wheat germination were evaluated. EOs were extracted from the plant samples using a Neo-Clevenger type apparatus where the plant material is subjected to hydro distillation. The chemical constituents of EOs were detected by gas chromatography-mass spectrometry. The experiments were conducted under laboratory conditions in 2019 and 2020. As a result of the study, it was determined that the fumigant activity changed according to the cultivars. While the LC_{90} value in the fumigant activity of the EOs of the Hemus was determined as 0.157 µl/ml air, this value was recorded as 0.139 µl/ml air and 0.118 µl/ml air in Raya and Yubileina, respectively. Six h after treatment, the highest repellent activity was 86% at 0.05 µl/cm^2 with Yubileina. Main EOs components of each cultivar were: Yubileina, linalool (36.0%), linalyl acetate (24.2%) and lavandulyl acetate (5.86%); Hemus, linalool (28.5%), linalyl acetate (23.1%) and lavandulyl acetate (6.59%); and Raya, linalool (42.5%), linalyl acetate (30.0%) and α-terpineol (5.45%). There was no negative effect on the germination of wheat with any of essential oils. These results show that lavender EOs could be useful for the control of *S. granarius*.

Keywords: Biplot analyses, fumigant activity, GC-MS, granary weevil, *Lavandula angustifolia*, repellent activity

Öz

Bu çalışma, ticari amaçlı üretimi yapılan üç farklı lavanta çeşidi Hemus, Raya ve Yubileina'dan izole edilen uçucu yağların *Sitophilus granarius* (L., 1758) (Coleoptera: Curculionidae)'un ergin dönemlerine karşı insektisidal ve davranışsal etkisini laboratuvar koşullarında belirlemek amacıyla yapılmıştır. Bitki uçucu yağlarının kontakt, fumigant ve repellent etkileri araştırılmış ve bu yağların buğdayın çimlenme gücü üzerine olası etkileri değerlendirilmiştir. Uçucu yağlar, bitki materyallerinden Neo-Clevenger tipi aparat kullanılarak hidrodistilasyon yöntemine göre elde edilmiştir. Uçucu yağların kimyasal bileşenleri gaz kromatografisi-kütle spektrometrisi ile tespit edilmiştir. Denemeler 2019 ve 2020 yıllarında laboratuvar koşullarında yürütülmüştür. Çalışma sonucunda bitki çeşitlerine göre fumigant aktivitesinin değiştiği tespit edilmiştir. Hemus uçucu yağının fumigant aktivitesindeki LC_{90} değeri 0.157 µl/ml hava olarak belirlenirken, bu değer Raya ve Yubileina'da sırasıyla 0.139 µl/ml hava ve 0.118 µl/ml hava olarak belirlenmiştir. Uygulamadan altı saat sonra en yüksek repellent etki, Yubileina'nın 0.05 µl/cm^2 uygulama dozunda %86 repellent etki ile gözlenmiştir. Yubileina'nın ana uçucu yağ bileşenleri, linalool (%36.0), linalil asetat (%24.2) ve lavandulil asetat (%5.86) olarak belirlenirken Hemus'un temel bileşenleri linalool (%28.5), linalil asetat (%23.1) ve lavandulil asetat (%6.59), Raya'nın ana bileşenleri ise linalool (%42.5), linalil asetat (%30.0) ve α-terpineol (%5.45) olarak belirlenmiştir. Her üç uçucu yağ için de buğdayın çimlenme gücü üzerinde olumsuz bir etki belirlenmemiştir. Bu sonuçlar, lavanta uçucu yağlarının *S. granarius*'un mücadelesinde önemli bir potansiyele sahip olabileceğini göstermektedir.

Anahtar sözcükler: Biplot analizi, fumigant aktivite, GC-MS, buğday biti, *Lavandula angustifolia*, repellent aktivite

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Introduction

The world population is increasing rapidly and as a result the growing demand for safe food demand is a major problem for agricultural systems with limited resources. Therefore, the current global agricultural production has to be protected from biotic and abiotic factors from harvest to the table. The largest insect order, Coleoptera, includes the major and significant stored product pests. These pests can live under in a wide range of environment conditions. The feeding behavior and levels of storage pests vary, so some are considered primary pests whereas others are classified as secondary pests. *Sitophilus granarius* (L., 1758) (Coleoptera: Curculionidae) is considered one of the primary pests. Qualitative and quantitative losses occur in the stored products because of the detrimental effects of this pest. Various cultural, physical and chemical control methods are used to protect the stored products. Stored product pests are potentially present in all warehouses and cause 10-30% damage to annual global cereal production (Singh et al., 2009). The most widely used approach to control stored product pests globally is pesticides and in particular fumigation (Mutungi et al., 2014). However, both the direct consumption of stored products and the increase in concerns about pesticide use in recent years have led to the development of more environmentally-friendly approaches.

Lavender spp. which is a member of the Lamiaceae are known to have a Mediterranean origin (Aslancan & Sarıbaş, 2011). The lavender is a semi-bushy, perennial herb that can grow up to 1 m. There are more than 30 species of lavender, but only a few are used commercially for essential oil (EOs). The essential oil obtained from the flowers and flower stalks is one of the 15 most traded EOs in the world (Aslancan & Sarıbaş, 2011). The essential oil of the lavender is extracted using water vapor distillation apparatus. Lavender essential oil is still in high demand around the world. In near future, over 200,000 ha are expected to be planted with lavender in Europe and the quality of the essential oil produced is crucial for medical, pharmaceutical and aromatherapy applications (Hassiotis et al., 2010).

Plants consist of very different chemical components that cause insecticidal effects. These secondary metabolites are complex mixtures and can be classified as alkaloids, glycosides, phenols, terpenoids, tannins and saponins (Shanker & Solanki, 2000). Lis-Balchin & Hart (1999) found that lavender oil from *Lavandula angustifolia* Mill. (Lamiaceae) flowers contains linalyl acetate, linalool, lavandulol, 1,8-cineole, lavandulyl acetate, camphor and borneol. The antimicrobial activity of EOs, along with their sweetener/aromatic properties, has been widely used in the pharmaceutical, cosmetic and food industries (Prashar et al., 2004). Lavender essential oil is used in the culinary industry to flavor drinks, ice cream, confectionery, baked goods and chewing gum (Kim & Lee, 2002). Recently, aromatherapy has become increasingly popular and lavender has been used as a sedative (Lis-Balchin & Hart, 1999). Lavender species is also used as pain relievers, antifungal and antibacterial agents in the treatment of burns and insect bites (Cavanagh & Wilkinson, 2002).

Many studies have been conducted in terms of insecticidal and some other biological activities of lavender species. Different activities of *Lavandula* spp. such as antioxidant (Yakoubi et al., 2021), cytotoxic (Siddiqui et al., 2020), antimicrobial (Leong et al., 2021), anti-acetylcholinesterase (Vairinhos & Miguel, 2020), antibacterial (Sayout et al., 2020), repellent (Huang et al., 2020), antifungal (Domingues et al., 2021), allelopathic (Nazemi et al., 2016) have been recorded recently.

In this study, the insecticidal and behavioral effects of EOs of lavender cvs Hemus, Raya and Yubileina were evaluated under laboratory conditions against *S. granarius* and also possible germination inhibitory effects of the EOs on wheat germination. In addition, essential oil components were determined by GC-MS.

Material and Methods

Plant material and essential oil extraction

Plant materials, *L. angustifolia* (cvs Hemus, Raya and Yubileina) cultivars were provided from the cultivation areas of Trakya Agricultural Research Institute (Edirne, Turkey). The four-year-old plants were harvested for each cultivar at the flowering time in cloudless sunny weather at midday in June 2019. EOs were extracted from 100 g dry flower samples of each cultivars using Neo-Clevenger apparatus by hydrodistillation for 4 h. Oils were kept in amber vials at -20°C until identified.

Gas chromatography-mass spectrometry analysis

Gas chromatography-mass spectrometry (GC-MS) analyses were performed using a Thermo Scientific Trace 1310 GC-MS system, equipped with HP-5MS capillary column (30 m x 0.25 mm and 0.25 m ID). 20 mg of EOs were solved in 1 ml of acetone and directly injected to GC-MS in 1 µl volumes. Helium (constant flow, 1.2 ml/min) was used as a carrier gas in split mode by 50:1. The injection site and mass transfer line temperature were set at 280°C. The column oven temperature was programmed accordingly: the initial column oven temperature was 60°C, held for 3 min then ramped to 200°C at a rate of 3°C/min and then immediately ramped to 240°C at a rate of 5°C/min and held for 5 min. The mass spectrometer conditions were: the ion source temperature was 280°C and the ionization energy was 70 eV in EI mode. The retention indexes were calculated for all the components using the Van den Dool & Kratz equation (Dool & Kratz, 1963) based on homolog n-alkane series retention times. Wiley and NIST2004 MS libraries were used to confirm the identities of the compounds. The relative peak area percentages of each compound were calculated based on the MS chromatograms. Relative peak area percentages were calculated by multiplying the division of the relevant peak area by the total peak area by 100.

Insect collection and rearing

The population of *S. granarius* population in the Plant Protection Central Research Institute (Ankara, Turkey) stock culture was used in the study. Soft wheat kernels which were stored in a freezer at -18°C for 72 h to eliminate the risk of possible contamination were used for rearing *S. granarius*. Mixed-sex adults were placed in 1 L glass jars and the monitoring of emerged adults was recorded daily. The adults that emerged between 7 and 28 days after first emergence were used in the study.

Fumigant toxicity bioassays

Glass tubes (10 ml) with airtight caps were used in the single concentration for fumigant activity assays. Discs of 10 mm in diameter were cut from Whatman No. 1 filter paper and attached to the glass tube caps with needle. EOs were diluted using acetone 0.1 (v/v) and 10 µl applied to each filter disc with a micropipette. The same amount of acetone was applied to the filter paper in the control groups. The tubes were placed in a fume hood for 5 min to evaporate the acetone, the tubes were closed using a motor creeper with a silicone septic cap afterwards. The tubes were placed in an incubator at 25±2°C and dead insects were recorded after 24 h (Polatoğlu et al., 2013). The insects were considered dead when they did not move when probing with a sable brush. The experiment was set up with a completely randomized design with 18 replicates and five adult insects were used in each replicate. Since 70-100% mortality was detected in the applications of Hemus, Raya and Yubileina EOs, all three EOs were included in the dose-response assays. In order to calculate the LC_{50} and LC_{90} values, bioassays were set up at doses ranging from 0.05 to 0.15 (v/v).

Contact activity bioassays

EOs which were diluted with acetone at a concentration of 0.15 (v/v) applied to the second segment of the dorsal surface of thorax of the insects (1 µl/insect) with a micro applicator (Hamilton Company, PB-

600, Reno, NV, USA) in single-dose contact activity assays. In the control group, the insects were treated with the same amount of acetone. Adult individuals were placed into Petri dishes (6 cm diameter) containing food and were maintained in the dark at 25±2°C and 65% RH in the incubator. After 24 and 48 h, the dead adults were counted. The insects were considered dead when they did not move when probing with a sable brush (Hossain et al., 2019). The experiment was conducted according to completely randomized design with 10 replicates. In each replicate, 20 mixed sexed adult insects were used and the whole experiment was repeated two times.

Repellent activity assays

The method developed by McDonald et al. (1970) was used in order to evaluate the repellent effect of lavender cultivars EOs. For this purpose, 9 cm discs were cut from Whatman No. 1 filter paper. Acetone was applied to half of the filter paper as a control and EOs at 0.05 and 0.25 µl/cm^2 concentrations were separately applied to the other half. The filter papers, which were kept under a fume hood for 5 min to evaporate the acetone, were then fixed to the bottom of Petri dishes and 20 adult individuals were placed in the middle of the filter. The plates were covered with Parafilm to prevent evaporation and were kept at 25±2°C and 65% RH in the incubator. The area where the insects were present was recorded after 2, 4 and 6 h. The experiment was conducted according to completely randomized design with three replications. In each replicate 20 mixed sexed adult insects were used and the whole experiment was repeated two times. The following formula (McDonald et al., 1970) was used to compute the percent repellent efficacy:

$$\text{Repellent activity (\%)} = (Nc - Nt) / (Nc + Nt) \times 100$$

where, Nc is the number of insects in control and Nt is number of insects in respective EOs treatment.

After the calculation of percentage repellent effectivity, it was scored on 5-point scale used by Juliana & Su (1983): 0, 0.1% repellent activity; 1, 0.1-20.0%; 2, 20.1-40.0%; 3, 40.1-60.0%; 4, 60.1-80.0%; and 5, 80.1-100%.

Grain germination assays

Wheat seeds [*Triticum aestivum* L. cv. Eser (Poaceae)] have >95% germination rate obtained from the Field Crops Central Research Institute (Ankara, Turkey) were used in the study. Wheat seeds were surface-sterilized by NaOCl (5%) and ethanol (96%) for 5 and 30 min respectively and rinsed with distilled water. EOs concentrations tested in wheat germination were determined according to LC_{50} values obtained as a result of fumigant activity tests. Accordingly, 0.4l, 0.8l, 1.6l and 3.2 µl/ml doses for Hemus EOs, 0.35, 0.7, 1.4 and, 2.8 µl/ml doses for Raya EOs, and 0.3, 0.6, 1.2 and, 2.4 µl/ml doses for Yubileina EOs were used. Twenty seeds were treated with EOs concentrations in closed 10 ml airtight glass bottles and the lids open after 2 days were kept under a fume hood for 1 day. Seeds placed in Petri dishes containing filter paper moistened with purified water were incubated at 15-20°C at room temperature. After 7 days, the number of germinated seeds was determined. Seeds were considered as germinated when the shoot and root development reached the half-size and the same size of the seed, respectively. The experiment was laid out in a completely randomized design with five replicates, and repeated two times.

Statistical analysis

The mortality data recorded in single-dose assays were converted to percent mortality and then transformed by arcsine transformation technique. One-way analysis of variance was used to test the significance and treatment means were separated by Tukey's multiple comparison test. The statistical analyses were performed using the MINITAB 18 computer program (Minitab Inc., PA, USA). The data from dose-response tests were analyzed using the Polo-PC probit package program and the LC_{50} and LC_{90} values, as well as confidence intervals, were calculated. The GenStat computer program (VSN International, Hemel Hempstead, UK) was used to perform principal component analysis.

Results

GC-MS analysis revealed 25, 20 and 24 compounds in Yubileina, Raya and Hemus EOs, respectively, and these represented 97.8, 97.1 and 97.2% of total EOs, respectively (Table 1).

Table 1. Essential oil components extracted from three lavender cultivars

RT[a]	RI[b]	RI Lit[c]	Essential oils component	Yubileina (% area)	Raya (% area)	Hemus (% area)	RI Lit.	IM[d]
4.98	956	954	camphene	-	0.3	0.3	Asuming et al., 2005	MS[e], RI[f]
5.46	980	981	1-octen-3-ol	0.4	0.4	1.9	Oliveira et al., 2006	MS, RI
5.62	987	988	3-octanone	3.4	2.1	-	Zhao et al., 2006	MS, RI
5.72	992	986	*α*-myrcene	1.0	1.2	1.1	Shang et al., 2002	MS, RI
5.80	996	994	3-Octanol	0.4	-	0.4	Juliani et al., 2004	MS, RI
6.60	1034	1032	limonen	0.6	-	0.8	Mevy et al., 2006	MS, RI
6.68	1037	1038	1,8-Cineole	1.4	1.1	0.5	Jalali-Heravi et al., 2006	MS, RI
6.75	1040	1040	*cis*-ocimene	1.6	1.4	2.1	Oliveira et al., 2006	MS, RI
7.00	1051	1050	*β*-ocimene	1.1	1.4	1.4	Karioti et al., 2003	MS, RI
7.64	1077	1078	linalool oxide	0.7	-	1.8	Liu et al., 2006	MS, RI
8.02	1092	1092	terpinolene	0.7	0.7	1.6	Novak et al., 2001	MS, RI
8.33	1105	1103	linalool	36.0	42.5	28.5	Bouzouita et al., 2003	MS, RI
8.54	1114	1112	1-octen-3-yl acetate	2.0	2.1	2.1	Benzo et al., 2007	MS, RI
9.48	1152	1153	camphor	0.4	-	0.6	Radulescu et al., 2004	MS, RI
10.01	1172	1171	borneol	2.4	-	2.7	Zeng et al., 2007	MS, RI
10.32	1183	1182	4-terpineol	-	-	7.7	Avato et al., 2004	MS, RI
10.54	1191	1192	cryptone	0.7	1.0	0.3	Lazarević et al., 2010	MS, RI
10.65	1195	1196	α-terpineol	5.4	5.5	6,0	Nickavar et al., 2002	MS, RI
11.58	1230	1229	nerol	1.1	0.6	1.3	Mevy et al., 2006	MS, RI
12.32	1255	1257	linalyl acetate	24.2	30,0	23.1	Quijano et al., 2007	MS, RI
13.19	1291	1293	lavandulyl acetate	5.9	3.3	6.6	Saroglou et al., 2006	MS, RI
15.07	1368	1364	neryl acetate	1.7	-	1.7	Saroglou et al., 2006	MS, RI
15.56	1379	1380	geranyl acetate	2.8	1.7	2.4	Bonaïti et al., 2005	MS, RI
16.59	1415	1419	caryophyllene	1.1	1.1	1.1	Benkaci-Ali et al., 2007	MS, RI
17.38	1461	1459	*β*-farnesene	1.2	0.6	-	Kundakovic et al., 2007	MS, RI
18.08	1478	1477	germacrene-D	0.4	0.4	-	Kundakovic et al., 2007	MS, RI
20.51	1590	1592	caryophyllene oxide	1.3	-	1.8	Kundakovic et al., 2007	MS, RI
21.78	1620	1623	*α*-cadinol	-	0.3	-	Pavlović et al., 2006	MS, RI
Monoterpene hydrocarbons				4.9	4.9	7.2		
Oxygenated monoterpenes				88.3	89.0	87.2		
Sesquiterpene hydrocarbons				2.7	2.10	1.1		
Oxygenated sesquiterpenes				1.3	0.3	1.8		
Total				97.8	97.12	97.2		

[a]RT, retention time (min); [b]RI, retention index; [c]RI Lit., RI of the compound at same GC column and similar GC-MS condition; [d]IM, Identification method; [e]MS; mass spectrometry match in database; and [f]RI, comparison of retention index from the literature.

It was concluded that the EOs of Hemus, Raya and Yubileina contain high amounts of linalool and linalyl acetate. It was found that Raya EOs contained 42.5% linalool whereas Yubileina and Hemus EOs contained 36.0 and 28.5% linalool, respectively. Similarly, the highest linalyl acetate content was determined in Raya EOs with 30.0%, followed by Yubileina and Hemus EOs with 24.2 and 23.1% contents, respectively. In addition, lavandulyl acetate content was determined in all three EOs, ranging from 3.33 to 6.59%.

Raya and Yubileina EOs generated a similar composition as pointed in Figure 1. Raya and Yubileina EOs have been closer to the center at the end of the biplot analysis.

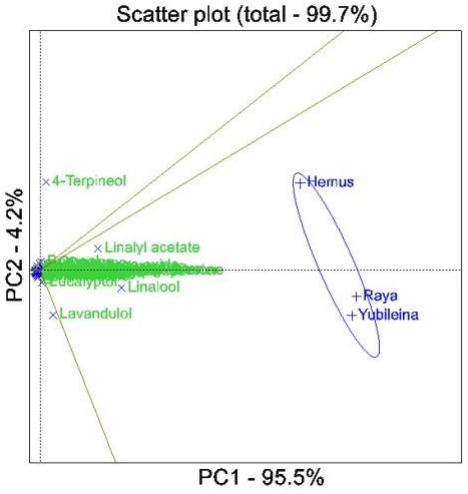

Figure 1. Principal component (PC) analysis of essential oil composition of lavender cvs Hemus, Raya, Yubileina.

As a result of the germination tests, no negative effects of EOs on wheat germination were observed. After 7 days, 100% germination was observed in all treatments.

Although there were significant differences between the EOs and their doses ($P < 0.05$) in terms of contact activity of the EOs evaluated in this study against *S. granarius* adults, the high-level effect was not observed (Table 2). The highest contact activity was determined at the 10% (v/v) application dose of Yubileina EOs with 27.9% ($F = 27.3$; $df = 6,28$; $P < 0.05$) and 62.2% ($F = 51.2$; $df = 6,28$; $P < 0.05$), respectively, after 24 and 48 h, while other EOs and doses did not show useful activity.

Table 2. Contact activities on *Sitophilus granarius* of essential oil obtain from lavender cvs Hemus, Raya, Yubileina

Treatment	Concentration (% v/v)	Mortality (%)±SEM	
		24 HAT	48 HAT
Control		0.0±0.0 c	0.0±0.0 c
Hemus	5	0.0±0.0 c	0.2±0.5 bc
	10	4.3±1.6 b	4.3±1.6 b
Raya	5	0.0±0.0 c	0.0±0.0 c
	10	1.8±0.7 bc	4.7±0.8 b
Yubileina	5	0.0±0.0 c	0.0±0.0 c
	10	27.9±0.1 a	62.2±0.4 a

Means followed by the same letter within a column are not statistically different (ANOVA $P < 0.05$ Tukey's test); HAT, hours after treatment; and SEM, standard error of the mean.

The fumigant effects of EOs isolated from three lavender cultivars showed similar fumigant effect on *S. granarius*, while the highest effect was shown in Yubileina EOs (Table 3). LC_{50} and LC_{90} values were 0.079 and 0.118 µl/ml air with Yubileina EOs and 0.094 and, 0.157 µl/ml air with Hemus EOs, 0.09, 0.139 µl/ml Raya EOs.

Table 3. The results of dose-response assays used to evaluate the fumigant activity of lavender cvs Hemus, Raya and Yubileina essential oils 24 h after treatment against *Sitophilus granarius*

Cutlivar	Slope±SE	LC_{50} (µl/ml air)	95% CI	LC_{90} (µl/ml air)	95% CI	χ2	Heterogeneity
Hemus	5.83±0.56	0.094	0.088-0.101	0.157	0.143-0.176	27.9	0.93
Raya	6.92±0.66	0.091	0.085-0.097	0.139	0.130-0.153	16.4	0.55
Yubileina	7.22±0.63	0.079	0.073-0.084	0.118	0.110-0.129	26.9	0.90

HAT, hours after treatment; SE, standard error; LC_{50}, 50% lethal concentration; LC_{90}, 90% lethal concentration; and CI, confidence interval.

Hemus, Raya and Yubileina EOs were in the same repellent group representing both doses, but the effects varied depending on the application time and dose. Similar to the fumigant effect, the highest average repellent activity was obtained with Yubileina EOs at 70 and 75% at both doses. In Raya and Hemus, 64 and 70% effect were at 0.25 µl/cm^2 dose, respectively, while 66 and 64% at 0.05 µl/cm^2 dose.

Table 4. Repellent effects of essential oil obtain from three lavender cultivars on *Sitophilus granarius*

Cultivar	Dose (µl/cm^2)	RA (%)±SE (95 % CI)			
		2 HAT	4 HAT	6 HAT	Mean (repellency score)
Raya	0.05	74.0±5.1 (52.3-95.7)	56.0±10.3 (34.9-77.1)	62.0±11.6 (44.2-79.8)	64.0 (4)
	0.25	64.0±16.9 (42.3-85.7)	64.0±8.7 (42.9-85.2)	70.0±7.7 (52.2-87.8)	66.0 (4)
Hemus	0.05	74.0±5.1 (52.3-95.7)	64.0±8.1 (42.9-85.2)	72.0±8.0 (54.2-89.8)	70.0 (4)
	0.25	64.0±11.7 (42.3-85.7)	72.0±12.4 (50.9-93.1)	56.0±6.8 (38.2-73.8)	64.0 (4)
Yubileina	0.05	78.0±10.2 (56.3-99.7)	62.0±8.6 (40.9-83.2)	86.0±6.0 (68.2-104)	75.3 (4)
	0.25	74.0±9.3(52.3-95.7)	72.0±12.4 (50.9-93.1)	64.0±10.3 (46.2-81.8)	70.0 (4)

HAT, hours after treatment; RA, repellent activity; and CI, Confidence interval.

Discussion

The main EOs components of lavender were 28.5-42.5% linalool and 23.1-30.0% linalyl acetate, which is consistent with earlier studies (Smigielski et al., 2018; Najibullah et al., 2021). Linalool, linalyl acetate, 1,8-cineole and borneol have already been identified as important components of EOs of various lavender cultivars, although in varying proportions (Cosimi et al., 2009; Fouad et al., 2021). It has been reported that there can be high variability in the component contents of lavender EOs and that some compounds such as linalyl acetate can be detected at highly variable concentrations depending on the production area and cultivar (Dušková et al., 2016). It is also known that biotic and abiotic factors affect the chemical composition of the plant species (Fernández-Sestelo & Carrillo, 2020) and the chemical composition can vary according to the part of the plant used (Smigielski et al., 2018). The other factors affecting chemical composition are the distillation time and method.

There are many studies on the potential use of plant EOs on controlling them against pests (Koul et al., 2008; Lopez et al., 2008). In this study, the possibilities of using lavender cultivars in the control of *S. granarius* were investigated. The biological activities of EOs of lavender species, as well as some of its main components, against stored product pests have been evaluated by different researchers (Al-Ansari et al., 2021; Al-Harbi et al., 2021; Fouad et al., 2021). Considering the results of contact activity, Yubileina EOs, whose main components were determined as linalool and linalyl acetate, showed the highest contact activity at the end of 48 h. However, no significant effect was detected with Hemus and Raya EOs, which have the same main components. This suggests that the activity detected in Yubileina EOs may be due to other compounds. Similar results were obtained with other insect species and EOs. Many researchers found that the same plant EOs or extract from the same genus, as well as various types of insects, react differently to these varying quantities (Guo et al., 2017, Liang et al., 2017).

Lavender cultivars used in this study showed fumigant activity against *S. granarius*, in parallel with the results of other studies against various stored product pests (Al-Ansari et al., 2021; Al-Harbi et al., 2021). Kheloul et al. (2020) reported that *L. angustifolia* EOs showed significant fumigant activity against *S. granarius* and the LC_{50} value was 1.57 mg/l. In addition, it has been reported that the main components of the EOs of *L. angustifolia* are linalool (23.8%), 1,8-cineole (12.0%) and borneol (10.7%). Similarly, the amount of linalool (28.5-42.5%) detected as the main component with a higher area, but 1,8-cineole was not detected in our study. Between the *L. angustifolia* cvs, borneol in Hemus (2.72%) and Yubileina (2.41%) EOs was lower, but not in Raya. Previous studies indicated that linalool or linalool-rich EOs have fumigant activity against stored product insects (Yang et al., 2014; Zhou et al., 2012). Our findings have confirmed these results.

Lavender EOs showed significant repellent activity against *S. granarius* depending on the application dose and time. There are many studies in parallel with this study, in which the effect of repellent activities of EOs obtained from plants in the Lamiaceae with storage pests were investigated (Mishra et al., 2012; Moazeni et al., 2014). The repellent activity of 10 plant EOs, including *L. angustifolia* Mill., was evaluated against *Sitophilus oryzae* (L., 1763) (Coleoptera: Curculionidae) and the EOs of *L. angustifolia* was the oil with the third highest repellent activity (Jayakumar et al., 2017). Similarly, Sabbour & El-Aziz (2020) were evaluated the repellent activity of lavender, *Nepeta* sp., *Geranium* sp. (Geraniaceae) and *Chamaemelum* sp. (Asteraceae) EOs against *Tribolium confusum* Jacquelin du Val, 1863 and *Tribolium castaneum* (Herbst, 1797) (Coleoptera: Tenebrionidae). They showed that the highest repellent activity in geranium EOs and reported that the repellent activity of this oil was followed by the activity of lavender EOs. However, there are conflicting results about which component provides the repellent activity. Fouad et al. (2021) reported that linalool, which was the most abundant component in our study, did not have a significant repellent activity, but eα-pinene, which was not detected in our study, showed significant repellent activity. Linalyl acetate was the second most abundant component detected in this study. Cosimi et al. (2009) was reported that *Citrus bergamia* Risso (Rutaceae) and *Lavandula* hybrid EOs rich in linalool and linalyl acetate showed significant repellent activity against *Sitophilus zeamais* (Motschulsky), 1855 (Coleoptera: Curculionidae). Repellent activity of linalyl acetate was also determined for *Rhodnius prolixus* (Sfara et al., 2009). Germinara et al. (2017) was also reported that the EOs of *L. angustifolia*, in which 23.8% linalool, 6.90% linalyl acetate, 12.0% 1,8-cineole and 10.7% borneol were detected, showed significant repellent activity against *S. granarius*. In our study, the components of the EOs we used were similar in terms of linalool content, while the linalyl acetate content was found at a high level, the borneol content at a very low level and 1,8-cineole was not detected. Obtaining similar results at different component densities suggests that the repellent activity is not caused by a single chemical component in plant EOs, but rather multiple EOs components act together on the repellant activity.

Studies on the use of EOs in controlling agricultural pests have recently gained momentum. In parallel with the high level of toxicity of pesticides used in pest control and increasing consumer awareness, interest in the use of agents derived from natural or natural products is increasing. One of the limiting factors for the use of EOs for pest control is their low stability in open areas and field conditions. The decrease in fumigant activities due to low vapor pressures is another factor limiting the use of EOs. However, this can be eliminated with new formulation studies such as slow-release mechanisms and propellant applications. Studies with EOs are mostly aimed at determining the biological activity of the total EOs composition and these studies will gain more meaning with studies on the determination of the biological activities of the main components and their purification. It is extremely important to increase the number of these studies in order for the biomolecules of EOs, whose effectiveness has been demonstrated, to be an alternative to synthetic fumigants. In this study, the insecticidal and behavioral of EOs obtained from lavender cultivars that can be produced commercially were tested against *S. granarius* under laboratory conditions. The results of the experiment show that these oils have a considerable potential for pest control. However, in order for this potential to be realized, application method and formulation need to be optimized.

References

Al-Ansari, M. M., A. M. Andeejani, E. Alnahmi, R. H. AlMalki, A. Masood, P. Vijayaraghavan & K. C. Choi, 2021. Insecticidal, antimicrobial and antioxidant activities of essential oil from *Lavandula latifolia* L. and its deterrent effects on *Euphoria leucographa*. Industrial Crops and Products, 170: 113740.

Al-Harbi, N. A., N. M. Al Attar, D. M. Hikal, S. E. Mohamed, A. A. H. Abdel Latef, A. A. Ibrahim & M. A. Abdein, 2021. Evaluation of Insecticidal Effects of Plants Essential Oils Extracted from Basil, Black Seeds and Lavender against *Sitophilus oryzae*. Plants, 10 (5): 829.

Aslancan, H. & R. Sarıbaş, 2011. Lavanta Yetiştiriciliği. Meyvecilik Araştırma Enstitüsü Müdürlüğü, Yayın No: 41, 4s. (in Turkish)

Asuming, W. A., P. S. Beauchamp, J. T. Descalzo, B. C. Dev, V. Dev, S. Frost & C. W. Ma, 2005. Essential oil composition of four *Lomatium* Raf. species and their chemotaxonomy. Biochemical Systematics and Ecology, 33 (1): 17-26.

Avato, P., F. Raffo, N. Aldouri & S. Vartanian, 2004. Essential oils of *Varthemia iphionoides* from Jordan. Flavour and Fragrance Journal, 19 (6): 559-561.

Benkaci-Ali, F., A. Baaliouamer, B. Y. Meklati & F. Chemat, 2007. Chemical composition of seed essential oils from Algerian *Nigella sativa* extracted by microwave and hydrodistillation. Flavour and Fragrance Journal, 22 (2): 148-153.

Benzo, M., G. Gilardoni, C. Gandini, G. Caccialanza, P. V. Finzi, G. Vidari, S. Abdo & P. Layedra, 2007. Determination of the threshold odor concentration of main odorants in essential oils using gas chromatography-olfactometry incremental dilution technique. Journal of Chromatography A,1150 (1-2): 131-135.

Bonaïti, C., F. Irlinger, H. Spinnler & E. Engel, 2005. An iterative sensory procedure to select odor-active associations in complex consortia of microorganisms: application to the construction of a cheese model. Journal of Dairy Science, 88 (5): 1671-1684.

Bouzouita, N., F. Kachouri, M. Hamdi & M. M. Chaabouni, 2003. Antimicrobial activity of essential oils from Tunisian aromatic plants. Flavour and Fragrance Journal,18 (5): 380-383.

Cavanagh, H. M. A. & J. M. Wilkinson, 2002. Biological activities of lavender essential oil. Phytotherapy Research, 16 (4): 301-308.

Cosimi, S., E. Rossi, P. L. Cioni & A. Canale, 2009. Bioactivity and qualitative analysis of some essential oils from Mediterranean plants against stored-product pests: Evaluation of repellency against *Sitophilus zeamais* Motschulsky, *Cryptolestes ferrugineus* (Stephens) and *Tenebrio molitor* (L.). Journal of Stored Products Research, 45 (2): 125-132.

Domingues, J., F. Delgado, J. C. Gonçalves & C. S. Pintado, 2021. Essential oils of *Lavandula stoechas* subsp. *luisieri* as antifungal agent against fungi from strawberry tree fruit. Journal of Pharmacy and Pharmacology, 9 : 98-106.

Dool, V. D. & H. Kratz, 1963. A generalization of the retention index system including linear temperature programmed gas-liquid partition chromatography. Journal of Chromatography, 11: 463-471.

Dušková, E., K. Dušek, P. Indrák & K. Smékalová, 2016. Postharvest changes in essential oil content and quality of lavender flowers. Industrial Crops and Products, 79: 225-231.

Fernández-Sestelo, M. & J. M. Carrillo, 2020. Environmental effects on yield and composition of essential oil in wild populations of spike lavender (*Lavandula latifolia* Medik.). Agriculture, 10 (12): 626.

Fouad, H. A., W. de Souza Tavares & J. C. Zanuncio, 2021. Toxicity and repellent activity of monoterpene enantiomers to rice weevils (*Sitophilus oryzae*). Pest Management Science, 77 (7): 3500-3507.

Germinara, G. S., M. G. Di Stefano, L. De Acutis, S. Pati, S. Delfine, A. De Cristofaro & G. Rotundo, 2017. Bioactivities of *Lavandula angustifolia* essential oil against the stored grain pest *Sitophilus granarius*. Bulletin of Insectology, 70 (1): 129-138.

Guo, S. S., W. J. Zhang, C. X. You, J. Y. Liang, K. Yang, Z. F. Geng, S. S. Du & C. F. Wang, 2017. Chemical composition of essential oil extracted from *Laggera pterodonta* and its bioactivities against two stored product insects. Journal of Food Processing and Preservation, 41 (2): e-12941.

Hassiotis, C. N., P. A. Tarantilis, D. Daferera & M. G. Polissiou, 2010. Etherio, a new variety of *Lavandula angustifolia* with improved essential oil production and composition from natural selected genotypes growing in Greece. Industrial Crops and Products, 32 (2): 77-82.

Hossain, F., P. Follett, S. Salmieri, K. D. Vu, M. Harich & M. Lacroix, 2019. Synergistic effects of nanocomposite films containing essential oil nanoemulsions in combination with ionizing radiation for control of rice weevil Sitophilus oryzae in stored grains. Journal of food science, 84 (6): 1439-1446.

Huang, K., D. Zhang, J. J. Ren, R. Dong & H. Wu, 2020. Screening of the repellent activity of 12 essential oils against adult German cockroach (Dictyoptera: Blattellidae): Preparation of a sustained release repellent agent of binary oil-γ-cd and its repellency in a small container. Journal of Economic Entomology, 113 (5): 2171-2178.

Jalali-Heravi, M., B. Zekavat & H. Sereshti, 2006. Characterization of essential oil components of Iranian geranium oil using gas chromatography-mass spectrometry combined with chemometric resolution techniques. Journal of Chromatography A, 1114 (1): 154-163.

Jayakumar, M., S. Arivoli, R. Raveen & S. Tennyson, 2017. Repellent activity and fumigant toxicity of a few plant oils against the adult rice weevil *Sitophilus oryzae* Linnaeus, 1763 (Coleoptera: Curculionidae). Journal of Entomology and Zoology Studies, 5 (2): 324-335.

Juliana, G. & H. C. F. Su, 1983. Laboratory studies on several plant materials as insect repellents for protection of cereal grains. Journal of Economic Entomology, 76 (1): 154-157.

Juliani, H. R., J. A. Zygadlo, R. Scrivanti, E. de la Sota & J. E. Simon, 2004. The essential oil of *Anemia tomentosa* (Savigny) Sw. var. *anthriscifolia* (Schrad.) Mickel. Flavour and Fragrance Journal, 19 (6): 541-543.

Karioti, A., H. Skaltsa, C. Demetzos, D. Perdetzoglou, C. D. Economakis & A. Salem, 2003. Effect of nitrogen concentration of the nutrient solution on the volatile constituents of leaves of *Salvia fruticosa* Mill. in solution culture. Journal of Agricultural and Food Chemistry, 51 (22): 6505-6508.

Kheloul, L., S. Anton, C. Gadenne & A. Kellouche, 2020. Fumigant toxicity of *Lavandula spica* essential oil and linalool on different life stages of *Tribolium confusum* (Coleoptera: Tenebrionidae). Journal of Asia-Pacific Entomology, 23 (2): 320-326.

Kim, N.S. & D. S. Lee, 2002. Comparison of different extraction methods for the analysis of fragrances from *Lavandula* species by gas chromatography-mass spectrometry. Journal of Chromatography, A 982 (1): 31-47.

Koul, O., S. Walia & G. S. Dhaliwal, 2008. Essential oils as green pesticides: potential and constraints. Biopesticides International, 4 (1): 63-84.

Kundakovic, T., N. Fokialakis, N. Kovacevic & I. Chinou, 2007. Essential oil composition of *Achillea lingulata* and *A. umbellata*. Flavour and Fragrance Journal, 22 (3): 184-187.

Lazarević, J., N. Radulović, R. Palić & B. Zlatković, 2010. Chemical analysis of volatile constituents of *Berula erec*ta (Hudson) Coville subsp. *erecta* (Apiaceae) from Serbia. Journal of Essential Oil Research, 22 (2): 153-156.

Leong, W. H., K. S. Lai & S. H. E. Lim, 2021. Combination therapy involving *Lavandula angustifolia* and its derivatives in exhibiting antimicrobial properties and combatting antimicrobial resistance: Current challenges and future prospects. Processes, 9 (4): 609.

Liang, J. Y., W. T. Wang, Y. F. Zheng, D. Zhang, J. Wang, S. S. Guo & J. Zhang, 2017. Bioactivities and chemical constituents of essential oil extracted from *Artemisia anethoides* against two stored product insects. Journal of Oleo Science, 66 (1): 71-76.

Lis-Balchin, M. & S. Hart, 1999. Studies on the mode of action of the essential oil of lavender (*Lavandula angustifolia* P. Miller). Phytotherapy Research, 13 (6): 540-542.

Liu, J., P. Nan, Q. Tsering, T. Tsering, Z. Bai, L. Wang, Z. Liu & Y. Zhong, 2006. Volatile constituents of the leaves and flowers of *Salvia przewalskii* Maxim. from Tibet. Flavour and Fragrance Journal, 21 (3): 435-438.

Lopez, M., M. Jord´an & M. Pascual-Villalobos, 2008. Toxic compounds in essential oils of coriander, caraway and basil active against stored rice pest. Journal of Stored Product Research, 44 (3): 273-278.

McDonald, L. L., R. H. Guy & R. D. Speirs, 1970. Preliminary Evaluation of New Candidate Materials as Toxicants, Repellents and Attractants against Stored Product Insect. Marketing Research Report No. 882. Agricultural Research Service. US Department of Agriculture, 183 pp.

Mevy, J., J. Bessiere, J. Rabier, M. Dherbomez, M. Ruzzier, J. Millogo & J. Viano, 2006. Composition and antimicrobial activities of the essential oil of *Triumfetta rhomboidea* Jacq. Flavour and Fragrance Journal, 21 (1): 80-83.

Mishra, B. B., S. P. Tripathi & C. P. M. Tripathi, 2012. Repellent effect of leaves essential oils from *Eucalyptus globulus* (Mirtaceae) and *Ocimum basilicum* (Lamiaceae) against two major stored grain insect pests of Coleopterons. Nature and Science, 10 (2): 50-54.

Moazeni, N., J. Khajeali, H. Izadi & K. Mahdian, 2014. Chemical composition and bioactivity of *Thymus daenensis* Celak (Lamiaceae) essential oil against two lepidopteran stored-product insects. Journal of Essential Oil Research, 26 (2): 118-124.

Mutungi, C., H. Affognon, A. Njoroge, D. Baributsa & L. Murdock, 2014. Storage of mung bean (*Vigna radiata* [L.] Wilczek) and pigeonpea grains (*Cajanus cajan* [L.] Millsp) in hermetic triple-layer bags stops losses caused by *Callosobruchus maculatus* (F.) (Coleoptera: Bruchidae). Journal of Stored Product Research, 58: 39-47.

Najibullah, S. N. M., J. Ahamad, A. A. Aldahish, S. Sultana & S. Sultana, 2021. Chemical characterization and α-glucosidase inhibitory activity of essential oil of *Lavandula angustifolia* flowers. Journal of Essential Oil Bearing Plants, 24 (3): 431-438.

Nazemi, A. H., G. A. Asadi & R. Ghorbani, 2016. Assessment of the allelopathic potential of extracts of *Lavendula officinalis*. Journal of Plant Protection, 30 (3): 468-480.

Nickavar, B., M. Salehi-Sormagi, G. Amin & M. Daneshtalab, 2002. Steam volatiles of *Vaccinium arctostaphylos*. Pharmaceutical Biology, 40 (6): 448-449.

Novak, J., K. Zitterl-Eglseer, S. G. Deans & C. M. Franz, 2001. Essential oils of different cultivars of *Cannabis sativa* L. and their antimicrobial activity. Flavour and Fragrance Journal, 16 (4): 259-262.

Oliveira, D. R., G. G. Leitao, S. S. Santos, H. R. Bizzo, D. Lopes, C. S. Alviano, D. S. Alviano & S. G. Leitao, 2006. Ethnopharmacological study of two *Lippia* species from Oriximiná, Brazil. Journal of Ethnopharmacology, 108 (1): 103-108.

Pavlović, M., N. Kovačević, O. Tzakou & M. Couladis, 2006. Essential oil composition of *Anthemis triumfetti* (L.) DC. Flavour and Fragrance Journal, 21 (2): 297-299.

Polatoğlu, K., Ö. C. Karakoç & N. Gören, 2013. Phytotoxic, DPPH scavenging, insecticidal activities and essential oil composition of *Achillea vermicularis*. *A. teretifolia* and proposed chemotypes of *A. biebersteinii* (Asteraceae). Industrial Crops and Products, 51: 35-45.

Prashar, A., I. C. Locke & C. S. Evans, 2004. Cytotoxicity of lavender oil and its major components to human skin cells. Cell Prolif, 37 (3): 221-229.

Quijano, C. E., G. Salamanca & J. A. Pino, 2007. Aroma volatile constituents of Colombian varieties of mango (*Mangifera indica* L.). Flavour and Fragrance Journal, 22 (5): 401-406.

Radulescu, V., S. Chiliment & E. Oprea, 2004. Capillary gas chromatography-mass spectrometry of volatile and semi-volatile compounds of *Salvia officinalis*. Journal of chromatography A, 1027 (1-2): 121-126.

Sabbour, M. M. & S. E. A. El-Aziz, 2020. Plant essential oils for the management of two serious stored pests in Egypt. Journal of Entomological Research, 44 (3): 377-384.

Saroglou, V., N. Dorizas, Z. Kypriotakis & H. D. Skaltsa, 2006. Analysis of the essential oil composition of eight *Anthemis* species from Greece. Journal of Chromatography A, 1104 (1-2): 313-322.

Sayout, A., A. Ouarhach, I. Dilagui, N. Soraa & A. Romane, 2020. Antibacterial activity and chemical composition of essential oil from *Lavandula tenuisecta* Coss. ex Ball. an endemic species from Morocco. European Journal of Integrative Medicine, 33: 101017.

Sfara, V., E. N. Zerba & R. A. Alzogaray (2009). Fumigant insecticidal activity and repellent effect of five essential oils and seven monoterpenes on first-instar nymphs of *Rhodnius prolixus*. Journal of medical entomology, 46 (3): 511-515.

Shang, C., Y. Hu, C. Deng & K. Hu, 2002. Rapid determination of volatile constituents of *Michelia alba* flowers by gas chromatography-mass spectrometry with solid-phase microextraction. Journal of Chromatography A., 942 (1-2): 283-288.

Shanker, C. & K. R. Solanki, 2000. Botanical insecticides: A historical perspective. India. Asian Agrihistory, 4 (2): 21-30.

Siddiqui, M., H. H. Siddiqui, A. Mishra & A. Usmani, 2020. Evaluation of cytotoxic activity of *Lavandula stoechas* aerial parts fractions against HepG2 cell lines. Current Bioactive Compounds, 16 (9): 1281-1289.

Singh, C. B., D. S. Jayas, J. N. D. G. Paliwal & N. D. G. White, 2009. Detection of insect-damaged wheat kernels using near-infrared hyperspectral imaging. Journal of stored products research, 45 (3): 151-158.

Smigielski, K., R. Prusinowska, A. Stobiecka, A. Kunicka-Styczyñska & R. Gruska, 2018. Biological properties and chemical composition of essential oils from flowers and aerial parts of lavender (*Lavandula angustifolia*). Journal of Essential Oil Bearing Plants, 21 (5): 1303-1314.

Vairinhos, J. & M. G. Miguel, 2020. Essential oils of spontaneous species of the genus *Lavandula* from Portugal: a brief review. Zeitschrift für Naturforschung C, 75 (7-8): 233-245.

Yakoubi, R., S. Megateli, T. H. Sadok, C. Bensouici & E. Bağci, 2021. A synergistic interaction of Algerian essential oils of *Laurus nobilis* L., *Lavandula stoechas* L. and *Mentha pulegium* L. on anticholinesterase and antioxidant activities. Biocatalysis and Agricultural Biotechnology, 31: 101891.

Yang, K., C. F. Wang, C. X. You, Z. F. Geng, R. Q. Sun, S. S. Guo, S. S. Du, A. L. Liu & Z. W. Deng, 2014. Bioactivity of essential oil of *Litsea cubeba* from China and its main compounds against two stored product insects. Journal of Asia-Pacific Entomology, 17 (3): 459-466.

Zeng, Y. X., C. X. Zhao, Y. Z. Liang, H. Yang, H. Z. Fang, L. Z. Yi & Z. D. Zeng, 2007. Comparative analysis of volatile components from *Clematis* species growing in China. Analytica Chimica Acta, 595 (1-2): 328-339.

Zhao, C. X., X. N. Li, Y. Z. Liang, H. Z. Fang, L. F. Huang & F. Q. Guo, 2006. Comparative analysis of chemical components of essential oils from different samples of Rhododendron with the help of chemometrics methods. Chemometrics and Intelligent Laboratory Systems, 82 (1-2): 218-228.

Zhou, H. Y., N. N. Zhao, S. S. Du, K. Yang, C. F. Wang, Z. L. Liu & Y. J. Qiao, 2012. Insecticidal activity of the essential oil of *Lonicera japonica* flower buds and its main constituent compounds against two grain storage insects. Journal of Medicinal Plants Research, 6 (5): 912-917.