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Original article

Fumigant and contact toxicity of *Ruta chalepensis* L. (Rutaceae) essential oil against five coleopteran stored product pests and its effects on cholinesterases

Ruta chalepensis L. (Rutaceae) uçucu yağının beş coleopteran depo ürün zararlısına karşı fumigant ve kontak toksisitesi ile kolinesterazlar üzerine etkileri

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ABSTRACT

The essential oil composition of aerial parts of *Ruta chalepensis* L. was analyzed with GC-MS. Seventy-nine compounds were detected representing 85.93 ± 1.08% (n = 3) of the essential oil. The major components of the essential oil were 2-undecanone 21.52 ± 0.21%, 2-nonanone 18.31 ± 0.27%, and 2-nonyl acetate 13.22%. The highest insecticidal contact toxicity of the oil was observed against *Rhyzopertha dominica* F. with 0.018 µl/insect LD50 and 0.039 µl/insect LD90 after 24h. Essential oil also produced considerably low 0.50 and 0.59 µl/insect LD50 values after 24h against *Sitophilus oryzae* L. and *Sitophilus granarius* L. respectively. The lowest contact toxicity was observed against *Tribolium castaneum* Herbst. and *Tribolium confusum* Jacquelin du Val. 0.138 and 0.078 µl/insect LD50 after 24h respectively. The highest fumigant toxicity was observed against *S. granarius* for the application concentration of 10 µl, 10% oil/acetone (v:v) in a 10 ml chamber which afforded 100.00 ± 0.00% mortality after 48h. The essential oil also produced high fumigant toxicity against *S. oryzae*, *T. castaneum* and *R. dominica* which were 95.47 ± 3.41%, 93.30 ± 5.54%, and 85.47 ± 3.41% mortality at 20 µl application concentration of the oil solution after 48h. The *R. chalepensis* essential oil produced low acetylcholinesterase enzyme 5.29 ± 1.20% (n=3) inhibition and mediocre butyrylcholinesterase enzyme inhibition 42.6 ± 0.71% (n=3). According to the insecticidal activity assays performed, the essential oil *R. chalepensis* seems to be a promising source that could yield natural compounds that could be employed in stored product pest management.

INTRODUCTION

The appropriate storage of agricultural products is very important to reduce product losses. Product loss during storage is caused by many factors, including pests such as insects, rodents, and microorganisms as well as the conditions during storage such as humidity, temperature, etc. (Bouajaj et al. 2014, Chebli et al. 2004, Eisen 2013, Shah et al. 2015, Shaaya et al. 1997, Yazdgerdian et al. 2015). The most significant damage among the factors listed that affect the product is caused by stored product insects. The primary (*S. granarius*, *S. oryzae*, *R. dominica*, etc.) and secondary (*T. confusum*, *T. castaneum*, *O. surinamensis*, etc.) stored product insect species cause serious damage to the product during the storage (Atay et al. 2023, Polatoğlu and Karakoç 2016, Prakash and Rao 1996, Rajendran and Sriranjini 2008). Unless they are controlled, they can produce serious losses in the product (Çam et al. 2012, Hernandez-Lambraño et al. 2015, Kamanula et al. 2010). Product losses can be between 40% and 100%, particularly if modern storage techniques are not used (Campbell and Sinha 1976, Shaaya et al. 1997, Yıldırım et al. 2001). Currently, synthetic chemical compounds with contact and fumigant activity—often consisting of organic phosphorus—are frequently utilized in large-scale pest control of stored products against insects (Alkan et al. 2023, Çam et al. 2012, Evlice and Alkan 2023, Martin et al. 2015, Udo 2005). Natural pest control methods are becoming more popular as a result of increased environmental and health concerns, as well as new laws implemented to address these issues, such as the European Pesticide Regulation (EC) No. 1107/2009 (Alkan and Atay 2023, Atay and Alkan 2023, Isman 2006, 2008, Villaverde et al. 2014).

Ruta species of Rutaceae is only represented by *Ruta chalepensis* L. in Cyprus (Viney 1994). *Ruta* species are plants native to the Mediterranean and they found many uses in traditional medicine (Pollio et al 2008). These medicinal uses include uses for dropsy, inflamed eyes, colics, spasms, diarrhoea, insect/scorpion/spider bites, snake bites, poisoning, headache, migraine, epilepsy, hysteria, internal parasites, catarrh, common cold, cough, toothache (Lardos 2006). Until now from the *Ruta* species, many different types of secondary metabolites including alkaloids, coumarins, flavonoids, triterpenes, sterols, saponins, and tannins were isolated (Chen et al. 2001, Hnatyszyn et al. 1974). The furocoumarins (psoralens) are specifically observed in the species of Rutaceae, Umbelliferae, Moraceae, Fabaceae, and Apiaceae. Psoralens cause phytophotodermatitis in humans (Pathak et al. 1962). *Ruta* species is also known for its quinolone alkaloids. The essential oils of the *Ruta* species were also investigated comprehensively. However, most of the reports only indicate a few identified substances in

the composition of the essential oil except for a couple of comprehensive reports that identify many substances in the composition of the oil (Ulubelen et al. 1986).

The *Ruta* essential oils are characterized with alcohols (2-nonanol), esters (nonyl acetate, 2-methyloctyl acetate), ketones (2-undecanone, 2-dodecanone, 2-decanone, 2-nonanone) of saturated fatty acids (Abbad et al. 2014, Akkari et al. 2015, Rustaiyan et al. 2002, Tounsi et al. 2011, Tzakou and Couladis 2001). Usually, the essential oils of *Ruta* species contain geijerene (2-Isopropenyl-1-methyl-1-vinyl-3-cyclohexane) and moskachan (psoralen) derivatives in minor quantities (Joulain et al. 1991, Stashenko et al. 1995, Stashenko et al. 2000, Yaacob et al. 1989). The *Ruta* essential oils are reported to have herbicidal, molluscicidal, nematocidal, antimicrobial, antibacterial, antifungal, anti-inflammatory, antipyretic, emmenagogue and antihelminthic effects (Bouabidi et al. 2015, Günaydin and Savcı 2005, Haddouchi et al. 2013, Hmamouchi et al. 2000, Iauk et al. 2004, Ntalli et al. 2011,). Additionally, the essential oils of *Ruta* species are known for their considerable insecticidal activity against a broad spectrum of insect species. *R. chalepensis* has already been reported to have larvicidal, repellent and biting deterrent activity against vectors of many diseases namely *Aedes albopictus* Skuse, *Aedes aegypti* (L.), *Culex pipiens pallens* Coquillett, *Anopheles quadrimaculatus* Say (Diptera: Culicidae) (Ali et al. 2013, Conti et al. 2013, Kim et al. 2002, Perez López et al. 2015). Furthermore, the essential oils of *Ruta* species were tested against agricultural product insects namely *Orgyia trigotephra*s Boisid. (Lepidoptera), *Tribolium confusum* Jacquelin du Val, *T. castaneum* (Herbst) (Coleoptera) and found active against these pests (Abbad et al. 2014, Akkari et al. 2015, Majdoub et al. 2014). The extracts of *Ruta* species were reported to have aphicidal, insecticidal, and nematocidal activity against *Alphitobius diaperinus* Panzer, *Trogoderma granarium* Everts (Coleoptera), *Aphis punicae* Passerini, *Bemisia tabaci* Gennadius (Hemiptera), *Hypsipyla grandella* Zeller (Lepidoptera), *Locusta migratoria* L. (Orthoptera), *Meloidogyne arenaria* Chitwood, *M. hapla* Chitwood, *M. javanica* (Treb) Chitwood, *M. incognita* (Kofoid and White) Chitwood (Tylenchida), *Pediculus humanus* L. (Phthiraptera), *Psammodermes hybostoma* Desneux (Blattodea), *Xiphinema index* Thorne and Allen (Dorylaimida) species (Abdellaoui et al. 2014, Al-mazr'awi and Ateyyat 2009, Alshehry et al. 2014, Jorge et al. 2009, Madkour et al. 2012, Mancebo et al. 2001, Marcomini et al. 2009, Moawad and Al-Barty 2011, Sasanelli 1992, Sasanelli and D'Addabba 1993, Soto Monterrosa et al. 2007). Natural substances isolated from *Ruta* species namely quinoline, quinoline-4-carbaldehyde, quinoline-3-carbaldehyde, and quinolone carboxylic acid derivatives were reported to have fumigant and contact toxicity against *S. oryzae* (Jeon et al. 2013). The 3-(2",2"-dimethylbutenyl)-

3'-hydroxyl-dihydrofuropsoralen and rutamine isolated from *R. chalepensis* were reported to have larvicidal activity against *Spodoptera littoralis* (Emam et al. 2009). The active essential oil component of *R. graveolens* 2-isopropyl-5-methylphenol and its derivatives namely 5-isopropyl-2-methylphenol, 4-isopropyl-3-methylphenol, 2-methylphenol, 3-methylphenol, 4-methylphenol, 2-isopropylphenol, 3-isopropylphenol and 4-isopropylphenol were tested against *S. oryzae*, *S. zeamais* and *Lasioderma serricorne* (Fabricius) with fumigant toxicity assay; highest activity was observed for 3-isopropylphenol (Jeon et al. 2015). *R. chalepensis* essential oils were also reported to have antifungal activity against the fungal species that produce damage to agricultural products such as species from *Alternaria*, *Aspergillus*, *Botrytis*, *Fusarium* and *Phytophthora* genus (Bouabidi et al. 2015, Bouajaj et al. 2014, Chebli et al. 2004, Haddouchi et al. 2013). Additionally, *R. chalepensis* essential oil was reported to have seed germination inhibition against *Triticum durum* Desf. and *Phalaris canariensis* L. (Bouajaj et al. 2014, Mejri et al. 2012).

Inhibition of the cholinergic system enzymes is reported to be one of the possible insecticidal action mechanisms of essential oils (Rattan 2010). Essential oil components with inhibitory properties against AChE were reported in the literature (López and Pascual-Villalobos 2010, Miyazawa et al. 1997, Miyazawa and Yamafuji 2005). Previously, AChE inhibitory properties of methanol and water extracts of *Ruta graveolens* L. were reported (Adersen et al. 2006). Another report shows that hexane extract of the same species inhibits AChE and BChE enzymes better than its methanol extract (Wszelaki et al. 2010). This report also indicates that non-polar substances which include components of the essential oil present in the plant could be responsible for the previously reported insecticidal activity.

A considerable number of reports regarding the insecticidal activity of the *Ruta* species as well as a lack of any reports on the chemical composition of *Ruta chalepensis* from Cyprus presented a high potential for discovering new insecticidal lead compounds from this genus which prompted us to do present research. Herein, it is aimed to determine insecticidal, AChE, and BChE inhibitory activity and essential oil composition of *R. chalepensis* as a part of our phytochemical and insecticidal activity screening study of Cypriot plants.

MATERIALS AND METHODS

Plant materials

Plant samples were collected on 25 May 2015 from St. Hilarion - Kyrenia. The voucher specimen has been placed at the Herbarium of the Near East University of Cyprus' Institute of Environmental Sciences (Voucher number.

6890). Dr. Salih Gücel from the Institute of Environmental Sciences, Near East University, Nicosia, identified the plant materials.

Isolation of the essential oils

Using a Clevenger-style device, aerial parts (100 g) of the air-dried plant were hydro distilled for 3 hours to obtain essential oils with yields of 1.27% (v/w). After being dried over anhydrous Na₂SO₄, oil was kept in an amber vial at -20 °C until the day it was tested.

Gas chromatography-mass spectrometry analysis

With Agilent 7890B GC and 5977B EI MSD equipment, the GC-MS analysis was carried out. Samples of essential oils were diluted by 1/10 (v/v) in n-hexane. Temperatures were set at 250 °C for the MS transfer line and injector. The 50:1 split ratio was chosen. Both GC/MS studies employed an Innowax FSC column (60 m x 0.25 mm, 0.25 μm film thickness) and helium as the carrier gas (1 ml/min). The temperature of the oven was set at 60 °C for 10 minutes before being increased to 220 °C at a rate of 4 °C/min. After being held at 220 °C for 10 minutes, the temperature was increased to 240 °C at a rate of 1 °C/min. At 70 eV, mass spectra covering the m/z range of 35 to 425 were captured. The integration of the peaks in MS chromatograms was used to determine the relative percentage quantities of the separated substances. The components of essential oils were identified. The commercial Wiley 8th Ed./NIST 05 Mass Spectra Library, the Adams Essential Oil Mass Spectral Library, and the Pallisade 600K Complete Mass Spectral Library were used for computer matching throughout the mass spectra comparison process. The findings of the analysis were presented as the mean standard deviation after being performed in triplicate. Figure 1 shows the GC-MS chromatogram for the study of *Ruta chalepensis* aerial parts oil.

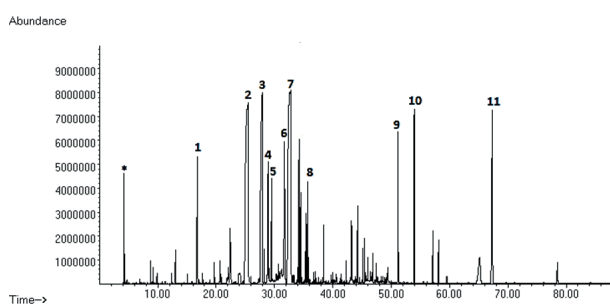


Figure 1. GC/MS Chromatogram of *Ruta chalepensis* aerial parts oil. Compounds: *. Solvent peak (n-hexane); 1. Limonene; 2. 2-Nonanone; 3. 2-Nonyl acetate; 4. 2-Decanone; 5. 2-Nonanol; 6. Pregeijerene; 7. 2-Undecanone; 8. 2-Dodecanone; 9. Moskachan A; 10. Moskachan B; 11. Moskachan D

Stored product insect cultures

Sitophilus oryzae, *Sitophilus granarius*, *Rhyzopertha dominica*, *Tribolium confusum* and *Tribolium castaneum* were collected from the infested stored products in Türkiye. Insect cultures were prepared at Çankırı Karatekin University, Yapraklı Vocational School, in the laboratory of the Animal and Plant Production Department according to the previous methods (Karakoç et al. 2006, Pimentel et al. 2008).

Insecticidal activity assays

Insecticidal contact toxicity assay

To create a 10% (v/v) stock solution, *Ruta chalepensis* essential oil was diluted with acetone. The dorsal side of the thorax of the insects was exposed to a stock solution of oil sample (1 µl) using a 50 µl Hamilton syringe (Gokce et al. 2010). The same volume of acetone was used in the empty controls. Following the sample or blank application, the insects were transferred to 60 mm glass petri plates containing 5 g of either whole wheat (for *S. granarius* and *S. oryzae*) or broken wheat (for *T. castaneum*, *T. confusum* and *R. dominica*) (n = 10 for each bug species and treatments). For 72h the insects were raised at 50 ± 10% relative humidity and 27 ± 2 °C. At 24, 48, and 72 hours, the samples and controls were observed, and the number of dead insects was noted. Each sample and blank treatment were made at least three times.

Insecticidal fumigant toxicity assay

The fumigant toxicity of *Ruta chalepensis* essential oil against the mentioned insects was determined according to a previous protocol (Çam et al. 2012). At the 48th hour, samples and controls were checked, and the number of dead insects was noted. Each sample and blank treatment were made at least three times.

Acetylcholinesterase (AChE) and butyrylcholinesterase (BChE) inhibitory activity

The previously mentioned technique was used to assess the *Ruta chalepensis* essential oil's inhibitory effect on AChE and BChE (Ellman et al. 1961). AChE (Electric Eel) or BChE was added to the reaction mixture at a concentration of 0.0325 U/ml to initiate reactions. The average and standard deviation of three parallel tests were used to display the experiment's results.

Statistical analysis

In order to calculate the arcsin values, the activity findings from the insecticidal activity assays were converted into % death values (Zar 1996). ANOVA was performed on the arcsin values before Tukey's multiple comparison tests

with a P<0.05 significance threshold. The software package Minitab Release 14 (McKenzie and Goldman 2005) was used for all of the statistical analyses.

RESULTS

The essential oil composition of aerial parts of *Ruta chalepensis* was given in Table 1. Seventy-nine compounds were detected representing 85.93 ± 1.08 % (n = 3) of *R. chalepensis* aerial parts oil. The essential oil was dominated by the derivatives of n-alkanes. The major components of the essential oil were 2-undecanone 21.52 ± 0.21%, 2-nonanone 18.31 ± 0.27%, and 2-nonyl acetate 13.22%.

However, there were differences in the relative percent amount of the major components of the essential oil as well as the composition of the secondary components of the oil. The essential oil investigated in the present study afforded moskachan derivatives as the components that have a relative percentage in the range of 2-5% very similar to a previous study (Joulain et al. 1991) unlike the other studies.

The insecticidal contact toxicity of the *Ruta chalepensis* oil against *S. granarius*, *S. oryzae*, *T. castaneum*, *T. confusum* and *R. dominica* was given in Table 2. *R. chalepensis* essential oil was active against all the insects with different LD50 and LD90 values. The highest activity of the oil was observed against *R. dominica* with 0.018 µl/insect LD50 and 0.039 µl/insect LD90 after 24h. The highest activity was observed after 24h of application against all insect species except for *R. dominica*. The oil showed the highest activity after 24h against *R. dominica* and there was no change after 48h and 72h of applications. *S. oryzae* and *S. granarius* were also affected considerably with 0.50 and 0.59 µl/insect LD50 after 24h respectively. However, this activity increased with longer exposure times and produced lower LD50 values. The lowest contact toxicity was observed against *T. castaneum* and *T. confusum* at 0.138 and 0.078 µl/insect LD50 after 24h respectively. The contact toxicity of the essential oil slightly increased at longer exposure periods for *T. confusum* and *T. castaneum*.

The fumigant toxicity of the *Ruta chalepensis* oil against *S. granarius*, *S. oryzae*, *T. castaneum*, *T. confusum* and *R. dominica* was given in Table 3. The highest fumigant toxicity was observed against *S. granarius* for the application concentration of 10 µl, 10% oil/acetone (v:v) in a 10 ml chamber which afforded 100.00 ± 0.00% mortality. The toxicity of the oil produced by the oil was decreased to 98.86 ± 3.41% mortality at 5 µl and to 0.00 ± 0.00% mortality at 2.5 µl application of the oil solution for *S. granarius*. The essential oil also produced high fumigant toxicity against *S. oryzae*, *T. castaneum* and *R. dominica* which were 95.47 ± 3.41%, 93.30 ± 5.54% and 85.47 ± 3.41% mortality at 20 µl

Table 1. The essential oil composition of aerial parts of *Ruta chalepensis*

No	RRI ¹	RRI Lit. ²	Compound	I ³	II	III	Ave ⁴	SD ⁵	IM ⁶	Literature
1	1023	1032	α -Pinene	0.24	0.21	0.22	0.22	0.02	RI ⁷ , MS ⁸	Başer et al. 2000a)
2	1027	1176	α -Thujene	0.02	0.02	0.02	0.02	0.00	RI, MS	(Tabanca et al. 2011)
3	1033	1035	2-Methyl-3-buten-2-ol	0.18	0.17	0.17	0.17	0.01	RI, MS	(Başer et al. 2000b)
4	1038	1042	Isopropyl butyrate	0.01	0.01	0.01	0.01	0.00	RI, MS	(Narain et al. 2007)
5	1051	1061	Isopropyl-2-methyl butyrate	0.13	0.12	0.13	0.13	0.01	RI, MS	(Başer et al. 2002a)
6	1069	1079	Ethyl isovalerate	0.01	0.01	0.01	0.01	0.00	RI, MS	(Kamatou et al. 2008)
7	1075	1083	Butanoic acid, 3-methyl-1methylethyl ester	0.02	0.02	0.02	0.02	0.00	RI, MS	(Kaya et al. 2003)
8	1087	1093	Hexanal	0.03	0.03	0.03	0.03	0.00	RI, MS	(Başer et al. 2000a)
9	1112	1118	β -Pinene	0.13	0.12	0.12	0.12	0.01	RI, MS	(Başer et al. 2000a)
10	1126	1132	Sabinene	0.4	0.38	0.38	0.39	0.01	RI, MS	(Başer et al. 2000a)
11	1168	1174	Myrcene	0.11	0.11	0.11	0.11	0.00	RI, MS	(Başer et al. 2001)
12	1184	1188	α -Terpinene	0.03	0.03	0.03	0.03	0.00	RI, MS	(Başer et al. 2000a)
13	1186	1192	2-Heptanone	0.03	0.03	0.03	0.03	0.00	RI, MS	(Başer et al. 2004)
14	1205	1203	Limonene	2.49	2.34	2.4	2.41	0.08	RI, MS	(Başer et al. 2000a)
15	1210	1213	1,8-Cineole	0.05	0.05	0.05	0.05	0.00	RI, MS	(Başer et al. 2001)
16	1215	1213	4-Methoxy-2-methyl-2-mercaptobutane	0.01	0.01	0.01	0.01	0.00	RI, MS	(Kumazawa and Masuda 1999).
17	1224	1232	(E)-2-Hexanal	0.11	0.1	0.1	0.10	0.01	RI, MS	(Kırimer et al. 2000)
18	1238	1244	2-Pentylfuran	0.03	0.03	0.03	0.03	0.00	RI, MS	(Başer et al. 2000a)
19	1251	1255	γ -Terpinene	0.06	0.06	0.06	0.06	0.00	RI, MS	(Başer et al. 2000a)
20	1258	1265	(E)- β -Ocimene	0.01	0.01	0.01	0.01	0.00	RI, MS	(Başer et al. 2002b)
21	1277	1278	m-Cymene	0.06	0.01	0.01	0.03	0.03	RI, MS	(Kılıç et al. 2010)
22	1290	1295	2-Octanone	0.3	0.29	0.29	0.29	0.01	RI, MS	(Başer et al. 2006)
23	1294	1296	Octanal	0.14	0.14	0.14	0.14	0.00	RI, MS	(Başer et al. 2001)
24	1318		Geijerene derivative I	0.08	0.08	0.08	0.08	0.00	MS	
25	1332	1338	Geijerene	1.64	1.53	1.72	1.63	0.10	RI, MS	(Tabanca et al. 2007)
26	1344	1348	6-Methyl-5-hepten-2-one	0.01	0.01	0.01	0.01	0.00	RI, MS	(Başer et al. 2000a)
27	1387	1391	(Z)-3-Hexen-1-ol	0.04	0.04	0.03	0.04	0.01	RI, MS	(Kırimer et al. 2000)

28	1410	1398	2-Nonanone	18.58	18.04	18.32	18.31	0.27	RI, MS	(Başer et al. 2006)
29	1423	1426	2-Octanol	0.08	0.08	0.08	0.08	0.00	RI, MS	(Van Vuuren et al. 2006)
30	1479	1470	2-Nonyl acetate	13.41	13.04	13.21	13.22	0.19	RI, MS	(Van Vuuren et al. 2007)
31	1486	1483	Acetic acid octyl ester	0.31	0.31	0.31	0.31	0.00	RI, MS	(Başer et al. 2000c)
32	1501	1493	Isogeijerene c	0.03	0.03	0.03	0.03	0.00	RI, MS	(Tabanca et al. 2007)
33	1505	1491	2-Decanone	2.3	2.21	2.23	2.25	0.05	RI, MS	(Bendimerad et al. 2005)
34	1509	1506	Decanal	0.23	0.24	0.23	0.23	0.01	RI, MS	(Başer et al. 2001)
35	1516	1516	Geyrene	0.06	0.06	0.06	0.06	0.00	RI, MS	(Das 2015)
36	1524	1521	2-Nonanol	1.98	1.93	1.93	1.95	0.03	RI, MS	(Kıvçak et al. 2004)
37	1528		Geijerene derivative II	0.14	0.14	0.14	0.14	0.00	MS	
38	1535		Geijerene derivative III	0.04	0.04	0.04	0.04	0.00	MS	
39	1556		Geijerene derivative IV	0.13	0.14	0.13	0.13	0.01	MS	
40	1577	1572	Pregeijerene B	0.92	0.9	0.88	0.90	0.02	RI, MS	
41	1591	1594	Pregeijerene	3.68	3.61	3.5	3.60	0.09	RI, MS	(Tabanca et al. 2005)
42	1628	1604	2-Undecanone	21.73	21.32	21.51	21.52	0.21	RI, MS	(Kırimer et al. 2000)
43	1635	1651	6-Methyl-1-octanol	0.07	0.07	0.07	0.07	0.00	RI, MS	(Wen et al. 2014)
44	1665	1664	1-Nonanol	0.23	0.23	0.23	0.23	0.00	RI, MS	(Başer et al. 2001)
45	1682	1674	2-Dodecanone	1.19	1.15	1.15	1.16	0.02	RI, MS	(Kawakami and Kobayashi 1991)
46	1688	1687	Decyl acetate	0.07	0.07	0.07	0.07	0.00	RI, MS	(Tabanca et al. 2006)
47	1694	1694	p-Vinylanisole	0.01	0.01	0.01	0.01	0.00	RI, MS	Başer et al. 2002b)
48	1698	1676	1,8-Menthadien-4-ol	0.03	0.03	0.02	0.03	0.01	RI, MS	(Kollmannsberger et al. 1992)
49	1707	1706	α -Terpineol	0.3	0.29	0.29	0.29	0.01	RI, MS	(Başer et al. 2001)
50	1715	1766	1-Decanol	0.73	0.71	0.71	0.72	0.01	RI, MS	(Kırimer et al. 2000)
51	1719	1719	1-Heptadecene	0.02	0.01	0.01	0.01	0.01	RI, MS	(Kamariah et al. 1999)
52	1724	1722	2-Undecanol	1.36	1.35	1.35	1.35	0.01	RI, MS	(Kırimer et al. 2000)
53	1735	1728	(Z,E)- α -Farnesene	0.05	0.05	0.05	0.05	0.00	RI, MS	(Özek et al. 2010)
54	1758	1758	(E,E)- α -Farnesene (= α -farnesene)	0.12	0.12	0.12	0.12	0.00	RI, MS	(Demirci et al. 2006)
55	1766	1766	1-Decanol	0.04	0.04	0.04	0.04	0.00	RI, MS	(Kırimer et al. 2000)
56	1770		2-Methyl-undecanal	0.11	0.11	0.11	0.11	0.00	MS	

57	1797	1800	Methyl salicylate	0.02	0.02	0.02	0.02	0.00	RI, MS	(Kırimer et al. 2000)
58	1819	1815	2-Tridecanone	0.62	0.67	0.6	0.63	0.04	RI, MS	(Demirci et al. 2006)
59	1837	1835	β -Damascenone	0.01	0.01	0.01	0.01	0.00	RI, MS	(Başer et al. 2000a)
60	1845	1845	trans-Carveol	0.01	0.01	0.01	0.01	0.00	RI, MS	(Başer et al. 2000a)
61	1901	1904	Ethyl dihydrocinnamate	0.1	0.1	0.1	0.10	0.00	RI, MS	(Polatoğlu et al. 2010)
62	1955	1958	(E)- β -Ionone	0.04	0.04	0.04	0.04	0.00	RI, MS	(Demirci et al. 2006)
63	2026	2029	Methyl eugenol	0.04	0.05	0.04	0.04	0.01	RI, MS	(Başer et al. 2001)
64	2093	2099	Hedycaryol	0.4	0.4	0.39	0.40	0.01	RI, MS	(Joichi et al. 2005)
65	2101	2181	Anthranilic acid	0.17	0.17	0.15	0.16	0.01	RI, MS	(Pino et al. 2009)
66	2134	2131	Hexahydrofarnesyl acetone	0.06	0.05	0.07	0.06	0.01	RI, MS	(Başer et al. 2000a)
67	2186	2185	γ -Eudesmol	0.07	0.07	0.06	0.07	0.01	RI, MS	(Başer et al. 2001)
68	2197	2192	τ -Cadinol	0.02	0.02	0.02	0.02	0.00	RI, MS	(Tümen et al.1998)
69	2241	2250	α -Eudesmol	0.06	0.06	0.06	0.06	0.00	RI, MS	(Başer et al. 2001)
70	2251	2257	β -Eudesmol	0.09	0.09	0.08	0.09	0.01	RI, MS	(Demirci et al. 2006)
71	2342		Moskachan A	1.95	1.93	1.91	1.93	0.02	MS	
72	2383	2181	1-Nonadecanol	0.03	0.03	0.03	0.03	0.00	RI, MS	
73	2400	2400	Tetracosane	0.02	0.02	0.02	0.02	0.00	RI, MS, Ac ⁹	
74	2449		Moskachan B	2.77	2.75	2.72	2.75	0.03	MS	
75	2504	2500	Pentacosane	0.04	0.04	0.04	0.04	0.00	RI, MS, Ac	
76	2551		4-(3,4-Methylenedioxy-phenyl)-2-butanone	0.77	0.77	0.77	0.77	0.00	MS	
77	2617	2622	Phytol	0.11	0.11	0.11	0.11	0.00	RI, MS	(Kırimer et al. 2000)
78	2700	2700	Heptacosane	0.03	0.02	0.01	0.02	0.01	RI, MS, Ac	
79	2793		Moskachan D	5.38	5.33	5.3	5.34	0.04	MS	
Monoterpenes				10.27	9.82	9.94	10.01	0.23		
Oxygenated Monoterpenes				0.44	0.43	0.42	0.43	0.01		
Sesquiterpenes				0.17	0.17	0.17	0.17	0.00		
Oxygenated Sesquiterpenes				10.80	10.70	10.61	10.70	0.10		
Diterpenes				0.11	0.11	0.11	0.11	0.00		
Others				65.34	63.82	64.36	64.51	0.77		
Total				87.13	85.05	85.61	85.93	1.08		

¹Relative retention index; ²Relative retention index of the compound given in the literature that uses similar methodology; ³The GC-MS analyses were performed in triplicate and results were given as relative percent amounts calculated from the chromatograms; ⁴Average relative percent amounts obtained from the analyses; ⁵Standard deviation of the relative percent amounts; ⁶Identification method used for the compound; ⁷RI: Relative retention index match of the compound with the literature that uses same/similar chromatographic conditions; ⁸MS: Mass spectra match of the compound; ⁹Ac: Relative retention index and mass spectra similarity of the compound with the authentic compound (co-injection).

Table 2. The contact toxicity of the *Ruta chalepensis* oil given for 24, 48, and 72h periods as LD50 and LD90

Insect Species	24 h				48 h				72 h			
	LD ₅₀ (µL/insect) (Fiducial limits)	LD90 (µL/insect) (Fiducial limits)	Slope±SE	X ²	LD ₅₀ (µL/insect) (Fiducial limits)	LD90 (µL/insect) (Fiducial limits)	Slope±SE	X ²	LD ₅₀ (µL/insect) (Fiducial limits)	LD90 (µL/insect) (Fiducial limits)	Slope±SE	X ²
<i>S. granarius</i>	0.059 (0.052-0.067)	0.107 (0.089-0.141)	5.02±0.77	7.98	0.052 (0.045-0.059)	0.096 (0.081-0.125)	4.76±0.66	11.37	0.052 (0.045-0.059)	0.096 (0.081-0.125)	4.76±0.66	11.37
<i>S. oryzae</i>	0.050 (0.040-0.061)	0.090 (0.072-0.138)	4.97±0.69	22.94	0.046 (0.038-0.053)	0.083 (0.069-0.110)	4.92±0.67	13.94	0.040 (0.034-0.045)	0.071 (0.061-0.088)	5.06±0.68	7.93
<i>T. castaneum</i>	0.138 (0.118-0.162)	0.327 (0.259-0.480)	3.42±0.49	11.65	0.116 (0.092-0.146)	0.316 (0.231-0.559)	2.95±0.39	22.57	0.111 (0.089-0.137)	0.311 (0.230-0.522)	2.86±0.38	19.43
<i>T. confusum</i>	0.078 (0.060-0.099)	0.340 (0.238-0.628)	2.01±0.31	11.90	0.078 (0.060-0.099)	0.340 (0.238-0.628)	2.01±0.31	11.90	0.073 (0.055-0.093)	0.324 (0.227±0.597)	2.011±0.31	12.48
<i>R. dominica</i>	0.018 (0.011-0.023)	0.039 (0.029-0.066)	3.83±0.59	11.63	0.018 (0.014-0.022)	0.038 (0.029-0.062)	3.92±0.60	11.31	0.018 (0.014-0.022)	0.038 (0.029-0.062)	3.92±0.60	11.31

application concentration of the oil solution in acetone. The fumigant toxicity of the oil was concentration-dependent. According to the results presented in Table 3, the most susceptible insect species was *S. granarius* to the essential oil of *R. chalepensis*. The essential oil did not produce any fumigant toxicity against *T. confusum*.

The contact toxicity study of the essential oil provided a different activity profile than the fumigant toxicity study. The highest contact toxicity was observed for *R. dominica* whereas the highest fumigant toxicity was observed for *S. granarius*. This fact states that active substances were less active in the vapor phase for *R. dominica*. In contact toxicity, this fact could be explained by different cuticle penetration amounts of active substances in different insects.

The AChE and BChE inhibitory activity of the oil was determined at 20 µl application volume of 10 mg/ml oil solution in methanol in the assay mixture. The oil produced 5.29 ± 1.20% inhibition of the AChE and 42.6

± 0.71% inhibition of BChE. The oil produced very low AChE inhibition and mediocre BChE inhibition, therefore observed insecticidal activity could be related to another mechanism than the action on the cholinergic system.

DISCUSSION

Previously insecticidal activity of 2-undecanone was reported against *Keiferia lycopersicella* (Walsingham) and *Spodoptera exigua* (Hübner) of Lepidoptera (Lin et al. 1987). Additionally, the biting deterrence and larvicidal activity of *R. chalepensis* essential oil, 2-undecanone, 2-nonanone and 2-nonyl acetate against *Aedes aegypti* L. and *Anopheles quadrimaculatus* Say. were documented in detail (Ali et al. 2013). The insecticidal activity observed for *R. chalepensis* essential oil could be related to the major components namely 2-undecanone, 2-nonanone, and 2-nonyl acetate.

The essential oil of *Ruta chalepensis* from Cyprus has very high fumigant insecticidal activity against *S. granaries* and

Table 3. The fumigant toxicity of the *Ruta chalepensis* oil given for 48 h period as % mortality

Test insects	Mortality±SD (%)				
	Doses (µl (10% (v/v) oil/acetone) ¹				
	Control ²	20	10	5	2.5
<i>Sitophilus granarius</i>	0.00±0.00	100.00±0.00	100.00±0.00	98.86±3.41	0.00±0.00
<i>Sitophilus oryzae</i>	0.00±0.00	95.47±3.41	56.69±0.34	25.00±5.54	N.A.
<i>Tribolium castaneum</i>	0.00±0.00	93.30±5.54	80.69±1.66	4.53±3.41	N.A.
<i>Tribolium confusum</i>	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	N.A.
<i>Rhyzopertha dominica</i>	0.00±0.00	85.47±3.41	53.35±1.35	6.70±5.54	N.A.

¹The volume (µl) of essential oil solution (essential oil is diluted with acetone 10% (v/v)) applied in the fumigant insecticidal activity assay (chamber size is 10 ml). ²Control (blank) is essential oil-free acetone.

S. oryzae, *Ryzopherta dominica* and *Tribolium castaneum*. The oil also produced considerably high insecticidal activity against *Ryzopherta dominica*, *S. granaries*, and *S. oryzae*. The major components of the *Ruta chalepensis* oil namely 2-undecanone, 2-nonanone, and 2-nonyl acetate were previously reported to have insecticidal and repellent activity against other insect species (Ali et al. 2013, Lin et al. 1987). Consequently, these substances are thought to be the cause of the insecticidal effects seen in the current study on the species under investigation. However, the insecticidal activity of 2-undecanone, 2-nonanone, and 2-nonyl acetate should be investigated in detail against *S. granarius*, *S. oryzae*, *T. castaneum*, *T. confusum* and *R. dominica* to prove their activity. Additionally, the *R. chalepensis* essential oils from different locations were reported to have seed germination inhibition against *Triticum durum* Desf. and *Phalaris canariensis* L. which contains 2-undecanone and 2-nonanone major compounds (Bouajaj et al. 2014, Mejri et al. 2012). The inhibitory activity of 2-undecanone and 2-nonanone on germination of radish was reported to be 38% and 30% respectively which could be related to low inhibition values (De Feo et al. 2002). The essential oil of *R. chalepensis* appears to be a promising source from which natural compounds can be extracted that can be used in the pest control of stored product.

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Author's Contributions

Authors declare the contribution of the authors is equal.

Statement of Conflict of Interest

The authors have declared no conflict of interest.

ÖZET

Ruta chalepensis L.'nin toprak üstü aksamından elde edilen uçucu yağ kompozisyonu GC-MS ile analiz edildi. Uçucu yağın 85.93 ± 1.08 ($n = 3$)'i temsil eden yetmiş dokuz bileşik tespit edildi. Uçucu yağın ana bileşenleri olarak 21.52 ± 0.21 oranında 2-undekanon, 18.31 ± 0.27 oranında 2-nonanon ve 13.22 oranında da 2-nonil asetat belirlendi. Uçucu yağı en yüksek insektisidal kontak toksisitesi 24 saat sonra $0.018 \mu\text{l}/\text{böcek}$ LD50 ve $0.039 \mu\text{l}/\text{böcek}$ LD90 değerleri ile *Rhyzopertha dominica* F. üzerinde gözlemlenmiştir. Uçucu yağ, ayrıca *Sitophilus oryzae* L. ve *Sitophilus granarius* L. karşısında 24 saat sonra sırasıyla 0.50 ve $0.59 \mu\text{l}/\text{böcek}$ değerleriyle oldukça düşük

denebilecek LD50 değerlerini gösterdi. En düşük kontak toksisite 24 saat sonra *Tribolium castaneum* Herbst. ve *Tribolium confusum* Jacquelin du Val'e karşı sırasıyla 0.138 ve $0.078 \mu\text{l}/\text{böcek}$ LD50 değerleri şeklinde gözlemlendi. *S. granarius*'a karşı en yüksek fumigant toksisite 48 saat sonra %100 ölüm oranını sağlayan ve 10 m^3 'lik haznede uygulanan $10 \mu\text{l}$, %10 yağ/aseton (v:v) konsantrasyonunda görüldü. Uçucu yağ ayrıca *S. oryzae*, *T. castaneum* ve *R. dominica*'ya karşı yüksek fumigant toksisiteyi de ortaya çıkardı. 48 saat sonra $20 \mu\text{l}$ yağ çözeltilisi uygulama konsantrasyonunda ölüm oranı sırasıyla 95.47 ± 3.41 , 93.30 ± 5.54 ve 85.47 ± 3.41 idi. *R. chalepensis* uçucu yağı ile 5.29 ± 1.20 'lik düşük bir asetilkolinesteraz enzim inhibisyonu ile 42.6 ± 0.71 'lik orta derecede bir bütirilkolinesteraz enzim inhibisyonu elde edildi. Yapılan insektisidal aktivite denemelerine göre, *R. chalepensis* uçucu yağının, depo ürün zararlılarına karşı mücadelede kullanılabilir doğa bileşiklerin elde edilmesi adına umut verici bir kaynak olduğu düşünülmektedir.

Anahtar Kelimeler: *Ruta chalepensis*, uçucu yağ analizi, depo ürün zararlıları, asetilkolinesteraz, bütirilkolinesteraz, enzim inhibisyonu

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