



Chemical composition and fumigant toxicity of essential oils from ten aromatic plants growing in Egypt against different stages of confused flour beetle, *Tribolium confusum* Jacquelin du Val

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Abstract

Essential oils may provide green and safe alternatives to currently used fumigants and insecticides for the management of stored-product insects. This study reports the chemical analysis and fumigant toxicity of 10 essential oils against *Tribolium confusum*. The essential oils were obtained by hydrodistillation. The chemical profile of essential oils was determined by gas chromatography-mass spectrometry. The essential oils consisted mainly of monoterpene hydrocarbons and oxygenated monoterpenes except the oil of *Artemisia monosperema* which was enriched with benzenes. The essential oil of *Schinus molle* ($LC_{50} = 2.22 \mu\text{l/L}$ air) revealed the highest toxicity against *T. confusum* eggs, followed by *Calistemon viminalis* ($LC_{50} = 5.38 \mu\text{l/L}$ air) and *Eucalyptus camaldulensis* ($LC_{50} = 5.80 \mu\text{l/L}$ air) after 7 days of exposure. The toxicity of essential oils against larvae improved significantly with increasing exposure time. The oils *Origanum vulgare* and *Cupressus macrocarpa* caused the highest larval mortality with LC_{50} values of 22.81 and 25.11 $\mu\text{l/L}$, respectively, after 7 days of exposure. Furthermore, *O. vulgare* essential oil was the most potent toxicant against adults after three exposure times displaying LC_{50} values of 5.59, 1.57 and 1.26 $\mu\text{l/L}$ air after 1, 3 and 7 days, respectively. Furthermore, the essential oils of *Citrus paradisi*, *A. monosperema*, *E. camaldulensis* and *S. molle* displayed promising fumigant toxicity against adults. Based on the obtained results, the essential oils of *O. vulgare*, *S. molle*, *C. viminalis*, *C. paradisi* and *A. monosperema* had potent fumigant toxicity against *T. confusum* different stages and they could be applicable for the management of this insect.

Keywords Natural products · Essential oils · Chemical composition · Insecticidal activity · Confused flour beetle

Introduction

Confused flour beetle, *Tribolium confusum* Jacquelin du Val (Coleoptera: Tenebrionidae), is a worldwide stored product insect. It causes extensive loss of stored grain products, such as flour, cereals, beans and spices (Rees 2004; Mediouni Ben Jemaa et al. 2012). The control of stored product insects, including *T. confusum*, relied largely

on the use of synthetic insecticides and fumigants. In spite of that, these fumigants and insecticides have several negative effects, such as resistance development, toxic residues and high application cost (Antonious et al. 2007). To compensate the withdrawal of methyl bromide that was the most effective fumigant for treatment stored products in storage and quarantine but it has been reported to induce ozone depleting effect (Carpenter et al. 2014). In addition, phosphine is widely applied for the management of stored product insects nowadays. However, the resistance of stored-product insects to phosphine becomes evidence in numerous countries (Nayak et al. 2020). Therefore, alternative approaches for stored product insect control are required. Extracts, essential oils and other secondary metabolites of plants have a potential to be used as alternatives for managing stored product insects.

Essential oils are plant secondary metabolites with important roles in plant communication and defense. They are mainly comprised of monoterpenes and

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sesquiterpenes (Pavela 2015). These two main groups of secondary metabolites present in the oils in several subclasses, including ketones, alcohols, esters, aldehydes, lactones and phenols. Essential oils are relatively safe for humans as they commonly added to foods and beverages as fragrances and flavoring additives (Isman 2000). The lipophilicity of essential oils enables their interaction with biochemical and physiological processes of insects. Moreover, essential oils have, in general, low boiling points and are relatively volatiles, therefore, they are commonly used as fragrances, fumigants and repellents (Nishimura 2001). Many researchers highlighted the biological activities of essential oils against insects, including insecticidal, antifeedant, repellent, growth inhibitory and antioviposition effects (Prajapati et al. 2005; Tomova et al. 2005; Carroll et al. 2011; Nenaah 2014; Yang et al. 2020).

The toxicity of essential oil vapors against the adults of common insect pests attacking stored products has been reported (Saroukolai et al. 2010; Mahmoudvand et al. 2011; Koutsaviti et al. 2018; Campolo et al. 2018). In contrary, few studies have described the fumigant toxicity of essential oils against eggs and larvae of these insects (Mondal and Khalequzzaman 2006; Chaubey 2008; Tarigan et al. 2016; Ajayi et al. 2018; Brari and Thakur 2018). Similarly, few reports were found in the literature on the fumigant toxicity of essential oils against *T. confusum* (Tunc et al. 2000; Isikber et al. 2006; Ziaee et al. 2014; Sener et al. 2009). Some of the tested essential oils have been described to possess insecticidal and repellent activities against stored products insects. For example, the essential oil of *Artemisia judaica* showed repellent and residual toxicity against *Callosobruchus maculatus* (Fab.) (Abd-Elhady 2012). Also, Ndomo et al. (2010) found that the essential oil of *Calistemon viminalis* had fumigant toxicity against *Acanthoscelides obtectus* Say and *C. maculatus* with LC_{50} values of 0.011 and 0.019 $\mu\text{l}/\text{cm}^3$, respectively. Moreover, the essential oils of *Citrus paradisi* and *Eucalyptus camaldulensis* have been shown to induce fumigant toxicity against *C. maculatus* (LC_{50} = 125.0 $\mu\text{l}/\text{L}$) and *Sitophilus oryzae* L. (LC_{50} = 17.49 $\mu\text{l}/\text{L}$), respectively (Moravvej and Abbar 2008; Nowrouziasl et al. 2014). It has been also reported the oil of *Origanum vulgare* had repellent and fumigant toxicity against *Ephestia kuehniella* Zellerlarvae (Bouzeraa et al. 2019) and the essential oils of *Schinus molle* leaf and fruit against *Trogoderma granarium* Everts and *Tribolium castaneum* (Herbts) (Abdel-Sattar et al. 2010). However, the fumigant toxicity of the tested essential oils was not reported against the different stages of *T. confusum*. Therefore, this work was undertaken to assess the fumigant toxicity of essential oils isolated from 10 plants grown in Egypt against different stages (egg, larva and adult) of *T. confusum*. Also, the chemical

constituents of essential oils were identified by GC–MS analysis to correlate the chemical composition of essential oils with insecticidal activity.

Materials and methods

Insect culture

A culture of *Tribolium confusum* was reared in the laboratory for several years on wheat flour free from insecticides. The insect was bred on wheat flour and yeast (10: 1, w/w). The culture rearing and bioassay experiments were done at 26 ± 1 °C, $65 \pm 5\%$ Rh and in a 12: 12 light: dark photoperiod.

Plant materials

Leaves of 9 plant species, *Artemisia judaica* L., *A. monosperma* Del., *Calistemon viminalis* (Sol.ex Gaertn.) G. Don, *Cupressus macrocarpa* Hartw. ex Gordon, *Eucalyptus camaldulensis* Dehnh., *Origanum vulgare* L., *Pelargonium graveolens* L'Her, *Schinus molle* L. and *Thuja orientalis* L., and fruit peels of *Citrus paradisi* Macfad were used in this study. The plant samples were collected from three Egyptian Governorates, Matrouh, Albehira and Alexandria. The plant sample collection was carried out during the reproductive stages of plants from April to August, 2013. The taxonomy of plants was confirmed by Prof. FathAllah Zaitoon of Botany Department and plant specimens (AS-1301, AJ-1302, CV-1303, CP-1304, CM-1305, EC-1306, OV-1307, PG-1308, SC-1309 and TO-1310) were kept at Pesticide Chemistry and Technology Department, Faculty of Agriculture, Alexandria University.

Extraction of essential oils

Extraction of essential oils was done via hydrodistillation of selected plant parts (2 kg) for 3 h. in a Clevenger-type apparatus. The leaves were partially dried before extraction, while the fruit peels were extracted without drying. Sodium sulfate anhydrous was used to remove water and the essential oils were stored at 4 °C. in brown glass vials.

Analysis of essential oils

Chemical analyses of essential oils were done on a gas chromatography (Hewlett Packard 5890)/mass spectrometry (Hewlett Packard 5989B) (GC–MS) apparatus. The essential oils were diluted in diethyl ether and 0.5 μl was injected. The GC column was a 30 m (0.25 mm i.d., film thickness 0.25 μm) HP-5MS (5% diphenyl) dimethylpolysiloxane (Agilent) capillary column. The GC conditions were as follows: injector temperature, 240 °C; column

temperature, isothermal at 70 °C and held for 2 min, then programmed to 280 °C at 6 °C/min and held at this temperature for 2 min; ion source temperature, 200 °C; detector temperature, 300 °C. Helium was used as the carrier gas at a constant rate of 1 ml/min. The effluent of the GC column was introduced directly into the ion source of the MS. Mass spectra were obtained in the EI mode with 70 eV ionization energy. The sector mass analyzer was set to scan from 40 to 400 amu for 5 s. The oil components were identified by comparison of their retention indices and mass spectra with the NIST Mass Spectral Library (Adams 2004). Essential oil components are reported as a relative percent of the total oil by peak area.

Fumigant toxicity assay

The fumigant toxicity of tested essential oils towards *T. confusum* different stages (eggs, larvae and adults) was determined by the method described by Abdelgaleil et al. (2009). The fumigation was carried out in one liter glass jars. Whatman No.1 filter papers were cut to small pieces (2×3 cm) and attached to the inner side of caps of fumigation jars. The essential oil volumes (μl) were added on filter paper pieces to give final concentrations of 0.1, 0.5, 1, 5, 10, 20, 40, 80 and 100 μl/L. The jars containing 50 eggs (24 h-old) or 25 third instar larvae or 25 adults (7d-old) of

T. confusum were tightly covered with their screw caps. Similar sets without essential oils were maintained for control treatments. The distance between essential oils and insects was 12 cm. To prevent insects, adults and larvae, from direct contact with essential oils, the jar's neck (inner side) was painted with Vaseline. Each concentration was replicated four times. All experiments were kept under the same insect rearing conditions. The fumigation was carried out for 24 h. Then the dead larvae and adults were recorded after 1, 3 and 7 days from the beginning of treatment, while the dead eggs were recorded after 7 days from the beginning of treatment. Insects were considered dead if their appendages did not move or respond after being touched with a fine metal probe under the microscope. The values of LC₅₀ (μl/L air) of each essential oil after three exposure times were determined from regression lines (Finney 1971) by using software program (SPSS 21.0, Chicago, IL, USA).

Results

Composition of essential oils

The major compounds of essential oils as identified by GC/MS analysis are shown in Table 1. The monoterpene hydrocarbons, limonene (74.29%), α-phellandrene (29.87%) and

Table 1 Major constituents of essential oils extracted from ten Egyptian plant species

<i>Artemisia judaica</i>		<i>Artemisia monosperma</i>		<i>Callistemon viminalis</i>		<i>Citrus paradisi</i>		<i>Cupressus macrocarpa</i>	
Compound	%	Compound	%	Compound	%	Compound	%	Compound	%
β-Thujone	49.83	Capillene	36.86	1,8-Cineole	71.77	Limonene	74.29	Terpinen-4-ol	20.29
Chrysanthenone	10.88	capillin	14.68	α-Pinene	11.47	Linalool	4.61	Sabinene	18.67
α-Thujone	8.21	γ-Terpinene	12.46	α-Terpineol	3.18	Linalool oxide	4.18	β-Citronellol	13.01
1,8-Cineole	4.91	β-Pinene	7.85	Octadecanoic acid	3.08	β-Citral	2.66	γ-Terpinene	7.59
L-Camphor	3.0	cis-Ocimene	3.26	1-Phellandrene	1.30	β-Fenchyl alcohol	1.99	Campher	6.66
Artemisia alcohol	2.20	4-Terpineol	2.59	Terpinen-4-ol	1.22	Nootkatone	1.78	α-Terpinene	4.50
Total compounds	79.03		77.7		92.02		89.51		70.72
<i>Eucalyptus camaldulensis</i>		<i>Origanum vulgare</i>		<i>Pelargonium graveolens</i>		<i>Schinus molle</i>		<i>Thuja orientalis</i>	
Compound	%	Compound	%	Compound	%	Compound	%	Compound	%
1,8-Cineole	45.47	Pulegone	77.45	β-Citronellol	35.92	α-Phellandrene	29.87	α-Pinene	35.49
(-)-Spathulenol	32.37	Menthone	4.86	Geraniol	11.66	β-Phellandrene	21.08	δ-3-Carene	25.42
Bicyclogermacrene	11.20	cis-Isopulegone	2.22	Citronellylformate	11.40	Elemol	13.0	α-Cedrol	9.05
Bicycloelemene	3.27	Piperitenone	2.13	Linalool	9.63	τ-Muurolol	5.35	α-Terpinolene	6.76
(-)-Isolatedene	1.47	dl-Limonene	1.08	(+)-Isomenthone	6.36	γ-Eudesmol	4.48	Limonene	4.91
1-Phellandrene	0.64	β-Myrcene	0.66	σ-Selinene	5.52	σ-Cadinene	3.99	β-Myrcene	2.77
Total compounds	94.42		88.4		80.49		77.77		84.4

α -pinene (35.49%) were the most abundant compounds in the essential oils of *C. paradisi*, *S. molle* and *T. orientalis*, respectively. The oxygenated monoterpenes, β -thujone (49.83%), 1,8-cineole (71.77%), terpinen-4-ol (20.29%), 1,8-cineole (45.47%) pulegone (77.45%) and β -citronellol (35.92%) were the main compounds in the essential oils of *A. judaica*, *C. viminalis*, *C. macrocarpa*, *E. camaldulensis*, *O. vulgare* and *P. graveolens*, respectively. A benzene-type compound, capillene (36.86%), was the major compound in the oil of *A. monosperma*. The results indicated that certain major components, such as 1,8-cineole, limonene, α -pinene, camphor, linalool, β -myrcene and terpinen-4-ol were detected in some essential oils, while other compounds, such as chrysanthenone, artemisia alcohol, capillene, capillin, nootkatone, elemol, τ -muurolol, γ -eudesmol, σ -cadinene (-)-spathulenol and bicyclogermacrene were detected in one essential oil. The compounds found in 10 essential oils belong to four chemical groups: monoterpene hydrocarbons (i.e. sabinene, γ -terpinene, limonene, β -pinene, phellandrene, α -pinene and δ -3-carene); oxygenated monoterpenes (i.e. chrysanthenone, terpinen-4-ol, α -thujone, β -thujone, linalool, camphor, linalool oxide, pulegone, β -citral and β -citronellol); sesquiterpene hydrocarbons (i.e. bicyclogermacrene, bicycloelemene, isodene, σ -selinene and σ -cadinene) and oxygenated sesquiterpenes (i.e. spathulenol, α -cedrol, nootkatone elemol τ -muurolol and γ -eudesmol).

Toxicity of essential oils against eggs of *T. confusum*

The fumigant toxic effect of the ten essential oils expressed as LC_{50} values against *T. confusum* eggs after 7 days of exposure is summarized in Table 2. All essential oils showed a

pronounced fumigant toxic effect against eggs. The essential oil of *S. molle* (LC_{50} =2.22 μ l/L air) revealed the highest toxicity, followed by *C. viminalis* (LC_{50} =5.38 μ l/L air), *E. camaldulensis* (LC_{50} =5.80 μ l/L air), *O. vulgare* (LC_{50} =5.84 μ l/L air), and *A. monosperma* (LC_{50} =7.69 μ l/L air). Similarly, the oils of *T. orientalis*, *A. judaica* and *P. graveolens* caused strong toxicity where LC_{50} values were less than 20 μ l/L air. While the oils of *C. macrocarpa* and *C. paradisi* had less toxicity with LC_{50} values close to 50 μ l/L air.

Toxicity of essential oils against third larval instar *T. confusum*

The LC_{50} values of the 10 essential oils against *T. confusum* third instar larvae within 1, 3 and 7 days exposure are presented in Table 3. The tested essential oils showed variable insecticidal activity. In general, the toxicity of oils improved with increasing exposure time. Among the tested essential oils, 5 oils (*C. paradisi*, *O. vulgare*, *A. judaica*, *T. orientalis* and *C. viminalis*) had obvious toxicity after 1 day of exposure as their LC_{50} values were lower than 100 μ l/L air, with *C. paradisi* oil (LC_{50} =61.34 μ l/L air) causing the highest toxicity. While the other tested oils showed a weak toxicity as their LC_{50} values were more than 100 μ l/L air. After 3 days of exposure, the toxicity of tested essential oils significantly increased except those of *E. camaldulensis*, *C. viminalis*, *P. graveolens*, *C. paradisi* and *S. molle*. The essential oil of *O. vulgare* (LC_{50} =25.18 μ l/L air) revealed the highest toxicity, followed by *C. macrocarpa* (LC_{50} =35.33 μ l/L air) and *A. judaica* (LC_{50} =41.94 μ l/L air). All tested essential oils caused higher mortality after 7 days than after 1 or 3 days

Table 2 Fumigant toxicity of the isolated essential oils against *T. confusum* eggs after 7 days of exposure

Oil	LC_{50} ^a (μ l/L)	95% confidence limits (μ l/L)		Slope ^b \pm SE	Intercept ^c \pm SE	(χ^2) ^d	p ^e
		Lower	Upper				
<i>Artemisia judaica</i>	19.56	12.93	28.81	0.77 \pm 0.13	-1.00 \pm 0.18	1.08	0.583
<i>Artemisia monosperma</i>	7.69	5.23	10.48	0.81 \pm 0.10	-0.71 \pm 0.13	0.33	0.988
<i>Calistemon viminalis</i>	5.38	3.66	7.30	0.94 \pm 0.11	-6.69 \pm 0.13	3.16	0.368
<i>Citrus paradisi</i>	51.99	37.32	78.27	1.02 \pm 0.19	-1.76 \pm 0.31	0.11	0.737
<i>Cupressus macrocarpa</i>	49.96	36.65	72.06	1.10 \pm 0.19	-1.87 \pm 0.31	0.51	0.476
<i>Eucalyptus camaldulensis</i>	5.80	4.69	6.98	1.58 \pm 0.15	-1.21 \pm 0.15	3.18	0.365
<i>Origanum vulgare</i>	5.84	4.33	7.46	1.13 \pm 0.11	-0.86 \pm 0.13	3.32	0.677
<i>Pelargonium graveolens</i>	27.93	21.24	38.48	0.90 \pm 0.10	-1.30 \pm 0.14	1.44	0.838
<i>Schinus molle</i>	2.22	0.70	4.26	0.63 \pm 0.11	-0.23 \pm 0.15	0.607	0.738
<i>Thuja orientalis</i>	11.50	9.12	14.18	1.62 \pm 0.14	-1.72 \pm 0.19	2.37	0.306

^aThe concentration causing 50% mortality

^bSlope of the concentration- mortality regression line \pm standard error

^cIntercept of the regression line \pm standard error

^dChi square value

^eProbability value

Table 3 Fumigant toxicity of the isolated essential oils against *T. confusum* larvae after 1, 3 and 7 days of exposure

Oil	Exposure time (days)	LC ₅₀ ^a (µL/L)	95% confidence limits (µL/L)		Slope ^b ± SE	Intercept ^c ± SE	(χ ²) ^d	p ^e
			Lower	Upper				
<i>Artemisia judaica</i>	1	81.61	73.12	108.13	3.25±0.86	-6.21±1.56	0.15	0.930
	3	41.94	17.19	66.74	1.92±0.19	-3.11±0.33	23.69	0.000
	7	35.71	23.54	46.93	1.96±0.19	-3.05±0.33	8.75	0.068
<i>Artemisia monosperma</i>	1	> 100	-	-	-	-	-	-
	3	61.18	44.59	93.31	1.09±0.19	-1.94±0.31	0.10	0.757
	7	44.46	34.22	58.77	1.34±0.19	-2.21±0.32	0.492	0.483
<i>Calistemon viminalis</i>	1	99.98	88.71	119.89	4.06±0.69	-8.12±1.30	0.003	0.960
	3	99.94	88.16	121.40	3.82±0.67	-7.66±1.26	0.007	0.935
	7	96.0	81.49	122.31	2.67±0.44	-5.29±0.82	0.94	0.331
<i>Citrus paradisi</i>	1	61.34	30.97	134.14	2.03±0.22	-3.63±0.39	27.28	0.000
	3	61.05	56.99	64.70	5.34±0.62	-9.54±1.13	1.35	0.508
	7	44.81	34.40	51.17	3.26±0.62	-5.39±1.14	4.26	0.235
<i>Cupressus macrocarpa</i>	1	> 100	-	-	-	-	-	-
	3	35.53	29.90	42.57	1.40±0.18	-2.18±0.27	4.76	0.313
	7	25.11	9.89	42.76	2.04±0.19	-2.85±0.29	29.58	0.000
<i>Eucalyptus camaldulensis</i>	1	> 100	-	-	-	-	-	-
	3	> 100	-	-	-	-	-	-
	7	> 100	-	-	-	-	-	-
<i>Origanum vulgare</i>	1	70.78	65.05	76.76	4.35±0.63	-8.04±1.18	1.22	0.000
	3	25.18	11.79	47.55	1.67±0.13	-2.34±1.18	27.02	0.000
	7	22.81	9.42	44.45	1.79±0.14	-2.43±0.20	33.55	0.000
<i>Pelargonium graveolens</i>	1	> 100	-	-	-	-	-	-
	3	> 100	-	-	-	-	-	-
	7	91.19	54.45	282.21	0.69±0.18	-1.35±0.30	1.35	0.690
<i>Schinus molle</i>	1	> 100	-	-	-	-	-	-
	3	> 100	-	-	-	-	-	-
	7	55.13	50.55	80.87	1.11±0.19	-1.93±0.31	0.23	0.632
<i>Thuja orientalis</i>	1	98.20	84.09	124.07	2.38±0.35	-4.73±0.63	1.83	0.400
	3	56.86	48.97	62.79	3.76±0.63	-6.59±1.16	1.16	0.281
	7	39.19	13.17	99.32	1.78±0.18	-2.83±0.30	18.31	0.000

^aThe concentration causing 50% mortality^bSlope of the concentration- mortality regression line ± standard error^cIntercept of the regression line ± standard error^dChi square value^eProbability value

except that of *E. camaldulensis*. After 7 days of exposure, the essential oils of *O. vulgare*, *A. judaica*, *C. macrocarpa* and *T. orientalis* revealed the highest fumigant toxicity with LC₅₀ values of 22.81, 25.11, 35.71 and 39.19 µL/L air, respectively.

Toxicity of essential oils against adults *T. confusum*

Toxicity of the 10 essential oils against adults of *T. confusum* after 1, 3 and 7 days of exposure in terms of LC₅₀ values is presented in Table 4. It was clear that the tested essential

oils possessed strong fumigant toxicity against the adults except the essential oil of *P. graveolens*. Also, the toxicity was highly dependent on exposure time. The *O. vulgare* oil had the lowest LC₅₀ values of 5.59, 1.57 and 1.26 µL/L air after 1, 3 and 7 days of exposure, respectively. Among the tested oils, *C. paradisi* oil showed promising fumigant toxicity against adults of *T. confusum* after the 1, 3 and 7 days of treatment with LC₅₀ values of 25.60, 20.03 and 13.54 µL/L air, respectively. Similarly, the essential oils of *A. monosperma*, *E. camaldulensis* and *S. molle* revealed pronounced fumigant toxicity after 3 and 7 days of exposure.

Table 4 Fumigant toxicity of the isolated essential oils against *T. confusum* adults after 1, 3 and 7 days of exposure

Oil	Exposure time (days)	LC ₅₀ ^a (µl/L)	95% confidence limits (µl/L)		Slope ^b ± SE	Intercept ^c ± SE	(χ ²) ^d	p ^e
			Lower	Upper				
<i>Artemisia judaica</i>	1	54.96	47.47	66.07	2.33 ± 0.40	-4.05 ± 0.67	0.92	0.337
	3	34.83	26.37	41.84	1.86 ± 0.38	-2.86 ± 0.63	0.14	0.704
	7	31.49	24.90	36.86	2.35 ± 0.39	-3.52 ± 0.65	0.26	0.611
<i>Artemisia monosperema</i>	1	91.79	68.12	143.56	1.31 ± 0.21	-2.58 ± 0.36	0.69	0.409
	3	18.17	11.80	24.71	1.19 ± 0.19	-1.50 ± 0.29	0.88	0.347
	7	12.91	8.51	17.31	1.45 ± 0.20	-1.61 ± 0.30	0.70	0.403
<i>Calistemon viminalis</i>	1	46.61	29.26	112.70	3.76 ± 0.32	-6.27 ± 0.51	26.89	0.001
	3	37.08	23.01	56.57	3.44 ± 0.31	-5.40 ± 0.50	14.49	0.001
	7	32.46	16.16	65.52	2.59 ± 0.21	-3.91 ± 0.32	43.82	0.000
<i>Citrus paradisi</i>	1	25.60	-	-	3.93 ± 0.33	-5.54 ± 0.51	7.87	0.005
	3	20.03	-	-	3.07 ± 0.26	-3.99 ± 0.38	3.04	0.081
	7	13.54	10.65	16.50	2.29 ± 0.24	-2.60 ± 0.34	0.83	0.361
<i>Cupressus macrocarpa</i>	1	85.99	-	-	1.47 ± 0.27	-3.36 ± 0.44	16.14	0.001
	3	49.14	24.37	699.11	1.90 ± 0.26	-3.21 ± 0.42	13.97	0.003
	7	37.52	35.38	39.57	6.58 ± 0.88	-10.36 ± 1.40	0.50	0.480
<i>Eucalyptus camaldulensis</i>	1	63.38	60.38	66.42	6.44 ± 0.88	-11.61 ± 1.60	1.91	0.386
	3	22.85	1.498	42.64	2.13 ± 0.19	-2.90 ± 0.32	22.94	0.000
	7	20.08	-	-	2.00 ± 0.18	-2.61 ± 0.30	25.67	0.000
<i>Origanum vulgare</i>	1	5.59	3.06	17.08	1.19 ± 0.13	-0.89 ± 0.09	7.51	0.057
	3	1.57	0.49	3.50	1.42 ± 0.13	-0.28 ± 0.07	15.15	0.002
	7	1.26	0.65	2.01	1.52 ± 0.14	-0.15 ± 0.07	7.07	0.070
<i>Pelargonium graveolens</i>	1	> 100	-	-	-	-	-	-
	3	> 100	-	-	-	-	-	-
	7	> 100	-	-	-	-	-	-
<i>Schinus molle</i>	1	46.89	-	-	1.72 ± 0.70	-2.87 ± 0.31	14.48	0.001
	3	22.04	8.56	55.22	1.53 ± 0.14	-2.06 ± 0.20	16.25	0.001
	7	15.79	3.99	36.52	1.30 ± 0.14	-1.56 ± 0.19	13.87	0.003
<i>Thuja orientalis</i>	1	50.93	38.84	63.0	15.97 ± 1.28	-27.25 ± 2.19	11.84	0.003
	3	48.26	24.09	65.64	14.17 ± 1.18	-23.86 ± 2.00	13.83	0.001
	7	47.29	-	-	11.19 ± 1.22	-18.75 ± 2.05	17.87	0.000

^aThe concentration causing 50% mortality^bSlope of the concentration- mortality regression line ± standard error^cIntercept of the regression line ± standard error^dChi square value^eProbability value

Discussion

The results of chemical analysis showed that the major compounds of essential oils were monoterpene hydrocarbons and/or oxygenated monoterpenes except two major components of *A. monosperma* which were benzene-type compounds. The chemical compositions of essential oils extracted from *C. paradisi*, *C. viminalis*, *S. molle*, *C. macrocarpa*, *E. camaldulensis* and *P. graveolens* were similar to those formerly reported on plants collected from other countries (Malizia et al. 2000; Tsiri et al. 2003; Viuda-Martos

et al. 2009; Bendaoud et al. 2010; Boukhatem et al. 2013; Mubarak et al. 2014). On the other hand, the major compounds of *A. judaica*, *A. monosperma* and *O. vulgare* essential oils were different from those reported earlier on the chemical analysis of these oils from other regions of the world (Sahin et al. 2004; Khan et al. 2012; Janackovic et al. 2015). Meanwhile, some of the major constituents of *T. orientalis* essential oil were similar to those of the oil of this plant collected from Tunisia (Amri et al. (2015)). The quantitative and qualitative changes of the compositions of essential oils may be due to numerous factors, such as

environment, plant age, season, location and plant nutritional status as well as sample handling, extraction technique and essential oil analysis method (Ozcan and Chalchat 2006; Harkat-madouri et al. 2015).

Essential oils isolated from plants are among the most studied natural products for their possible use as alternative tools for managing numerous insect pests (Tripathi et al. 2009). Phytochemicals could be used in integrated pest management programs of insects attacking stored products in several ways, such as prevention, detection and control (Lopez et al. 2008). In the current study, the essential oils exhibited promising fumigant toxicity against the different stages of *T. confusum*, particularly eggs and adults. Although essential oils from several plants were studied for their fumigant toxicity against stored product insects (Ogendo et al. 2008; Rajendran and Sriranjini 2008; Saroukolai et al. 2010; Mahmoudvand et al. 2011; Kim et al. 2013; Koutsaviti et al. 2018; Campolo et al. 2018) there were no reported studies on the fumigant toxicity of the tested essential oils against *T. confusum*. The tested oils were more potent toxicants to the eggs than other essential oils, such as *Rosmarinus officinalis* L., *Cuminum cyminum* L., *Pimpinella anisum* L. and *Origanum syriacum* L. which have been evaluated against the eggs of this insect and induced 60, 70, 100 and 70% mortality at 196.9 $\mu\text{L/L}$ air, respectively, after 4 days of exposure (Tunc et al. 2000). The results of current study showed that the oil of *S. molle* ($\text{LC}_{50}=2.22 \mu\text{L/L}$ air) was potent toxicant against *T. confusum* eggs. This finding indicated that this oil is one the most potent natural fumigants, such as the oils of cinnamon and cardamom showing LC_{50} values of 1.05 and 2.92 mg /L air, respectively, against the related insect *T. castaneum* (Herbst) (Tarigan et al. 2016). The toxicity of essential oils to eggs may be due to their effects on the development of nervous system inside the egg (Michaelides and Wright 1997). Instead, the permeability of the chorion and/or vitelline membrane may be changed during embryogenesis facilitating the diffusion of essential oils vapours into eggs. The diffused essential oils may affect vital biochemical and physiological processes which causing egg death (Gurusubramanian and Krishna 1996).

Likewise, the results showed that all essential oils induced pronounced toxic effect against *T. confusum* larvae after 7 days of exposure except the oil of *E. camaldulensis* which had weak toxicity with LC_{50} values greater than 100 $\mu\text{L/L}$ air. Among the tested oils, *O. vulgare* oil ($\text{LC}_{50}=22.81 \mu\text{L/L}$ air) had the strongest fumigant toxicity against *T. confusum* larvae. The tested oils showed higher fumigant toxicity than other oils, such as *Cymbopogon citratus* L. and *Syzygium aromaticum* (L.) Merr. & L. M. Perry evaluated against the larvae of *T. castaneum*. The essential oils of *C. citratus* and *S. aromaticum* caused 30% and 41% larval mortality, respectively, at the fumigation dose of 150 $\mu\text{L/L}$ (Ajayi et al. 2018). Similarly, the tested oils were more toxic than the oils of

Rabdosia rugosa (Wall. ex Benth.) H. Hara and *Artemisia maritime* L. which induced 80.03 and 76.01% mortality, respectively, against 8–10 day-old larvae of *T. castaneum* at 100 $\mu\text{L/ml}$ (Brari and Thakur 2018).

The toxicity results of tested essential oils against *T. confusum* adults revealed that the tested oils possessed potent fumigant activity except *P. graveolens* oil. The oil of *O. vulgare* ($\text{LC}_{50}=1.26 \mu\text{L/L}$ air) had the highest toxicity after 7 days of exposure. This essential oil is one of the most effective natural fumigant toxicants evaluated against other stored product insects, such as ZP51 oil ($\text{LC}_{50}=0.71 \mu\text{L/L}$ air), *Mentha microphylla* C. Kock oil ($\text{LC}_{50}=0.21 \mu\text{L/L}$ air), *Carum copticum* L. oil ($\text{LC}_{50}=0.91 \mu\text{L/L}$ air) and *Artemisia scoparia* Waldst. & Kit. (1.87 $\mu\text{L/L}$ air) (Shaaya et al. 1997; Negahban et al. 2006; Sahaf et al. 2007; Mohamed and Abdelgaleil 2008). In addition, the results showed that the oils of *A. monosperema*, *C. paradisi*, *S. molle*, and *E. camaldulensis* showed outstanding insecticidal activity as fumigants. These oils caused fumigant toxicity against the adults of *T. confusum* higher than those essential oils isolated from *Vitex pseudo-negundo*, *Anethum graveolens*, *Carum carvi* and *Cuminum cyminum* (Sahaf et al. 2008; Kim et al. 2013).

The fumigant toxicity of essential oils within 7 days of exposure varied with insect stage. Generally, eggs were more susceptible to the essential oils than larvae except the oils of *C. macrocarpa* and *C. paradisi*. Similarly, the oils were more potent toxicants against eggs than adults except the oils of *O. vulgare*, *C. macrocarpa* and *C. paradisi*. Moreover, the adults were more susceptible than larvae to essential oils except *C. macrocarpa*, *T. orientalis* and *P. graveolens*. Similar findings were reported by Isikber et al. (2006) on the toxicity of *R. officinalis* and *Laurus nobilis* L. essential oils against life stages of *T. confusum*. They found that *L. nobilis* oil was more effective against egg than adult and larva, while *R. officinalis* oil was more effective against adult than larva. Likewise, the essential oils of *Elettaria cardamomum* (L.) Maton., *Cinnamomum aromaticum* Nees and *S. aromaticum* were more effective fumigants against adults of *T. castaneum* than larvae (Mondal and Khalequzzaman 2006). In contrary, the oils of *Piper nigrum* L., *C. cyminum*, *Anethum graveolens* L., *Myristica fragrans* Houtt., 1774, *Illicium verum* Hook.f., *Trachyspermum ammi* (L.) Sprague and *Nigella sativa* L., were shown to be more toxic to larvae of *Callosobruchus chinensis* (L.) than adults (Chaubey 2008).

The insecticidal activities of many essential oils are mostly attributed to their monoterpenoidal contents (Bakkali et al. 2008). Monoterpenoids are volatile and lipophilic molecules which can penetrate quickly into insect body and interfere with physiological functions. In this study, *O. vulgare* oil was the most effective oil against eggs, larvae and adults. The promising insecticidal activity of this oil may be attributed to its main

component, pulegone which represents 77.45% of oil. In fact, pulegone has been shown to possess strong insecticidal activity against different insect species (Rossi et al. 2011; Herrera et al. 2015). Likewise, the potent fumigant toxicity of *E. camaldulensis*, *C. viminalis* and *C. paradisi* oils could be linked with their main constituents, 1,8-cineole and limonene which are widely known by their insecticidal activities (Abdelgaleil et al. 2009; Abdelgaleil 2010).

Although the modes of toxic effect of essential oils are not completely known several studies showed their neurotoxic actions (Jankowska et al. 2017). Essential oils have been reported to inhibit acetylcholinesterase (AChE) which is one of the most important enzymes in insect nervous system (Liao et al. 2016; Nattudurai et al. 2017). Moreover, the essential oils were shown to inhibit adenosine triphosphatases (ATPases) suggesting that their effects on energy chain reactions (Guo et al. 2009; Campolo et al. 2018). In addition to their inhibitory effects on AChE and ATPases, the essential oils have been described to inhibit GABA receptor and modify the insect octopaminergic system (Enan 2001; Jankowska et al. 2017). These different probable modes of action of essential oils are perhaps the reason for their wide spectrum insecticidal activities.

Conclusion

In conclusion, the present study showed that among the tested essential oils, *O. vulgare*, *S. molle*, *E. camaldulensis*, *A. monosperma*, *C. viminalis* and *C. paradisi* oils have the potential to be alternatives to conventional fumigants for managing population of *T. confusum* in enclosed spaces, such as storage buildings and bins. In addition, the availability of plant essential oils and their common use as flavours in food and drinks give these natural products some advantages to be used in crop protection and pest management.

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Declarations

Conflict of interest The authors declare that they have no conflict of interest.

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