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Chemical profile, characterization and acaricidal activity of essential oils of three plant species and their nanoemulsions against *Tyrophagus putrescentiae*, a stored-food mite

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Abstract

Essential oils of *Ocimum basilicum* (L.), *Achillea fragrantissima* (Forssk.) and *Achillea santolina* (L.) were obtained by hydrodistillation and analyzed using gas chromatography (GC) and GC/mass spectrometry (MS). Oil-in-water nanoemulsions (10% active ingredient) were prepared through a high-energy (ultrasonication) emulsification process. Nanoemulsions were characterized by viscosity, pH, thermodynamic stability, droplet size, polydispersity index (PDI) and scanning electron microscopy (SEM) measurements. The plant oils and their nanoemulsions showed considerable acaricidal activity against the mold mite, *Tyrophagus putrescentiae* (Schrank) (Sarcoptiformes: Acaridae). In a contact toxicity bioassay and 48 h post treatment, *O. basilicum* oil was the most toxic, followed by *A. fragrantissima* and *A. santolina*, where LC_{50} values were 8.4, 14.1 and 21.8 µl/cm^2, respectively. LC_{50} for benzyl benzoate, a standard acaricide was 9.8 µl/cm^2. Upon fumigation, responses also varied according to the test oil. Based on the 48-h LC_{50} values, the same manner of activity was also observed, where *O. basilicum* was the most toxic followed by *A. fragrantissima* and *A. santolina*. When prepared as nanoemulsions (particle size from 78.5 to 104.6) and tested as fumigants, toxicity of the oils was increased drastically with LC_{50} values of 2.2, 4.7, and 9.6 µl/l air for *O. basilicum*, *A. fragrantissima* and *A. santolina*, respectively. The oils showed a moderate to strong residual acaricidal activity, where *O. basilicum* oil was the most effective. The results suggest that appropriate nanoemulsions containing the tested oils can be developed to control *T. putrescentiae* after the required toxicological assessments.

Keywords Essential oils · Nanoemulsions · Acaricidal activity · *Tyrophagus putrescentiae*

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Introduction

In the storage ecosystem, mites play an important role in the deterioration process and are intimately associated with agricultural production. One of the various acarines associated with the stored grain ecosystem is the mold mite, *Tyrophagus putrescentiae* (Schrank) (Sarcoptiformes: Acaridae). It is a cosmopolitan post-harvest pest of durable stored foods with a high fat and protein content, such as dry cured ham, grains, aged cheese, spices, mushroom, dried fruit, dried eggs, nuts, and other stored foods (Hughes 1976; Brazis et al. 2008). In addition to destroying food, there is evidence that *T. putrescentiae* may be a source of allergens affecting farmers and workers handling heavily infested stored products, and causes acute enteritis, dermatitis (Mueller et al. 2005), and systemic anaphylaxis (Matsumoto et al. 1996) when contaminated food is ingested. *T. putrescentiae* also acts as a carrier of bacteria and toxigenic fungi such as *Aspergillus* spp. and *Penicillium* spp. in stored grain kept under warm and moist conditions (Franzolin et al. 1999). Mold mites, including *T. putrescentiae*, may be found at several points along the food storage, processing, and distribution system, including raw grain through to finished food products. Once a mite outbreak has been detected, it can be difficult to fully control the infestation (Gill et al. 2011).

Tyrophagus putrescentiae is one of the most difficult pests to control in the dry environment because of its morphological, ecological, physiological and behavioral characteristics (Boczek 1991). However, control of this mite depends heavily on the use of chemical methods such as fumigation, spraying with organophosphorus compounds, or treatment with benzyl benzoate because ecological control methods with high humidity and temperature cause alterations in food quality (Hagstrum et al. 2012). The repeated use of chemical acaricides, however, has resulted in the development of resistant strains of mites, showed undesirable effects on non-target organisms, and has fostered environmental and human health concerns (Park et al. 2014). These problems have highlighted the need for the development of new strategies for selective storage mite control using biorational tools, which do not affect human health as well as the organoleptic characters of food products. Natural plant products may provide ecofriendly alternatives to currently used pest control agents (Nenaah and Ibrahim 2011).

In the literature, much effort has been focused on the plant essential oils as potential sources of commercial storage mite control agents, especially against *T. putrescentiae* (Macchioni et al. 2002; Kim et al. 2003a, b, 2004; Lee et al. 2006; Sung et al. 2006; Park et al. 2014; Song et al. 2016). Plant-derived essential oils, in general, are considered minimum-risk pesticides and are exempt from Environmental Protection Agency registration under Sect. 25 (b) of the Federal Insecticide Fungicide and Rodenticide Act. However, the performance of most phytochemicals, including plant oils in pest control protocols, is inadequate for major practical use due to concerns associated with extraction, formulation and application (Nenaah 2014c; Nenaah et al. 2015). The urgent need now is the search for innovative strategies for developing and strengthening the use of plant-based products as alternative pest control agents (Almadiy et al. 2018).

In this context, nanotechnology has emerged as a promising area for developing and utilizing nano-sized particles with a wide range of uses in material science, medicine, pharmacology and pesticide applications (Khot et al. 2012). When transformed into nanoparticles, materials could acquire novel biological properties, as they offer large specific surface area and hence increased affinity to the target, penetrate rapidly and are selectively accumulated in biosystems enhancing various activities within the living cells (Weiss et al. 2006). The current

study was designed to evaluate the chemical composition and the acaricidal activity of the essential oils of three plant species namely, *Ocimum basilicum, Achillea fragrantissima*, and *A. santolina* and their nanoemulsions against the mold mite, *T. putrescentiae.*

Materials and methods

Test mites

A stock culture of *T. putrescentiae* was established from infested samples of dry stored grains obtained from a governmental drying store in Kafr Elsheikh Governorate, Egypt. Mites were identified according to the key described by Robertson (1959). Mites were maintained in the laboratory for 2 years without exposure to any pesticidal contamination. Mites were reared in plastic containers (15 × 10.5 × 6 cm) containing 30 g of sterilized diet (fry feed No. 1: dried yeast, 1: 1 (wt/wt); Korea Special Feed Meal, Inchon, Korea), each covered with a round plastic plate with a 3-cm-diameter hole in the center, which was sealed with a filter paper disk for ventilation. The rearing cages were kept at 25 ± 2 °C and 70 ± 5% r.h. in darkness. Adults were sexed by observing their secondary sexual characters (Hughes 1976).

Collection and preparation of the test plants

The aerial parts of *A. fragrantissima* and *A. santolina* were collected from Sinai Peninsula and Allamain desert, respectively, whereas *O. basillicum* were collected from local gardens at Gharbiya Governorate, Egypt, at the flowering period (May 2017). Plant samples were identified and authenticated by botanists of the Botany Department, Faculty of Science, Tanta University, Egypt, where voucher specimens were deposited for further reference (voucher numbers are *Ob* 01, *Af* 01 and *As* 01). The fresh plant samples were air-dried in the shade for 5 days at environmental temperature (28–32 °C daytime). The dried parts were powdered mechanically using an electric blender (Multiquick Immersion Hand Blender, B White Mixer MR 5550 CA, Braun, Germany), then sieved through a mesh size of 0.5 mm. The resulting fine powders were maintained in tightly closed dry bags until used for the extraction of the essential oils.

Extraction of essential oils

Powdered samples, 1 kg each, from the tested plants were hydrodistilled using a modified Clevenger-type apparatus to produce the plant oils. The extraction condition was as follows: 50 g powders, 500 ml distilled water, and 6 h distillation. Anhydrous sodium sulphate was utilized to remove water after extraction. The oil yield (% wt/wt) was calculated on a dry weight basis. The extracted oils were stored in a refrigerator at 4 °C until analyzed and tested.

Preparation of oil nanoemulsions

Oil-in-water nanoemulsion from the test plants was prepared by using the high-energy ultrasonic method (Badawy et al. 2017). The nanoemulsions were prepared in two phases. Firstly, EOs were emulsified using a high energy ultrasonic process, then mixed with surfactant (Tween 80) as a non-ionic surfactant and deionized water at a ratio of 1:2:7,

respectively, with a final concentration of 10% yielding coarse emulsion formulations. Emulsions were prepared by dropping organic phase containing oil to water phase (water and surfactant) using a magnetic stirrer for 30 min at 4000 rpm. The emulsions formed were then subjected to ultrasonic emulsification for 15 min with sonication power of 10 kHz (9 cycle/sec) controlled by the software of the device (Ultrasonic Homogenizers HD 2070) with HF generator (GM 2070), ultrasonic converter UW2070, booster horn (SH 213 G) and probe microtip MS 73, Ø 3 mm). The difference of temperature from the initial coarse emulsion to the final emulsion was not more than 25 °C (Anjali et al. 2012).

Characterization of the oil nanoemulsions

Physicochemical characterization and stability of the prepared nanoemulsions were evaluated to different stress, such as thermodynamics, centrifugation, heating, cooling, and freeze cycles (Ghosh et al. 2013). Nanoemulsions were centrifuged at 5000 rpm for 30 min at 25 °C using Heraeus Labofuge 400R (Kendro Laboratory Products, Germany) and observed for phase separation, creaming, and cracking if any. This method has been widely used to determine the stability of nanoemulsions (Golemanov et al. 2006). Then, successful formulations that did not show any phase separation were subjected to the heating–cooling test, which contains six cycles between refrigerator temperatures at 4 °C for 48 h and 40 °C for 48 h. Then, formulations that did not show any phase separation were taken for the freeze–thaw stress test. For this purpose, nanoemulsions were kept alternatively at two temperatures (−21 and 21 °C) for each temperature test of at least 24 h. Finally, the stable formulation nanoemulsions were stored for about 8 weeks at room temperature in closed tubes for additional observation like phase separation or creaming. The pH values of the stable nanoemulsions were measured at 25 ± 0.1 °C using an Adwa (AD 8000) pH meter. The dynamic (absolute) viscosity (μ) of the nanoemulsions was measured by a Rotary Myr VR 3000 digital viscometer with L4 spindle at 200 rpm at 25 °C without further dilution. Each reading was taken after the equilibrium of the sample for 2 min. All experiments are carried out in triplicates.

Droplet size and polydispersity index

The particle size distribution, mean particle diameter (Z-averages), and polydispersity index (PDI) of the samples were determined using dynamic light scattering (DLS) at 25 °C on a Zetasizer Nano-ZS90 (Malvern Instruments, Malvern, UK). The samples were diluted before measurement to 10% with deionized water to avoid multiple scattering effects. From the DLS data, the average particle size (z-diameter), which was determined by cumulate analysis of the intensity–intensity autocorrelation function (Stepanek 1993), and the PDI, which gives an indication of the width of the droplet size distribution, were determined. The analysis was performed at a scattering angle of 90°, and each recorded measurement was an average of three replicates.

Scanning electron microscopy (SEM)

Morphology of the oil nanoemulsions was investigated by SEM (JEOL, Model JFC-1600, Tokyo, Japan). A drop of each oil nanoemulsion (25 μL) was diluted with deionized water, transferred into a carbon-coated copper grid, then stained by phosphotungstic acid solution

(2%, pH = 6.7) for 1 min. The replica was lifted to drying at room temperature (28 °C), and then, the image was visualized with SEM at 80-kV accelerating voltages.

Gas chromatography–mass spectrometry

Gas chromatography–mass spectrometry (GC–MS) was carried out using an Agilent 6890 GC equipped with a 5973 N mass selective detector and an HP-5 (5% phenyl methylpolysiloxane) capillary column. The temperature of the oven was programmed from 50 to 280 °C at a rate of 4 °C/min and held at this temperature for 5 min. The temperatures of the inlet and interface were 250 and 280 °C, respectively. Helium, at a flow rate of 1.0 ml/min (constant flow), was used as the carrier gas. The sample (0.2 ml) was injected using a split of 20:1. Electron impact mass spectrometry was performed at 70 eV. The temperatures for the ion source and quadrupole were maintained at 230 and 150 °C, respectively. Compounds were identified by comparing their retention indices and mass spectra with those found in the NIST 98.1 and Mass Finder 3.1 commercial libraries. The integration area of the chromatographer was used to calculate the concentration of each component of the analyzed oils.

Contact acaricidal activity

An impregnated fabric disc bioassay was used for measuring the acaricidal activity of test plant oils and against *T. putrescentiae* (Kim et al. 2004). Test solutions of the plant oils were prepared by dissolving 3.2, 1.6, 0.8, 0.4, 0.2 and 0.1 ml of each material in 5 ml acetone. Each test concentration was applied uniformly to a black cotton fabric piece filter paper disc (6 cm diameter pieces, 28.3 cm^2) using a Hamilton micro-applicator (Bonaduz, Switzerland) to obtain gradient concentrations of 113.2, 56.6, 28.3, 14.2, 7.1 and 3.5 µl/cm^2. Control fabric discs received 5 ml acetone. After drying in a fume hood for 1 min, each disc was placed in the bottom of a Petri dish (6 cm diameter). Groups of 25 unsexed adults (7–10 days old) were placed separately in each Petri dish and covered with a lid. Treated and control mites were kept in the dark at 25 ± 2 °C and 70 ± 5% r.h. All treatments were set up in four replicates along with control. Benzyle benzoate, the conventional acaricide was used for comparison. Mortality was determined 48 h post treatment under a binocular microscope (20×), thereafter the end-point mortality was reached. The contact toxicity was expressed as µl/cm^2. Adults were considered dead if their appendages did not move when they were prodded with a pin.

Fumigant acaricidal activity

The susceptibility of *T. putrescentiae* adults to the fumigant action of the plant oils and nanoemulsions was investigated according to Kwon and Ahn (2002). A fabric cotton piece (6.0 cm diameter) was impregnated with 25 µl of six graded concentrations of each oil or nanoemulsion to obtain equivalent fumigant concentrations of 45.85, 22.9, 11.5 5.7, 2.9 and 1.45 µl/l air or acetone only (control). After evaporating the solvent in a fume hood, the cotton piece was attached to the undersurface of the screw cap of a glass vial (54 ml volume), thus preventing direct contact of test adults with the test oils. Mites were transferred to the vials in groups of 25 unsexed (7–10 days old) adults. The vials were covered with fine steel gauze secured with adhesive tape. Treated and control mites were kept in

the dark at 25 ± 2 °C and 70 ± 5% r.h. All treatments were set up in four replicates along with control. Mortality was determined 48 h post treatment under a binocular microscope (20×), thereafter the end-point mortality was reached. The fumigant toxicity was expressed as µl/l. Adults were considered dead if their appendages did not move when they were prodded with a pin.

Persistence activity assessment

This experiment was done in order to determine how long acaricidal activity of the plant oils was retained over the time. Each oil was diluted in acetone and admixed with sterilized crushed wheat grains in 1 l glass jars, at a concentration equal to the LC_{95} fraction of each oil in the contact toxicity bioassay. Jars containing the treated media were hand-shacked to ensure complete mixing. After evaporation of the solvent, the treated media were packed in tightly closed jute sacks (30 × 30 cm) and stored at darkness under the same laboratory conditions described before. Samples, each of 20 g of the stored food were withdrawn at various intervals (10, 20, 30, 40, 50, and 60 days) and offered 25 adults of *T. putrescentiae* (7–10 days old) in a Petri-dish (9 cm diameter). Control sets were made, where the same number of mites were offered food treated with acetone. Each experiment was replicated four times and mortality was recorded and corrected for mortality in control using Abbott's formula (Abbott 1925).

Data analysis

Mortality data were recorded and corrected for that in the control using Abbott's formula (Abbott's 1925). The dose-mortality response was analyzed by probit analysis (SAS 1990) then the LC_{50} and LC_{95} values and their fiducial limits were estimated. Lethal concentrations at the 50% and slope levels were considered significantly different if their associated confidence intervals did not overlap. Significance of mean differences between treatments and control were compared using ANOVA ($\alpha = 0.05$) followed by individual pairwise comparisons with Tukey's honestly significant differences (HSD) test using SPSS v.15.0 software. LT_{50}'s and their fiducial limits were estimated.

Results

Chemical composition of the plant oils

Data in Table 1 show the amount yielded and the chemical composition of each oil. A total of 30 compounds constituting 98.7% were identified in *O. basilicum* oil. The major components were methyl eugenol (71.3%), α-cubebene (6.4%), and linalool (4.1%). It is indicated that *cis*-thujone (24.9%), 3,3,6-trimethyl-1,5-heptadien-4-one (Artemisia ketone) (19.8%), 2,5-dimethyl-3-vinyl-4-hexen-2-ol (Santolina alcohol) (14.3%), and *trans*-thujone (13.5%) were the major constituents of *A. fragrantissima* oil. For *A. santolina*, fragranyl acetate (26.1%), 1,6-dimethyl-1,5 cyclooctadiene (12.6%), 1,8 cineole (11.8%), and *cis*-thujone (9.4%) were the major components.

Table 1 Chemical composition of essential oils of *Ocimum basilicum*, *Achillea fragrantissima* and *A. santolina*

Compound[a,b]	$R_I(K_i)^c$	R_t^*	Concentration (%)		
			O. basilicum	*A. fragrantissima*	*A. santolina*
2-Hexanal	856	8.2	–	0.1	–
Isopentyl acetate	878	8.7	–	–	0.2
Santolina triene	910	9.3	–	1.6	0.3
α-Pinene	937	10.2	–	0.4	0.5
Camphene	954	10.7	–	–	0.9
Sabinene	976	11.5		–	0.4
β-Pinene	980	11.7	0.1	–	0.2
Myrcene	984	12.4	0.1	–	–
Yomogi alcohol	998	12.6	–	3.2	0.4
α-Terpinene	1017	13.2	–	–	1.1
P-Cymene	1026	13.4	–	–	2.7
1,8 Cineole	1034	14.4	2.9	3.7	11.8
2,5-Dimethyl-3-vinyl-4-hexen-2-ol (Santolina alcohol)	1038	14.7	–	14.3	–
3,3,6-Trimethyl-1,5-heptadien-4-one (Artemisia ketone)	1046	15.1	–	19.8	–
γ-Terpinene	1049	15.4	0.1	–	–
cis-Sabinene hydrate	1055	15.7	–	–	0.8
trans-Linalool oxide	1073	15.9	0.2	–	–
Linalool	1086	16.4	4.1	–	0.3
Fenchone	1088	16.8	0.4	–	–
n-Octanol	1090	17.4	0.4	–	0.3
cis-Thujone	1092	17.9	–	24.9	9.4
trans-Thujone	1098	18.4	–	13.5	–
cis-p-Ment-2-en-1-ol	1122	19.6	–	–	0.5
Octan-3-yl acetate	1128	19.8	0.7	–	–
trans-p-Ment-2-en-1-ol	1141	20.1	–	–	0.2
Camphor	1145	20.7	0.3	–	5.7
Pinocarvone	1162	21.4	0.1	4.7	–
Borneol	1164	21.1	–	–	1.7
Lavandulol	1166	21.5		2.4	
Terpinen-4-ol	1178	21.8	0.1	1.1	1.1
α-Terpineol	1190	22.3	0.1	–	2.3
3,7-Dimethyl-2,6-octadien-1ol	1192	22.1	0.3	–	–
Myrtenol	1194	22.6	–	–	–
Fragranol	1196	23.5	–	–	3.2
trans-Carveol	1223	24.1	–	–	2.1
Nerol	1230	24.4	0.8	–	
Linalyl acetate	1249	25.2	0.2	–	–
Piperitone	1258	26.1	–	–	0.4
Bornyl acetate	1285	26.9	–	0.7	0.6
Thymol	1288	27.1	–	–	0.6
trans-Sabinyl acetate	1290	27.4	0.2	1.2	5.2

Table 1 (continued)

Compound[a,b]	$R_I(K_i)^c$	R_t^*	Concentration (%)		
			O. basilicum	*A. fragrantissima*	*A. santolina*
Carvacrol	1296	27.8	0.1	–	–
Fragranyl acetate	1334	29.3	–	–	26.1
α-Cubebene	1351	30.5	6.4	–	–
Geranyl acetate	1366	30.6	–	–	–
Methyl eugenol	1402	32.1	71.3	–	–
β-Caryophyllene	1415	32.8	0.2	1.3	1.1
α-Humulene	1452	33.2	0.3	0.2	–
Germacrene D	1480	33.5	0.8	2.1	0.5
Bicyclogermacrene	1492	33.8	–	1.1	0.1
1,6-Dimethyl-1,5-cyclooctadiene	1502	34.5	–	–	12.6
Lavandylyl-2-methylbutyrate	1510	36.1	–	–	1.2
β-Sesquiphellandrene	1512	36.3	–	1.4	–
γ-Cadinene	1516	36.4	1.1	–	–
δ-Cadinene	1524	36.8	0.6	–	–
cis-Nerolidol	1538	38.3	0.2	–	–
Dendrolasin	1556	39.2	–	–	0.2
Spathulenol	1574	40.1	–	0.3	0.4
Caryophyllene oxide	1582	40.6	2.4	0.2	0.3
10-epi-γ-Eudesmol	1620	42.8	1.2	–	1.2
α-Muurolol	1632	43.2	2.6	–	0.3
β-Eudesmol	1648	44.7	0.1	0.2	0.2
α-Bisabolol	1684	46.5	–	–	0.3
% Peaks identified			98.7	98.4	96.8
Total yield % (ml/100 g)			0.67	0.83	0.59

[a]Compounds are listed in the order of their elution

[b]Identification based on R_f values and mass spectra relative to standards, data found in the NIST 98.1 and Mass Finder 3.1 commercial libraries and literature

[c]Kovats index based on *n*-alkane series (Adams 2007)

*Retention time

Formation and physicochemical characteristics of the oil nanoemulsions

The experimental design and the optimum conditions for the preparation of nanoemulsions containing EOs from *O. basillicum, A. fragrantissima* and *A. santolina* are shown in Table 2. Oil in water nanoemulsions were prepared in the proportions as follows: the active ingredient (10%), water phase (70%), surfactant (20%, lipophilic emulsifier), sonication time (15 min), sonication cycle (five cycles per second), and sonication power (10 kHz). The results of nanoemulsions preparation also include the viscosity (mPa.s) and the pH values. Nanoemulsions were exposed to extreme storage conditions to evaluate their stability during storage. The results of the thermodynamic characterization studies (centrifugation at 5000 rpm, temperature stability at 25 °C, heating–cooling cycle at 4–40 °C, and freezing cycle at −4 °C of nanoemulsions are also presented in Table 2. All of the prepared oil nanoemulsions exhibited thermodynamic stability during the extreme storage conditions.

Table 2 Mean (±SE; n=3) size and polydispersity index (PDI) of nanoemulsions prepared from essential oils of *Ocimum basilicum*, *Achillea fragrantissima* and *A. santolina*

Nanoemulsion	Viscosity (mPa.s)	pH	PDI	Size (nm)
O. basilicum	4.1	6.1	0.18±0.04	78.5±11.2
A. fragrantissima	4.5	6.4	0.20±0.02	91.3±9.6
A. santolina	5.1	6.3	0.26±0.01	104.6±14.1

Visual appearance and particle size

The appearance of nanoemulsions was visualized before and after the sonication process by the naked eye. The nanoemulsion formulations were transparent after sonication. The particle size and PDI of the prepared nanoemulsions are presented in Table 2 and illustrated in Fig. 1. The data indicated that the droplet size varied from 78.5 to 104.6 nm with PDI values ranged between 0.18 and 0.26. Measurements showed that the polydispersity indices obtained from *O. basilicum* and *A. fragrantissima* nanoemulsions were lower than that obtained from *A. santolina*. This result proved that all these liquid formulations were successful in their preparation in the nanometric size range. SEM study showed that the obtained nanoemulsions consisted of dispersed spherical shape nanoparticles with different particle sizes (Fig. 2). On average, the transparent nanoemulsions obtained from *O. basilicum* and *A. fragrantissima* samples had smaller particles (78.5 and 91.3 nm, respectively) than the translucent nanoemulsion obtained from *A. santolina* (104.6 nm).

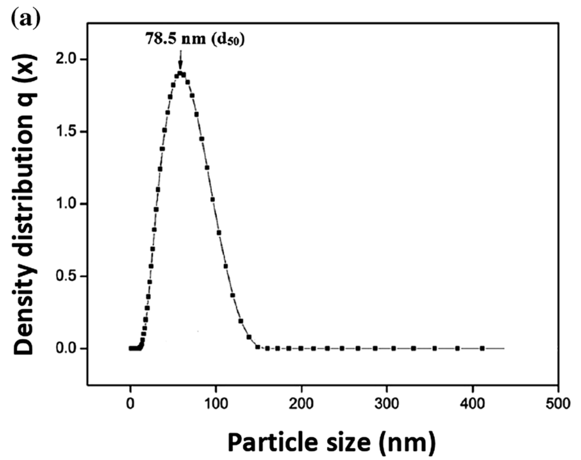
Acaricidal activity

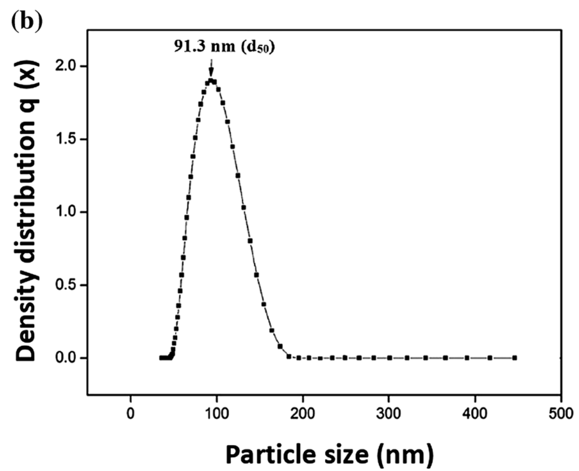
The contact toxicity bioassay of the test oils against adults of *T. putrescentiae* is recorded in Table 3. The widely used benzyl benzoate served as a standard control for comparison. Based on 48 h LC_{50} values, *O. basilicum* was the most toxic, followed by *A. fragrantissima* and *A. santolina*, where $LC_{50's}$ were 8.4, 14.1, and 21.8 µl/cm^2, respectively. LC_{50} for benzyl benzoate was 9.8 µl/cm^2. In this case, the oil of *O. basilicum* was more toxic than the standard acaricide, benzyl benzoate. Upon fumigation, response of *T. putrescentiae* adults to the tested oils also varied according to the test oil and the formulation used (Table 4). Based on the 48-h LC_{50} values, the same manner of activity was also observed, where *O. basilicum* was the most toxic with $LC_{50}=6.7$ µl/l air, followed by *A. fragrantissima* and *A. santolina*. When prepared as nanoemulsions and tested as fumigants, toxicity of the plant oils was increased drastically with LC_{50} values of 2.2, 4.7, and 9.6 µl/l air for *O. basilicum*, *A. fragrantissima* and *A. santolina*, respectively.

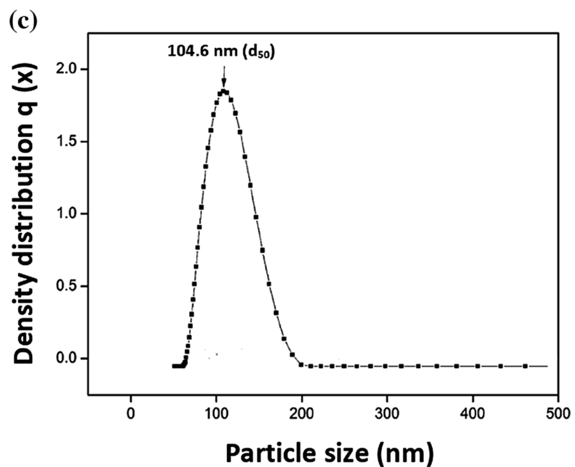
Persistence activity of essential oils

Results in Tables 5 and 6 show the residual acaricidal effect of the test oils against adults of *T. putrescentiae*, where the oil of *O. basilicum* showed the potent residual protecting activity with LT_{50} value of 63.2 days (adult mortality reached 54.6 after 60 days of storage). The essential oil of *A. fragrantissima* showed a moderate residual activity with LT_{50} values of 46.6 days, whereas *A. santolina* oil showed a weak activity. As shown in Fig. 3, all of the

Fig. 1 Particle size distribution of the oil nanoemulsions of **a** *Ocimum basilicum*, **b** *Achillea fragrantissima*, and **c** *A. santolina*







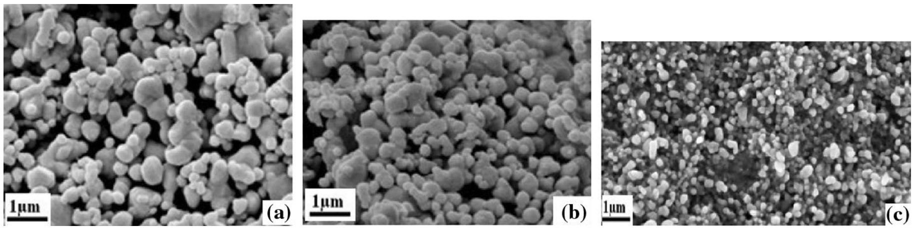


Fig. 2 SEM images of oil nanoemulsions derived from **a** *Ocimum basilicum*, **b** *Achillea fragrantissima*, and **c** *A. santolina*

Table 3 Contact toxicity of the essential oils of *Ocimum basilicum*, *Achillea fragrantissima* and *A. santolina*, and a standard acaricide (benzyl benzoate) as a positive control, against *Tyrophagus putrescentiae* 48 h post treatment, using the impregnated fabric disc bioassay

Plant oil	LC_{50} (95% fl)	LC_{95} (95% fl)	Slope (± SE)	χ^2 (df = 4)
O. basilicum	8.4 (6.6–10.3)	15.1 (13.4–19.5)	2.84 ± 0.24	2.55
A. fragrantissima	14.1 (12.5–17.6)	25.3 (22.6–33.1)	2.13 ± 0.35	2.87
A. santolina	21.8 (18.6–27.3)	47.0 (42.5–59.3)	3.08 ± 0.27	3.66
Benzyl benzoate	9.8 (7.55–11.22)	26.3 (23.4–29.1)	1.44 ± 0.23	2.05

Concentrations (mean of 4 replicates, each based on 25 mites) are expressed as LC_{50} and LC_{95} (μl/cm^2) with 95% fiducial limits in parentheses

Table 4 Fumigant toxicity of the plant oils and nanoemulsions of *Ocimum basilicum*, *Achillea fragrantissima* and *A. santolina* against *Tyrophagus putrescentiae* 48 h post treatment, using the fumigation bioassay

Plant species	Formulation	LC_{50} (95% fl)	LC_{95} (95% fl)	Slope (± SE)	χ^2 (df = 4)
O. basilicum	Oil	6.7 (5.2–8.9)	13.8 (11.5–17.8)	2.14 ± 0.35	2.85
	Nanoemulsion	2.2 (1.2–3.4)	5.3 (3.8–8.4)	2.06 ± 0.28	1.32
A. fragrantissima	Oil	11.5 (9.1–14.8)	22.7 (17.0–26.6)	2.44 ± 0.22	3.43
	Nanoemulsion	4.7 (3.2–6.8)	10.6 (7.0–12.3)	2.08 ± 0.40	1.65
A. santolina	Oil	16.8 (14.7–22.1)	43.1 (35.7–49.5)	3.04 ± 0.30	2.32
	Nanoemulsion	9.6 (7.1–13.3)	24.4 (19.7–33.1)	2.70 ± 0.34	2.25

Concentrations (means) are expressed as LC_{50} and LC_{95} (μl/l air) with 95% fiducial limits in parentheses

tested oils undergo degradation over time, whereas those of *O. basilicum* and *A. fragrantissima* showed moderate stability, with a promising residual activity against *T. putrescentiae* during storage.

Table 5 Residual toxicity of *Tyrophagus putrescentiae* fed food treated with plant oils of *Ocimum basilicum*, *Achillea fragrantissima* and *A. santolina* at their LC_{95} values (mean [±SE] % mortality, of four replicates, each based on 25 mites), after storage for 10–60 days

Plant oil	% Mortality					
	10	20	30	40	50	60
O. basilicum	96.2±2.0a	92.1±3.2a	84.0±2.4a	74.8±2.5a	66.6±2.7a	54.6±2.1a
A. fragrantissima	95.3±1.6a	89.7±2.6a	75.6±3.2b	60.3±3.1b	48.2±2.8b	34.3±1.6b
A. santolina	90.4±2.1b	74.8±3.5b	46.0±2.2c	32.4±1.6c	24.4±1.9c	15.8±1.3c
F	210.2	967.9	1154.1	2016.8	2065.9	1608.5
P	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001

Means within a column followed by the same letter are not significantly different (ANOVA followed by Tukey's HSD test: $p<0.05$)

Table 6 Mean (±SE) LT_{50} values (i.e., the time (days) elapsed after storage, required to achieve 50% mortality) against adults of *Tyrophagus putrescentiae* fed grains treated with plant oils of *Ocimum basilicum*, *Achillea fragrantissima* and *A. santolina* at their LC_{95} values

Plant species	LT_{50}	95% fiducial limits	χ^2 (df=3)	Slope
O. basilicum	63.2±4.1	57.6–69.1	2.04	2.08±0.34
A. fragrantissima	46.6±2.8	41.0–55.4	2.44	2.33±0.28
A. santolina	27.4±2.5	23.3–34.2	3.18	2.86±0.42

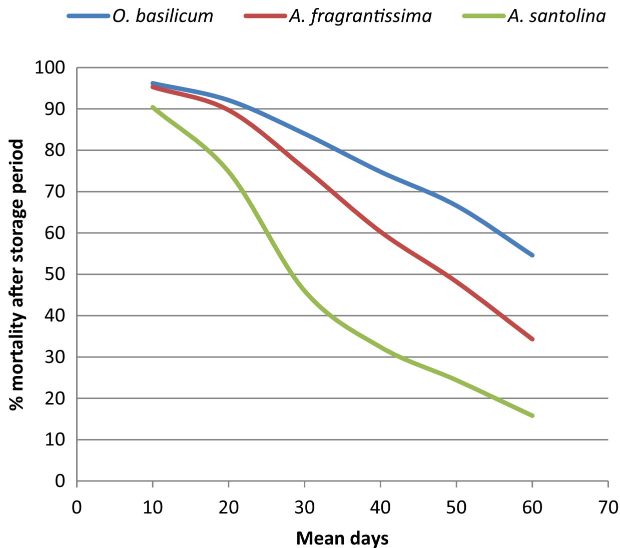


Fig. 3 Residual toxicity of the plant oils against *Tyrophagus putrescentiae* showing stability over time

Discussion

Composition of the plant oils was similar to previous reports concerning the same plant species (Özcan and Chalchat 2002; Bader et al. 2003; Nenaah 2014a, b, c; Almadiy et al. 2016). Still, differences were observed both in the composition and abundance of the major components of *A. santolina* (Mohamed and Abdelgaleil 2008) and *O. basilicum* (Sartoratto et al. 2004). Sources of compositional variability include the plant part extracted, phenological state of the plant, environmental conditions (climatic, seasonal and geographical), genetic and chemotype differences, soil variations and nutritional status of plants (Nenaah 2014b, c; Nenaah et al. 2015; Isman 2016).

The plant oils showed a marked acaricidal activity against the mold mite, *T. putrescentiae*. Superior activity was achieved by the essential oils of *O. basilicum* and *A. fragrantissima*. In the literature, these plants showed insecticidal and acaricidal activity against insects and mites of stored food products. In a related study, *A. fragrantissima* growing in Saudi Arabia exhibited acaricidal activity against *Hyalomma dromederi*, a prevalent tick species of camels (Al-Harbi et al. 2015). Essential oil of *Achillea millefolium* was 2.62 times more potent than that of benzyl benzoate as a contact acaricide against *T. putrescentiae* (Song et al. 2016). Ebadollahi (2017) obtained similar results with *Achillea filipendulina* oil against the spider mite *Tetranychus urticae*. In the current study, *O. basilicum* (contatining a high percentage of eugenol) showed the strongest acaricidal activity against *T. putrescentiae*. These findings are in accordance with those of Hüe et al. (2015) who studied the acaricidal activity of essential oils from three *Ocimum* species on 14- to 21-day-old larvae of the cattle tick *Rhipicephalus microplus*. The essential oils of *O. urticaefolium* and *O. gratissimum* were the most effective with matching LC_{50} values. Both oils contain high amounts of eugenol (33.0 and 22.3%, respectively). Assis et al. (2011) reported a direct relationship between high concentration of eugenol and high mortality values when testing the acaricidal activity of essential oils of *Cinnamomum zeylanicum* and *Suidasia pontifica* against *T. putrescentiae.* According to many studies, *O. basilicum* showed a marked acaricidal activity against a wide range of species such as *Rhipicephalus sanguineus* (Manzoor et al. 2013) and *R.* (*Boophilus*) *microplus* (Veeramani et al. 2014). Many authors such as Macchioni et al. (2002) and Kim et al. (2003b) studied the control of *T. putrescentiae* using plant oils. Chemical profile of the plant oils tested herein revealed their high content of monoterpenoids. It is well known that the acaricidal activity of many plant oils are mainly due to their monoterpenoid constituents (Sànchez-Ramos and Castanera 2001; Kim et al. 2003a, b, 2004; Lee et al. 2006; Jeong et al. 2008; Jeon et al. 2009). Kim et al. (2004) suggest that hydrophobicity appears to play a crucial role in *T. putrescentiae* toxicity and that the presence of a hydroxyl moiety in the ortho position may have a considerable influence on toxicity.

Nanoemulsions (particle size 78.5 to 104.6 nm) were prepared from the plant oils through a green procedure without using toxic chemical solvents. Optimum conditions for the preparation of nanoemulsions depend on the active ingredient (10%), water phase (70%) and the lipophilic emulsifier (surfactant) (20%). Nanoemulsions were exposed to extreme storage conditions to judge their stability during storage. Stabilization of nanodroplets in the emulsion with a 1:3 ratio of oil and surfactant would be due to the surfactant, which reduces interfacial free energy and provides a mechanical barrier to coalescence (Reiss 1975). The sonication time (15 min), sonication cycle (five cycles per second), and sonication power (10 kHz) are also determinant factors for nanoemulsion stability (Anjali et al. 2012; Badawy et al. 2018). Centrifugation can accelerate the rate of

cremation or sedimentation, demonstrating that the rupture of an emulsion may be related to the action of the gravity force. The results showed that all nanoemulsions passed from the centrifugation test at 5000 rpm. Stability at 25 to 4 °C indicated that all samples were stable without phase separation up to 8 weeks. These findings are consistent with other studies that reported an increase in surfactant concentration and the emulsification time had a direct relationship to the stability of the emulsion (Ghosh et al. 2013; Badawy et al. 2017). In non-equilibrium systems, emulsions tend to reduce their interfacial areas and free energy through several breakdown processes, such as creaming, sedimentation, flocculation, Ostwald ripening, and coalescence (Taylor 2003; Tadros et al. 2004). Compared to conventional emulsions, nanoemulsions have a good stability against creaming, sedimentation, flocculation, and coalescence due to the small size of the droplets (Reddy and Fogler 1981). The stability of emulsions can also improve considerably with increasing the surface charge due to the repulsive forces produced between droplets against flocculation and coalescence (Stachurski and Michalek 1996). In our study, the overall pH value of the various formulations is around 6.0. The pH can exert a vital effect on the stability of nanoemulsions. Variations at different levels of pH cause a change in the surface charge of the globules and thus their stability during storage. According to Badawy et al. (2017), an increase in the surface charge of the globules promotes electrostatic repulsion and reduces the flocculation and dissolution of the nanoemulsions. The polydispersity index (PDI) is a measure of the uniformity and stability of the droplet size in the formulation. Low polydispersity results in high uniformity of droplet size. According to the current study, PDI of the oil nanoemulsions ranged between 0.18 and 0.26. It has been reported that PDI values lower than 0.25 indicate a narrow particle size distribution, proving good physical stability of the nanoemulsion, due to the reduced Ostwald ripening (Hoeller et al. 2009). Shakeel et al. (2007) stated that PDI value remaining below 0.2 reflects the relative homogeneity of the nanoemulsion, and PDI $>$ 0.3 indicates system heterogeneity. On the other hand, an increase in viscosity of the continuous phase reduces oil droplet mobility, which delays instability phenomena, resulting in oil droplets with a more homogeneous particle size (Arancibia et al. 2016). Similar results have been reported by other studies on nanoemulsions of plant oils such as cinnamon oil (Ghosh et al. 2013), neem oil (Anjali et al. 2012), and basil (Ghosh et al. 2014). The spherical form of the nanoemulsions reported herein and their size range (78.5–104.6 nm) are in agreement with the findings of many authors. Li and Chiang (2012) stated that nanoemulsions containing D-limonene by ultrasonic emulsification had a droplet size of less than 100 nm. It is reported that small nanoemulsion droplets can be found when the hydrophilic–lipophilic balance (HLB) value of the surfactant pair coincides with the required HLB value of the oil (Fernandes et al. 2014).

Results of the current study revealed that the oil nanoemulsions showed increased fumigant acaricidal activity against *T. putrescentiae*. When transformed into nanoparticles, materials could acquire novel biological properties, as they offer large specific surface area and hence increased affinity to the target, penetrate rapidly and are selectively accumulated in biosystems enhancing various activities within living cells (Weiss et al. 2006; Nenaah 2014c; Nenaah et al. 2015; Almadiy et al. 2016, 2018). It is possible that a reduction in droplet size, and hence an increase in surface area of the droplets, increases the rate of accumulation by the mites of the acaricidal component of the oil nanoemulsions (Margulis-Goshen and Magdassi 2013). The stability of the prepared nanoemulsions, the high acaricidal activity, and the absence of organic toxic solvents make these formulations suitable as biorational acaricidal products. In a related study, Sarapothong et al. (2017) studied the contact acaricidal activity of nano essential oil of black pepper, *Piper nigrum*, against the African red mite, *Eutetranychus africanus*. After 24 h, and a test concentration of 1%,

96% mortality was recorded (LC_{50} was 0.34%). Nanoemulsions from *Callistemon viminalis* and *Origanum vulgare* and two monoterpenes (R-limonene and pulegone) exhibited high acaricidal activity against *T. urticae* with 100% reduction at 5 mg/l after 2–3 days of application (Badawy et al. 2018). Garlic oil nanoemulsion with droplet size 93.4 nm showed a high acaricidal activity against injurious eriophyid mites *Aceria oleae* and *Tegolophus hassani* with LC_{50} 298.3 and 309.63 µg/ml, respectively (Mossa et al. 2018). As many of the pesticides known today are organic compounds with poor water solubility, development of nanomaterials, including nanoemulsions appear to solve this problem by enhancing water solubility of poorly water-soluble substances. Their bioavailability results in stable formulations without utilization of organic toxic solvents (Nenaah et al. 2015). These formulations should also degrade rapidly with residue levels below the regulatory criteria in foodstuffs and environment (Khot et al. 2012).

The molecular targets associated with the mechanism of action of essential oils include inhibition of acetylcholinasterase (AChE), antagonism with the receptors of tyramine and/or octopamine neurotransmitter that modulate vital functions ranging from metabolism to behavior. Closure of the chloride channels by GABA is also confirmed (Blenau et al. 2012; Isman 2016). Based on our results, the test plant oils and their nanoemulsions could be a very promising control strategy to protect stored food from mite infestation. However, challenges still have to be overcome for the effective use of plant oils as natural acaricides. These include scarcity of the natural resource, the need for chemical standardization and quality control, in addition to some challenges associated with formulation, application, stability, and storage of these compounds. It is also necessary to extend the residual life of oil-based pesticides in open areas. Innovative technologies in the formulation of acaricides, such as microencapsulation and nanoformulation, provide clues to solving problems with essential oil-based products. However, many of the plant essential oils studied herein are used as pharmaceuticals, therefore considered less harmful to humans than most conventional insecticides and they can be used as relatively safe fumigants and/or contact acaricides after experiments required to validate various limitations about their mammalian and environmental safety.

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Author contributions GN and BA conceived and designed the experiments. All authors conducted bioassays. GN achieved GC–Ms. All authors collected and analyzed data. GN and BA wrote the manuscript. All read and approved manuscript.

Compliance with ethical standards

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

Ethical approval All applicable international, national, and/or institutional guidelines for the care and use of animals were followed. This article does not contain any studies with human participants performed by any of the authors.

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