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Efficacy of different formulations of essential oils against the cotton mealybug, *Phenacoccus solenopsis* (Hemiptera: Pseudococcidae) under laboratory conditions

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Abstract

During recent decades, one of the most important methods of replacing synthetic pesticides is using of new formulations based on plant essential oils (EOs) that can improve their quality and effectiveness. Due to restrictions in application of EOs in pure form, preparation of their commercial formulations is essential. In this research, the contact toxicity of fifteen different plant EOs on 1st instar nymphs of *Phenacoccus solenopsis* was examined at 25±1°C, 65±5% RH, and a photoperiod of 16:8 h = L:D. Three EOs including; *Mentha longifolia* (L.), *Mentha piperita* (L.), and *Oliveria decumbens* (Vent.) had the highest contact toxicity and were considered for the next experiments. According to GC and GC/MS analysis, pulegone (51.49%), menthone (22.75%), and 1,8-cineole (11.69%) were the principal components of *M. longifolia*; menthone (36.51%), menthene (28.51%), menthol (8.12%), and 1, 8-cineole (7.66%) were the principal components of *M. piperita* and the main components of *O. decumbens* EO were thymol (43.99%), γ-terpinene (13.96%), and *p*-cymene (12.62%). Moreover, contact toxicity of the EOs were evaluated on 1st instar nymphs of *P. solenopsis* under laboratory conditions, before and after formulation. Based on lethal concentration trials, LC_{50} values of pure and formulated EOs of *M. longifolia*, *M. piperita*, and *O. decumbens* on 1st instar nymphs were 113.49, 129.74, 149.93, and 48.22, 55.55, 61.68 ppm, respectively; after 48 hours. Therefore, the contact toxicity of formulated EOs of *M. longifolia*, *M. piperita*, and *O. decumbens* were 2.34, 2.36, and 2.43-fold higher than the pure EOs. Based on physicochemical trials, the prepared formulations were stable under the experimental conditions. Therefore, formulation of EOs of examined plants can be considered as new environmentally friend pesticides for controlling of the pests.

Key words: *Phenacoccus solenopsis*; Essential oils; Botanical insecticides; Contact toxicity; GC-MS analysis.

کارایی فرمولاسیون‌های مختلف اسانس‌ها روی شپشک آردآلود پنبه *Phenacoccus solenopsis* (Hemiptera: Pseudococcidae) در شرایط آزمایشگاهی

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چکیده

در دهه‌های اخیر، یکی از مهمترین روش‌های جایگزینی آفت‌کش‌های مصنوعی، استفاده از فرمولاسیون‌های جدید بر پایه اسانس‌های گیاهی است، که می‌تواند کیفیت و میزان تاثیر آن‌ها را افزایش دهد. به دلیل محدودیت‌های کاربرد اسانس‌ها به شکل خالص، تهیه فرمولاسیون تجاری آن‌ها ضروری است. در این تحقیق، ابتدا سمیت تماسی ۱۵ اسانس مختلف گیاهی روی پوره‌های سن اول شپشک آردآلود پنبه *Phenacoccus solenopsis* (Tinsley) (Hemiptera: Pseudococcidae) در دمای ۱±۲۵ درجه سلسیوس، رطوبت نسبی ۵±۶۵ درصد و دوره نوری ۱۶ ساعت روشنایی و ۸ ساعت تاریکی بررسی شد. اسانس‌های نعناع فلفلی *Mentha piperita* (L.) (Lamiaceae)، پونه *Mentha longifolia* (L.) (Lamiaceae) و لعل

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کوهستان (Umbelliferae) (Vent.) *Oliveria decumbens* ، دارای بیشترین سمیت تماسی بودند و برای آزمایش‌های بعدی در نظر گرفته شدند. بر اساس آنالیز GC و GC/MS، ترکیبات مهم اسانس پونه شامل پولگون ۵۱/۴۹، منتون ۲۲/۷۵ و ۱و۸ سینئول ۱۱/۶۹ درصد؛ اسانس نعناع فلفلی شامل منتون ۳۶/۵۱ ، منتن ۲۸/۵۱ ، منتول ۸/۱۲ و ۱و۸ سینئول ۷/۶۶ درصد و لعل کوهستان شامل تیمول ۴۳/۹۹، گاما ترپینن ۱۳/۹۶ و پی_سیمن ۱۲/۶۲ درصد بودند. پس از تهیهٔ فرمولاسیون، سمیت تماسی اسانس‌ها قبل و بعد از فرموله شدن، روی پوره‌های سن اول شپشک آردآلود پنبه در شرایط آزمایشگاهی مورد بررسی قرار گرفت. بر اساس نتایج آزمایش‌های تعیین غلظت کشنده، مقادیر LC_{50} سمیت تماسی برای اسانس پونه، نعناع فلفلی و لعل کوهستان فرموله نشده و فرمولاسیون اسانس پونه، نعناع فلفلی و لعل کوهستان روی پوره‌های سن اول شپشک آردآلود پنبه *P. solenopsis* پس از ۴۸ ساعت، به ترتیب ۱۱۳/۴۹، ۱۲۹/۷۴، ۱۴۹/۹۳ و ۴۸/۲۲، ۵۵/۵۵ و ۶۱/۶۸ پی‌پی‌ام محاسبه شد. بنابراین، سمیت تماسی فرمولاسیون اسانس پونه، نعناع فلفلی و لعل کوهستان نسبت به اسانس خالص پونه، نعناع فلفلی و لعل کوهستان به ترتیب ۲/۳۴، ۲/۳۶ و ۲/۴۳ برابر بیشتر بود. بر اساس آزمایش‌های فیزیکوشیمیایی، فرمولاسیون‌های تهیه شده پایداری خوبی از خود در شرایط آزمایشگاهی نشان دادند. بنابراین اسانس‌های فرموله شدهٔ این سه گیاه می‌تواند به عنوان حشره‌کش‌های جدید دوست‌دار محیط زیست برای کنترل آفات مورد توجه قرار گیرند.

واژه‌های کلیدی: شپشک آردآلود پنبه، اسانس‌های گیاهی، حشره‌کش‌های گیاهی، سمیت تماسی، آنالیز جی‌سی- مس.

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Introduction

The cotton mealybug, *Phenacoccus solenopsis* (Tinsley) (Hemiptera: Pseudococcidae) is an invasive, polyphagous pest of global distribution (Fand *et al*., 2014). During 2005–2009 the cotton mealybug attacked cotton, *Gossypium hirsutum* (L.) in Pakistan and India, and made severe economic losses (Wang *et al*., 2010). Seasonal and annual population growth data of *P. solenopsis* from nine locations in its native range in the United States, and distribution of this mealybug worldwide, were analyzed using the CLIMEX model. Findings indicated that tropical regions of worldwide were highly suitable for *P. solenopsis* (Wang *et al*., 2010). Damage of the cotton mealybug is reported on more than 200 plant species from about 24 countries of tropical and subtropical regions of the world (Fand & Suroshe, 2015). Adults and nymphs weaken the plants by sucking sap from leaves, twigs, stems, and fruiting bodies. Honeydew secreted by the pest encourages the development of black sooty mold, adversely affecting the photosynthetic activity (Joshi *et al*., 2010). Plants infested by mealybugs during their vegetative phase exhibit symptoms of distorted, bushy shoots, crinkled and twisted bunchy leaves, and stunted plants that desiccate completely in severe cases. Late season infestations during the reproductive crop stage result in reduced plant vigor and early crop senescence (Nagrare *et al.*, 2011). For the first time, *Phenacoccus solenopsis* was reported on *Hibiscus rosa-sinensis* L. (Malvales: Malvaceae) from Iran (Moghadam & Bagheri, 2010; Mossadegh *et al*., 2012b; Mossadegh *et al*., 2015). *Hibiscus rosa-sinensis* is widely planted in parks and green space of Iran. Damage by *P. solenopsis* on *H. rosa-sinensis* results in cutting shrubs and significant damage to urban green space. Efficacy of the insecticides to control of *P. solenopsis* on sweet pepinos, showed that the pest effectively controlled by spraying chlorpyrifos and carbofuran (Larrain, 2002). Important natural

enemies of *P. solenopsis* in Iran are *Aenasius bambawalei* Hayat (Hymenoptera: Encyrtidae), *Promuscidea unfasciativentris* Girault (Hymenoptera: Aphelinidae), *Hyperaspis polita* Weise (Coleoptera: Coccinellidae), *Nephus arcuatus* Kapur (Coleoptera: Coccinellidae) and *Cryptolaemus montrouzieri* Mulsant (Coleoptera: Coccinellidae) are (Mossadegh *et al*., 2012a; Mossadegh *et al*., 2013, Mossadegh & Kocheyle, 1992).

Use of pesticides can lead to environmental pollution, affecting human health and causing death of non-target organisms (Biswas *et al*., 2014). Scientists found that a number of plants possess pesticidal activity. Plant extracts and essential oils (EOs) are eco-friendly and more compatible with environmental components compared with synthetic pesticides (Rahman *et al*., 2016). In detail, plant secondary metabolites lead to toxicity against insect pests in low concentrations in addition to ovicidal, larvicidal, anti-feedant, and sterilizing properties (Isman, 2006). However, EOs have some drawbacks on their use such as volatility, rapid oxidation, and chemical instability in the presence of light, moisture, and high temperature. To increase efficiency of EOs, the use of formulations of EOs would be the best option (Emamjomeh *et al*., 2018). *Minthostachys verticillata* (Griseb.) and *Eucalyptus globulus* (Labill.) (Myrtales: Myrtaceae) EOs were evaluated as insecticidal products on *Planococcus ficus* (Signoret) under laboratory conditions. The results revealed that *M. verticillata* (LC_{50} 39.60 $\mu L.L^{-1}$) was more toxic than *E. globulus* (LC_{50} 63.97 $\mu L \cdot L^{-1}$) (Peschiutta *et al*., 2017). Based on Prishanthini & Vinobaba (2014), laboratory studies were carried out to evaluate the efficacy of botanical extracts from *Azadirachta indica* A. Juss. (Rutales: Meliaceae), *Ocimum sanctum* L. (Lamiales: Lamiaceae), *Calotropis gigantean* L. (Gentianales: Apocynaceae), *Nicotina tabacum* L. (Solanales: Solanaceae) and *Alium sativum* L. (Asparagales: Amaryllidaceae) against *P. solenopsis* on *H. rosa-sinensis.* Among the treated botanicals, *O.sanctum* was effective significantly ($p<0.05$) at lower concentrations and has the 0.6% concentration as LC_{50}. Repelling effects of *Prunus persica* L. (Rosales: Rosaceae), *E. globulus*, *Polyalthia longifolia* (Magnoliids: Annonaceae), *Silybum marianum* (Asterales: Asteraceae), and *Sonchus oleraceus* (Asterales: Asteraceae) extracts each in petroleum ether, acetone and ethanol were evaluated at the concentration of 1000, 500 and 250 ppm against *P. solenopsis*. The ethanol extract was the most effective against cotton mealy bug by having highest repellency 72.5% at 500 ppm. The lowest average repellency was 26.3 % observed in acetone extract of at 500 ppm dose (Roonjho *et al*., 2013). Recently, there has been an increasing interest in studying and evaluating the botanical insecticides (e.g. EOs) for pest management in both developing and developed countries as a result of insect resistant to the traditional insecticides (Mossa, 2016). Present study attempts to evaluate the efficacy of EO formulations from *Mentha longifolia* L. (Lamiacea) (Wild mint), *Mentha piperita* L. (Lamiacea) (Peppermint) and *Oliveria decumbens* Vent. (Umbelliferae) (Denak) against the 1st instar nymphs of *P. solenopsis* under laboratory conditions.

Materials and Methods

Collection of the plants and preparation of EOs

A preliminary trial was conducted to select *M. longifolia*, *M. piperita*, and *O. decumbens* plant for EO extraction among of the fifteen examined plants (Table 1). The results showed that three plants *M. longifolia*, *M. piperita*, and *O. decumbens* were the most effective with LC_{50} 113.49, 129.74, 149.93 ppm, respectively (Table 6). Then leaves of *M. longifolia* and *M. piperita* and aerial parts of *O. decumbens* were dried at temperatures up to 40°C. Their EOs were extracted via steam distillation using a clevenger apparatus. Distillation took about 3h to obtain the EOs. Finally, the EOs were dehydrated by sodium sulphate and kept at 4°C for less than a month before onset of bioassays (Mahmoodi Sourestani, 2016).

Table 1. Characteristics of collected plants for essential oils extraction

Scientific name	Family	Used part of plant	Collection place
Mentha piperita L.	Lamiaceae	Leaves	Cultivated (Dezful)
Teucrium polium Boiss.	Lamiaceae	Leaves	Wild (Ilam)
Rosmarinus officinalis L.	Lamiaceae	Leaves	Cultivated (Ahvaz)
Nepeta cataria L.	Lamiaceae	Leaves	Cultivated (Ahvaz)
Mentha longifolia L.	Lamiaceae	Leaves	Cultivated (Ahvaz)
Ocimum basilicum L.	Lamiaceae	Leaves	Wild (Ilam)
Mentha spicata L.	Lamiaceae	Leaves	Cultivated (Ahvaz)
Dracocephalum moldavica L.	Lamiaceae	Leaves	Cultivated (Dezful)
Satureja khuzistanica Jamzad.	Lamiaceae	Leaves	Cultivated (Ahvaz)
Origanum vulgare L.	Lamiaceae	Leaves	Wild (Ilam)
Myrtus communis L.	Myrtaceae	Leaves	Wild (Ilam)
Eucalyptus camaldulensis Dehnh.	Myrtaceae	Leaves	Cultivated (Fars)
Callistemon viminalis Gaertn.	Myrtaceae	Leaves	Cultivated (Dezful)
Prangos ferulacea L.	Apiaceae	Leaves	Cultivated (Ahvaz)
Oliveria decumbens Vent.	Umbelliferae	Aerial parts	Cultivated (Dezful)

Gas Chromatography (GC) and Gas Chromatography-Mass Spectrometer (GC/MS)

Analysis of EOs was performed using a Gas Chromatography and Gas Chromatography interfaced to Mass Spectroscopy. Applied GC was Varian 3800 and column was CP-Sil8-CB (30 m. length, 0.32 mm. internal diameter, 0.25 µm. film thickness). For *O. decumbens* EO temperature was programmed to increase from 60°C to 260°C at a rate of 5°C/min and then held isothermally for 2 min, injector and detector temperatures were set at 265ºC and 275ºC, respectively. Also, for Eos of *M. longifolia* and *M. piperita*, temperature increased from 40°C to 300°C at a rate of 5°C/min and then held isothermally for one min, injector and detector temperatures were set at 280ºC and 300ºC, respectively. Helium was used as the carrier gas at a flow rate of 1.5 ml/min. For GC interfaced to MS using an Agilent 5975 was equipped with an HP-5ms capillary column (30m length, 0.25mm internal diameter, 0.25µm. film thickness). Injector and detector temperature and column temperature and carrier gas was similar to GC. The MS transfer line temperature maintained at 280°C, whereas the ion source

temperature was 180°C. Scan time 1s and ionization energy 70 eV (Central Laboratory of Shahid Chamran University of Ahvaz, Iran).

Insect rearing

To establish a laboratory colony of *P. solenopsis*, the adult females were collected from Chinese hibiscus shrubs from campus of Shahid Chamran University of Ahvaz and were transferred to the laboratory. The insects were fed on potato *Solanum tuberosum* (L.) buds at 25 ± 1°C, 65 ± 5% RH and 16L: 8D of photoperiod to get stock population. Potatoes were replaced every three weeks and insects reared at the F_{10} generation.

Formulation of EOs

Materials used in preparation of oil-in-water formulation were emulsifier agent (polyarylphenyl ether sulphate; 4-6%) (soprophor® 4D384), binding agent (polyvinyl pyrrolidone; 3-6%) (PVP-K30), methanol 70%, active ingredient EOs (5-10%) and sesame oil (1-3%). Therefore, six formulations were prepared by reducing and adding different materials during formulation as follows; For each formulation, at first the binding agent was dissolved in 50 ml of methanol 70% by a laboratory digital stirrer at 500-1000 rpm (Microstar 15 digital, IKA, Germany). Then emulsifier, EOs and sesame oil were added to the solution. Finally, the solution was reached to volume of 100% with methanol 70%. Experimental concentrations were prepared by diluting formulations with distilled water (Table 2) (Riazi *et al*., 2015; Ardakani & Heydari Alizadeh, 2017).

Table 2. Materials and their quantities used for preparation of examined formulations

		Compounds (%)				
Essential Oils	Formulations	Methanol 70%	Emulsifier	Binding agent	Essential oil	Sesame oil
Mentha longifolia	F1	85	5.5	3	5	1.5
	F2	83	4.5	5.5	5	2
Mentha piperita	F3	79	4	4	10	3
	F4	77	6	6	10	1
Oliveria decumbens	F5	78	4.5	4.5	10	3
	F6	77	5	5.5	10	2.5

F1, F2, … & F6 are indices for examined formulations**.**

Physicochemical tests of EOs formulations

Stability of EOs formulations was studied for physicochemical properties according to Poucher (1993) with some modifications. Formulations were tested with different stress factors such as cooling and heating trial, centrifugal trial, freeze and thaw trial, creaming and coalescence trial and pH changing. Formulations were centrifuged at 2,000 rpm (FX-P4, FENIX, India). After waiting 5, 15, 30 and 60 min from the time of centrifugation, the stability of the formulations was evaluated. Creaming and coalescence are signs of instability of formulations. So, it necessary to study the formulations in these states. Samples were stored for 48h at 45-50°C, then stored at 4°C for 48h (heating and cooling trial). Also, samples were stored for 48h at -8 °C, then were stored at 25°C for 48h (freeze and thaw trial). The cycles were repeated 6 times for each test. After the end of 6 periods, the quality of

formulations with respect to appearance was evaluated. The pH value was measured by a pH meter (DA600, Hana, Japan) from time preparing them to 1-week later (FAO, 2006).

Laboratory bioassays

Preliminary experiment for screening EOs

Bioassays were conducted under laboratory conditions in Petri dishes (diameter = 8 cm) that had lids with openings (diameter = 3 cm) covered with fine muslin. Four concentrations were tested for each EO and formulated EOs. At first a preliminary experiment was conducted to assess the insecticidal activity of fifteen plants EOs. The concentrations for *M. longifolia*, *M. piperita* and *O. decumbens* were 110, 150, 200, 300, *N. cataria* and *M. spicata* were 130, 190, 280, 400, *D. moldavica, R. officinalis* and *O. vulgare* 260, 330, 400, 500, *E. camaldulensis, S. khuzistanica*, *P. ferulacea* and *M. communis* 150, 220, 330, 500, and for *T. polium*, *O. basilicum* and *C. viminalis* EOs, the concentrations were 350,390, 440 and 500 ppm. Methanol was used as a solvent to prepare EOs solutions. Leaves of Chinese hibiscus of approximately the same size were dipped in desired concentrations for 15s and air-dried for 30 min. Control leaves were dipped only in methanol (70%). Control and treated leaves were placed on a layer of agar in the Petri dishes, then ten *P. solenopsis* 1st instar nymphs (same size class life stage) were released at the center of leaf discs in the Petri dishes. Petri dishes were kept inside the incubator with the above mentioned conditions at 25±1°C and 65±5% RH, with a 16:8-h L:D photoperiod. (Kaveh *et al*., 2014). Each concentration and control replications were tested three times (Amirmohammadi & Jalali Sendi, 2013; Sohrabi & Kohanmoo, 2016; Mostafa *et al*., 2018). After 48h, the number of the dead 1st instar nymphs in control and treatment was recorded using a stereomicroscope (B-810BF, OPTIK, Italy). The mortality percentages was calculated based on control mortality accounted for using Abbott's formula (Abbott, 1925).

Contact toxicity of three plant EOs and their emulsion formulations

According to the previous experiment, EOs of *M. longifolia*, *M. piperita* and *O. decumbens* exhibited a high degree of efficiency against *P. solenopsis* 1st instar nymphs. So, the concentrations 110, 150, 200, 300 ppm for pure EOs were prepared. Also, due to additives in EOs and their more effectiveness, lower concentrations (60, 80, 110, 150 ppm) than pure EOs were prepared for formulated EOs. The conditions of the experiment was the same as above and the mortality was counted 48h after exposure of 1st instar nymphs to the treated leaves.

Data analysis

The experiments were conducted in a completely randomized design. Mortality data obtained from each dose-response trial were subjected to probit analysis and LC_{50}, LC_{90} and LC_{95} values and 95% confidence intervals were estimated. LC_{50}, LC_{90} and LC_{95} values were compared using respective confidence intervals (Finney, 1971). Also, comparison of these values was done by calculating the relative toxicity parameter, Relative Median Potency

(RMP) (Robertson & Preisler, 1992). Statistical analysis was conducted using SPSS ver. 22 (SPSS, 2019), and ANOVA were performed, and the means were compared using Tukey's test at 5% level.

Results

Chemical composition of plant EOs

The qualitative and quantitative compounds of plant EOs are shown in (Table 3). Focusing on the most abundant components of the EOs, *M. longifolia* consisted primarily of pulegone (51.49%), menthone (22.75%), and 1,8-cineole (11.69%). *M. piperita* EO contained menthone (36.51%), menthene (28.51%), menthol (8.12%), and 1, 8-mineole (7.66%). The main components of *O. decumbens* EO were thymol (43.99%), γ-terpinene (13.96%), and *p*-cymene (12.62%) (Table 3).

Physicochemical tests

For each EO, two formulations were prepared based on different amounts of used materials. All formulations except No. 4 were stable after the physicochemical studies. So, between the 5 stable formulations, No. 1, 3 and 5 due to their more suitable organoleptic properties were selected for the bioassay studies (Tables 4, 5).

By using cycles between –8°C and 25°C, it was possible to establish freeze-thaw stability. Also, it was found that +4°C to 45-50°C cycles proved sufficient to effectively evaluate formulations stability. In creaming and coalescence trial, formulations were able to maintain their quality well during 8 weeks storage at 25°C. Also, maintain their stability in centrifugal trial. Formulations pH ranged from 6.4-6.8. Formulation No. 1, 48h after its preparation had more stability (df = 2, 8; F=8.61; P=0.017).

Laboratory bioassays

In contact toxicity trials, LC_{50} values of pure EOs of *M. longifolia*, *M. piperita*, and *O. decumbens* on 1st instar nymphs after 48h, were 113.49, 129.74, and 149.93 ppm, and for formulated EOs of *M. longifolia*, *M. piperita*, and *O. decumbens*, LC_{50} values were 48.22, 55.55, and 61.68 ppm, respectively (Table 6, 7).

Table 3. Chemical composition (%) of essential oils derived from *Mentha longifolia*, *Mentha piperita*, and *Oliveria decumbens* by GC-MS.

Components	Percentage Composition		
	Mentha longifolia	*Mentha piperita*	*Oliveria decumbens*
α-Pinene	1.19	0.66	0.28
Sabinene	0.20	0.45	0.08
β-Pinene	3.04	1.04	2.26
β-Myrcene	0.86	0.28	
Limonene	0.52		1.54
1, 8-Cineole	11.69	7.66	
α-Terpinolene	0.24	1.41	
Menthone	22.75	36.51	
Isomenthone	3.40		
Isopulegone	1.28		
Pulegone	51.49	1.03	
Piperitone	0.12	0.11	
Bornyl acetate	0.13		
Piperitenone	0.91		0.04
trans-Caryophyllene	1.03	3.77	
α-Humulene	0.12		
α-Cubebene	0.30		
γ-Cadinene	0.10	0.10	
Azulene		0.66	
p-Cymene		0.45	12.62
Ocimene		0.28	
Menthol		8.12	
Menthene		28.51	
Carvone		0.84	
Carene		2.08	
Eugenol		0.24	
β-Cubebene		0.14	
Germacerene D		0.50	
Cadina-4,9-diene		3.71	
α-Cadinene		0.80	
Methyl chavicole		0.51	
alpha-Thujene			0.48
Myrcene			0.76
δ-3-Carene			0.07
Terpinene - α			0.18
Terpinene - γ			13.96
Sabinene hydrate -cis			0.04
Terpinolene			0.08
Linalool			0.07
Terpinen-4-ol			1.03
Terpineol - α			0.1
Thymol			43.99
Carvacrol			0.48
Selinene - β			0.05
Selinene - α			0.04
Spathulenol			0.08
Elemicin			0.22
Myristicin			4.3
Total	99.37	99.86	82.75

Table 4. Comparison of means (± SE) related to pH formulations at different times

Time	Formulations					
	F1	F2	F3	F4	F5	F6
Time preparing	$0.1^{a\ (A)}$±6.4	$0.1^{a\ (A)}$±6.5	$0.1^{a\ (A)}$±6.6	$0.2^{a\ (A)}$±6.5	$0.2^{a\ (A)}$±6.5	$0.1^{a\ (A)}$±6.7
48h later	$0.1^{b\ (A)}$±6.8	$0.2^{a\ (A)}$±6.4	$0.3^{a\ (A)}$±6.5	$0.2^{a\ (A)}$±6.5	$0.1^{a\ (A)}$±6.7	$0.1^{a\ (A)}$±6.7
1-Week later	$0.1^{ab\ (A)}$±6.5	$0.3^{a\ (A)}$±6.4	$0.2^{a\ (A)}$±6.4	$0.1^{a\ (A)}$±6.4	$0.2^{a\ (A)}$±6.6	$0.2^{a\ (A)}$±6.6

* Means within each column and row followed by the same letter are not significantly different (P<0.05).

Table 5. Results of physicochemical trials of examined formulations

Trials	Formulations					
	F1	F2	F3	F4	F5	F6
Creaming and Coalescence	+	+	+	+	+	+
Centrifugal	+	+	+	+	+	+
Freeze and Thaw	+	+	+	-	+	+
Cooling and Heating	+	+	+	-	+	+

Signs are expressed as results of stability trial.
Stability formulation: (+)
Instability of formulation: (-)

Table 6. LC_{50}, LC_{90} and LC_{95} values for contact toxicity of the fifteen pure essential oils on first instar nymphs of *Phenacoccus solenopsis.*

Essential Oils	n	X^2 (df=10)	Slope ± SE	LC_{50} (ppm)	LC_{90} (ppm)	LC_{95} (ppm)
Mentha piperita	120	1.38	0.77±2.55	129.74 (89.99-155.01)	411.40 (283.76- 1578.93)	570.70 (352.17- 3512.72)
Teucrium polium	120	2.11	2.25±8.62	363.75 (315.74-385.74)	511.40 (465.17- 662.05)	563.50 (498.35- 803.87)
Rosmarinus officinalis	120	1.84	1.19±3.87	282.10 (213.07-320.96)	604.40 (478.33- 1424.30)	750.00 (551.69- 2435.94)
Nepeta cataria	120	2.24	0.69±2.99	193.05 (158.90-225.06)	518.40 (380.96- 1097.71)	685.30 (466.12- 1812.10)
Mentha longifolia	120	0.71	0.85±3.19	113.49 (80.48-134.92)	285.30 (223.60- 553.56)	370.80 (269.72- 942.82)
Ocimum basilicum	120	1.87	2.26±8.40	357.84 (304.93-380.69)	507.40 (461.23- 663.63)	560.50 (494.88- 815.51)
Mentha spicata	120	1.20	0.69±2.76	163.04 (121.16-194.08)	474.00 (347.72-1077.01)	641.50 (431.28- 1935.92)
Dracocephalum moldavica	120	1.57	1.20±4.78	308.39 (254.88-349.24)	571.30 (474.20- 938.80)	680.50 (536.71- 1316.74)
Satureja khuzistanica	120	2.35	0.66±2.84	205.25 (151.64-242.18)	580.40 (427.77- 1211.13)	778.90 (529.87- 2055.19)
Origanum vulgare	120	1.08	1.18±3.89	292.93 (230.00-339.98)	612.90 (486.02- 1365.25)	756.10 (559.18- 2242.06)
Myrtus communis	120	1.11	0.64±2.66	211.07 (154.59-261.47)	640.90 (456.41- 1532.30)	877.30 (571.10- 2759.70)
Eucalyptus camaldulensis	120	2.26	0.64±2.59	215.03 (156.04-266.23)	671.40 (469.96- 1729.28)	927.10 (590.43- 3207.47)
Callistemon viminalis	120	2.53	2.16±7.65	371.90 (331.63-395.94)	546.60 (485.52- 795.18)	609.70 (522.99- 1012.02)
Prangos ferulacea	120	2.20	0.66±2.85	205.74 (151.26-242.36)	578.50 (426.68- 1201.44)	775.70 (528.34- 2032.95)
Oliveria decumbens	120	0.02	1.80±0.73	149.93 (62.35-206.45)	771.42 (385.42-3026.54)	1228.37 (503.14-3069.03)

n: number of tested insects

Table 7. LC_{50}, LC_{90} and LC_{95} values of contact toxicity of formulated essential oils of *Mentha longifolia*, *Mentha piperita*, and *Oliveria decumbens* on 1st instar nymphs of *Phenacoccus solenopsis* after 48 h.

Essential Oils	X^2 (df)	Slope ± SE	LC_{50} (ppm) 95% Confidence interval	LC_{90} (ppm) 95% Confidence interval	LC_{95} (ppm) 95% Confidence interval
Mentha longifolia	2.05(10)	2.82±0.93	48.22 (22.83-62.09)	136.98 (109.91-252.28)	184.15 (135.91-473.98)
Mentha piperita	1.44(10)	2.70±0.87	55.55 (30.71-69.20)	165.68 (127.41-355.97)	225.84 (158.09-683.19)
Oliveria decumbens	0.59(10)	2.35±0.83	61.68 (33.51-76.68)	216.39 (151.60-746.94)	308.85 (191.85-1726.21)

The LC_{50} values did not reveal any significant differences among pure EOs of tested plants. Based on (RMP) calculations, LC_{50} values for pure EOs were significantly greater

than the LC_{50} of formulated EOs (Table 8). Therefore, toxicity of formulated EOs of *M. longifolia*, *M. piperita*, and *O. decumbens* were 2.34, 2.36 and 2.43-fold higher than the pure EOs (Table 8).

Table 8. Comparison relative contact toxicity of formulated essential oils and pure essential oils of *Mentha longifolia*, *Mentha piperita*, and *Oliveria decumbens* on 1st instar nymphs of *Phenacoccus solenopsis.*

Essential Oils	RMP LC_{50} (pure): LC_{50} (formulated)	95% Confidence interval
Mentha longifolia	2.34	1.39-3.92*
Mentha piperita	2.36	1.49-3.74*
Oliveria decumbens	2.43	1.49-3.96*

RMP: Relative Median Potency, *: Shows significant difference between the LC_{50} values compared at 5% probability level.

Discussion

Overall EOs of *M. longifolia*, *M. piperita*, and *O. decumbens* were equally toxic against the 1st instar nymphs of *P. solenopsis*. Insecticidal properties of different *Mentha* species EO have been reported on various insect pests. Fumigant and repellent toxicities of *Ricinus communis* (L.) and *Mentha pulegium* (L.) EOs were assessed toward two major stored product beetles: *Lasioderma serricorne* (F.) and *Tribolium castaneum* (Herbst). The effectiveness of *M. pulegium* EO against the coleopteran insects showed potential fumigant impact particularly against *L. serricorne* with LC_{50} = 8.46 µL/L air. Moreover, significant pest repellent activity was demonstrated with *R. communis* and *M. pulegium* where the repellency effects reached 80 and 60% after 1 and 24h of exposure against *T. castaneum* at doses of 0.31 µL/cm^2 and 0.078 µL/cm^2, respectively (Salem *et al*., 2017). Based on Saeidi & Moharramipour (2013), *M. longifolia* proved to be fumigant toxicity less than *Artemisia khorassanica* (Podl.) and *R. officinalis* on *Tribolium confusum* (Duval). In contrast to their low fumigant properties, the EO of *M. longifolia* had significantly higher repellency to *T. confusum* adults than the other two. Efficacy of *M. piperita* EO, with four different solvents namely: acetone, ethanol, n-hexane and chloroform, was screened against the green peach aphid *Myzus persicae* Sulzer (Homoptera: Aphididae). As the result shown, *M. piperita* EO with chloroform and ethanol was the most effective against 1st and 2nd nymph of *M. persicae* with the LC_{50} of 0.004 (v/v) and LC_{90} of 0.090 and 0.070 (v/v) (Al – Antary *et al*., 2017). Also, the most efficient EOs were obtained from *M. pulegium* and *Thymus mastichina* (L.), with $LC_{50\,(90)}$ estimated as 3.1(3.8) and 3.6 (4.6) mg/L air, respectively against *Frankliniella occidentalis* (Perg.) (Stepanycheva *et al*., 2019). Based on Mostafa *et al*. (2018), ten EOs of seven different families including *Thymus vulgaris* L. (Lamiaceae), *Artemisia absinthium* L. (Asteraceae), *Pluchea dioscoridis* L. (Asteraceae), *Cyperus articulatus L.* (Cyperaceae), *M. longifolia*, *Anethum graveolens* L. (Apiaceae)) and *Lantana camara* L.(Verbenaceae) were extracted and examined for their insecticidal activity against adult females of *P. solenopsis.*

Results showed that *T. vulgaris*, *M. longifolia* L. and *C. articulates* exhibited a high degree of efficiency as insecticide with the LC_{50} values 29.03, 34.32 and 54.69 ppm, respectively after 24h, while, after 72h of treatments were 15.04, 24.93 and 29.21 ppm, respectively. Also, in the present study, *Mentha* species (*M. longifolia* with LC_{50} =113.49 ppm, *M. piperita* with LC_{50} =129.74 ppm, *Mentha spicata* with LC_{50} =163.04 ppm) had a significant insecticidal effect. The EOs of *M. piperita*, *Satureja thymbra* (L), *Lavandula angustifolia* (Mill.), and *O. basilicum* were tested for their insecticidal activity against *P. ficus*. According to the results, the main components of *M. piperita* EO consisted of menthol (34.6%), menthone (14.6%), *α*-pinene (0.7%), and menthyl acetate (12.4%) (Karamaouna *et al*., 2013). Based on Bolandnazar *et al*. (2017), mentone (22.55%), menthol (34.81%) and menthyl acetate (10.64%) were the principal components of *M. piperita*. They studied the effects of some micro and nanoemulsified EOs (*R. officinalis, M. piperita* and *E. globulus*) on *Bemisia tabaci* Genn. (Hem.: Aleyrodidae) under laboratory condition and found that nano-emulsion treatment containing all tested EOs was the most toxic for controlling population of the whitefly. Comparison among compounds of the EOs in both studies, with our study showed that menthene and 1, 8-mineole did not exist in EO of *M. piperita*. While in our investigation menthone (36.51%) was one of the major components of EO along with menthone (36.51%), menthene (28.51%), menthol (8.12%) and 1, 8-mineole (7.66%).

The insecticide activity of the EO of *M. longifolia*, consisting mainly 1-8-cineole (25.46%), menthone (17.85%), pulegone (29.93%) was found to be effective against *Sitophilus zeamais* (Motschulsky) (Odeyemi *et al*., 2008). In another study, Azarkish *et al*. (2016), investigated of the compositions of EO of *M. longifolia*, a rich source of polygon in five habitats of Fars province. The results showed high amount of pulegone in EO. Also, in our study, polygon (51.49%) had the highest percentage of composition for EO of *M. longifolia*. The results obtained from *O. decumbens* EO analysis showed that the EO contained thymol (43.99%), γ-terpinene (13.96%), and *p*-cymene (12.62%). Another investigation which has been done on this plant from Iran showd that thymol was one of the major components of the EO (Najafpour Navai & Mirza, 2002; Amin *et al*., 2005; Mahboubi *et al*., 2008; sajjadi & Hoseini, 2011). But, in a study performed by Hajimehdipoor *et al*., (2010), γ-terpinene (23.33) and *ρ*-cymene (19.40) were higher than other components of *O. decumbens* essential oil.

The differences in detailed findings may be attributed to the place where plants cultivated, harvesting time, drying temperature, drying period and etc. (Mahmoodi Sourestani, 2016). However, the chemical composition of plants is known to be influenced by several external factors including climate, as some compounds may be accumulated at a particular period to respond to environmental changes (McKay & Blumberg, 2006). Also, the timing of harvest and the number of harvests during the year are factors that greatly influence herbage and EO yields, EO content, and composition of plants (Hussain *et al*.,

2010). There is no information about preparing formulation from *M. longifolia, M. piperita* and *O. decumbens* EOs and their effects on *P. solenopsis* until now. There are some data about different formulations EOs including *M. longifolia* and *M. piperita* on various pests.

In a research by Louni *et al*. (2018), the contact toxicity of *M. longifolia* EO compared with its nanoemulsion on *Ephestia kuehniella* (Zeller.) was investigated. Their results showed that the nanoemulsion formulation increased the effect of EO contact toxicity and its durability. Laing *et al*. (2012) increased stability and bioavailability of *M. piperita* oil starch based on the nanoemulsion.

In this study, samples were stable after the physicochemical studies. Oil-in-water emulsions are, however, inherently unstable and all emulsions will eventually degrade and separate. The evaluation of emulsion stability is consequently of significant importance. The known mechanisms for emulsion degradation are separation (creaming) and coalescence. Historically, stability testing has been done by centrifugation and isothermal storage at elevated temperatures. Centrifugation can be considered effective at predicting creaming instability, but does not evaluate coalescence effectively. Isothermal testing at 25°C is very commonly used to speed evaluation of coalescence. This test is inherently slow, often requiring 8 weeks or more. It was found that four or less +3 °C to 50 °C cycles lasting 2-3 days proved sufficient to effectively evaluate emulsion stability. Formulations pH ranged from 6.4 - 6.8 indicating good stability of the formulations because many changes in the formulations can be a sign of the activity of fungal or microbial agents that causes formulations to degrade (Poucher, 1993).

The present study provides a first screening on the insecticidal activity of pure EOs on 1st instar nymphs of *P. solenopsis*. In contrast, some problems (e.g. volatility, solubility and oxidation) of EO-based insecticides were recorded which plays an important role in the EOs activity, application and persistent. For this reason, new formulations can resolve these problems and offer numerous advantages. In this paper, formulations were prepared by selecting and testing different materials, and investigating their chemical effects. The EOs were incorporated in absorption bases and after preliminary studies 6 formulations were prepared.

Based on the results, formulated EOs had a stronger insecticidal effect when compared with pure EOs. According to the results, production of formulation with this new technique results in considerable decrease of the required EO concentrations. The additives in the EOs formulation may act as synergist and lead to increase the toxicity of EOs. Also, EOs showed a significant relative percentage of monoterpenes. This suggests that their presence may responsible for the highly insecticidal properties against *P. solenopsis* 1st instar nymphs.

This paper suggests plants that are rich sources of secondary metabolites with pesticidal properties which can be a suitable alternative to chemicals and can be used in *P. solenopsis* management program. Also, extensive field experimentations are needed to determine the

insecticidal efficiency of formulated EOs against 1st instar nymphs of *P. solenopsis* and the toxic effect on its natural enemies under normal cropping conditions.

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