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Original article

Fumigant activity of some plant essential oils and their components against *Stegobium paniceum* (L.) and *Lasioderma serricorne* (F.) (Coleoptera:Anobidae)

Bazı bitki uçucu yağlarının ve bileşenlerinin *Stegobium paniceum* (L.) ve *Lasioderma serricorne* (F.) (Coleoptera:Anobidae)'ye karşı fumigant aktivitesi

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ABSTRACT

In this study, fumigant activity of essential oils obtained from five plants [*Mentha piperita* L. (Lamiaceae), *Micromeria fruticosa* (L.) Druce (Lamiaceae), *Anethum graveolens* L. (Apiaceae), *Salvia officinalis* L. (Lamiaceae) and *Mentha spicata* L. (Lamiaceae)] were tested against *Stegobium paniceum* (L.) and *Lasioderma serricorne* (F.) (Coleoptera: Anobidae) in the laboratory conditions. For this purpose, essential oils obtained by using Neo-Clevenger apparatus were applied at 20% (v/v) concentration, dead and live individuals were recorded at the end of 24 hours. Also, fumigant activities of plant essential oils at different temperatures (10-15-20-25-30 °C) were determined. In addition, fumigant activities of eight essential oil components (menthone, eugenol, carvone, limonene, menthol, dillapiol, methyl-eugenol, linalool) were tested against *S. paniceum*. At the end of the study, *A. graveolens* and *M. spicata* essential oils showed the highest toxicity for *L. serricorne* with 97.63% and the highest toxicity was found in *M. spicata* essential oil with a mortality rate of 97.76% for *S. paniceum*. Among the essential oil components, eugenol had the highest fumigant toxicity of with 60.79% mortality rate. These results indicate that the *M. spicata* essential oil and eugenol could have significant potential as fumigant in the control of *L. serricorne* and *S. paniceum*.

INTRODUCTION

The growth of human population in the world is not parallel with agricultural production area. Therefore, it is very important to protect the quality and quantity of the products in the field and warehouse conditions. This

goal is very important for both developed and developing countries. Synthetic chemicals are used intensively today in the control of stored product pests. However, this excessive use of insecticide leads to problems such as resistance and

residue. One of the synthetic fumigant substances used was methyl bromide and its usage was prohibited under the Montreal Protocol. Studies are being carried out all over the world to find new molecules, especially of plant origin, which may be an alternative to these synthetic chemicals. Nowadays, research on the use of plant extracts in the control of pests causing the loss in agricultural production has gained a significant impetus. In particular, plant essential oils have started to be studied in the all over the world since the 1980s, and these works have continued to increase as important as every day. Essential oils and their constituents are intensively studied materials and tested in laboratory conditions against pest damages, especially against Curculionidae and Bruchidae family pests (Ayvaz et al. 2010, Bachrouch et al. 2010, Eliopoulos et al. 2015, Erler 2005, Karakoç et al. 2006, Keita et al. 2001, Kim et al. 2003, Papachristos and Stamopoulos 2004, Shaaya et al. 1991, Tripathi et al. 2000). Plant essential oils were evaluated aspects of contact activity (Huang and Ho 1998, Huang et al. 2002), fumigant activity (Boukaew et al. 2017, Işıkber et al. 2009, Karabörklü et al. 2011, Maedeh et al. 2012), antifeedant activity (Kiran et al. 2017, Papachristos and Stamopoulos 2002, Prajapati et al. 2005) and some behavioral effect (González et al. 2014, Plata-Rueda et al. 2017, Silva et al. 2017). In this study, essential oils of naturally growing plants and their chemical consistents were evaluated against important stored product pests. In addition, the experiments were based on different storage conditions and the relationship between temperature and toxicity was examined.

MATERIALS AND METHODS

Insect rearing

Insect cultures used in the experiment were obtained from stock cultures of Gaziosmanpaşa University, Faculty of Agriculture, Plant Protection Department. *Lasioderma serricornis* (F.) and *Stegobium paniceum* (L.) were grown in 0.5 liter jars containing whole wheat flour and yeast in $75 \pm 5\%$ humidity and 12:12 dark photoperiod. Adults of mixed sex were kept in the food for 3 days to get adults of the same age, and then the adults were sifted from the diet and the individuals who were used in this environment were created.

Plant materials

Mentha piperita L. (Lamiaceae), *Micromeria fruticosa* (L.) Druce (Lamiaceae), *Anethum graveolens* L. (Apiaceae), *Salvia officinalis* L. (Lamiaceae) and *Mentha spicata* L. (Lamiaceae) were used in the study. The plants used in the experiments were grown at the experimental area

of Gaziosmanpaşa University, Faculty of Agriculture. Harvested plant samples were dried to a constant weight (11-13% humidity) without expose to sunlight environment. Dried plant materials were stored at room temperature, in dark conditions, and in pouch paper until essential oils were obtained.

Essential oils

Plant essential oils were obtained by the previously described method (Telci et al. 2006). According to this method, fifty grams of dry plant material was transferred to a 1000 ml shaker flask and 500 ml of water was added. They were then subjected to hydrodistillation for two hours on a Neo-Clevenger and the essential oils obtained were transferred in glass bottles and stored in a refrigerator at 4 °C until used.

All essential oil components used in the experiments were purchased from Sigma-Aldrich. (menthone CAS Number: 10458-14-7, eugenol CAS Number: 97-53-0, carvone CAS Number: 2244-16-8, limonene CAS Number: 5989-27-5, menthol CAS Number: 89-78-1, dillapiole CAS Number: 484-31-1, methyl-eugenol CAS Number: 93-15-2, linalool CAS Number: 78-70-6.)

Fumigant activity tests of plant essential oils and components

In the experiments of fumigant effect of plant essential oils against *S. paniceum* and *L. serricornis*, the method mentioned in Çam et al. (2012) was used. Experiments were carried out in 10 ml volumetric, rubber-covered, gas-tight glass tubes. For this purpose, 10 adult individuals were left in glass tubes with approximately 5 g wheat/broken wheat. Filter papers cut to a diameter of 10 mm were fixed to the bottom of the rubber covers. Essential oils were diluted with acetone at concentration of 200 µl/L and in a volume of 10 µl were applied to the filter papers with the aid of a micropipette. The acetone used as the solvent was allowed to stand for 5 minutes and then the tubes were sealed. The applied tubes were incubated for 72 hours at 65% relative humidity at 25 °C. Dead individuals at the end of 72 hours were recorded. In addition, the activity of plant essential oils in *S. paniceum* at different temperatures (10-15-20-25-30 °C) has been tested according to the above mentioned method. Some of the major components (menthone, eugenol, carvone, limonene, menthol, dillapiole, methyl-eugenol, linalool) of the *Mentha* species and *A. graveolens* have been evaluated for fumigant activity against *S. paniceum*. Pure acetone was used in the control groups. Experiments were set up in 3 replications according to random block design and repeated 3 different times.

Statistical analysis

The data obtained at the end of the study were first converted to % mortality and then subjected to arcsine transformation. Variance analysis was performed with the obtained data, and in addition, the differences between the treatments were analysed by the Tukey multiple comparison tests. All statistical analysis were carried out using the MINITAB Release 16 package program.

RESULTS AND DISCUSSION

Fumigant activity of essential oils against different stored product pests

When fumigant activities on two important pests of plant essential oils were examined, fumigant activity was observed in oils at varying rates depending on the pest (Table 1). The highest toxicity for the *L. serricornis* was determined with 97.63% mortality rate in the *A. graveolens* and *M. spicata* essential oils. For this pest, *S. officinalis* (91.84%) and *M. piperita* (86.76%) essential oils showed high activity while *M. fruticosa* showed no significant activity. *M. spicata* was showed the highest mortality rate on the *S. paniceum* with 97.76%. *M. piperita* was the second most effective essential oil with 73.37% mortality rate. Mortality rates significantly between the treatments ($F=49.18$; $df=5.12$; $P<0.05$). Some of the synthetic fumigants used today are forbidden due to damage to the environment and toxicity to mammals. Ovicidal, repellent, fumigant, antifeedant and toxic effects of plant essential oils are known (Isman 2006, Nawrot and Harmatha 1994). The plants used in this study are known

to have acaricidal properties (Rajendran and Sriranjini 2008) as well as information on the fumigant activity of *M. piperita* (Kim et al. 2004). Previous studies have demonstrated the activity of this plant's volatile oil against different storage pests and different agricultural pests (Choi et al. 2003, Kumar et al. 2011, Nerio et al. 2010, Oka et al. 2000). It has been reported that 1,8-cineole, menthone, and menthol are identified as the major components in a study of the main components of this plant's essential oil (Rohloff 1999). The fumigant activity and ovicidal activity of these essential oil components, especially menthol and 1,8-cineole, on different stored product pests were demonstrated (Erler 2005). These results are in line with the information obtained as a result of this study. In our study, *M. spicata* and *M. piperita* showed similar results. This is important because it shows that different essential oils and their components have different effects on the same pest, suggesting that the main component of the plant plays an important role in determining its effectiveness. In addition, plant essential oils used in this study had fumigant activity at varying rates against different storage pests. It is thought to be caused by physiological or biochemical differentiation of the insects. In this study, *S. officinalis* showed significant insecticidal activity. Hosseini et al. (2013) tested the fumigant activity of essential oil from *Salvia leriifolia* (Benth) (Lamiaceae) against *Sitophilus granarius* (L.) (Coleoptera: Curculionidea) and *Rhyzopertha dominica* (F.) (Coleoptera: Bostrichidae) and found activity up to 50%. These results are in parallel with our results. In addition, these results indicate that plants entering the

Table 1. Fumigant toxicities on *Lasioderma serricornis* and *Stegobium paniceum* after 24 hours of essential oil applications

Treatment	Mortality (%) \pm SE	
	<i>Lasioderma serricornis</i>	<i>Stegobium paniceum</i>
Control	0.00 \pm 0.00c ¹	0.00 \pm 0.00d
<i>Mentha piperita</i>	86.76 \pm 0.06b	73.37 \pm 0.03b
<i>Micromeria fruticosa</i>	0.56 \pm 0.56c	11.14 \pm 0.33c
<i>Anethum graveolens</i>	97.63 \pm 2.37a	70.00 \pm 0.00b
<i>Salvia officinalis</i>	91.84 \pm 0.10a	70.08 \pm 0.10b
<i>Mentha spicata</i>	97.63 \pm 2.37a	97.76 \pm 0.56a

¹ Different letters in the same column indicate statistically different from each other (Anova $P<0.05$. Tukey test). SE: Standart Error

Table 2. Fumigant toxicity of essential oils on *Stegobium paniceum* at different temperatures at the end of 24 hours

Treatment	Mortality (%) ± SE				
	10 °C	15 °C	20 °C	25 °C	30 °C
Control	0.00±0.00c ¹	0.00±0.00c	4.32±0.07d	1.49±0.37c	0.37±0.37c
<i>Mentha piperita</i>	4.32±0.07bD ²	36.61±0.12bC	35.55±0.00cC	64.60±0.26bB	81.42±0.25aA
<i>Micromeria fruticosa</i>	0.00±0.00cC	0.00±0.00cC	5.98±0.40dB	10.31±0.16cAB	21.09±0.02bA
<i>Anethum graveolens</i>	12.82±0.34bD	39.91±0.26bC	69.52±0.09bB	73.38±0.05bB	95.59±1.22aA
<i>Mentha spicata</i>	63.63±0.58aB	96.73±0.91aAB	85.24±0.17aAB	95.75±1.32aAB	97.63±2.37aA

¹ Different letters in the same column indicate statistically different from each other (Anova P<0.05. Tukey test).

² Different letters in the same line indicate statistically different from each other (Anova P<0.05. Tukey test).
SE: Standart Error

Salvia, *Mentha* and *Anethum* genus have significant fumigant activities against two different pests, with the broad spectrum of substances or substances present in the chemical composition of these plants.

Fumigant toxicities of essential oils on Stegobium paniceum at different temperatures

It has been found that the activity of essential oils has a significant effect on the activity of *S. paniceum* in the study in which the activities at different temperatures are determined (Table 2). *M. spicata* essential oil was showed the highest fumigant activity with 63.63% mortality rate against *S. paniceum* at 10 °C. Mortality rates significantly between the treatments (F=73.67; df=4.10; P<0.05). The highest activity at 15 °C was found in the *M. spicata* essential oil (96.73%) followed by the activity of *A. graveolens* (39.91%) and *M. piperita* (36.61%) essential oils, respectively (F=129.92; df= 4.10; P<0.05). The highest activity was determined at 30 °C. In this temperature, *M. spicata* essential oil was showed the highest fumigant activity with 97.63% mortality rate. The fumigant activity rates were 95.59% and 81.42% for *A. graveolens* and *M. piperita*, respectively (F=41.367; df= 4.10; P<0.05). When the results are examined, it is seen that temperature has significant effects on toxicity. It is observed that toxicity increases due to temperature increase and that 30 °C are reached for all essential oils of plants which are tried to the highest toxicity. When the temperature is increased, the rate of metabolism of the insects increases and accordingly the transition of the active substance or substances to the insect environment is accelerated (Nakakita and Ikenaga

1997). Similarly, Chaudhry et al. (2004) measured the susceptibility of *L. serricornis* to magnesium phosphine at different temperatures and reported that when the temperature was increased from 5 °C to 25 °C fumigant activity was increased. Karakoç et al. (2006) reported that essential oils from six plants were tested against *S. granarius*, *Sitophilus oryzae* (L.) (Coleoptera: Curculionidea) and *Acanthoscelides obtectus* Say. (Coleoptera: Bruchidae). The researchers have reached the highest mortality rate at mortality rate at 30 °C.

Fumigant toxicity of the essential oil components on Stegobium paniceum

In the study in which the fumigant activities on the *S. paniceum* of the essential oil components were determined. The highest activity was found in the eugenol compound with a mortality rate of 93.33% and this component was statistically found in a different group than the other compounds (F = 78.12; df= 9.20; P < 0.05) (Table 3). In parallel with the toxicity results obtained from eugenol, Rozman et al. (2007) found that eugenol showed fumigant toxicity at 85% on *S. oryzae* and 80% on *R. dominica*. In a study conducted by Regnault-Roger and Hamraoui (1995) monoterpenic substances were tested in laboratory conditions against *A. obtectus*. In the study, the researchers have reported that eugenol (approximately LC₅₀ 0.5 mg/L) have fumigant toxicity. Similarly, Ogendo et al. (2008) examined the fumigant activities of some stored product pests of eugenol. In this experiments, LD₅₀ values for *S. oryzae*, *Oryzaephilus surinamensis* (L.) (Coleoptera: Silvanidae), *R. dominica* and *Callosobruchus chinensis* (L.)

Table 3. Fumigant toxicity of pure essential oil components on *Stegobium paniceum*

Treatment	Mortality (%)±SE
control	0.00±0.00g ¹
menthone	50.57±0.23bc
eugenol	93.33±0.00a
d-carvone	25.36±0.21de
limonen	6.74±0.62f
menthol	37.21±0.02cd
dillapiole	28.88±0.02de
methyl-eugenol	60.79±0.45b
linalool	27.20±0.03de

¹ Different letters in the same column indicate statistically different from each other (Anova P<0.05, Tukey test).

(Coleoptera: Bruchidae) were calculated as 16.8 µl/L air, 0.53 µl/L air, 0.50 µl/L air and 0.20 µl/L air, respectively. Other essential oil components have lower activity compared to eugenol. However, the fumigant activity of methy-eugenol was 60.79% and the activity of the menthone essential oil component was 50.57%.

One of the limiting factors in the use of essential oils to control a harmful pest is that the vapor pressures of natural fumigant molecules are very low compared to synthetic fumigants. This situation appears to be a major problem. Such deficiencies are thought to be eliminated by formulation studies. In formulation studies, slow release mechanisms and propellant applications are a potential application tools. Another methodological problem with essential oils is that the most of the work is done with total essential oils and that no active molecule or molecules are detected. In the near future it is thought that these problems can be overcome and that new molecules which are alternative to synthetic fumigants will be synthesized. In this study fumigant activities of essential oils from five different plant species were tested against the two storage pests causing significant damage at the storages in a single-dose screening test in laboratory conditions and it was determined that the fumigant activity of these oils against *S. paniceum* changed depending on the temperature. In addition, fumigant activities of pure essential oil components have been tested against *S. paniceum*.

ÖZET

Bu çalışmada beş bitkiden [*Mentha piperita* L. (Lamiaceae), *Micromeria fruticosa* (L.) Druce (Lamiaceae), *Anethum graveolens* L. (Apiaceae), *Salvia officinalis* L. (Lamiaceae) ve *Mentha spicata* L. (Lamiaceae)] elde edilen uçucu yağların fumigant aktivitesi *Stegobium paniceum* (L.) ve *Lasioderma serricornis* (F.) (Coleoptera: Anobidae)'ye karşı laboratuvar koşullarında test edilmiştir. Bu amaçla Neo-Clevenger aparatıyla elde edilen uçucu yağlar %20 (v/v) konsantrasyonda uygulanmış, ölü ve canlı bireyler 24 saat sonunda kaydedilmiştir. Aynı zamanda bitki uçucu yağlarının fumigant aktivitesi farklı sıcaklıklarda (10-15-20-25-30 °C) belirlenmiştir. Ek olarak sekiz uçucu yağ bileşeninin (menthone, eugenol, carvone, limonene, menthol, dillapiole, methyl-eugenol, linalool) fumigant aktivitesi *S. paniceum*'a karşı test edilmiştir. Çalışma sonunda *A. graveolens* ve *M. spicata* uçucu yağları *L. serricornis* üzerinde %97.63 ile en yüksek toksisiteyi göstermiş, *S. paniceum*'dan ise en yüksek toksisite %97.76 ölüm oranı ile *M. spicata* uçucu yağından elde edilmiştir. Test edilen uçucu yağ bileşenleri arasında eugenol %60.79 ölüm oranı ile en yüksek toksisiteye sahip olmuştur. Bu sonuçlar *M. spicata* ve eugenol'ün *L. serricornis* ve *S. paniceum*'ün mücadelesinde fumigant olarak önemli potansiyele sahip olduğunu göstermiştir.

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