Egypt. J. Plant Prot. Res. Inst. (2019), 2 (2): 291 - 300

Egyptian Journal of Plant Protection Research Institute

www.ejppri.eg.net

Insecticidal activity of citrus peel oil of navel orange against the striped mealybug *Ferrisia virgata* (Hemiptera: Pseudococcidae) and the mango shield scale *Milviscutulus mangiferae* (Hemiptera: Coccidae)

Ahmed, A. Elhefny; Sahar, A. Attia ; Samah, M.Y. Helmy and Wafaa, M.M. El-Bradey

Plant Protection Research Institute, Agricultural Research Centre, Dokki, Giza, Egypt.

ARTICLE INFO

Article History

Received:25 / 4 / 2019

Accepted: 20 / 6 /2019

Keywords

Navel orange, essential oil, *Ferrisia virgata*, *Milviscutulus mangiferae* and insecticidal activities.

Abstract:

Essential oil extracted from peels of citrus fruit namely navel orange (*Citrus sinensis* L.), belonging to family Rutaceae was tested for its insecticidal activity at four different concentrations (500, 1000, 2500, and 5000ppm) against nymphs and adults of the striped mealybug *Ferrisia virgata* Cockerell (Hemiptera, Pseudococcidae) and mango shield scale or mango soft scale *Milviscutulus mangiferae* (Green) (Hemiptera: Coccidae). Formulated oils of navel orange was bioassayed against mealybug *F. virgata* and *M. mangiferae*. The results revealed that, the two formulated oils of navel orange achieved high toxicity against nymphs and adults of *F. virgata* and *M. mangiferae* . The essential oil of navel orange was isolated by hydrodistillation and the analysis of essential oil by GC/MS revealed the presence of 35 peaks, approximately all peaks were identified. The chemical composition showed that limonene was the main constituent in citrus oil (78.15%). The results of the present study suggested that, formulated navel orange oil used as safe, potential natural products for control of *F. virgata* and *M. mangiferae* infesting mango and guava trees and may be used as alternatives to the reference products after application of these results in the semifield and field experiments.

Introduction

Fruits trees are liable to be infested with many serious pests during their growth stages, including striped mealybug *Ferrisia virgata* Cockerell (Hemiptera: Pseudococcidae) infested guava trees and *Milviscutulus mangiferae* (Green) (Hemiptera: Coccidae) infested mango trees. Mango trees (*Mangifera indica* L.) are considered of the most popular and economic fruit trees in Egypt, it occupy the third rank from the commercial point of view (Attia, 2010). Mango is tropical/sub tropical fruit with highly significant economic importance (Sivakumar and Yahia, 2011). Guava trees (*Psidium guajava*) has major commercial importance in India, Egypt, South Africa, Barazil, Colombia and the Caribbean region. The fruits are eaten fresh or as preserves and processed for use in dairy and baked products (Richard, 2005).

The striped mealybug *F. virgata* is one of the most important pest attacking many different host plant, belongs to several plant families, it infests mulberry, fig, guava , pear, apple, grape and olive in Egypt (EL-Minshawy *et al*., 1972). The mealybug *F. virgata* can be found throughout the world, is known to feed on more than 100 plant species grown throughout the world. This mealybug mainly attacks the foliage, sucks a great amount of plant sap for its protein requirement and secretes honey dew. Most mealybug individuals are accumulated around the branches, foliage, leaves, twigs and at the base of fruits. Many of these species are covered with white wax and have a distinct fringe of waxy filaments around the circumference of their bodies and the long tails and the presence of two stripes on the body. This species produces an egg mass or ovisac (Ghose and Ghosh, 1990).

The mango shield scale or mango soft scale, Milviscutulus mangiferae (Green) (Hemiptera: Coccidae), a serious pest of mango trees in various parts of the world and is reported on *M. indica* in Egypt , represents the first record of this species in the country (Abd-Rabou and Evans, 2018). *M. mangiferae* an invasive coccid infested mango orchard profusely in Qaliobiya Governorate caused yellowing, defoliation, reduction in fruit set and loss in plant vigor, the insect excrete large amounts of honeydew which encourage the growth of sooty mould and the infested parts acquire the dirty black appearance that affect on photosynthesis (Attia *et al.,* 2018).

In general, control of the mealybugs and soft scale insects around the world relies heavily on use of insecticides and mineral oils. However, continuous and heavy use of these synthetic pesticides has created serious problems such as environmental pollution, toxicity to non-target organisms (parasitoids and predators), pest resistance and pesticide residues (Mohan and Fields, 2002). Therefore, there is an urgent need to develop new, convenient and safer alternatives to synthetic pesticides. Essential oils and their major constituents, attracted research attention in recent years as potential alternatives to synthetic insecticides (Aslan *et al*., 2004).

The genus *Citrus* includes several important fruits such as oranges, mandarins, lime, lemons and grapefruits. The essential oils of some citrus species have been reported to have insecticidal properties against insect pests (Elhag, 2000). The major active component of citrus oil is limonene and using 1% limonene mixture was safe for most plants and provided good control of mealybugs and scale insects (Hollingsworth, 2005).

The aim of this research work is to study the efficacy of Egyptian citrus peel essential oil of navel orange (*Citrus sinensis*) against nymphs and adults of mealybug *F. virgata* and soft scale insect *M. mangiferae* on mango and guava trees, respectively. Also, extraction and chemical analysis of essential oil was studied.

Materials and methods

1. Tested citrus species:

The experimental citrus species, navel orange (*C. sinensis*), belonging to family Rutaceae was selected for this study. This citrus species was obtained from a private citrus orchard.

2. Insects source:

Insects culture of *F. virgata* and *M. mangiferae* for laboratory experiments were obtained from a private orchards at El Mansouria region in Giza Governotare . Samples were collected randomly from each of the four cardinal directions (East, West, North and South). Leaves were packed in paper bags and transferred to the laboratory and they were maintained at laboratory temperature about 25±1°C and 65±1% relative humidity. In the laboratory, *F. virgata* and *M. mangiferae* were identified by Department of Mealybug and Scale Insects, Plant Protection Researches Institute, Agriculture Researches Centre.

3. Extraction of citrus oil:

Citrus oil was extracted by Cavalcanti *et al.* (2004). The essential oil was extracted from the fresh peels (200g weight 400 ml of distilled water) by hydrodistillation using a modified Clevenger type apparatus for 4 h. The distilled was extracted with diethyl ether after saturation with Sodium Chloride. The extracted oil was dried over anhydrous Sodium Sulfate, then packed in dark container and stored at 4°C until used for GC-MS analysis and bioassays.

4. Chemical analysis of essential oil:

4.1. Chemical analysis of citrus peel oil constituents:

The extracted citrus oil was subjected to GC/MS analysis using Shimadzu GC/MS–QP-2010 Plus (Japan). Column: DB5 MS (30 m length, 0.25 mm thickness, 0.25mdiameter, 1.5µm film). Carrier gas: Helium (flow rate 1.2 ml/Min.). Ionization mode: (70ev). The injection volume was 0.5µl (split ratio of 1:100), temperature program: 50^oC (static for 2 Min) with gradually increasing (a rate of 4^oC/ Min) up to 200^oC then (10^oC/ Min) to 280^0C. The detector temperature was 290^0C, while, the injector temperature was 250^0C.

4.2. Identification of the chemical constituents:

Qualitative identification of the essential oil was achieved by library searched data base Willey 229 LIB as well as by comparing their retention indices and mass fragmentation patterns with those of the available references and with published data, (Adams, 2007). The percentage composition of components of the volatile was deterMined by computerized peak area measurements.

5. Preparation of formulated orange essential oil:

Four concentrations (500, 1000, 2500 and 5000ppm) of formulated citrus oil of navel orange were prepared by two emulsifiers, Triton-100 (TE) and local emulsifier (LE).

6. Toxicity bioassays:

Laboratory bioassays were conducted to determine the bioactivity of formulated citrus oil of navel orange against nymphs and adults of *F. virgata* and *M. mangiferae*.

The toxicity bioassay was conducted to evaluate toxicity of formulated citrus oil of navel orange to nymphs and adults of mealybug *F. virgata* and soft scale insect *M. mangiferae* at four different concentrations (500, 1000, 2500 and 5000ppm). In spray toxicity assay, guava and mango trees leaves containing nymphs and adults of mealybug *F. virgata* and mango shield scale *M. mangiferae* , respectively were placed into plastic petri dishes (10cm dia × 2cm ht). Ten infested leaves were sprayed with 1ml for five seconds of the two formulated citrus oils of navel orange (Triton-x100 and local emulsifier), then, kept at room temperature. Control insects were sprayed with Triton-x100 and local emulsifiers alone (without oil). Five replicates were used and the experiment was repeated for three times and mortality was recorded after 24, 48 and 72 hrs.

7. Statistical analysis:

The percentage of the mortality was recorded and the mortality was corrected with Abbot formula (Abbott, 1925). LdP-line program was used to deterMinate LC_{50} values. Data of all experiments were evaluated statistically using ANOVA and means compared using Duncan's Multiple Range Test (Duncan, 1955) at P<0.05). All statistical analyses were done using the software package Costat.

Results and discussion

1. Toxicity bioassay:

The obtained two formulated citrus oil of navel orange species in this study were mainly conducted to investigate a relationship between the oil constituents and their potency towards nymphs and adults of *F. virgata* and *M. mangiferae*.

1.1. Toxicity of formulated essential oil of navel orange against *Ferrisia virgata*:

The results of toxicity assays (spray toxicity) as represented in Tables (1 and 2), showed that essential oil of citrus peel exhibited toxicity rate with concentration and time dependent. Formulated peel essential oil of (TE) achieved higher mortality percentages against nymphs and adults than formulated peel essential oil of (LE) at the different concentrations (500, 1000, 2500, 5000 ppm). The highest toxicity rates against nymphs and adults was recorded with formulated peel orange oil of (TE) were 98.00±4.00 and 95.70±4.00 %, respectively, at the maximum concentration (5000ppm) and the last day (72hr) of assay. The percentages of mortality achieved by formulated peel essential oil of (LE) were 92.30±4.00% and 87.50±4.95 % respectively, at the same concentration and time.

1.2.Toxicity of formulated essential oil of navel orange against *Milviscutulus mangiferae*:

The results of toxicity assays as represented in Tables (3 and 4), showed that essential oil of citrus peels exhibited toxicity rate with concentration and time dependent. Formulated peel essential oil of (TE) achieved higher mortality percentages against nymphs and adults than formulated peel essential oil of (LE) at the different concentrations (500, 1000, 2500 and 5000 ppm). The highest toxicity rates of formulated navel orange essential oil of (TE) against nymphs and adults were 81.69±2.48 and 83.47±2.48%, respectively, at the maximum concentration 5000ppm and the last day 72hr. While, the percentages of mortality of navel orange essential oil of (LE) were 81.43±7.08% and 73.33±2.45%, respectively, at the same concentration and time. The lowest mortality against nymphs and adults of the two insects was obtained with the lowest concentration (500 ppm) and at the first day of assay (24hr). Generally, the two formulated citrus oils were toxic to nymphs and adults of *F. virgata* and *M. mangiferae* at all concentrations. There were significant differences in mortality between the tested concentrations after 24hr, but, non-significant differences in mortality after 48 and 72hrs were observed. Also, there were significant differences in mortality between control and treated variants (P< 0.05).

Insecticidal effect of citrus species essential oils against mealybugs and soft scale insects were studied by many workers, Pumnuan *et al*. (2015) showed that, fresh peels essential oils of four citrus species recorded moderate toxicity at 2 ml/L air (fumigation) and high toxicity at 2 ml/L air against larvae of mealybug *Pseudococcus jackbeardsleyi* Gimpel and Miller (Hemiptera: Pseudococcidae) at 24hr. These findings are confirmed by Karamaouna *et al*. (2013), who showed that the citrus peel essential oils of lemon (*Citrus limon*) and navel orange (*C. sinensis*) were the most toxic of all the tested essential oils against 3rd instar nymphs and female adults of the vine mealybug *Planococcus ficus* (Signoret) (Hemiptera: Pseudococcidae). Also, El-Badawy (2015) found that, all tested citrus oils specially navel and baladi oranges achieved high insecticidal and repellent activities against mealybug *Icerya seychellarum* (Westwood) (Hemiptera:Monophlebidae).

2.Effect of formulation on potency of navel orange essential oil:

From the data of LC_{50} values presented in Tables (1, 2, 3 and 4), it could be demonstrated that, the formulated peel citrus oil of (TE) was more potent nymphicidal and adultscidal effect than the formulated peel essential citrus oil of (LE) against *F. virgata* and *M. mangiferae* after 24, 48 and 72hr of assay. From the data presented in Tables (1 and 2), it could be demonstrated that, the LC_{50} values of the formulated orange oil of (TE) against *F. virgata* ranged from 22.44 (Last day 72hr) to 778.70ppm (1^{st}day 24 hr) for nymphs and from 36.79 to 807.66ppm for adults after the same times, while, the formulated orange oil of (LE) recoded LC_{50} values ranged from 44.56 ppm (Last day 72hr) to 934.61 ppm (1^{st}day 24 hr) for nymphs and from 81.27 to 1146.49ppm for adults after the same time.

The data presented in Table (3 and 4), reported that, the LC_{50} values of the formulated citrus oil of (TE) against *M. mangiferae* ranged from 155 (Last day 72hr) to 790.20 ppm (1^{st} day 24 hr) for nymphs and from 193.29to 815.23ppm for adults, while, the formulated citrus oil of (LE) recoded LC_{50} values ranged from 230.74 (Last day 72hr) to 1044.73ppm (1^{st}day 24 hr) for nymphs and from 474.83 to 8105.53ppm for adults after the same time. The variation of the LC_{50} values of citrus oil against *F. virgata* and *M. mangiferae* depending on the toxicity of the formulation of citrus oil, the mealybug and scale insect life stage. LC_{50} values of each formulated citrus oil reveal significant differences between nymphs and adults.

These findings are confirmed by Karamaouna *et al*. (2013) who showed that, the LC_{50} values of citrus (*C. sinensis* and *C. limon*) oils ranged from 2.7 to 8.1mg/ml depending on the essential oil and the mealybug life stage. These LC_{50} values were significantly lower than the LC_{50} of the reference paraffin oil in the respective *P. ficus* life stages. Results of El-Badawy (2015) revealed that the oil of navel orange achieved the highest toxicity against nymphs and adults of mealybug *I. seychellarum* with LC_{50} values of (406.97 and 370.04 ppm), respectively.

3. Chemical analysis of citrus peel essential oil:

The essential oil yield of fresh citrus peels of *C. sinensis* was 4.30%. The chemical composition of the essential oil of citrus peels are presented in Table (5). The essential oil analysis by GC/MS revealed that, the presence of 35 peaks, all peaks were identified, representing 99.46 % of the essential oil of navel orange. The major constituents of this essential oil mainly belonged to two groups: Monoterpenes and oxygenated monoterpenes hydrocarbons, while the minor constituents belonged to: sesquiterpene and oxygenated sesquiterpene hydrocarbons. Oxygenated monoterpenes with contribution of 3.08% constituted the second major portion of the essential oil after monoterpenes (86.41%) from peel oil. Sesquiterpene hydrocarbon was present at very low levels in the oil of navel orange.

The chemical analysis of the citrus oil showed limonene as the main constituent (78.15%) for navel orange. The monoterpene hydrocarbons α-Phellandrene, β- Phellandrene, *α*-pinene, β-pinene, β-Myrcene, 3-Carene β-Ocimene and γ-Terpinene are present in studied citrus oil. The qualitative and quantitative composition of the essential oil of fresh citrus peels showed that, the most abundant ingredients beside to limonene, were β-myrcene (4.30%), linalool (1.59%) and *α*-pinene (1.55%) in the citrus peels oil of navel orange. Among other than monoterpenes, Bis (2-ethylhexyl) phthalate (7.60%) was present in oil. Our results of the chemical composition of citrus peel oil are in agreement with many other studies (Mansour *et al*., 2004; Ahmad *et al*., 2006; Asekun *et al*., 2007 and El-Badawy ,2015). All these studies showed that, limonene was the main component with high variation in all citrus peel oils and also, there are considerable variations in the other constituents of the chemical composition of citrus oils. Such variation in chemical composition (Limonene content and other constituents) in citrus peel oils may be related to the time of harvesting, the degree of freshness, genetic makeup and the size of the fruit. Also, geographical location, fruit variety and method of extraction (Ahmad *et al*., 2006).

Regarding to potency of citrus oil against nymphs and adults of *F. virgata* and *M. mangiferae* the data presented in Tables (1-4) indicated that the potency of the tested formulated oils was related to the major component limonene content of that oils. These results are confirmed by El-Badawy (2014 and 2015), who showed that the toxic effect of five citrus oils on *I. seychellarum* could be related to the high content of limonene. Also, these results are in agreement with these obtained by Ibrahim *et al*. (2001) who stated that the monoterpene limonene showed deterrent and insecticide properties, which might used in pest control in organic agriculture. The best limonene mixture (1% limonene, 0.75% emulsifier APSA-80 and 0.1% surfactant Silwet) controlled from 69 to 100% of mealybugs and scale insects, depending on the species, insect stage and application method (Hollingsworth, 2005). Also Ware (2000) showed that, orange oil contains the monoterpenoid d-limonene, and the mode of action of d-limonene is similar to that of pyrethrum, affecting sodium flux in the peripheral neurons.

Formulated citrus oil of navel orange achieved high insecticidal activity against striped mealybug *F. virgata* and mango shield scale *M. mangiferae*, so, it can be used as an effective natural alternative to mineral oils and insecticidal soap. Overall results indicated that the toxic effects of citrus oil on *F. virgata* and *M. mangiferae* could be related to the high content of limonene. There are synergistic or antagonistic effects between limonene and the other minor constituents in the citrus oils.

It is recommended to expand such laboratory experiments to semifield and field conditions and determine the efficacy of orange citrus essential oil against *F. virgata* , *M. mangiferae* and other scale insects

Table (1): Toxic effect of formulated essential oil (Triton- x100 emulsifier) of navel orange peels against *Ferrisia virgata* nymphs and adults at different concentrations.

Corrected mortality(%)±SE						
Conc. (ppm)	**Nymph**			**Adult**		
	24hr	**48 hr**	**72 hr**	**24 hr**	**48 hr**	**72 hr**
500	36.70±2.45^{c}	79.00±3.43^{a}	83.67±4.00^{a}	38.70±5.09^{c}	72.69±1.37^{a}	80.00±8.90^{a}
1000	56.67±5.09^{b}	86.11±4.75^{a}	88.00±4.90^{a}	58.67±5.09^{b}	80.00±4.33^{a}	88.00±4.90^{a}
2500	83.33±2.45^{a}	90.00±2.45^{a}	92.00±4.90^{a}	66.67±5.09^{ab}	85.70±4.87^{a}	92.00±4.90^{a}
5000	86.67±2.45^{a}	95.00±2.45^{a}	98.00±4.00^{a}	76.70±2.45^{a}	88.89±2.45^{a}	95.70±4.00^{a}
Control	0.00	0.00	00.00	0.00	0.00	00.00
LC_{50}	778.70	35.87	22.44	807.66	46.23	36.79
F value	20.10***	2.78ns	1.27ns	19.10**	2.48ns	1.30ns
$LSD_{0.05}$	17.19	17.19	13.41	16.20	27.91	17.99

Table (2): Toxic effect of formulated essential oil (Local emulsifier) of navel orange peels against *Ferrisia virgata* nymphs and adults at different concentrations.

Corrected mortality(%)±SE						
Conc. (ppm)	**Nymph**			**Adult**		
	24hr	**48 hr**	**72 hr**	**24 hr**	**48 hr**	**72 hr**
500	53.33±5.09[b]	57.14±5.39[b]	70.00±6.32[b]	36.70±3.00[c]	63.16±8.43[a]	70.83±8.00[a]
1000	60.0±2.74[b]	83.33±4.43[ab]	84.62±9.80[ab]	60.00±3.19[b]	66.70±2.91[a]	76.00±6.86[a]
2500	76.60±2.13[b]	85.71±9.24[a]	91.60±8.00[ab]	66.70±2.45[ab]	77.70±6.53[a]	80.00±6.32[a]
5000	83.33±2.13[a]	95.00±2.77[a]	92.30±4.00[a]	76.70±4.00[a]	83.30±7.13[a]	87.50±4.95[a]
Control	0.00	0.00	0.00	0.00	0.00	0.00
LC_{50}	934.61	335.52	44.56	1146.49	218.67	81.27
F value	6.93*	4.01 ns	2.42ns	11.6**	1.11 ns	1.02ns
$LSD_{0.05}$	16.31	27.43	22.03	16.31	36.21	19.84

Table (3): Toxic effect of formulated essential oil (Triton- x100 emulsifier) of navel orange peels against *Milviscutulus mangiferae* nymphs and adults at different concentrations.

Corrected mortality(%)±SE						
Conc. (ppm)	**Nymph**			**Adult**		
	24hr	**48 hr**	**72 hr**	**24 hr**	**48 hr**	**72 hr**
500	54.17±6.00[b]	58.58±0.90[c]	69.64±8.70[a]	41.76±7.12[c]	58.94±7.50[b]	60.66±6.12[b]
1000	63.06±2.54[b]	67.02±3.39[b]	71.43±1.66[a]	70.22±2.65[b]	64.62±3.21 [b]	73.33±0.96[ab]
2500	73.75±1.92[a]	76.97±3.40[a]	76.67±2.63[a]	75.66±3.31[a]	75.61±1.70 [a]	78.60±1.92[a]
5000	74.40±1.92[a]	80.91±0.60[a]	81.69±2.48[a]	76.35±4.17[a]	76.60±2.87 [a]	83.47±2.48[a]
Control	0.00	0.00	0.00	0.00	0.00	0.00
LC_{50}	790.20	317.20	155.00	815.23	502.87	193.29
F value	7.98	16.8**	1.46	10.60**	6.89	2.62
$LSD_{0.05}$	10.59	7.44	14.32	13.86	13.22	10.42

Table (4): Toxic effect of formulated essential oil (Local emulsifier) of navel orange peels against *Milviscutulus mangiferae* nymphs and adults at different concentrations.

Corrected mortality(%)±SE						
Conc. (ppm)	**Nymph**			**Adult**		
	24hr	**48 hr**	**72 hr**	**24 hr**	**48 hr**	**72 hr**
500	42.33±9.37[b]	42.78±7.32[b]	61.54±8.16[b]	38.78±7.12[a]	32.26±3.68[a]	53.45±4.60[b]
1000	44.79±4.61[b]	51.83±7.42[ab]	69.51±7.41[ab]	41.48±5.25[a]	48.05±9.99[a]	63.75±10.20[ab]
2500	64.68±8.42[a]	67.22±6.90[a]	72.34±4.77[ab]	43.76±5.39[a]	51.14±3.21[ab]	71.22±1.92[a]
5000	71.38±3.08[a]	73.32±4.33[a]	81.43±7.08[a]	50.00±9.09[a]	53.17±2.76[a]	73.33±2.45[a]
Control	0.00	0.00	0.00	0.00	0.00	0.00
LC_{50}	1044.73	1005.63	230.74	8105.53	3671.55	474.83
F value	5.25**	2.52ns	1.74ns	0.173ns	3.11ns	3.45ns
$LSD_{0.05}$	19.82	20.64	20.911	20.66	17.17	18.235

Table(5):Chemical composition of essential oil from peels of Navel orange citrus species.

No.	Components	RT(Min.)	Ratio (%)
1	α-Phellandrene	6.901	0.03
2	α-Pinene	7.125	1.55
3	β-Phellandrene	8.344	0.63
4	β-Pinene	8.45	0.63
5	β-Myrcene	8.85	4.3
6	Octanal	9.223	0.53
7	3-Carene	9.443	0.72
8	Benzyl alcohol, p,.alpha.-dimethyl-	9.632	0.03
9	D-Limonene	10.115	78.15
10	β-Ocimene	10.241	0.08
11	γ-Terpinene	10.846	0.16
12	1-Octanol	11.163	0.07
13	β-Terpinolene	11.604	0.02
14	α-Terpinolene	11.665	0.14
15	β-Linalool	11.952	1.59
16	Nonanal	12.066	0.06
17	Limonene oxide	12.999	0.01
18	β-Citronellal	13.333	0.18
19	α-Terpineol	14.005	0.43
20	Decanal	14.596	0.56
21	n-Octyl acetate	14.708	0.06
22	Carveol	14.993	0.04
23	β-Citronellol	15.14	0.18
24	β-cis-Citral	15.477	0.27
25	1-Carvone	15.602	0.03
26	Geranial	15.739	0.35
27	Lauraldehyde	18.979	0.08
28	Caryophyllene	19.417	0.12
29	α-Farnesene	19.61	0.20
30	β-Farnesene	19.924	0.01
31	Germacrene	20.613	0.04
32	Eremophila-1or Eremophila-1(10),8,11-triene	20.903	0.42
33	Cyclohexene,1-methyl-4(5-methyl-1-methylene-4-hexenyl	20.994	0.13
34	δ- Cadinene	21.343	0.06
35	Bis (2-ethylhexyl) phthalate	39.438	7.60
	Monoterpene Hydrocarbons		86.41
	Oxygenated Monoterpene Hydrocarbons		3.08
	Sesquiterpene Hydrocarbons		0.85
	Aldehydes		1.23
	Others		7.89
	Total		99.46

References

Abbott, W. S. (1925): A method of computing the effectiveness of an insecticide. J. Econ. Entomol., 18: 265-267.

Abd-Rabou, S. and Evans, G. A. (2018): The mango shield scale, Milviscutulus mangiferae (Green) (Hemiptera: Coccidae) – A new invasive soft scale in Egypt. Acta Phytopathologica et Entomologica Hungarica, 53 (1): 91-96.

Adams, P.R. (2007): Identification of essential oil components by gas chromatography/ mass spectromety, 4th. Ed. Allured Publishing Corp. Carol Stream, Illinois, USA.

Ahmad, M. M.; Rehman, S.; Iqbal, Z.; Anjum, F.M. and Sultan, J. I. (2006): Genetic variability to essential oil composition in four citrus fruit species. Pak. J. Bot., 38:319-324.

Asekun, O. T.; Grierson, D. S. and Afolayan, A. J. (2007): Effects of drying methods on the quality and quantity of the essential oil of *Mentha longifolia* L. subsp. Capensis. Food Chem., 101: 995-998.

Aslan, I.; Ozbek, H. ; Calmasur, O. and Sahin, F. (2004): Toxicity of essential vapours to two greenhouse pests, *Tetranychus urticae* Koch and *Bemisia tabaci* Genn. Indust. Crops Prod., 19: 167–173.

Attia, S. A. (2010): Ecological studies of scale insects infesting mango and guava trees and their control in Qaliobiya Governorate . Ph. D. Thesis Fac. Science Ain Sams Universty.

Attia, S. A.; El-Sayid, M. I. and Abd-ELAziz, S. Y. (2018): Abundance and generation determination of the mango shield scale *Milviscutulus mangiferae* (Green) (Coccidae: Homoptera) an invasive coccid infest mango orchards at Qaliobiya Gevernorate. J. Plant Prot. and Path., Mansoura Univ., 9 (3): 209-213.

Cavalcanti, E. S. B.; Morais, S. M.; Lima , M. A. A. and Santana, E. W. P. (2004): Larvicidal activity of essential oils from Brazilian plants against *Aedes aegypti* L. Mem. Inst. Oswaldo Cruz, 99:541–544.

El-Badawy, S. S. (2014): Interpretation phenomenon of infestation susceptibility of some mango cultivars with mealybug *Icerya seychellarum* (Westwood) (Hemiptera: Monophlebidae). Egypt. J. Agric. Res., 92 (4): 1235-1252.

El-Badawy, S.S. (2015): Insecticidal and repllenet activities of citrus peel oils against mealybug *Icerya seychellarum* (Westwood). Egypt. J. Agric. Res., 93 (3): 791-808.

El- Minshawy, A.M.; El-Sawaf, S.K.; Hammad, S.M. and Donia, A. (1972): The Biology of *Hemiberlesia lataniae* (Signoret) in Alexandria district. Bull. Ent. Soc. Egypt, Economic, Ser., 65: 461- 467.

Elhag, E. A. (2000): Deterrent effect of some botanical products on oviposition of the cowpea bruchide *Callosobruchus maculatus* (F.) (Coleoptera:Bruchid). Intern. J. Pest Manag., 46 (2): 109-113.

Ghose, S.K. and Ghosh, A.B. (1990): Morphology of different instars of some mealybugs (Pseudococcidae, Homoptera). Environment and Ecology, 8(1): 137-142.

Hollingsworth, R. G. (2005): Limonene, a citrus extract for control of mealybugs and scale insects. Ecotoxicol., 98:772–779.

Ibrahim, M. A.; Kainulainen, P.; Aflatuni, A.; Tilikkala, K. and Holopainen, J. K.(2001): Insecticidal, repellent, antimicrobial activity and phytotoxicity of essential oils: With special reference to limonene and its suitability for control of insect pests. Agric. Food Sci. Finland, 10: 243-259.

Karamaouna, F.; Kimbaris, A.; Michaelakis, A.; Papachristos, D.; Polissiou, M.; Papatsakona, P. and Tsora, E. (2013): Insecticidal activity of plant essential oils against the vine mealybug, *Planococcus ficus*. J. Insect Sci., 13:1-13.

Mansour, S. A.; El-Sharkawy, A. Z. and Amina, R.A. (2004): Botanical biocides .12. Mosquitocidal activity of citrus peel oils with respect to their limonene content. Egyptian J. Nat. Toxins, 1: 111-134.

Mohan, S. and Fields, P.G. (2002): A simple technique to assess compounds that are repellent or attractive to stored product insects. J. Stored Products Res., 38:23-31.

Pumnuan . J.; Khurnpoon, L. and Insung, A. (2015): Effects of insecticidal essential oil fumigations on physiological changes in cut *Dendrobium* Sonia orchid flower Songklanakarin. J. Sci. Technol., 37 (5): 523-531.

Richard E. L. (2005): Biotechnology of fruit and nut crops. Biotechnology in Agriculture Series, 29: 1-731.

Sivakumar, D. and Yahia, E. (2011): Maintaining mango (*Mangifera indica* L.) fruit quality during the export chain. Food Research International, 44 (5) :1254-1263.

Ware, G. W. (2000): The pesticide book, 5th ed. Thomson Publications, Fresno, California. 415 pp.