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ORIGINAL RESEARCH ARTICLE

Effect of *Citrus limonum* essential oil against granary weevil, *Sitophilus granarius* and its chemical composition, biological activities and energy reserves

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Abstract

Stored grains could lose substantially their nutritional quality, and consequently its value, between storage and consummation because of insect's infestation. One of the most frequent pests of wheat is the grain weevil (*Sitophilus granarius* L.). Cleverly hidden, the immature stages of this destructive pest are very difficult to identify. Protecting stored grains has become an agricultural challenge. In the present study, the essential oil (EO) distilled from *Citrus limonum* (L.) (Burm. F., 1768) and analyzed by gas chromatography-mass spectrometry (GC-MS) revealed the presence of 47 compounds with (*Z*)-citral (30.74%), dl-limonene (19.81%) and geranyl propionate (16.28%) as major components. In a second series of experiment, its fumigant (using filter paper), repellent (by semi circles joined discs) and residual (persistent insecticidal activity) activities were examined against *Sitophilus granarius* (L.) adults. The total nutrition (carbohydrates and lipids) depletion index (NDI) was also calculated. Residual toxicity and repellency of EO were demonstrated after fumigation against *S. granarius* with a dose-response relationship. The effectiveness of EO was exposure time dependent and its residual activity persisted up to 30 h following mortality. Moreover, data showed that treatment of *S. granarius* adults had adverse effects on the depletion of their energy reserves. *C. limonum* EO declined the lipid and carbohydrate contents in adults. Obtained data prove that botanical essential oils could be considered as potent biorational alternatives to synthetic insecticides for pest control of stored products in a sustainable manner.

Keywords *Sitophilus granarius* · *Citrus limonum* · Gas chromatography-mass spectrometry (GC/MS) · Fumigant toxicity · Repellency · Persistence

Introduction

Economy is continuously harmed by infestation of stored pests (Özberk 2018). Developing countries are mostly affected by crop pests infestation (Hengsdijk and de Boer 2017; Kumar and Kalita 2017). Several recent studies were conducted to decrease these adverse effects and prevent food insecurity (Abass et al. 2018). There are 1663 insect species associated with stored products (Hagstrum and Subramanyam 2016). These pests could change the storage microenvironment, which makes it more likely suitable for the rapid development of fungi and other microorganisms (Hubert et al. 2018).

Usually, producers rely on chemical insecticides such as phosphine to prevent these losses during storage. But their exhaustive use has led to serious problems like insecticide resistance (Pavlidi et al. 2018), environmental pollution and toxicity to humans and non-target organisms (Sarwar et al. 2009). *Rhyzopertha dominica* (Afful et al. 2018), *Tribolium castaneum* (Cato et al. 2019), *Sitophilus zeamais* and *Sitophilus oryzae* have shown resistance to chemical insecticides (Haddi et al. 2018). The use of essential oils (EOs) of aromatic plants in crop protection becomes important because

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of their safety, effectiveness and ecofriendly properties (Polatoğlu and Karakoç 2015; Priya et al. 2016; Jayakumar et al. 2017; Ebrahimifar et al. 2020). The genus *Citrus* (Rutaceae) is represented in Algeria by several species such as *C. limonum* (L.) (Burm. F., 1768), *C. sinensis* (Osbeck, 1765), *C. medica* (L., 1753), *C. reticulate* (Blanco, 1837), *C. aurantifolia* (Swingle, 1913) and *C. aurantium* (L., 1753) (Swingle 1967). The chemical composition and insecticidal activity of different *Citrus* species such as *C. sinensis* and *C. aurantium*, *C. grandis*, *C. medica* and *C. sinensis* have been the subject of several studies (El-Akhal et al. 2014; Abdelgaleil et al. 2015; Akono et al. 2016; Mossa 2016). *Citrus limonum* is recognized for its antifeedant, antimicrobial, antioxidant and antifungal activities (Ali et al. 2017; Ben Hsouna et al. 2017; Gucwa et al. 2018).

The granary weevil, *Sitophilus granarius* (L.) (Coleoptera: Curculionidae), is one of the major stored product insect pests (Kučerová et al. 2003). Moreover, the propagation of this species depends mostly on the transport of infested grains (Plarre 2010). *S. granarius* causes changes in several wheat properties affecting the bread quality characteristics (Keskin and Ozkaya 2015). In Algeria, aluminium phosphide (Phostoxin$^{(R)}$) is commonly used to control infestations. Due to environmental concerns, plant derived products are considered better alternatives to synthetic fumigants (Isikber et al. 2019). By the way, *F. angulata* EO did not affect wheat germination (Ebrahimifar et al. 2020). The aim of this study was a) to determine the chemical composition of *C. limonum* EO and b) to investigate its insecticidal, repellency and residual efficacy against *S. granarius* under laboratory conditions. The energy reserves play important roles in development and reproduction of insects (Howard and Blomquist 2005; Arrese and Soulages 2010; Smykal and Raikhel 2015). Therefore, the third objective of our current study is the evaluation of *C. limonum* EO on main biochemical content (carbohydrates, proteins and lipids) of surviving individual *S. granarius* adult. Obtained data provided a better understanding of the potential of EOs in the management of *S. granarius* during the storage.

Materials and methods

Collection and rearing of insects

Sitophilus granarius colonies obtained from the wheat storage farm in El Aouinet (Tébessa, Algeria) in 2017 (35°52′0“ N and 7°54’0” E; elevation 634 m) have been reared under laboratory conditions (26 ± 1 °C, 65 ± 5% R.H and 12:12 L:D photoperiod) at laboratory of Water and Environment, Larbi Tebessi University (Tébessa, Algeria) in 2 kg capacity kilner jar containing sterilized intact wheat grains of *Triticum turgidum* (50 g). Newly emerged adults (unseparated sex) were used in all experiments.

Plant materials and extraction of essential oil

Natural *C. limonum* was harvested in January, February, and March of 2017, in Tebessa (Northeast Algeria: 35°24′15″ North, 8°07′27″ East) and identified by plant taxonomist Dr. S. Hioune (Department of Biology, Larbi Tebessi University, Algeria). After washing with tap water and shade drying at laboratory temperature, leaves were shade dried, cut into tiny pieces then milled into powder (Grinder Electric Moulinex DJ305110, France). Sample of 50 g were subjected to hydrodistillation for 3 h with 500 mL distilled water using a Clevenger-type apparatus. The obtained oil was collected and dried over anhydrous sodium sulfate and stored in amber bottle and kept at 4 °C until use. The yield based on the dried weight of the samples was calculated (Dris et al. 2017).

Gas chromatography-mass spectrometry analysis

The *C. limonum* EO was analyzed by gas chromatography-mass spectrometry (GC-MS) as previously described (Dris et al. 2017). An HP Agilent 6890 Plus gas chromatograph with a HP-5MS column (30 m × 0.25 mm, 0.25 μm film thickness) was used. The injector temperature was set at 60 °C for 8 min, and then increased to 250 °C at rate of 2 °C/min. The injector and detector temperatures were kept respectively at 250 and 270 °C. Carrier gas was helium, the flow through the column was 0.5 ml/min, and the split ratio was set to 50:1 with injection of 0.2 μl of oil sample. The GC/MS analysis was performed with a Quadrupole mass spectrometer that operated at 70 eV. The identification of components was made based on retention index related to the n-alkane series and then the elution order based on both the index and the reference mass spectra were compared.

Fumigant assay

The fumigant toxicity of *C. limonum* EO against *S. granarius* adults (unseparated sex) (1 to 7d-old) was tested using glass jars (60 mL) (Huang et al. 2000). Pieces of filter paper (2 cm in diameter) were soaked with a series of concentrations of EO (20, 40, 80, 100, 200 and 400 μL/L air), then were attached to the bottom surface of the screw caps of the glass jars. The jars containing 10 insects were closed tightly by their screw caps. All treatments as well as control were repeated five times. Insect mortality rate was recorded after exposure time of 24, 48 and 72 h. After correction of observed mortality (Abbott 1925) (Mc = [(Mo-Mt) / (100-Mt)]*100 with Mc: Corrected mortality rate, Mo: Mortality rate in treated series, Mt: Mortality rate observed among control series), sublethal and lethal concentrations (LC_{10}, LC_{25} and LC_{50}) with their 95%

confidence interval (CI) and the slopes were determined (Swaroop et al. 1966). Four replicates each containing 25 individuals have been used for each concentration.

Repellency assay

The repellent activity of the EO against adults of *S. granarius* was evaluated using the procedure of McDonald et al. (1970). Briefly, the filter paper discs of 9 cm diameter have been cut into two semi circles. Four doses diluted with acetone were prepared (1, 2, 4 and 8 µL/mL). After that, 0.5 ml of each solution was spread uniformly over one-half of the disc. Once the solvent has evaporated, the two semi circles were joined using duct tape, and then placed in a box. A batch of 10 adult insects was placed in the center of each disk. The number of insects on the part of filter paper treated with essential oil (P) and the number of those present on the treated only with acetone (G) part were recorded after 15 and 30 min. The percentage of repulsion (RP) was calculated using the following formula:

$$PR = [(P-G)/(P+G)] \times 100$$

The repellent effect categorization of EO was estimated as described (Tapondjou et al. 2005; Ebrahimifar et al. 2020) and five groups were formed based on the mean of percent repellency (PR):

Class 0: PR = 0–0.1%; Class I: PR = 0.1–20%; Class II: PR = 20.1–40%; Class III: PR = 40.1–60%; Class IV: PR = 60.1–80%, and Class V: PR = 80.1–100%.

Residual activity assay

Experiments were designed to test the persistent insecticidal activity of tested EO. The assay was performed according to Ngamo et al. (2007). The LC_{50} of EO was applied on Whatman filter paper (No.1) discs (2.5 cm diameter) in plastic vials (2.5 cm diameter). Six hours later, 10 adults (unseparated sex aged 1 to 7 days old) were introduced separately into vial. The mortality was recorded until no mortality was observed in 6 h intervals. Four replications, each containing ten individuals, were conducted for each time interval.

Extraction and estimation of energy reserves

Proteins, carbohydrates and lipids were extracted in 1 mL of trichloracetic acid (20%) (Shibko et al. 1966) and quantified as previously described (Hamaidia et al. 2018). In brief, quantification of proteins was carried using the Coomassie Brilliant Blue G-250 dye-binding method (Bradford 1976). Carbohydrates and lipids were determined following the anthrone method (Duchateau and Florkin 1959) and the vanillin method (Goldsworthy et al. 1972), respectively. The contents were expressed as µg per individual. The values of carbohydrate, lipid and protein in µg were converted into joules (Clements 1992). The total nutrition (carbohydrates + lipids) depletion index (NDI) was calculated as follows:

$$NDI = [(C-T)/(C+T)] \times 100.$$

Where: **C** is the control total energy reserve and **T** is the total energy reserves present in treated adult.

The **NDI** is considered important when it is greater than 75%, moderate when it is between 50 and 75%, and low when it is less than 50%.

Data statistical analysis

Data are presented as the mean ± standard error mean (m ± SEM). The normality of data was verified using the Kolmogorov-Smirnov test, and the homogeneity of variances was checked by Levene's test. Repetitions and numbers of individuals were also cited. One-way analysis of variance (ANOVA at $P \leq 0.05$) followed by a post-hoc honestly significant difference (HSD) Tukey's test were used to compare between the different series. All statistical analyses were performed using MINITAB Software (Version 16, PA State College, USA) and $p \leq 0.05$ was considered to be a statistically significant difference.

Results

Yield and chemical composition

The oil yield of *C. limonum* by weight was 0.94%. The chemical composition by GC-MS (Table 1) revealed 47 compounds with total percentage of 99.84%. Three major components were identified: (*Z*)-citral (30.74%), dl-limonene (19.81%) and geranyl propionate (16.28%).

Fumigant toxicity

Table 2 shows the corrected mortality of *S. granarius* after exposure to different concentrations of the tested EO. The highest mortality was observed at 400 µL/L air concentration of *C. limonum* at 24, 48 and 72 h after treatment. Highest mortality (92.52%) was reached at 72 h after exposure at 400 µl/L air. All differences between used concentration were significant (24 h series $P = 0.001$; 48 h: $P < 0.001$; 72 h: $P < 0.001$) using Tukey's test. After fumigation tests, sublethal and lethal concentrations (LC_{10}, LC_{25} and LC_{50}) of the EO with their respective confidence intervals (95%) are calculated and given in Table 3. For each exposure period LC_{10}, LC_{25} and LC_{50} were determined. Data probit analysis demonstrated that concentrations of 1630 µl/L air, 160.69 µl/L air

Table 1 Chemical composition of *C. limonum* oil, their retention time (Rt) and concentration percent of constituents

N.	Compound	Rt (min)	Percent
1	2-hexenal	6.705	0.05
2	α-pinene	10.648	0.32
3	bicyclo[3.1.1]heptanes, 6,6-dimethyl-2-methylene	13.380	6.96
4	6-methyl-5-hepten-2-one	14.094	0.47
5	ß-myrcene	14.296	0.63
6	l-phellandrene	15.106	0.06
7	Δ.3-carene	15.487	0.30
8	**dl-limonene***	17.116	**19.81**
9	ß-trans-ocimene	17.568	1.53
10	γ-terpinene	18.923	0.13
11	cis-sabinene hydrate	19.564	0.04
12	α-terpinolene	21.005	0.10
13	linalool	22.089	1.43
14	nonanal	22.321	0.23
15	cis-limonene oxide	24.292	0.04
16	trans-limonene oxide	24.619	0.06
17	methanone	25.217	0.09
18	citronella	25.935	2.27
19	7-methyl-bicyclo[2,2,1]heptane	26.673	0.33
20	terpinene-4-ol	27.439	0.20
21	cyclohexane,ethenyl	27.979	0.45
22	α-terpineol	28.480	0.44
23	decanal	29.559	0.07
24	nerol	31.800	6.02
25	**(Z)-Citral***	32.591	**30.74**
26	geraniol	33.608	1.97
27	undecanal	36.611	0.07
28	methylester	37.734	0.11
29	cis-2,6-dimethyl-2,6-octadiene	39.709	0.53
30	**geranyl propionate***	40.818	**16.28**
31	trans-methanol	41.204	0.05
32	camphene	41.396	0.04
33	geranyl acetate	41.893	4.56
34	trans-caryophyllene	43.777	1.63
35	α-bergamotene	44.770	0.22
36	α-humulene	45.806	0.13
37	butanoic acid	46.023	0.06
38	pyrrolidine	46.871	0.04
39	bicyclogermacrene	48.447	0.20
40	ß-bisabolene	49.252	0.40
41	Δ.cadinene	50.086	0.03
42	farnesol	52.539	0.04
43	(−)spathulenol	53.266	0.20
44	(−)-caryophylene oxide	53.570	0.34
45	isospathulenol	56.713	0.02
46	α-cadinol	57.648	0.09
47	α-bisabolol	59.368	0.07

*major components are highlighted in bold

and 105.80 μl/L air, recorded 50% mortality after 24 h, 48 and 72 h respectively. It has been observed that the effectiveness of EO was time-dependent.

Repellent activity

As shown in Table 4, the findings revealed the repellency (%) effect of EO against *S. granarius* adults depended on concentration and exposure time. The maximum repellency rate was 70% with 8 μL/mL at 30 min.

Effect on energy reserves

Concerning total energy (Table 5), results from ANOVA showed a significant decrease in the treated series with both tested concentration LC_{25} and LC_{50} as compared to control series at 24 h ($F_{2,\ 6} = 7.07$, $P = 0.026$), 48 h ($F_{2,\ 6} = 20.32$, $P = 0.002$) and 72 h ($F_{2,\ 6} = 26.90$, $P = 0.001$) respectively. However, comparison by Tukey test indicated no significant difference between the two tested concentrations (LC_{25} vs LC_{50}: 24 h: $q = 0.208$, 48 h: $q = 2.839$, 72 h: $q = 0.476$).

Effect on whole body protein contents

Changes in main biochemical components (proteins) were determined in whole body of *S. granarius* adults at different times of exposure (24, 48 and 72 h) to tested concentrations (LC_{25} and LC_{50}) (Table 6).

ANOVA revealed a significant effect of treatment ($P < 0.001$) in the protein contents in both treated series (LC_{25} and LC_{50}) as compared to controls during all tested exposure time with a concentration-response relationship (24 h; $F_{2,\ 6} = 1697.60$, $P < 0.001$, 48 h; $F_{2,\ 6} = 259.94$, $P < 0.001$, 72 h; $F_{2,\ 6} = 462.45$, $P < 0.001$). Furthermore, Tukey test indicated a significant difference between the three series ($q > 4.339$).

Nutrition depletion index

Nutrition depletion index (NDI %) in treated and untreated adults was determined in order to investigate the effectiveness of *C. limonum* EO (Table 7). Treatment had no significant effect ($P > 0.05$) on this parameter during the same exposure time. According to ANOVA, the decrease in NDI was concentration-dependent with a maximum depletion in LC_{50} treated series ($F_{2,\ 6} = 13.81$; $P = 0.005$). *C. limonum* EO induced a low nutritional depletion. In addition, Tukey test indicated a significant difference between the three series (24 h vs 48 h: $q = 7.890$; 24 h vs 72 h: $q = 7.745$).

Residual activity

Up to 30 h of exposure; the toxicity of *C. limonum* EO decreased with time (Fig. 1); after 6 h its toxicity was 38% and decreased to 14% after 24 h to disappear after 30 h of exposure.

Table 2 Fumigant effects (mean ± SEM) of essential oil of *C. limonum* on adult of *S. granarius*: corrected mortality

Time (h)	20 μL/L air	40 μL/L air	80 μL/L air	100 μL/L air	200 μL/L air	400 μL/L air	*P* value
24 h	5.00 ± 5.00a	7.50 ± 3.75b	10.00 ± 0.00c	17.50 ± 3.75d	22.50 ± 3.75e	27.50 ± 7.50f	0.001
48 h	15.00 ± 5.00a	20.00 ± 5.00b	32.50 ± 7.50c	37.50 ± 3.75d	52.50 ± 3.75e	75.00 ± 7.50f	<0.001
72 h	22.50 ± 3.75a	32.50 ± 12.50b	37.50 ± 7.50c	42.50 ± 3.75d	60.00 ± 5.00e	92.50 ± 3.75f	<0.001

In the same exposure time, different letters denote a significant difference between corrected mortality (HSD Tukey test ($P = 0.05$)

Discussion

The dry matter yield of *C. limonum* EO was 0.94% (dry weight). This yield is lower compared to 1.02% (Hamdani et al. 2015) and 3% (volume/fresh weight) (Ben Hsouna et al. 2017*)*. Several climatic factors can influence EO yield (Shams et al. 2016). Thus, these differences between yields observed may be due to the arid climate of Tebessa. Also, the yields in *Citrus* EOs vary between 0.28 and 0.45% depending on species (Javed et al. 2014). Indeed, studies reported yield values ranging from 0.2 to 1.30% for the essential oil of *Citrus* (Bourgou et al. 2012; Abdelgaleil et al. 2016). Seasons, plant part and age, geographical regions and method of extraction influence essential oil characteristics (Fathi and Sefidkon 2012; Rocha et al. 2014; Verma et al. 2015; Dosoky et al. 2016; Da Silva et al. 2017). About 47 compounds were identified in *C. limonum* EO by GC/MS with (*Z*)-citral (30.74%) as major component. It has been reported that the lemon species are distinguished by the presence of β-pinene (21.2%), γ-terpinene (17.4%), α-pinene (9.8%) (Vekiari et al. 2002). In another study, EO of *C. limon* grown in Iran contained 17 compounds, the main ones being: limonene (73.25%), α-pinene (8.44%), α-terpinene (6.21%) and geraniol (2.53%) (Saeidi et al. 2014). *Citrus* EOs contained more volatile (a mixture of monoterpene, sesquiterpene hydrocarbons, aldehydes, ketones, acids, alcohols and esters) than non-volatile compounds (Djenane 2015; Abdelgaleil et al. 2016; Sajid et al. 2016).

The insecticidal constituents of EOs are mainly monoterpenoids (Ahn et al. 1998) which gives them significant acute fumigant and contact toxicity (Campolo et al. 2014; Jayakumar et al. 2017), repellent (Wu et al. 2019), antifeedant (Ali et al. 2017), and development and growth inhibitory activities to insects (El-Sabrout et al. 2018). Essential oils distilled from aromatic plants have been the subject of many recent studies on stored products pest (Campolo et al. 2018; Francikowski et al. 2019; Ebrahimifar et al. 2020). Inhalation, ingestion and skin absorption are the main penetration pathways in insect body**.** As result of their high volatility, in the fumigation application, they act by penetrating through respiratory tract of the target. Our results indicate that fumigation of *C. limonum* EO showed an insecticidal activity against adult of *S. granarius* with concentration and exposure time dependence. This dependence was revealed by Haouas et al. (2012). Fumigant potential of some botanical essential oils has been investigated against pests of stored products (El Makarem et al. 2017; Idouaarame et al. 2018; Abd El-Salam et al. 2019). Şimşek et al. (2016) found that essential oils of *Achillea millefolium* (Asteraceae) and *Prangos ferulacea* (Apiaceae) have fumigant toxicity against *S. granarius* and *S. oryze*. Abdelgaleil et al. (2015) reported toxic effects of various Egyptian essential oils on *Tribolium castaneum* and *Sitophilus oryzae*. Repellent effects of *Ferulago angulata* EO have been exhibited against *T. castaneum* and *Rhyzopertha dominica* using three different techniques of filter paper, leaky glass, and olfactometry (Ebrahimifar et al. 2020**)**. Moreover, in real storage conditions, *O. basilicum* EO combined with diatomaceous earths significantly reduced *Sitophilus granarius* infestation on wheat (Pierattini et al. 2019). Furthermore, EOs or some of their components such as monoterpenoids exhibit an insecticidal activity by inhibiting AChE activity on *Sitophilus* species (López and Pascual-Villalobos 2010; Polatoğlu et al. 2016, 2017).

Table 3 Lethal and sublethal concentrations of *C. limonum* EO against adult of *S. granarius*

Time	Hill slope	R^2	Sublethal and Lethal concentrations (μl/L air)		
			LC_{10} (LCL-UCL)	LC_{25} (LCL-UCL)	LC_{50} (LCL-UCL)
24 h	0.64	0.94	52.91 (25.00–87.80)	293.76 (214.10–505.20)	1630 (830.10–6866)
48 h	0.99	0.98	17.63 (11.08–27.20)	53.21 (39.62–68.01)	160.69 (134.60–195.50)
72 h	1.01	0.89	12.07 (0.90–38.35)	35.73 (8.80–72.80)	105.80 (60.70–184.50)

LCL Lower confidence Limit, *UCL* Upper confidence Limit

Table 4 Repellent percentage (RP %) of different concentrations of *C. limonum* EO against adults of *S. granarius* at different exposure times

Exposure time (min)	Concentrations (µl/ml)	RP (%)	Class
15	1	30 ± 10	II
	2	45 ± 7.5	III
	4	55 ± 7.5	III
	8	65 ± 7.5	IV
30	1	35 ± 7.5	II
	2	50 ± 10	III
	4	70 ± 10	IV
	8	70 ± 10	IV

Because of their repellent efficacy, volatile essential oils were considered as excellent materials against stored grain pests and had a significant use in the traditional postharvest storage (Mishra et al. 2012). The present study revealed a repellent activity of *C. limonum* EO against *S. granarius* with all tested concentrations even the lowest which was not high enough to cause significant fluctuations at the physiological level. A study proved that EO at sub-lethal concentration behaved as repellent to pest (Rahayu and Mairawita 2018). It was suggested that exposure to EO may alter insects behavior and by consequence may affect exposure and insecticidal efficiency (Francikowski et al. 2019). In the same context, Wang et al. (2014) showed a repellency of 66.5, 80.3, 71.5 and 90.4% of *C. limonum, Litsea cubeba, Cinnamomum cassia* and *Allium sativum,* respectively against *Alphitobius diaperinus* (Coleoptera: Tenebrionidae). Previous studies have proven the repellent activity of EOs against stored product pests (Chen et al. 2018; Francikowski et al. 2019). Similar observations have been made after application of certain plant extracts which have revealed significant repellent effects on several insect pests: such as *Pimpinella anisum, Cinnamomum zeylanicum, Cymbopogon nardus, Eucalyptus globulus, Pelargonium graveolens, Lavandula officinalis, Rosmarinus officinalis, Vetiveria zizanioides* (Jayakumar et al. 2017).

Table 5 Lethal and sublethal effects of *C. limonum* EO on total energy (joules/individual) at different times in adults of *S. granarius* (mean ± SEM; n = 3 pools each containing 10 individuals)

Exposure time (hours)	Control	LC_{25}	LC_{50}
24	8.30 ± 0.17 a	6.73 ± 0.15 b	6.66 ± 0.71 b
48	10.62 ± 1.67 a	6.01 ± 0.28 b	3.82 ± 0.48 c
72	10.28 ± 1.48 a	4.11 ± 0.53 b	3.77 ± 0.26 b

The means ± SEM followed by different letters in each row are statistically statistically different at $P \leq 0.05$ (Tukey test)

Table 6 Effect of fumigation of *C. limonum* EO on protein contents (joules /individual) at different times in the adults of *S. granarius* (mean ± SEM; *n* = 3 pools each containing 10 individuals)

Exposure time (hours)	Control	LC_{25}	LC_{50}
24	3.61 ± 0.04 a	0.99 ± 0.07 b	0.49 ± 0.03 c
48	3.61 ± 0.05 a	1.31 ± 0.14 b	0.86 ± 0.11 c
72	3.61 ± 0.00 a	1.36 ± 0.12 b	1.14 ± 0.05 c

The means ± SEM followed by different letters in each row are statistically different at $P \leq 0.05$ (Tukey test)

Green pesticides present a safer alternative to synthetic insecticides in agriculture and public health sectors. Nevertheless, despite rigorous studies on plant-based products, the main type of botanical insecticide registered and commercialized successfully is neem seed extract (azadirachtin) (Isman et al. 2011). The chemical composition of an EO as well as the target sensitivity controlled the duration of its insecticidal activity (Obeng-Ofori et al. 1997). Monoterpenes are the most active compounds that maintain essential oil insecticidal efficiency (Ngamo et al. 2007). Because of their high volatility, the activity of EOs decreased with exposure time. In the present study, lethal effect of *C. limonum* EO persisted up to 30 h. Also, lethal concentrations (LC_{50} and LC_{90}) of *Thuja orientalis* EO were decreased by increasing the exposure periods against *Sitophilus granarius* (Hamza et al. 2016). But, enhancing concentrations and exposure times increased the repellency rates of *F. angulata* EO (Ebrahimifar et al. 2020).The speed of the oxidation of hydrogenated monoterpenes is greater for compounds as sabinene and 1,8 cineole (Kim et al. 2003). But volatility of EOs and other few limitations as their photosensitivity and volatilization restricts their shelf life making difficult their use on a large scale (Campos et al. 2016). New formulations with nanotechnology could improve their stability and sustainability with a considerable perspective as commercial insecticide products (Pasquoto-Stigliani et al. 2017; Athanassiou et al. 2018; Bipin 2019).

Table 7 Nutrition Depletion Index (%) of *S. granarius* adult after treatment with *C. limonum* EO at 24, 48 and 72 h (mean ± SEM; *n* = 3 pools each containing 10 individuals)

Exposure time (hours)	LC_{25}	LC_{50}
24	10.44 ± 0.95aA	11.27 ± 6.21aA
48	26.83 ± 8.57aA	46.48 ± 6.36aB
72	42.68 ± 4.33aB	45.84 ± 3.22aC

(The means ± SEM followed by different capital letters in each column and small letters in each row are statistically different at $P \leq 0.05$ (Tukey test)

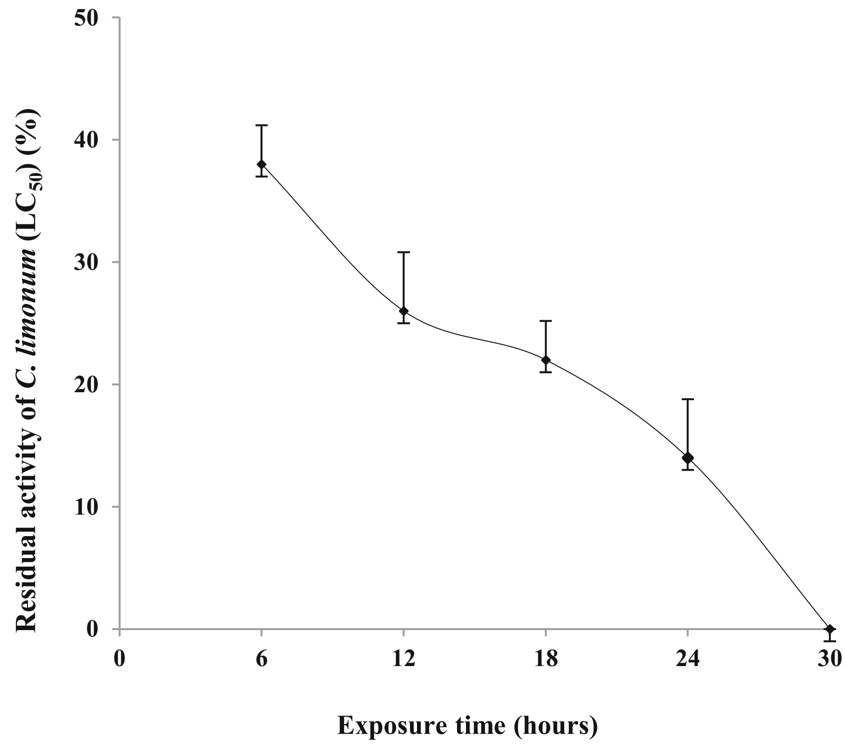

Fig. 1 Residual activity of *C. limonum* essential oil (LC_{50}) against *S. granarius* adults (mean ± SEM, *n* = 4 pools each containing 10 individuals)

In insects, biochemical fluctuations were associated with development stages (Cohen 2010; Sugumaran 2010). The energy reserve levels were high during larval instars, then decrease at the beginning of wandering (Phalaraksh et al. 2008; Mishra et al. 2010). In insect, various physiological processes (nymphosis, molt, metamorphosis…) controlled these fluctuations (Zhou and Miesfeld 2009). The mobilization of energetic reserves is important particularly during flight, embryogenesis, starvation and immune response (Arrese and Soulages 2010). Also they have semiochemical roles in number of social insects, viz. dominance and fertility cues (Howard and Blomquist 2005). Beside development, they play a key role in female reproductive events by regulation of sufficiency of nutrients for developing eggs and secretion of juvenile hormone and ecdysone (Smykal and Raikhel 2015).

Carbohydrates are considered as important energy elements playing a crucial role in the insect physiology, such as the molt process and the reproduction (Kaufmann and Brown 2008). The carbohydrate content was reduced in larvae of *Spodoptera littoralis* after treatment with essential oil of *Azadirachta indica* and *Citrullus colocynthis* methylene chloride extract and was increased with garlic and lemon EOs (Ali et al. 2017). Lipids are also an important source of acetyl groups needed to synthesize the enzymes from constitutive amino acids (Rivero et al. 2010). In the present study, *C. limonum* EO decreases the lipid contents in *S. granarius* adults. A reduction of total lipids in *S. littoralis* larvae treated with garlic and lemon oils was noted (Ali et al. 2017). Dris et al. (2017) and Bouguerra et al. (2018) also reported a decrease in the total protein, lipid and carbohydrate contents in *Culex pipiens* mosquito treated with *O. basilicum* and *Thymus vulgaris* essential oils respectively. The decline of lipid levels might be due to the effect of these oils on the mobilization of lipid reserves for energy production as a result of induced stress (Canavoso et al. 2001). Protein synthesis is necessary particularly for the maintenance of body growth and reproduction. They enter in various reactions such as the hormonal regulation and they integrated in the cell as a structural element at the same time as carbohydrates and lipids (Cohen 2010; Sugumaran 2010). In the present investigation, after treatment of *S. granarius* adults with *C. limonum* essential oil, an inhibitory action on proteins was generally exhibited. Previous observations were reported a reduction in the total proteins content of *S. littoralis* larvae when treated with garlic and lemon extracts (Ali et al. 2017). Such decrease in energetic resources can have extreme consequences for insects (Rivero et al. 2010). This depletion might be due to their degradation for metabolic purposes or to an impaired incorporation of amino acids into polypeptide chains or inhibition of protein synthesis (Sharma et al. 2011) or to the breakdown of these proteins into amino acids used in the compensatory mechanism as energy source to compensate stress (Ali et al. 2014).

Conclusion

The EO of *C. limonum* exhibited fumigant toxicity against *S. granarius* adults confirming its potential as a natural alternative to synthetic insecticides for the control of stored-product pests. In addition, a strong repellent activity able to disrupt pest orientation indicate possible applications to flush out insect infestation from empty stores before introduction of fresh grain (Germinara et al. 2017). Moreover, the essential oil of *C. limonum* was persistent on *S. granarius*. Our results provide an interesting opportunity to develop bioinsecticides and repellent formulations based on botanical extracts.

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Compliance with ethical standards

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

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