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Preparation and evaluation of botanical and mineral oils EC formulations against the black (*Parlatoria ziziphi*) on navel orange trees (*Citrus sinensis*)

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ABSTRACT

This study aimed to develop, and assess the insecticidal effectiveness of mineral and botanical oils as alternatives in environmentally friendly integrated pest management. These oils were evaluated as bio-insecticides for controlling the black parlatoria scale insect, *Parlatoria ziziphi* infesting navel oranges trees under field conditions. To assess the physical and chemical properties of three tested (orange, citronella, and mineral) oils they were prepared as emulsifiable concentrate (EC) formulations and combined at half the concentration and half application rate. The physical parameters tested include accelerated storage, surface tension, viscosity, pH, acidity or alkalinity, flash point, density, specific gravity, and the active ingredient content both before and after storage. The population reduction treatment was carried out on August 2023 and 2024 in the garden of the Agricultural Research Center in Giza Governorate, Egypt *P. ziziphi* showed insecticidal responses across various tested treatment stages, with the nymph stage being the most susceptible, followed by adult females and gravid individuals. All treatments effectively increased the reduction of nymphs, adult females, and gravid stages of over a four-week period in each season. During the first and second seasons, the average reduction rates with unmixed oils ranged between 60.9% and 66%. Based on the aforementioned results, the use of mixed oil formulations resulted in higher reduction rates, ranging from 75.1% to 78.1%. Botanical and mineral oils were valuable environmentally conscious trend for *(P. ziziphi)* on Navel oranges trees (*Citrus sinensis),* aligning perfectly with the principle of sustainable of integrated pest management.

Keywords: Green Emulsifiable concentrate, oils, *Parlatoria ziziphi*, Insecticidal efficiency and Navel oranges.

INTRODUCTION

In marketing year (MY) 2023/24, was estimated 2.0 million metric tons (MMT) up from 1.6 MMT of citrus produced in 2023, where the value of it reached more than USD 920 million primarily of the Valencia and Navel varieties oranges (FAO, 2023). Egypt ranked among the top ten countries in the world for local consumption or export important economic fruit crops, as the third highest producer of citrus among countries in the Mediterranean basin, mainly various types of oranges (Abu Habib *et al.,* 2020 and FAO, 2023). The black parlatoria scale, *Parlatoria ziziphi* (Lucas 1853), (Hemiptera : Diaspididae) is the most destructive pest in tropical and subtropical regions of the world on numerous hosts especially, attacking citrus trees (Assouguem *et al.,* 2021, Awadalla *et al* ., 2021; Salem *et al.,* 2024). *P. ziziphi* damages citrus trees by feeding on the plant sap through leaves and fruits, causing defoliation and drying up the young twigs. These insects have the ability to transmit plant pathogens, inject toxins, affecting the general physiological processes of the trees, Thus, the effect on the quality and quantity, as well as affects the fruit market value and their export potential consequently, reduce productivity (Faskha *et al.,* 2021 and Bragard *et al.*, 2023). The excessive use of traditional insecticides to control black parlatoria scale has resulted as insect resistance with also, the environmental risks moreover, had negative effects on human health and non-target pest in recent decades, rendering them ineffective and dangerous (Dassanayake *et al.*, 2021). Pesticides were formulated for increasing effectiveness in the field and enhancing each of the safety features and handling qualities. Emulsifiable concentrates (EC) (the conventional solvent-based insecticides) consist of an oil-soluble active ingredient dissolved in an appropriate oil-based solvent, emulsifying agent was added and mixed with water, which are subsequently sprayed. In recent trend, increasingly need for safer and more appropriate pesticide formulations instead of the conventional solvent-based insecticides (EC), which cause various problems for plants and the environment (Salem *et al.,* 2016). Researchers are currently interested in finding alternative materials are eco-friendly, economic, target-specific and biodegradable as bio-insecticides, (ecological pesticides) "Green pesticides" natural and bio-safe pesticides and Plant-based natural products have insecticidal, , antifeedant, and repellent activities against scale insects and mealy-bugs. Where the chemical composition of these naturally oils lipophilic nature can enter into insect and cause biochemical dysfunction and mortality (Mwanauta *et al.*, 2021; Zhou *et al.*, 2022). In integrated pest management (IPM) program, botanical pesticides have garnered a lot of attention due to their superior insect control capabilities and environmental safety such as botanical oils and mineral oils are considered one of the safest methods and show a broad spectrum of activity against pest insects (Jyotsna *et al.,* 2024; Qasim *et al.,* 2024). Mineral oils are regarded as one of the safest strategies for controlling pests, specifically scale insects and mealybugs, mineral oil "Super Misrona" play an important part in IPM programs for many pests due to their safety on the environment, wildlife, domestic animals and human health with reducing the population of soft scale insects to a satisfactory level. Avila *et al.,* (2022). Aim of the work was to develop and evaluate the physicochemical properties and insecticidal efficacy of botanical and mineral oil-based EC formulations against *P. ziziphi* under field conditions to ensure their effectiveness and sustainability with, contributing to integrated pest management strategies for citrus production.

MATERIAL AND METHODS

1. insecticides used: -
1.1. Orange oil 24 % EC application rate 50 cm / 100 L
1.2. Mineral oil 95 % EC application rate 1.5 L / 100 L

All from the daily sample received by Central agricultural pesticide laboratory

2. Oils Utilized: -
2.1. Orange oil (*Citrus aurantium*) (*Citrus sinensis*) CAS Number 8028-48-6 supplied by Borg Al Arab for Industry.
2.2. Citronella oil (*Cymbopogon nardus*) CAS number 8000-29-1 purchased by Borg Al Arab for Industry.
2.3. Mineral oil Mineral oil CAS number 8042-47-5 from Alexandria Mineral Oils Company (AMOC).
3. Surfactant applied: -
3.1. Rhodia GERONOL FF/4: Blended emulsifier (calcium dodecyl benzene sulphonate in 2-methylpropan-1-ol and tristryphenol ethoxylates), HLB number 9.6 supplied by Rhodia-Home, Personal Care &Industrial Ingredients, Milano, Italy
3.2. Solvay ALKAMULS® RC Surfactant: nonionic surfactant Castor oil ethoxylate with HLB number 10.5, CAS number: 61791-12-6. Purchased from Solvay S.A.
3.3. Solvay GERONOL® MS Surfactant: mixture of anionic and nonionic surfactant ethoxylated oleil amine, dodecyl benzene sulphonic salt, provided by Solvay S.A.
3.4. Solvay RHODACAL® 60/BE Surfactant: with HLB number 8.3, mixture base on calcium dodecylbenzene sulphonate, Sourced from Solvay S.A.
4. Solvent used: -
4.1. AMESOLV CME: Methyl oleate – Vegetable oil fatty acid methyl ester, CAS number: 112-62-9.
4.2. Soybean oil methyl ester epoxidized soybean oil CAS number 8013-07-8, used as a stabilizer, sticker, and solvent.
4.3. Castor oil, CAS number 8001-79-4.

Theses all green solvents provided by Oleo Misr Sadat city- Egypt

5. Equipment
5.1. A magnetic stirrer (Terrey Pines Scientific) from the USA
5.2. 4-digit sensitive balance (RADWAG) with a maximum load of 220 g was used to prepare the oil emulsifiable concentrate formulations.

6. Preparation of emulsifiable concentrate (EC) formulation for three oils:

The three tested oils were formulated by mixing each oil with a mixture of nonionic and anionic surfactants in different ratios to create a stable emulsion based on their Hydrophilic-Lipophilic Balance (HLB) values for optimal compatibility. After all, add natural solvent derivative of vegetable oils, was then incorporated, with the choice of solvent playing a crucial role in determining the stability and effectiveness of the formulation. The final result was a clear, stable, homogeneous liquid free from sediment or suspended particles (Ruidas *et al.,* 2022).

7. Storage stability at elevated temperature (After storage at 54 ±2 °C for 14 days)

Each emulsifiable concentrate (EC) formulation was transferred into a 100 ml dark glass bottle with a sealed cap and stored in an oven set at 54 ± 2°C for 14 days. This procedure was performed in accordance with CIPAC MT 46.3 (1999).

After the storage period completed, several essential parameters were assessed both before and after storage for all emulsifiable concentrate (EC) formulations. This included examining their physical properties as well as those of their spray solutions to evaluate the stability of the formulations under extreme temperature conditions. The evaluations covered active ingredient content, viscosity, surface tension, flash point, refractive index, density, foam generation, emulsion stability, and pH levels.

8. Physicochemical properties of oils emulsifiable concentrate (EC) formulation

8.1. Viscosity: Viscosity of the prepared formulation was determined using a ”Brookfield DV II+ PRO” digital Viscometer (Brookfield, USA), equipped with ULA rotational adaptor. The measurement was conducted at a controlled temperature of 25°C, maintained with the help of a water bath (Model: TC-502, USA). Viscosity values, expressed in centipoise (cP), were recorded in accordance with the ASTM D2196-20 (2020) guidelines.

8.2. Surface tension: The surface tension of the formulation was measured using Force tensiomate sigma 700 USA through the Whilmy plate method. For accurate measurement, the sample must be clean, homogenous and free of bubbles and possess a stable surface, and the dial reading surface tension is (dyne/cm) recorded from the tensiometer ASTM D1331-20 (2020).

8.3. Flash point: The lowest temperature at which a volatile substance vaporizes and becomes a flammable gas is determined through testing. For this analysis, 2 ml of the sample was transferred using a glass syringe into the Kolchler closed cup flash tester, and the flash point was measured in accordance with ASTM D3828-21 (2021) specifications.

8.4. Density and specific gravity: The measurement was conducted using a Rudolph Densitometer 2910 (USA) equipped with an autosampler took 2ml of the sample automatically provided a reading based on the test procedure outlined in ASTM D- 4052 (2022).

8.5. Refractive Index: The refractive index is an optical parameter used to assess the purity of the sample by measuring the transmittance of a light beam using a DR-A1 ATAGO digital refractometer (Japan) by applying drops of the EC formulation onto the lower prism surface at a temperature of 25°C. The refractive index is then read from the scale, according to the specifications outlined in ASTM D - 1218 (2021).

9. Physicochemical properties of oils emulsifiable concentrate (EC) formulation and spray solution

9.1. Emulsion stability and foam test

To assess the stability of the emulsion, a 100 ml measuring cylinder was utilized, containing 95 ml of WHO hard water prepared in accordance with (WHO, 1979). This hard water was formulated by dissolving 0.304 g of anhydrous calcium chloride and 0.139 g of magnesium chloride hexahydrate in distilled water, then using diluting the solution to one liter to achieve a water hardness of 342 ppm. Then using measuring cylinders (100 ml) were filled to 95 ml mark with WHO hard water at 30 ± 1 ºC, with pouring 5 ml of the test EC formulation on to the surface of the water. The cylinder was inverted 30 times to ensure mixing. After 5 minutes, the foam volume generated on the emulsion's surface was measured according to CIPAC MT 47.1 (1995). Following 30 minutes, the volume of any free oil or cream layer present in the cylinder was also recorded. For compliance with CIPAC MT 36.3 (2000), the foam volume should not exceed 5 ml, and the emulsion stability test must reveal no more than 2 ml of free oil, cream, or solid residues.

9.2. pH test

The test was conducted following CIPAC MT 75.3 (1995). A one-gram sample of the tested formulation was weighed and placed into a measuring cylinder containing 100 ml of distilled water. The mixture was shaken vigorously for one minute and then left to settle. The pH of the resulting supernatant liquid was determined using a Jenway pH meter 3510, UK, equipped with a HANNA pH electrode.

9.3. Electrical Conductivity

The conductivity of the spray solutions was assessed using the Conductivity and Salinity Meter Thermo Orion Model 115A+, USA. Measurements were conducted at a temperature of 25°C ± 2, following the procedure outlined in CIPAC MT 32 (1995). For each measurement, one gram of the sample was dissolved in 100 ml of distilled water in a beaker, ensuring thorough mixing by stirring. The probe was then immersed in the solution and left for one minute to allow the conductivity reading to stabilize.

10. Active ingredient content of tested oils EC formulations

The chemical stability for active ingredient content was measured before and after storage for orange oil formulation by HPLC, citronella oil formulation by GLC and mineral oil by CIPAC MT 57 (2007).

- The orange oil content was determined using high-performance liquid chromatography (HPLC). The analysis was performed on an Agilent Technologies 1260 Infinity system equipped with a UV detector and an Eslips plus C18 column. D-limonene, the primary component of the oil, was eluted using a mixture of acetonitrile and methanol (70:30) at a flow rate of 1 ml/min, a temperature of 30ºC, a detection wavelength of 235 nm, and a total elution time of 10 minutes (Braddock, 1999; Zheng *et al.,* 2023)
- The active ingredient content in the citronella oil 24% EC was analyzed using gas-liquid chromatography (Agilent 7890 B) with an auto sampler and a flame ionization detector (FID) set at 250ºC. The analysis was performed on a capillary column (HP-50+), utilizing nitrogen as the carrier gas with a constant flow rate of 4 ml/min. The oven temperature was initially set at 60ºC and maintained for 3 minutes before being incrementally increased at a rate of 5ºC/min until reaching 240ºC. The total runtime for the procedure was 44 minutes, with an injection volume of 1 µl (Kang *et al.,* 2023)
- The CIPAC MT 57 (2007) method is a standardized procedure used to assess the quality and purity of oil-based formulations. It measures the percentage of oil that remains unreacted when mixed with sulfuric acid. This is done using a Babcock bottle, into which 5 mL of oil is added. The process involves introducing 20 ml of concentrated sulfuric acid in four equal portions. After each addition, the mixture is carefully observed, and the bottle is securely clamped in an upright position at 60ºC for 1 hour. Observations are made every 10 minutes for approximately 10 seconds. Once this step is completed, the bottle and its contents are centrifuged for 5 minutes at 1250 rpm, ensuring the volume stabilizes. The unsulfonated portion of the oil is then isolated and analyzed based on the determined formula.

$$volume\ of\ unslphonated\ residue = \frac{\text{vol. of oil } \times \text{specific gravity } \times 100}{\text{Mass of oil taken}}$$

11. Evaluation of emulsifiable concentrate (EC)-formulated oils against the black parlatoria scale (*Parlatoria ziziphi*):

The experiment was conducted outdoors on approximately 63 navel orange trees, grafted onto bitter orange rootstock, in the garden of the Agricultural Research Center in Giza Governorate, Egypt. These trees, aged 13 to 15 years, were heavily infested with black parlatoria scale insects (*P. ziziphi*) on their leaves. It included seven treatments, with six subjected to tested insecticides applied at recommended rates, while the seventh treatment was left untreated as a control.

Seven treatments were applied on three replicates for one treatment, with three trees per replicate making nine orange trees per treatment, five leaves for every tree, approximately having similar size, shape, height, and vigor. The tested insecticides were applied as complete coverage. Spraying was carried out on August 2023 and 2024. When the temperature and relative humidity were 30-33 °C and 59-92 RH, respectively. Forty-five leaves from each treatment including control were collected immediately before spraying and after 1, 2, 3 and 4 weeks (for *P. ziziphi*) of application to the laboratory for counting of the insect. The reduction of population density percentage *P. ziziphi* nymphs, adult and gravid females were estimated according (Henderson and Tilton, 1955).

$$\%Reduction = 1 - \frac{(Ta \times Cb)}{(Tb \times Ca)} \times 100$$

Where Ta and Tb are number of insects in the treatment after and before application, respectively; Cb and Ca are number of insects in the control before and after application, respectively.

The treatments used were as follows.

- Treatment 1: Orange oil = (24 % EC) 10 cm / 20 L (F1)
- Treatment 2: Citronella oil = (24 % EC) 10 cm / 20 L (F2)
- Treatment 3: Mineral oil = (95 % EC) 300 cm / 20 L (F3)
- Treatment 4: Orange oil 12 % + Citronella oil 12 %= (24 % EC) 10 cm / 20 L (F4)
- Treatment 5: Orange oil 12 % + Mineral oil 12 %= (24 % EC) 155cm / 20 L (F5)
- Treatment 6: Citronella oil 12 % + Mineral oil 12 %= (24 % EC) 155cm / 20 L (F6)
- Treatment 7: Control

RESULTS

1-Physicochemical properties of prepared oils as emulsifiable concentrate (EC) formulation compared with registered oils at different storage conditions:

Data in (Table 1) represented the physicochemical properties of tested oils formulated as emulsifiable concentrate (EC) formulation at different storage conditions. These data revealed that storage temperature recorded bounded impact on all tested physicochemical properties of the evaluated (EC) formulation. Viscosity of tested formulations slightly increased due to the effect of storage condition temperature and recorded its highest values for (F3) formulation as (14.293 and 16.717 cp) for initial and hot storage sample respectively, while the lowest recorded viscosity was (2.057 and 2.260 cp) for (F1) formulation for initial and hot storage sample, respectively.

Density and refractive index of any pesticide used as measuring tools to its spontaneity on mixing with irrigation water as well as its concentration as related to optical transparency of emulsifiable concentrate (EC) formulation. The obtained results declared that, tested formulations recorded density from (0.8488–0.9157 g/cm^3) which is intelligible since all of prepared formulations are oils and less dense than water it affected formulation spontaneity. All the same, the highest density record (0.9157 g/cm^3) for (F6) formulation hot storage sample, while the lowest density recorded (0.8488 g/cm^3) for (F3) formulation initial sample. Refractive index on the other hand is slightly affected by storage temperature because of formulation components stability. However, the lowest recorded was (1.4654) for (F3) formulation initial sample and the highest recorded was (1.4849) for (F1) EC formulation initial sample. Flash point was an important concept in fire investigation and protection during pesticide storage since it was the lowest temperature at which fire risk may exist with the stored liquid formulation. The higher prepared formulation flash point is the more safety to store even at harsh climatic conditions. However, the lowest recorded flash point was (37 ℃) for (F1) formulation sample while the highest recorded flash point was (> 60 ◦C) for (F3) sample. It was also clear that, storage temperature has no noticed effect on flash point recorded of all tested (EC) samples.

From the table also it is cleared that, surface tension decreased as a result of storage temperature for all tested samples and this usually intelligible due to the decreased in cohesive force with increasing in molecular thermal activity. However, the result showed that, the highest decrease observed from (27.404 dyne/cm) to (24.542 dyne/cm) for (F2) formulation initial and hot storage samples respectively. While the lowest depression noticed from (29.137 dyne/cm) to (29.124 dyne/cm) for (F4) formulation initial and hot storage samples on sequence. On the other hand, all sample had acidic behavior for all stored samples and the acidic strength of the tested samples increase as a result of storage temperature this may be due to the fact that, as the temperature increase pH of the sample decrease. The highest increase in acidity percent of tested sample was (0.158) for (F5) formulation its record was (0.316 and 0.474) for initial and hot stored sample, respectively.

Table 1. Physicochemical properties of emulsifiable concentrate (EC) formulated oils compared with registered (EC) oils at different storage temperature.

Formulation	Storage state	Physicochemical properties					
		Viscosity (cp)	Density (gm/cm³)	Flash point(℃)	Refractive index	Surface tension (dyne/ cm)	Acidity (%)
Orange oil 24 % EC (F1)	Initial	2.057	0.8924	37.0	1.4849	24.217	0.388
	Hot	2.260	0.8937	37.0	1.4842	24.166	0.404
Citronella 24 % EC (F2)	Initial	5.050	0.9075	41.0	1.4831	27.404	0.134
	Hot	5.410	0.9116	41.0	1.4829	24.542	0.162
Mineral oil 95% EC (F3)	Initial	14.293	0.8488	> 60	1.4654	28.951	0.076
	Hot	16.717	0.8504	> 60	1.4652	28.809	0.085
Orange oil + Citronella oil 24% EC (F4)	Initial	11.004	0.9116	38.0	1.4749	29.137	0.282
	Hot	13.205	0.9126	38.0	1.4734	29.124	0.302
Mineral + Orange oil 24% EC (F5)	Initial	5.442	0.9037	42.0	1.4759	28.649	0.316
	Hot	6.709	0.9043	42.0	1.4748	28.597	0.474
Mineral + Citronella oil 24% EC (F6)	Initial	10.796	0.9125	43.0	1.4738	29.110	0.291
	Hot	12.155	0.9157	43.0	1.4726	29.048	0.416

Data in (Table 2) showed the physicochemical properties of spray solution for prepared formulation. It was well known that; surface tension of agrochemical is one of the important properties of liquid that cause them spread and penetrate through surface of plants and soil. The highest surface tension record was (29.046 dyne/cm) for hot storage sample of (F6) while the lowest record was (27.341dyne/cm) for (F3) initial sample. From this table it was clear that, electrical conductivity of hot storage sample showed higher records as compared with initial samples record for all tested prepared formulations. The highest record of electrical conductivity was (779 µs) for hot stored sample of (F6). While the lowest record was (582 µs) of initial stored sample of (F1). All tested formulations had acidic nature; the highest pH values were (6.57 and 6.50) for (F3) hot stored and initial sample respectively. While the lowest pH records were (3.99 and 3.89) for (F1) initial and hot storage sample on sequence and this, also clear from records of formulation acidity in (Table 1). All tested formulations showed no creamy separation or sediments, as well as formed foam not exceed 2 ml, which improve, the physical stability of oil prepared formulations.

Table 2. Physicochemical properties of spray solution of (EC) formulated oils compared with those of registered (EC) oils.

Formulation	Storage state	Physicochemical properties of spray solution				
		Surface tension (dyne/ cm)	Conductivity (µs)	pH	Emulsion stability (ml)	Foam (ml)
Orange oil 24 % EC (F1)	Initial	28.015	582	3.99	-----	-----
	Hot	28.052	598	3.89	-----	-----
Citronella 24 % EC (F2)	Initial	27.725	694	4.58	-----	1.5
	Hot	28.028	731	4.60	-----	2
Mineral oil 95% EC (F3)	Initial	27.341	737	6.50	-----	1.5
	Hot	27.497	757	6.57	-----	2
Orange oil + Citronella oil 24% EC (F4)	Initial	28.668	693	4.41	-----	-----
	Hot	28.847	674	4.44	-----	-----
Mineral + Orange oil 24% EC (F5)	Initial	28.765	656	4.49	-----	-----
	Hot	28.959	677	4.50	-----	-----
Mineral + Citronella oil 24% EC (F6)	Initial	28.319	732	5.35	-----	2
	Hot	29.046	779	4.35	-----	2

The data in (Table 3) showed the active ingredient content of the prepared formulations before and after accelerated storage (54°C for 14 days) as detected by using high-performance liquid chromatography (HPLC) for orange oil, gas-liquid chromatography) with a flame ionization detector (FID) for citronella oil and measure un-sulphonated residue of mineral oil. Which were in the acceptable range of active ingredient content. Which showed that Mineral oil (mineral oil 95 % EC) (F3) = ±2.5 percentage from the percentage of active ingredient content, and the tolerance of others formulations are = ±6 percentage from the percentage of active ingredient content.

Table 3. The chemical stability of active ingredient content for the oils and their mixture emulsifiable concentrate formulations

Formulation code	Storage condition	Percentages of active ingredient content
Orange oil 24 % EC (F1)	Before	25.10
	After	24.26
Citronella 24 % EC (F2)	Before	24.23
	After	24.65
Mineral oil 95% EC (F3)	Before	94.20
	After	93.87
Orange oil + Citronella oil 24% EC (F4)	Before	12.12+12.16
	After	12.03+12.24
Mineral + Orange oil 24% EC (F5)	Before	12.62+12.10
	After	12.43+12.02
Mineral + Citronella oil 24% EC (F6)	Before	12.62+12.15
	After	12.53+12.20

2- Reduction percentages of the black parlatoria nymphs applying with oils formulations

Data illustrated in (Table 4) the reduction percentage in nymph stage of *P. ziziphi* with six treatments as mentioned in tables, material, and methods. The results achieved over four weeks of treatment targeting the nymph stage of *P. ziziphi.* After the first week, all oil formulations showed nearly identical reduction percentages, while in the second week, mixed oil formulations (F4, F5, and F6) demonstrated reduction levels comparable to those of the registered formulations (F1 and F3). However, by the third week, the mixed oil formulations (F4, F5, and F6) outperformed the registered oils, achieving reduction percentages of 92.0 %, 90.3%, and 92.2 for (F4, F5 and F6) respectively, despite their reduced oil concentrations half or less in some cases, such as mineral oil, this trend of results for the second season also. In the second season, there was change in the outcomes regarding the reduction percentage and the competition between registered oil formulations and blended oils persisted, even with the decrease in oil percentages in the new blended preparations. The findings highlighted the effectiveness of blended oils in tackling this stage, with the highest reduction rates observed during the second, third, and fourth weeks, similar to the results from the first season. General mean reduction on nymphal stage of *P. ziziphi* recorded the lowest by treatment (F2) (65.0% and 63.8%) and the highest by treatment (F4) (81.1% and 79.8%) in both year (2023 and 2024) consecutively (Table 4).

Table 4. Reduction percentages of the black parlatoria nymphs applying oils formulations on Navel orange *C. sinensis* at Giza in both 2023 and 2024.

		Reduction percentages of *P ziziphi* nymph					
Treatments'		F1	F2	F3	F4	F5	F6
First season	**One week**	68.3	62.2	61.9	65.5	64.7	65.7
	Two Weeks	79.7	73.8	78.6	90.1	87.3	88.1
	Three Weeks	74.5	70.6	72.2	92.0	90.3	92.2
	Four Weeks	61.5	53.2	58.4	76.6	78.6	77.7
	Mean	71.0	65.0	67.8	81.1	80.2	80.9
Second eason	**One week**	66.9	60.9	60.7	64.2	63.5	64.3
	Two Weeks	78.5	72.6	77.4	88.7	86.1	86.7
	Three Weeks	73.4	69.5	71.2	90.7	89.1	90.8
	Four Weeks	60.5	52.3	57.4	75.4	77.4	76.4
	Mean	69.8	63.8	66.7	79.8	79.0	79.6

Orange oil 24 % EC (F1), Citronella 24 % EC (F2), Mineral oil 95% EC (F3), Orange oil + Citronella oil 24% EC (F4), Mineral + Orange oil 24% EC (F5), Mineral + Citronella oil 24% EC (F6).

Data in (Table 5) reported the effect of the six tested formulations on the reduction percentage of *P. ziziphi* in its adult stage. During the first week, all tested formulations whether newly developed oil mixtures or registered oils exhibited identical reduction percent. However, starting in the second week, the oil mixture formulations began to surpass the registered oil formulations in performance. This trend persisted, with the newly formulated oil mixtures maintaining higher reduction rates throughout the third and fourth weeks. A similar pattern was observed during the trial period in the second season, where the oil mixtures consistently outperformed the registered oils. The overall mean reduction in the adult female stage of *P. ziziphi* was lowest with treatment F2 (60.5% and 59.1%) and highest with treatment F4 (81.1% and 79.5%) across both 2023 and 2024 seasons successively.

Table (6), offers a different perspective on the comparison between mixed oil formulations and standard oil formulations, highlighting their effects on the population of gravid female black scale insects recorded during the 2023 and 2024 seasons. This table reveals that, during the first week, there was no distinct variation in the percentage reduction of gravid females across treatments with the six formulations, whether use individually or blended.

Table 5. Reduction percentage of the black *P. ziziphi* adult famales applying oils formulations on navel orange *C. sinensis* at Giza in both 2023 and 2024.

	Treatments'	F1	F2	F3	F4	F5	F6
		Reduction percentages of the black *P. ziziphi* adult females					
First season	One week	66.3	59.5	59.4	62.7	61.6	63.4
	Two Weeks	77.0	71.1	75.7	86.9	85.0	85.6
	Three Weeks	65.7	58.3	60.6	90.6	87.9	89.1
	Four Weeks	55.8	52.9	52.4	84.3	81.8	82.1
	Mean	66.2	60.5	62.0	81.1	79.1	80.1
Second season	One week	64.6	57.9	57.9	61.1	60.2	61.8
	Two Weeks	75.4	69.6	74.2	85.1	83.5	83.9
	Three Weeks	64.4	57.1	59.4	88.9	86.4	87.4
	Four Weeks	54.7	51.9	51.3	82.8	80.4	80.7
	Mean	64.8	59.1	60.7	79.5	77.6	78.5

Orange oil 24 % EC (F1), Citronella 24 % EC (F2), Mineral oil 95% EC (F3), Orange oil + Citronella oil 24% EC (F4), Mineral + Orange oil 24% EC (F5), Mineral + Citronella oil 24% EC (F6).

However, oil formulation F1 stood out as slightly more effective, achieving a reduction rate of 66.5%. During the second week, change was observed in the number of gravid females, as the oil mixture formulations started demonstrating their pronounced effects. Formulation F4 showed the highest impact, achieving a reduction rate of 84%. This was followed by formulation F5 with a reduction of 83.1% and formulation F6 that recorded a reduction rate of 82.8%. Over time, the performance of the registered oil formulation declined, hitting its lowest point by the fourth week, during which sample formulation F3 showed the minimum reduction rate of 36.6%. On the other hand, the oil mixture formulations demonstrated competitive reduction rates, with sample formulation F4 achieving the highest rate at 75% in the same timeframe.

During the second season, there was little change in the similarity of reduction rates for the number of gravid females during the first week. However, a decline in these rates was observed for the registered oil formulations, reaching their lowest level in the fourth week, particularly for formulation F3 at 33.3%. At the same time, the new mixture formulations showed an increase in reduction rates, achieving their highest value in the second week with sample formulation F4, and recording 81.6%. Overall, registered formulation F3 showed the lowest reduction rates across both seasons of the experiment, with values of 57.2% and 55.3%, respectively. In contrast, mixed oil formulation F4 demonstrated the highest efficiency, achieving the greatest reduction in the number of gravid females during both seasons, with values of 72.2% and 70.4%.

Table 6. Reduction percentages of the black *P.* gravid applying with some oils formulations on navel orange *C. sinensis* at Giza in both 2023 and 2024.

	Treatments'	F1	F2	F3	F4	F5	F6
		Reduction percentages gravid of the black *P. ziziphi*					
First season	One week	66.5	58.5	57.5	61.7	60.2	62.2
	Two Weeks	76.5	70.0	69.6	84.0	83.1	82.8
	Three Weeks	62.9	63.9	64.9	67.9	65.9	66.9
	Four Weeks	37.6	40.4	36.6	75.0	71.5	71.3
	Mean	60.9	58.2	57.2	72.2	70.2	70.8
Second season	One week	64.1	56.3	55.4	59.4	58.2	59.9
	Two Weeks	74.4	67.9	67.6	81.6	81.1	80.4
	Three Weeks	62.9	63.9	64.9	67.9	65.9	66.9
	Four Weeks	35.3	38.3	33.4	72.7	69.6	69.2
	Mean	59.2	56.6	55.3	70.4	68.7	69.1

Orange oil 24 % EC (F1), Citronella 24 % EC (F2), Mineral oil 95% EC(F3), Orange oil + Citronella oil 24% EC (F4), Mineral + Orange oil 24% EC (F5), Mineral + Citronella oil 24% EC (F6).

Table (7) presents the percentage reductions in the population of black scale insects (*P. ziziphi*) across all their developmental stages nymphs, adults, and gravid females during the two experimental seasons of 2023 and 2024. This table clearly demonstrates that throughout the two experimental seasons, aimed at comparing registered oil formulations with novel oil mixtures featuring reduced oil concentrations, the new formulations consistently outperformed in reducing black scale insect populations across all stages. Among these, formulation F4 proved to be the most effective, achieving average reduction ratios of 78.1% and 76.6% across the two seasons respectively. It was closely followed by formulation F6 which recorded reductions of 77.3% and 75.7%, and formulation F5, with results of 76.5% and 75.1% for the respective seasons. Conversely, formulation F2 exhibited the lowest reduction percentages, with averages of 61.2% and 59.8% across both seasons.

Table 7. Average reduction percentages in insect population of *P. ziziphi* as a result of applying some oil formulations on navel orange *C. sinensis* at Giza in both 2023 and 2024.

Treatments		F1	F2	F3	F4	F5	F6
First season	Nymph	71	65	67.8	81.1	80.2	80.9
	Adult	66.2	60.5	62.3	81.1	79.1	80.1
	Gravid	60.9	58.2	57.2	72.2	70.2	70.8
	Mean	66.0	61.2	62.4	78.1	76.5	77.3
second season	Nymph	69.8	63.8	66.7	79.8	79	79.6
	Adult	64.8	59.1	60.7	79.5	77.6	78.5
	Gravid	59.2	56.6	55.3	70.4	68.7	69.1
	Mean	64.6	59.8	60.9	76.6	75.1	75.7

Orange oil 24 % EC (F1) , Citronella 24 % EC (F2) , Mineral oil 95% EC(F3) , Orange oil + Citronella oil 24% EC (F4), Mineral + Orange oil 24% EC (F5), Mineral + Citronella oil 24% EC (F6).

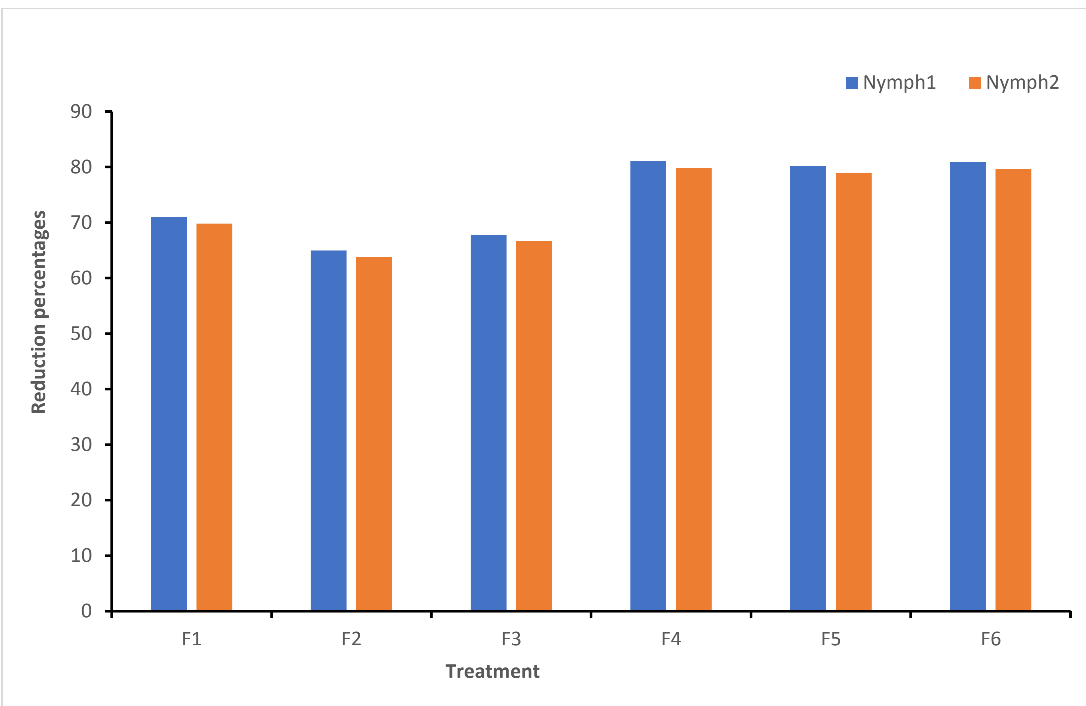
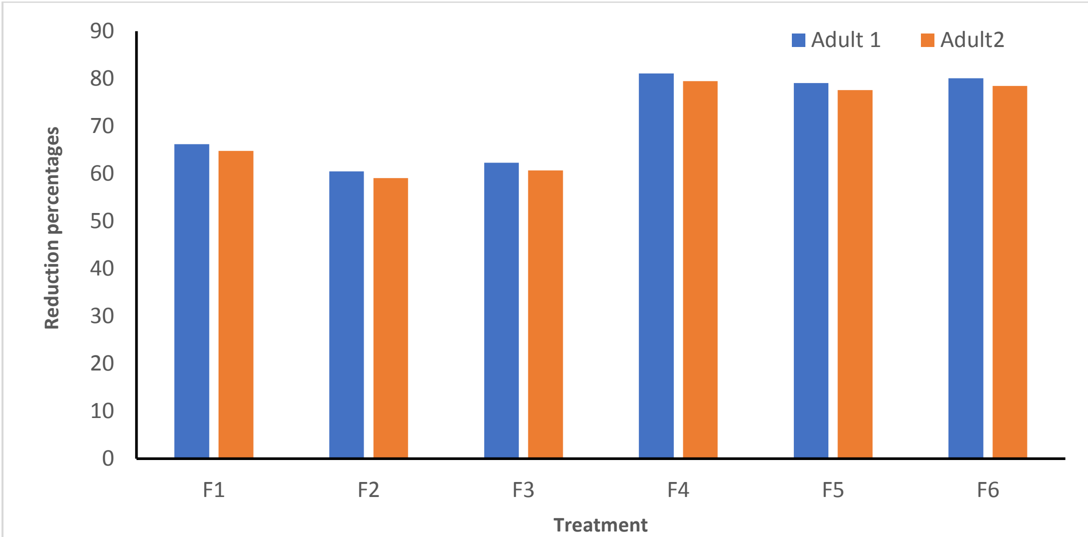


Fig. 1. Comparison between reduction percentages in the *P. ziziphi* nymph during 2023-2024.



Fig, 2. Comparison between reduction percentages in the *P. ziziphi* adult during 2023-2024**.**

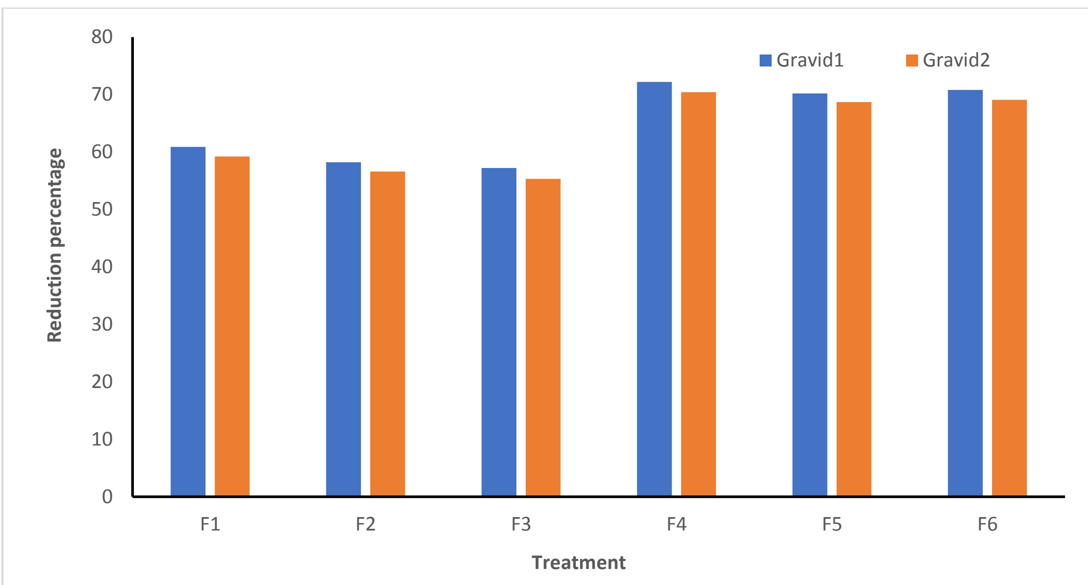


Fig 3. Comparison between reduction percentages in the gravid of *P. ziziphi* during 2023-2024.

DISCUSSION

This research paper focuses on the development of emulsifiable concentrate (EC) formulations using citronella oil, orange oil, and mineral oil, in addition combined at half the concentration and half application rate a blend of citronella oil with orange oil and mineral oil, and a combination of orange oil with mineral oil by. The formulations were analyzed for their thermal stability and physical compatibility as well as oil content, and the results were compared against commercial products Orange Oil 24% EC and Mineral Oil 95% EC.

Needs for alternative insecticides: Efforts in Egypt's agricultural sector have recently shifted toward minimizing pesticide usage in pest control due to the numerous adverse impacts these chemicals have. Pesticides harm agricultural crops and degrade soil fertility, making it less suitable for cultivating key crops. Furthermore, they contribute to pest resistance, diminishing the effectiveness of pest control measures over time. Their harmful effects extend to the climate, water resources, and, most notably, human health, highlighting the urgent need for alternative approaches. A widely advocated option is the application of oils, either mineral or vegetable, specifically to tackle insect pests. Several of these oils were already registered and actively utilized in pest control initiatives within the agricultural sector (Mohamed, 2023). Oils as bio pesticides: Botanical and mineral oils were considered among the safest methods for managing pests, particularly scale insects and mealybugs. Mineral oil, such as Super Misrona, is also highly valued in Integrated Pest Management (IPM) programs for various pests. Its environmental safety, along with minimal impact on wildlife, domestic animals, and human health, makes it an effective solution for controlling soft scale insect populations to an acceptable level (Avila *et al.,* 2022; Jyotsna *et al.,* 2024; Qasim *et al.,* 2024).

Physical stability: The new formulations exhibited a medium viscosity, a crucial factor in evaluating pesticide quality. Viscosity plays a significant role, as it affects the mixing, pumping, and spraying processes, all of which are essential for ensuring adequate coverage and improving overall effectiveness (Abdel-Aziz *et al.*, 2018). Flashpoint was a critical property of emulsifiable concentrates, particularly under the influence of climate change and elevated temperatures during pesticide storage. The findings indicated that the flashpoints of all formulations varied between 37 and above 60, which falls within the established safety thresholds by (WHO, 2011; El-Sayed and Mohammad, 2019). All the oil formulations tested, encompassing both prepared mixtures and registered products, demonstrated acidic properties. The spray solutions had pH values ranging from 3.59 to 6.60, which help safeguard formulation components against degradation. These results were consistent with (WHO, 2016).

The surface tension measurements of the tested formulations and spray solutions for all preparations were found to be below 30 dynes/cm. These low values contribute to increased thermal stability and improve the physical compatibility of oil mixtures within the new formulations. The storage temperature leads to a slight increase in viscosity, which can be attributed to the self-assembly of surfactants into aggregates above their (CMC). Alternatively, it may result from changes in the molecular chain's structure at the sample surface, which reduces electrostatic forces between particles. This crimping of the molecular chain diminishes its size, ultimately influencing both its density and viscosity (Ghosh *et al*., 2020; Yu *et al.,* 2020; Kadiri *et al.*, 2023; Man and Wu, 2024). From this table we showed that, surface tension of hot storage is higher than that of initial samples for all tested formulation this may be explained in terms of the presence of divalent hardness cations (Mg^{+2}, Ca^{+2}) (Dzienis. *et al.,* 2021) not to mention the self-assembly effect of surfactant (Ghosh *et al*., 2020; Kadiri *et al*., 2023; Man and Wu, 2024).

Field efficacy: The moderate viscosity of the formulation ensures stability on plant surfaces while maintaining smooth application during spraying. Moreover, its lower surface tension boosts the pesticide's ability to spread and cling to surfaces, resulting in enhanced coverage of the treated area. This improved distribution increases its efficiency in targeting and eliminating the specified insects. This aligns with the findings of (Dario *et al*., 2023). Conductivity was another factor that can affect the performance of the formulation; specifically, the high recorded conductivity (the presence of ions), when combined with surface tension and pH, influences both the size of spray droplets and their adhering force on the treated surface this finding complies with (Borra, 2018; Assuncao *et al.,* 2019). (FAO specifications, 2016) for emulsion stability in emulsifiable concentrates, (EC) formulations require the formulation to be a stable, homogeneous transparent liquid, free from visible suspended matter and sediment. Upon dilution with water, the resulting emulsion must also exhibit stability, with limited separation of solid or liquid phases not exceed 2ml as we find that no creamy or solid separation was noticed after dilution with water, which affects the stability of formulation and increases its effectiveness in the field.

The EC formulations, derived from botanical oils such as Laury 3% EC and Artemisia 5% EC, serve as alternatives to conventional insecticides for managing the scale insect *P. ziziphi.* These emulsifiable concentrates act by forming a thin, oil-based film around the insect's waxy coating, effectively suffocating them by blocking their oxygen intake (Salem *et al.*, 2016; Abdel-Aziz *et al.*, 2022). Orange oil operates through a physical mechanism, disrupting flying insects by deteriorating the protective layers of their wings. Additionally, it dries out the cuticles of mites and soft-bodied insects, leading to their swift demise. Citronella oil serves a dual purpose as an insect and animal repellent while also acting as a feeding deterrent. Moreover, it induces morphological alterations in the midgut and diminishes stored resources in the fat body, which can negatively influence insect reproduction and survival (Mahmud *et al.,* 2022). Causing phytotoxicity from vegetable or mineral oils is unwarranted, particularly because these oils are derived from natural sources and are used in formulated products at low concentrations. Additionally, their inclusion helps reduce production costs, making their use both practical and beneficial.

CONCLUSION

The research demonstrated that these formulations, applied at half the concentration and half the application rate, were effective against black parlatoria scale insect *(Parlatoria ziziphi*) on Navel oranges *(Citrus sinensis)* .This finding is particularly as it shows that used Vegetable oil fatty acid instead of organic solvent can still achieve satisfactory pest control. The effectiveness of these novel formulations at reduced rates highlights their potential for seamless integration into national Integrated Pest Management (IPM) programs. The successful preparation and evaluation of these formulations represent a significant step forward in developing greener and more sustainable agricultural practices, This not only lowers production costs for farmers but also reduce the pesticide residue on the fruit and in the ecosystem. Ultimately contributing to a healthier environment and safer food production.

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تجهيز وتقييم مستحضرات المركزات القابلة للأستحلاب للزيوت النباتية و المعدنية ضد الحشرة القشرية السوداء *(Parlatoria ziziphi)* علي أشجار *البرتقال ابو سرة* *(Citrus sinensis)* .

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تهدف هذه الدراسة إلى تطوير وتقييم الفعالية الحشرية للزيوت النباتية والزيوت المعدنية كبدائل مهمة في الأدارة المتكاملة للآفات الصديقة للبيئة. تم تقييم هذه الزيوت كمبيدات حشرية حيوية لمكافحة الحشرة القشرية السوداء *Parlatoria ziziphi)* والتي تصيب أشجارالبرتقال أبو سرة تحت الظروف الحقلية. لتقييم الخصائص الفيزيائية والكيميائية للزيوت محل الدراسة (زيت البرتقال، زيت السترونيلا والزيت المعدني) تم تحضيرها كمستحضرات مركزة قابلة للاستحلاب مع خلطها بمعدل نصف التركيز ونصف معدل التطبيق. تشمل الصفات الطبيعية التي تم اختبارها التخزين الحار، التوتر السطحي، اللزوجة، الرقم الهيدروجيني، الحموضة أو القلوية، نقطة الوميض، الكثافة، الوزن النوعي ومحتوى المادة الفعالة قبل وبعد التخزين. أجريت تجربة الزيوت لخفض أعداد الحشرة القشرية السوداء *(Parlatoria ziziphi)* عبر مختلف مراحلها المختلفة محل الدراسة بمزرعة مركز البحوث الزراعية بالجيزة خلال عامى 2023 و 2024 ، فكانت مرحلة الحوريات الأكثر أنخفاضاً، ثم جاءت الإناث البالغات يليها الأفراد الحاملة للبيض. وفي هذه التجربة زادت فاعلية جميع المعاملات بتقليل أعداد الحوريات ثم الإناث البالغات والأفراد الحاملة للبيض للحشرة القشرية السوداء (*Parlatoria ziziphi*) على مدار فترة أربعة أسابيع في كل موسم.خلال الموسمين الأول والثاني، تراوحت متوسط معدلات الانخفاض للزيوت المنفردة بين 60.9% و66%. بناءً على النتائج المذكورة أعلاه، استخدام تركيبات الزيوت المخلوطة إلى تحقيق معدلات انخفاض أعلى، تراوحت بين 75.1% و78.1%. وبناءاً علي ما تقدم فإن الزيوت النباتية والمعدنية تعد خيارات قيمة وصديقة للبيئة لمكافحة الحشرة القشرية السوداء علي أشجارالبرتقال أبو سرة ، والتي تتوافق مع مبادئ الإدارة المستدامة لمكافحة الافات.

الكلمات المفتاحية : المركز القابل للأستحلاب ، الزيوت ، الحشرة القشرية السوداء ، الكفاءة الأبادية الحشرية و البرتقال أبو سرة.