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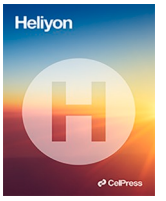

Research article

Chemical profiling and bioactivities of essential oils from *Thymus capitatus and Origanum compactum* against *Tribolium castaneum*

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ABSTRACT

The use of essential oils has emerged as an ecofriendly solution for controlling different pests, particularly insects of stored products. Essential oils (EOs) from *Thymus capitatus* (TC) and *Origanum compactum* (OC) have received less attention for these bioactivities. Therefore, our study aimed to assess the repellent, antifeedant and contact toxicity of their EOs against a major stored product pest *Tribolium castaneum*. Besides, GC-MS was also carried out to determine the compounds responsible for the observed bioactivities. Regarding contact toxicity, LC_{50} values were 0.58 and 0.35 μL/cm^2 for TC and OC after 24 h of exposure, respectively. For the repellent effect, the percentage of repellency (PR) was variable across different concentrations and exposure durations. TC exhibited the best PR (98%) after 3 h of exposure at 0.031 μL/cm^2. For prolonged repulsive effect (24 h), TC sustained its repulsive efficacy with a PR of 90% at 0.062 μL/cm^2 followed by OC with a PR of 88% at 0.125 μL/cm^2. As for the antifeedant effect, both EOs had a significant impact on nutritional indexes, especially the feeding deterrent index and relative consumption rate. OC displayed a notable effect, causing 59% of feeding deterrence at 1.92 μL/pellet. These multifaced effects can be explained by the high content of carvacrol in both EOs (OC: 90% and TC: 78%). These multifaced effects demonstrated through different exposure routes and bioassays promote the use of *T. capitatus* and *O. compactum* EOs as a sustainable management strategy to control *T. castaneum*.

1. Introduction

Stored products, such as grains and flour, constitute a vital source of carbohydrates globally. However, they are frequently vulnerable to infestations by various types of insect pests, leading to a decline in both quantity and quality [1]. Among these storage insect pests, *Tribolium castaneum* (Herbst, 1797), stands out as one of the most destructive, posing a threat to stored food. This secondary cosmopolitan species can attack a broad range of stored food items, such as flour, maize, oat, and sorghum, causing substantial weight loss [2,3].

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Phosphine, malathion and deltamethrin have proven great efficiency as conventional insecticide for the control of this pest [1,4]. However, the excessive use of these chemicals has led to the appearance of multiple cases of resistance in *T. castaneum* to phosphine, deltamethrin and malathion. Consequently, this urged the development of new strategies to minimize the environmental damage and reduce the possibility to evolve insecticide tolerance [5–7].

The essential oils extracted from Medicinal and Aromatic Plants (MAP) contain a rich mixture of biomolecules that have been documented for their potential use as insecticides with low risks to both humans and the environment [8]. Several essential oils belonging to different plant families, including *Mentha pulegium*, *Ocimum basilicum* L., *Origanum vulgare* L., *Cuminum cyminum* demonstrated promising insecticidal activities and eco-friendly candidates for the control of several insect pests [9,10]. Furthermore, studies have shown that these oils are effective against a wide range of stored product across different life stages, from eggs to adults, and using various exposure routes and bioassays, including fumigation, contact, direct application, repellency, and antifeedant [11, 12]. Essential oils have also been reported to significantly inhibit certain enzymes such as acetylcholinesterase, and other neurotransmitters. Additionally, they exhibit a notable effect on oxidative stress enzyme markers and influence lipid metabolism [13–15].

However, some essential oils have received less attention for their use in controlling insects such as *Thymus capitatus* and *Origanum compactum*. Those two species are abundantly distributed in the North of Morocco and are currently cultivated by local cooperatives in sustainable quantities. Their valorization can offer a ludic solution to develop a biopesticide to overcome the previously mentioned problems of chemical insecticides. On the first hand, *Thymus capitatus* (TC), a compact woody shrub, exclusively found in the Mediterranean region [16]. Despite several studies that have examined its pharmacological and biological activities in treating various diseases, as an antimicrobial agent, as well as its use as a food additive [17], only a few of them have focused on the insecticidal or behavioural effects of its essential oil against stored insects, including *T. castaneum*. On the other hand, *O. compactum* (OC) is a Moroccan and Andalusian (Spain) endemic plant species belonging to the Lamiaceae family [18]. This plant is rich in bioactive compounds found in its extracts and essential oil, which have been reported to exhibit numerous beneficial effects such as antimicrobial, anticancer, and antioxidant activities [19]. However, there are no studies investigating insecticidal activity against insect pests using the essential oil of this oregano species.

The use of the essential oils from *T. capitatus* and *O. compactum* has been less explored compared to the well-studies species *Origanum vulgaris* and *Thymus vulgaris* [20]. Furthermore, their antifeedant, repellent and contact effects were not explored against *Tribolium castaneum*. Therefore, our study aimed to assess the insecticidal and behavioral effect of the two sustainably produced essential oils extracted from *T. capitatus* and *O. compactum*, on the adults of red flour beetle *T. castaneum*. Additionally, gas chromatography-mass spectrometry (GC-MS) analysis was carried out to determine their chemical composition.

2. Material and methods

2.1. Plant material and essential oil extraction

Thymus capitatus and *Origanum compactum* fresh aerial parts were obtained from a local cooperative "Aghssane" specialized in the domestication and culture of aromatic and medicinal plants in Ain Zerka, Tetouan, Morocco. Both plants are domesticated and locally produced in large amounts every year in sustainable quantities. The confirmation of plant species was carried out by Dr. Homrani Abdelmonaim, Ecologist and Pastoralist at the Regional Agronomic Research Center of Errachidia. Essential oils were extracted using steam distillation [21]. Plant material (400 g) was fit into the still and the steam generator was heated to 120–130 °C with a pressure up to 4 bar. The steam was vehiculated to the still to extract the essential oils from the desired aromatic plant. After 4 h, the essential oil was separated from the hydrolat, kept in dark bottles, and kept at 4 °C until used for chemical characterization, insecticidal, and behavioural effects.

2.2. Chemical characterization of essential oils using GC-MS

The chemical composition of OC and TC essential oil was carried out using GC Shimadzu, Nexis 2030 instrument attached with TQ8040 NX mass spectrometer with Restek RTX-5MS column (30 0.25 mm, 0,25 μm film thickness). The initial temperature program was set at 50C for 2 min, increased to 300 with a rate of 5.5C/min, and stabilized for 3 min at 300 °C. Helium was used as a carrier gas with a 1.5 mL/min flow rate. 1 μl of the sample was injected in split mode (Injector HTA 2800T, HT, 250C). The scan of the mass range was from *m/z*:50–500. The essential oils identification was made based on their retention indices (RI) determined with reference to homologues series of C_5–C_{24} (n-alkanes), by comparison of their mass spectra with the reports in the literature using NIST and Wiley version libraries [22].

2.3. Insect culture

Adults of the red flour beetle (*T. castaneum*) were obtained from a laboratory culture at the Faculty of Science and Technology in Tangier, Morocco. The beetles were raised in a mixture of wheat flour and dried yeast in a 1:19 ratio and were kept in bottles at a temperature of 30 °C and humidity of 60 ± 5% in the dark [23]. Only 7–14 days old adults were used for all bioassays.

2.4. Contact toxicity

The contact toxicity consisted of exposing the adults of both sexes to a filter paper loaded with essential oil concentrations. Bioassay

was assessed following a method with some modification [24]. Three concentrations (1.6, 2.4, and 3.2% v/v) were prepared in acetone for both essential oils and 300 μl of each dilution was added to a 5 cm diameter filter paper (Whatman 1) giving a final concentration of 0.24, 0.37 and 0.49 μL/cm^2 respectively expressed as volume of essential oil per filter paper surface. Treated papers were left at room temperature for 3 min to allow the evaporation of acetone. The filter paper was then placed in a Petri dish (5 cm diameter) and 10 unsexed 7–14 days old adults were added to the Petri dish (5 cm diameter) and incubated in the same breeding conditions. The control received 300 μL of 100% acetone. This bioassay was conducted in a completely randomized design (CRD) with five replicates per concentration for each treatment. The mortality was observed 24-, 48-, and 72-h post-exposure and the insect was considered dead when no movement was observed.

2.5. Antifeedant bioassay

2.5.1. Flour pellets preparation

The antifeedant effect of essential oils was carried out using a method with some modifications [25]. The flour pellets were prepared according to the method of [26] with some slight modifications. In brief, the wheat bran was ground using a mixer and sifted through a sieve. Flour obtained after sieving was mixed with Wheat flour in a 1:1 w/w proportion. Dried yeast was added to this mixture in 1:19 w/w proportion. This mixture (25 g) was added to 100 mL of distilled water and magnetically stirred. Aliquots of 300 μL were then pipetted onto a plastic Petri dish and left overnight to dry. The following day, dry flour pellets were equilibrated at 30 °C for 24 h, and only the ones weighing 61 mg were used.

2.5.2. EO concentration preparation and application

Essential oil concentration was prepared in acetone. 30 μL of each EO concentration (0.8, 1.6, 3.2, 4.8, and 6.4%) were pipetted into flour pellets, resulting in final concentrations of 0.24, 0.48, 0.96, 1.44, 1.92 μL/pellet, respectively (Expressed as volume of essential oil per pellet). The treated flour pellets were then left at room temperature for 20 min to ensure the evaporation of acetone. After weighing, the pellets were placed in a Petri dish containing 10 adults previously starved for 24 h. Control pellets received acetone only and followed the same procedure previously mentioned. Three days later, adults and flour pellets weight were measured, and the nutritional indexes were calculated using Equations (1)–(4) according to Manuwoto, and Farrar et al. [27,28]. This bioassay was conducted in a completely randomized design (CRD) with 5 replicates per concentration for each treatment.

$$\text{Relative Growth Rate (RGR)} : \mathbf{RGR} = \frac{(\mathbf{A} - \mathbf{B})}{\mathbf{B\ x\ day}} \tag{1}$$

where: **A:** weight of live insect after the experiment (mg to each insect); **B:** weight of insect before the experiment (mg to each insect).

Relative Consumption Rate (RCR):

$$\mathbf{RCR} = \frac{\mathbf{D}}{\mathbf{B\ x\ day}} \tag{2}$$

where: **D:** weight of food consumed by the insect (mg).

Efficacy of Conversion of Ingested Food (ECI):

$$\mathbf{ECI} = \frac{\mathrm{RGR}}{\mathrm{RCR}} \boldsymbol{X}\ \mathbf{100} \tag{3}$$

Feeding Deterrence Index (FDI):

$$\mathbf{FDI} = \left[\frac{\mathbf{C} - \mathbf{T}}{\mathbf{C}}\right] \mathbf{X\ 100} \tag{4}$$

where: **C**: Consumption of control diet and **T:** Consumption of treated diet.

2.6. Repellency bioassay

The repellent effect was assessed through the behavioural choice test conducted in Petri dishes according to Jilani et al. [29] using circular filter paper halves of each one is treated with essential oil concentration and the other with acetone. Experimentally, 7 cm filter paper circles were cut into two halves of which one received 300 μL of each concentration (0.1, 0.2, 0.4 and 0.8%) dissolved in acetone giving a final concentration of 0.016, 0.031, 0.062, 0.125 μL/cm^2, respectively expressed as volume of essential oil per filter paper surface, and the other half was treated with 300 μl of acetone. Then after, treated and control halves were left to dry for 3 min and then attached edge to edge with duct tape and placed into a Petri dish (7 cm in diameter). Twenty unsexed adults were released in the middle. The number of individuals that settled on each half of the filter paper disc was counted after 1,2, 3, 4 and 24 h. The average count was converted to a percentage of repellency (PR) using **Equation (5)** according to [30]:

$$\boldsymbol{PR} = \left[\frac{\boldsymbol{Nc} - \boldsymbol{Nt}}{\boldsymbol{Nc} + \boldsymbol{Nt}}\right] \boldsymbol{X}\ \mathbf{100} \tag{5}$$

Nc: Number of adults counted in the control half.
Nt: Number of adults counted in the treated half.
Five replicates were reproduced of each treatment.

2.7. Statistical analysis

All data were analyzed using one-way ANOVA followed by Tukey Test as post-hoc at a significance level of $p < 0.05$. Probit analysis was conducted to determine the LC_{50}. All statistical analyses were conducted using IBM's software SPSS V25.0.

3. Results

3.1. GC-MS profiling

The essential oils (EOs) extracted from the aerial parts of OC and TC were analyzed by GC-MS. The chromatographic analysis, including the composition and the relative abundance, is summarized in Fig. 1 and Table 1. Twenty-two compounds were identified in the EO of OC (Fig. 1A), while thirty-two compounds were identified in the EO of TC (Fig. 1B) . Fifteen compounds were found to be common between the two EOs. Carvacrol was detected as a major compound in both EOs constituting 78.29 and 90.02% in TC and OC, respectively. TC was characterized by the presence of *p*-cymene, linalool, (E)-caryophyllene and caryophyllene oxide, accounting for 4.89%, 3.10%, 4.57% and 1.89%, respectively. Meanwhile, OC also contained *p*-cymene and thymol with 3.33% and 3.32%, respectively.

3.2. Contact toxicity

Significant mortality was observed in *T. castaneum* adults upon exposure to both EOs, demonstrating concentration- and time-dependent activities, **Fig. 2**. The highest mortality rates, recorded at the highest concentration (0.49 μL/cm^2), were 72% and 78%

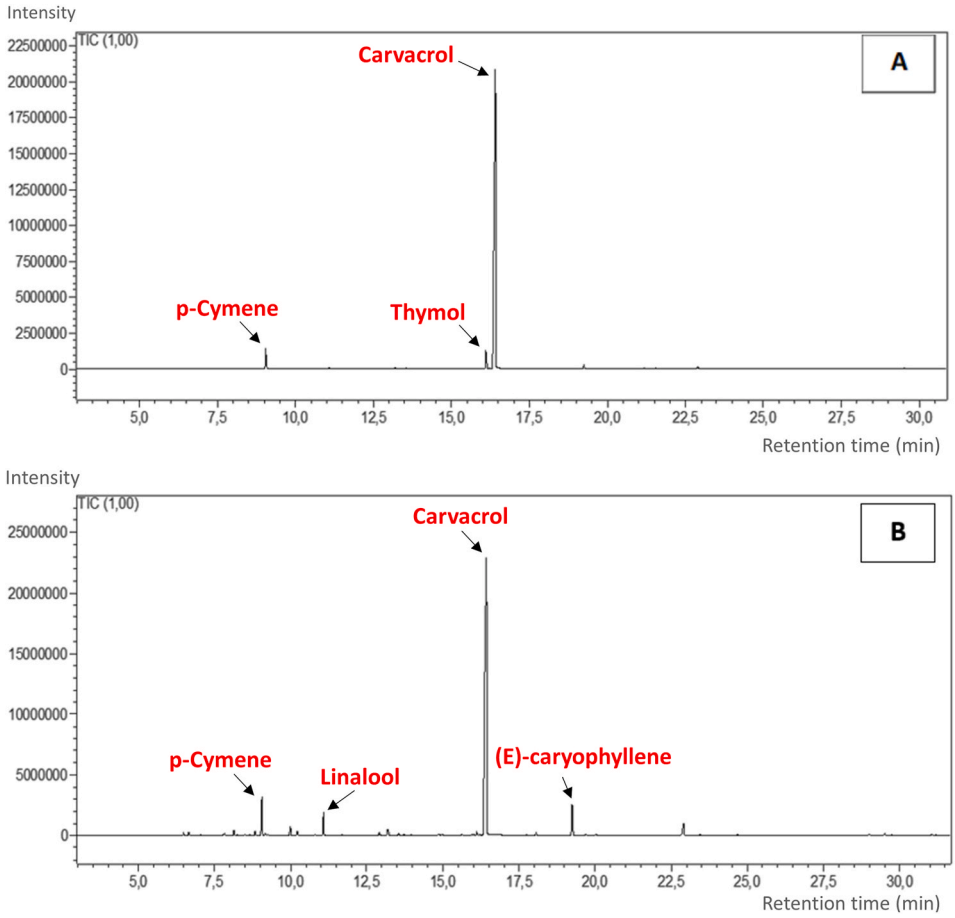

Fig. 1. GC-MS Chromatograms of **A**: *O. compactum;* **B**: *T. capitatus* essential oils.

Table 1
Chemical profile of *O. compactum* and *T. capitatus* essential oils using GC-MS.

Compound	Retention index (RI)		Relative abundance (%)	
	Calculated	Reported	*O. compactum*	*T. capitatus*
α-Thujene	918	924	–	0.37
α-Pinene	925	932	0.12	0.38
Camphene	940	946	–	0.09
β-Pinene	967	974	–	0.07
Myrcene	984	988	0.09	0.66
3-Octanol	984	988	–	0.05
α-Phellandrene	997	1002	–	0.10
δ-3-Carene	1000	1008	–	0.06
α-Terpinene	1006	1014	–	0.51
p-Cymene	1014	1020	**3.33**	**4.89**
Limonene	1020	1024	0.07	0.28
1,8-Cineole	1021	1026	–	0.11
γ-Terpinene	1048	1054	–	1.05
cis-Sabinene hydrate	1060	1065	–	0.61
Terpinolene	1081	1086	–	0.09
ρ-Cymenene	1084	1089	0.07	–
Linalool	1092	1095	0.30	**3.10**
Camphor	1144	1141	–	0.05
Borneol	1167	1165	0.11	0.38
Terpinen-4-ol	1179	1174	0.33	0.85
ρ-Cymen-8-ol	1184	1179	0.10	–
α-Terpineol	1193	1186	0.18	0.28
cis-Dihydro carvone	1196	1191	0.07	0.07
trans-Dihydro carvone	1200	1200	–	0.04
Pulegone	1239	1233	–	0.26
Carvone	1247	1239	0.06	0.10
Thymol	1296	1289	**3.32**	0.09
Carvacrol	1305	1298	**90.02**	**78.29**
Eugenol	1361	1356	–	0.07
Carvacrol acetate	1374	1370	–	0.39
(E)-caryophyllene	1422	1417	0.81	**4.57**
α-Humulene	1453	1452	0.07	0.21
β-Bisabolene	1499	1505	0.18	–
δ-Amorphene	1505	1511	0.07	–
γ-Cadinene	1508	1513	–	0.04
δ-Cadinene	1518	1522	0.13	–
Spathulenol	1582	1577	0.08	–
Caryophyllene oxide	1589	1582	0.42	**1.89**
epi-α-Cadinol	1643	1638	0.06	–

after 24 h and 48 h for OC, respectively (Fig. 2**A**), and 62% and 84% after 48 h and 72 h for TC, respectively (Fig. 2**B**). Notably, OC demonstrated higher activity than TC at 24 and 48 h at the same concentration (0.49 μL/cm^2), while TC exhibited increased mortality at the same concentration after 72 h, Fig. 2.

The calculated LC_{50} values at 24 h for OC and TC were 0.35 μL/cm^2 and 0.58 μL/cm^2 respectively, Table 2. Remarkably, these LC_{50} values decreased significantly to 0.22 μL/cm^2 and 0.37 μL/cm^2, respectively, at 72-h exposure period.

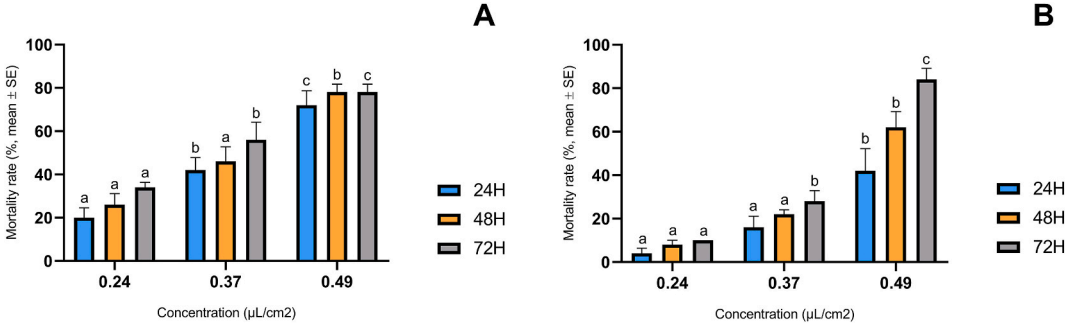

Fig. 2. Contact toxicity of *O. compactum* (OC) (**A**) and *T. capitatus* (TC) (**B**) essential oils against adults of *Tribolium castaneum*. Values expressed as mean of mortality percentage ± Standard Error for five replicates. Results are considered significant when the letters are different at a $p < 0.05$ at different essential oils concentrations.

3.3. Antifeedant effect

The incorporation of TC and OC EOs in flour resulted in significant disturbances in two key nutritional indexes, FDI and RCR, among *T. castaneum* adults, Table 3. Notably, OC demonstrated the most pronounced decrease in both indexes at the highest tested concentrations. For instance, the RCR values decreased from 0.143 mg/mg/day (control) to 0.055 mg/mg/day at 1.92 μL/pellet, indicating a 2.6-fold reduction. Similarly, the FDI dropped to 67.2% at 1.92 μL/pellet. On the other hand, TC essential oil also led to a significant decrease in RCR with values declining from 0.143 mg/mg/day (control) to 0.091 mg/mg/day at 1.92 μL/pellet. Additionally, the maximum FDI recorded was 44.3% at the highest used concentration (1.92 μL/pellet).

Statistical analysis revealed a significant difference in both FDI and ECI (essential oil concentration) for both EOs. Particularly, OC essential oil exhibited the strongest significance for RCR ($p < 0.05$ and $F = 28.68$) and FDI ($p < 0.05$ and $F = 32.2$) nutritional indexes. However, no significance was observed for RGR and ECI for TC and OC.

3.4. Repellent effect

Again, both essential oils exhibited concentration- and time- dependent repellent effects on *T. castaneum* adults, Table 4. TC exhibited the best repellent effect after 3 h of exposure at 0.031 μL/cm^2, with a percentage of repellency (PR) of 98%. Furthermore, OC exhibited a good repulsive potential (92%) at 0.062 μL/cm^2 of essential oil after 4 h. For prolonged repulsive effect (24 h), both essential oils caused an excellent percentage of repellency exceeding 85% at different concentrations. TC exerted the highest repellency (90%) at 0.062 followed by OC (88%) at 0.125 μL/cm^2.

4. Discussion

Our findings demonstrate the diverse insecticidal and behavioral effects of the EOs derived from *O. compactum* (OC) and *T. capitatus* (TC). Both EOs act as repellent, feeding deterrent, and toxic agents against adults of *T. castaneum*. These results provide the initial evidence of the antifeedant and repellent effect of both EOs on an insect model. Furthermore, both EOs were assessed for their contact toxicity for the first time. Overall, this research offers a comprehensive understanding of the multifaceted effects of these essential oils on *T. castaneum*, encompassing repellency, antifeedant properties, and contact toxicity. Our study highlights the high content of carvacrol of both species that surpasses 75%; this fact boosts the capacity of this molecule using different exposure routes to alter the appetite, attractant/repulsive behavior, as well as the viability of *T. castaneum*.

Our results highlight the contact toxicity of both EOs at different concentrations. This toxicity can be due to the main component of both EOs, carvacrol. This compound was reported to cause significant mortality against four stored product species. LC_{50} was reported to be 17.15 and 21.16 μg/cm^2, respectively, for *Sitophilus oryzae* and *T. castaneum* [31]. Significant toxicity at lower concentration was observed against *Rhyzopertha dominica and Lasioderma serricorne* with LC_{50} of 0.012 and 0.019 mg/cm^2, respectively [32]. The contact toxicity can also be due to the contribution of other compounds such thymol, *p* cymene, linalool and caryophyllene documented in literature for this kind of toxicity. For instance, *p* cymene, present in both EOs, was documented as well for its contact toxicity against *Sitophilus oryzae* giving an LC_{50} of 0.8 mg/cm^2 [33]. Thymol, present in both EOs, was documented as well for its contact against *Rhyzopertha dominica* and *Sitophilus oryzae* with an LC_{50} of 8.8 and 24.07 μg/cm^2, respectively [31]. Linalool, present in TC, was reported for its potent contact toxicity of 4 stored products pests namely *S. oryzae, S. zeamais, Lasioderma serricorne and T. castaneum* as well with LC_{50} of 66.74 μg/cm^2, 2.45 μL/cm2, 27.41 μg/cm^2 and 45.96 μg/cm^2 respectively [34–36]. Although no reports for the contact toxicity of caryophyllene were documented in the literature. These molecules could act as synergistic molecules boosting the activity of the essential oil as well as acting as antagonists to diminish their activity.

The current study also showed a strong repellency property of both EOs against adults of *T. castaneum* in mid- and long-term exposure. No studies have documented the repellency effect of TC. Despite absence of reports concerning TC essential oil, other

Table 2
Lethal concentrations *(LC)* of *O. compactum (OC)* and *T. capitatus (TC)* after 24, 48, and 72 h of exposure using probit analysis at $p < 0.05$.

	Time of Exposure	LC[a]	LC value[a]	Probit Value[b]	Z	Sig.	Chi-Square	df[c]	Sig.
T. capitatus (TC)	24 h	LC_{50}	0.58 (0.47–0.97)	3.47 ± 0.27 (2.93–4.00)	12.65	0.000	185.1	13	.00[c]
		LC_{90}	0.95 (0.71–1.99)						
	48 h	LC_{50}	0.47 (0.40–0.59)	3.64 ± 0.23 (3.19–4.09)	15.80	0.000	126.0	13	.000[c]
		LC_{90}	0.82 (0.67–1.17)						
	72 h	LC_{50}	0.37 (0.30–0.45)	4.52 ± 0.23 (4.08–4.97)	19.91	0.000	126.0	13	.000[c]
		LC_{90}	0.65 (0.54–0.89)						
O. compactum (OC)	24 h	LC_{50}	0.35 (0.27–0.45)	2.87 ± 0.19 (2.51–3.24)	15.40	0.000	124.3	13	.000[c]
		LC_{90}	0.79 (0.63–1.18)						
	48 h	LC_{50}	0.29 (0.20–0.38)	2.74 ± 0.18 (2.38–3.09)	15.14	0.000	125.3	13	.000[c]
		LC_{90}	0.76 (0.60–1.15)						
	72 h	LC_{50}	0.22 (0.11–0.30)	2.36 ± 0.18 (2.01–2.70)	13.40	0.000	93.6	13	.000[c]
		LC_{90}	0.77 (0.60–1.15)						

[a] Lethal concentration expressed in μL/cm^2 using 95% Confidence Limits.
[b] Probit Value estimation ± Standard error using 95% Confidence Limits.
[c] Degree of freedom, Statistics based on individual cases differ from statistics based on aggregated cases.

Table 3
Antifeedant effect of *O. compactum* (OC) and *T. capitatus* (TC) essential oils against adults of *T. castaneum*.

Oils	Concentration (μL/pellet)	RGR[a] (mg/mg/day)	RCR[a] (mg/mg/day)	ECI[a] (%)	FDI[a] (%)
O. compactum (OC)	0	0.023 ± 0.006^{a}	0.143 ± 0.004^{a}	16.7 ± 4.4^{a}	0.0 ± 3.3^{a}
	0.24	0.015 ± 0.002^{a}	0.148 ± 0.004^{a}	10.4 ± 1.2^{a}	0.0 ± 2.2^{a}
	0.48	0.010 ± 0.005^{a}	0.147 ± 0.008^{a}	6.6 ± 3.7^{a}	-7.4 ± 6.0^{a}
	0.96	0.010 ± 0.003^{a}	0.159 ± 0.014^{a}	9.1 ± 2.1^{a}	-17.2 ± 8.6^{a}
	1.44	0.010 ± 0.019^{a}	0.070 ± 0.007^{b}	6.6 ± 22.0^{a}	50.8 ± 4.6^{b}
	1.92	0.003 ± 0.020^{a}	0.055 ± 0.010^{b}	-16.8 ± 29.2^{a}	59.6 ± 7.6^{b}
	F	0.338	28.68	0.58	31.21
	p	0.885	<0.05	0.72	<0.05
T. capitatus (TC)	0	0.023 ± 0.006^{a}	0.143 ± 0.004^{ab}	16.7 ± 4.4^{a}	0.0 ± 3.3^{ab}
	0.24	0.014 ± 0.002^{a}	0.142 ± 0.007^{ab}	9.7 ± 1.3^{a}	-1.4 ± 5.4^{ab}
	0.48	0.016 ± 0.004^{a}	0.164 ± 0.013^{a}	10.4 ± 2.5^{a}	-15.8 ± 9.2^{a}
	0.96	0.020 ± 0.002^{a}	0.166 ± 0.008^{a}	12.2 ± 1.2^{a}	-15.5 ± 5.2^{a}
	1.44	0.022 ± 0.017^{a}	0.101 ± 0.013^{bc}	17.9 ± 12.5^{a}	25.5 ± 9.6^{b}
	1.92	0.024 ± 0.017^{a}	0.091 ± 0.011^{c}	9.6 ± 16.3^{a}	37.2 ± 7.1^{bc}
	F	0.17	10.27	0.18	9.70
	p	0.97	<0.05	0.968	<0.05

Values expressed as mean ± Standard Error of five replicates. Results are considered significant when the letters differ at a $p < 0.05$ at different essential oils concentrations using One-way ANOVA and Tukey Post-hoc.

[a] RGR = Relative Growth Rate | *RCR = Relative consumption Rate | *ECI = Efficacy of Conversion of Ingested Food | *FDI = Feeding Deterrence Index.

Table 4
Repellent effect of *O. compactum* (OC) and *T. capitatus* (TC) essential oils against adults of *T. castaneum*.

Essential oil	Concentration (μL/cm^2)	Period of Exposure (h)				
		1	2	3	4	24
O. compactum (OC)	0.016	–	–	–	–	–
	0.031	46 ± 5.1^{a}	42 ± 3.7^{a}	48 ± 2.0^{a}	52 ± 3.7^{a}	42 ± 13.3^{a}
	0.062	84 ± 6.8^{b}	70 ± 5.5^{b}	80 ± 5.5^{b}	92 ± 3.7^{b}	86 ± 5.1^{b}
	0.125	86 ± 5.1^{b}	80 ± 10.5^{b}	88 ± 2.0^{b}	88 ± 3.7^{b}	88 ± 2.0^{b}
T. capitatus (TC)	0.016	60 ± 7.1^{a}	80 ± 4.5^{a}	70 ± 9.5^{a}	82 ± 2.0^{a}	64 ± 4.0^{ab}
	0.031	82 ± 4.9^{a}	88 ± 7.7^{a}	98 ± 2.0^{a}	94 ± 4.0^{a}	72 ± 3.7^{ab}
	0.062	84 ± 6.8^{a}	92 ± 5.8^{a}	92 ± 3.7^{a}	86 ± 5.1^{a}	90 ± 4.5^{a}
	0.125	78 ± 9.7^{a}	84 ± 6.8^{a}	80 ± 9.5^{a}	82 ± 9.2^{a}	46.0 ± 18.1^{b}

Values expressed as mean of Repellency percentage (PR) ± Standard error for five replicates. Results are considered significant when the letters are different at a $p < 0.05$ at different tested concentrations using One-way ANOVA and Tukey Post-hoc.

thymus species were reported in the literature for their repellent potential. For instance, *Thymus vulgaris* was a highly studied species, and was reported as a potent repellent against stored products insects such as *Sitophilus zeamais, Plodia interpunctella* [20,37,38] as well as other phytophageous species (*Bemisia tabaci,* and *Meligethes aeneus*) [38,39]. *Thymus quinquecostatus* was generally repellent against three stored products *T. castaneum, Lasioderma serricorne,* and *Liposcelis bostrychophila* [40]. *Thymus persicus* EO exhibited strong repellency against *Tribolium castaneum* (Herbst) and *Callosobruchus maculatus* [41]. As for OC essential oil, Bounoua-Fraoucene et al. [42], reported its pronounced repellent activity against two stored products insects *Rhyzopertha dominica* and *Sitophilus oryzae* in the repellent bioassay. Additionally, another study by Aimad et al. [43] using the same *Origanum* species reported a moderate repellent activity with an average of 39% of repellency. The observed variation might be attributed to the difference in carvacrol concentration (38.7% in the previous study vs. 90% in the current study). Carvacrol has been documented to repel two stored product insects, *Rhyzopertha dominica* and *Lasioderma serricorne,* where repellency rates of 96, and 76%, respectively, were observed after 3 h of exposure at 12.5 μg/cm^2 [32]. Linalool, another volatile present in OC essential oil, was also reported for its repellency potential against *Tribolium castaneum* with RD_{50} value of 0.11 μL/cm2 [44]. Another study by Cao et al. [36] demonstrated its strong repellency against *Lasioderma serricorne* (84.0% PR at 15.83 μL/cm^2) and *Liposcelis bostrychophila* (64.0% PR at 78.63 μL/cm^2) after 2 h. While thymol has not been reported for repellency against stored products insects, it has been reported for mosquito repellency [45].

For the antifeedant effect, both EOs resulted in a significant decrease in the nutritional indexes of *T.* castaneum. This notable effect can be due to the high content of carvacrol. The latter demonstrated significant antifeedant effects against different orders of insect pests, and mostly against stored product coleopteran species. For instance, it exhibited 30% of feeding deterrence at a maximum concentration of 1 mg/g against *Rhyzopetha dominica* [31]. However, the same research found no evidence of feeding deterrent against *T. castaneum* and *S. oryzae*. The same study found that thymol was least lethal to *S. oryzae* but more toxic to *T. castaneum* and *R. dominica* when compared to carvacrol and eugenol [31]. The potent feeding deterrence was reported against different field insect pests, such as larvae of *Helicoverpa armigera*, *Spodoptera litura* and *Chilo partellus* with FI_{50} of 128.8, 122.3, and 230.1 μg/cm^2, respectively [46]. A 100% of FDI was reported on the 3rd instar larvae of *Plutella xylostella* using carvacrol at 50 μL/ml [47]. Another

study reported 55% of feeding deterrence against the invasive cotton moth *Spodoptera littoralis* at 100 μg/cm^2 [48]. Similarly, *Leptinotarsa decemlineata* was also reported to be sensitive to carvacrol, exhibiting 90.9% feeding index at 50 μg/cm^2, and thymol, showing 90.9% feeding index at the same concentration (50 μg/cm^2) [49]. Carvacrol also exhibited its feeding deterrence against mosquitos of *Aedes aegypti* at a concentration of 22.51 μg/cm^2, producing 50% of feeding repellency [50]. The observed antifeedant effect might be attributed to other major compounds of both EOs, as they have shown feeding deterrence. For instance, linalool and thymol deterred feeding of larvae of *Spodoptera littoralis* with a value of 45.3 and 52.4% of feeding reduction, respectively [48]. Thymol was reported in another study as the most effective feeding intake inhibitor out seven tested monoterpenes [51]. The antifeedant effect of both EOs might be attributed to the reduction in digestive enzymes, such α-amylase, protease, and lipase, which are crucial for insects [52]. Additionally, the antifeedant effect might involve the implication of olfactory and gustative binding protein, which exhibit a repellent effect on insects, preventing them from feeding [53,54].

The *in vitro* assessment of the insecticidal and behavioral effects of both EOs has opened door to harness the potential of volatile substances. However, these results may encounter limitation related when translated into real-life applications, due to the volatility of EOs. Therefore, gaining deeper understanding of the behavior of these essential oils in storing facilities might enhance our understanding of their efficacy. Furthermore, exploring bioformulations may offer a solution by protecting these substances from evaporation and ensuring prolonged efficacy for sufficient periods [55,56].

5. Conclusion

Our study unveils compelling evidence regarding the insecticidal and behavioral properties of *O. compactum* and *T. capitatus* EOs. Both EOs demonstrated remarkable effects, including approximately 50% feeding deterrence, over 80% repellency, and mortality. This research provides the first evidence of the effectiveness of both EOs against *T. castaneum* and supports their potential use as a sustainable and eco-friendly strategy for managing stored product insects. Further studies are needed to explore the involved mechanisms and to determine the appropriate form and frequency of application.

CRediT authorship contribution statement

Houssam Annaz: Writing – review & editing, Writing – original draft, Methodology, Investigation, Conceptualization. **Hassan Annaz:** Writing – original draft, Investigation. **Ayoub Ajaha:** Writing – review & editing. **Noureddin Bouayad:** Writing – review & editing, Methodology, Conceptualization. **Karim El Fakhouri:** Writing – review & editing. **Amin Laglaoui:** Writing – review & editing. **Mustapha El Bouhssini:** Writing – review & editing. **Mansour Sobeh:** Writing – review & editing. **Kacem Rharrabe:** Writing – review & editing, Validation, Methodology, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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