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Effect of Three Formulations Derived from Plant Origin on Two-spotted Spider Mite *Tetranychus urticae* Koch (Acari: Tetranychidae)

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Abstract

Jojoba oil has an acaricidal effect on *Tetranychus urticae* adult female mortality, as well as antifeedant, larvicidal, and physiological effects on other insects. The goal of this study was to formulate jojoba oil to compare its effectiveness with Baraka oil, and citric acid formulations against the two-spotted Spider Mite *T. urticae*. A new local jojoba oil formulation was developed as an emulsifiable concentrate (EC). Three formulations for (jojoba oil, Baraka oil and citric acid) were put to the test on eggs of *T. urticae*. Jojoba oil and Baraka oil were used as emulsifiable concentrates (ECs) while citric acid was used as soluble powder (SP). Jojoba oil EC showed the highest result followed by Baraka oil EC, and citric acid SP. The EC_{50} values were 376 ppm for jojoba oil and 1305 ppm for Baraka oil (ECs), respectively. Under greenhouse conditions, jojoba oil EC was evaluated on egg hatching and individuals of *T. urticae* at 1000 and 2000 ppm. After 14 days of treatment, egg hatching was totally suppressed, whereas individual inhibition was 84.4 and 91.4 % for both concentrations, respectively. As a result, the two-spotted spider mite can be combated with the new jojoba oil EC formulation.

Keywords: Biological efficiency, Emulsifiable concentrate, Plant origin, Spider mite.

1. Introduction

The two-spotted spider mite *Tetranychus urticae* Koch, 1836 is a chelicerate herbivore [1] and one of the most important agricultural pests, not only because of the damage it does, but also because it has a broad host range, infesting soybeans, cotton, leafy greens and beans among other major crops [2]. *T. urticae* feeds on the mesophyll cells on the underside of the leaves, which are UV-protected, on a regular way [3-4]. As a result, the mite causes mechanical damage in the form of empty cells, resulting in a dull coloring in the damaged organ, as the number of necrotic cells grows, it may turn black. Additionally, the feeding activity affects cell contents, resulting in reduced nitrogen, phosphorus, and protein concentrations, as well as disrupting cell physiology, lowering photosynthesis and injecting phytotoxic chemicals that reduce yields [5]. This mite can cause serious economic damage during harvest if more than 90 adults are identified on a hop leaf [6].

Many insect and mite species have developed resistance to insecticides and acaricides as a result of widespread use. Important crop pests, animal parasites, prevalent urban pests, and disease vectors have all evolved resistance to the point where controlling them has become extremely difficult [7]. Resistance to acaricides in phytophagous mites is on the rise, particularly in spider mites, which have a high intrinsic capacity for rapid resistance evolution [8]. *T.urticae* has the ability to develop resistance to acaricides in a short period of time, which makes control challenging [9]. After a few years of usage, their populations frequently developed a high level of resistance to a newly introduced chemical, with cross-resistance to other compounds with the same mode of action. However, because of its short life cycle, large progeny, and arrhenotokous reproduction, it can rapidly evolve resistance to these substances. As a result, it has acquired the unfavorable designation of "most resistant species" in terms of the total number of chemicals to which populations have developed resistance, and controlling it has become a challenge in many parts of the world [7]. TSSM's toxins reactivity, or capability to detoxify a wide range of

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phytochemicals, is linked to its potential to build pesticide resistance quickly [10].

Natural pesticides generated from plants and microorganisms are gaining popularity [11-12] since they are thought to be safer than synthetic pesticides. Jojoba oil, Baraka oil, and citric acid are examples of these compounds which are classified as plant-derived chemicals. At different temperatures, jojoba oil showed good acaricidal effect on the mortality of adult females of *T. urticae* [13], in addition it showed antifeedant, larvicidal and physiological activities on other insects (*S. littoralis* larvae) [14].

Nigella sativa is a herbal plant that thrives in Mediterranean regions, and its seed is known as black seed, HabbatuSawda, and HabatulBaraka ‘‘the Blessed Seed’’. Essential oils are among the most well-known compounds tested against insects, and *Nigella sativa* oil is an essential oil [15]. These compounds have been used as fumigants [16], contact insecticides [17], repellents [18], and antifeedants [19]. The insecticidal activity of *Nigella sativa* essential oil was also tested [20].

Pesticide compounds in their pure or purified form (also known as the active ingredient or a.i) are difficult to apply to crops. The concentration of many pure chemicals makes them unsuitable for use. It is possible that the active ingredient won't mix well with water, is too volatile to transport, or is too dangerous for humans to manage. As a result, chemical active constituents are blended with inert/inactive chemicals so that they can be safely applied to crops by the user. The pesticide formulation is the combination of the chemical and inert/inactive components [21].

The objective of this research paper was to determine the acaricidal efficacy of three formulations for three safe compounds derived from plants, jojoba oil 90 % EC and the previously prepared Baraka oil 75 % EC [22] and citric acid 90 % SP [23-25] on *T. urticae*. In order to move a new step in our ongoing program for obtaining new, safe and eco-friendly active ingredients and formulating it in a local formulation to be used for pest control after completing the necessary required studies.

2. Materials and Methods

Tested chemicals:

a) Jojoba oil and Baraka oil were supplied by supermarket.

b) Surface active agents were supplied by EL - Gomhoria Co., Cairo, Egypt.

Physico-chemical properties of formulation ingredients:

Active ingredient

a) Free acidity or alkalinity: It was determined using WHO guidelines [26].

b) Solubility: It was measured according to [27] with the following equation

% solubility = W/V x 100 ……………………… (1)

Where; W= Weight of active ingredient, V= Solvent amount required for complete solubility.

Surface active agents

a) Hydrophilic-lipophilic balance (HLB): It was determined according to [28].

b) Critical micelle concentration (CMC): It was calculated using [29].

c) Free acidity or alkalinity: It was determined, as before.

d) Surface tension: It was calculated according to ASTM D-1331 [30], it was determined using a Du-Nouy tensiometer.

Jojoba oil local prepared emulsifiable concentrate formulation (EC)

It was prepared according to [31]

a) Emulsion stability: It was performed according to [33].

b) Accelerated storage: It was determined according to Collaborative International Pesticides Analytical Council (CIPAC) [34].

Jojoba oil spray solution at field dilution rate (0.5 %)

a) Electrical Conductivity: It was calculated with Cole-Parmer PH/Conductivity meter 1484-44, where µmhos is the unit of electrical conductivity measurements [35].

b) Surface tension: It was determined as previously mentioned.

c) PH: It was measured by using conductivity Cole-Parmer PH meter 1484-44 [35].

d) Viscosity: It was determined using Brookfield viscometer Model DVII+Pro, where Centipoise is the unit of measurement according to ASTM D-2196 [36].

Bioassay

Rearing of two-spotted spider mite:

T.urticae mites used were reared in Department of Vegetables and Ornamental Mites Research, Plant Protection Research Institute, Agricultural Research Center, Dokki, Egypt, for more than 40 generations on *Acalypha marginata* at 25±2 ˚C and 60 ± 10 percent relative humidity and 14:10 hours photoperiod (Light: Dark). To keep the culture, fresh plants were provided at regular intervals.

Under laboratory conditions:

Ovicidal efficacy

A caricidal effect of oils (Jojoba oil and Baraka oil) and citric acid formulations on the eggs of *T. urticae* were made according to [37] with little modifications as follows: The toxicity on the egg stages was determined by dipping the *Acalypha marginata* leaf disks (2.5 cm in diameter) in five concentrations (10, 100, 1000, 10000 and 100000 ppm) for 10 seconds and the excess solution was dried off with filter paper. One-day-old eggs were applied to the lower surface of leaf discs with a hairbrush. Each concentration and control were repeated three times (each with 50 eggs), with the control being made by using water. The hatchability of the eggs was measured after 14 days of treatment in laboratory, where it was kept.

Under greenhouse conditions

Under greenhouse conditions, the efficacy of jojoba oil formulation on egg hatchability and individuals of the two-spotted spider mite (TSSM) was tested as follows: twelve pots were planted and infested artificially. The population was allowed to grow to appropriate scale. Two concentrations of jojoba oil formulation (1000 and 2000 ppm) were made, with each treatment replicated three times. After spraying the prepared pots, the results were obtained after 1, 3, 5, 7, and 14 days of treatment. Before and after treatment, the population of eggs and individuals were determined.

Statistical analysis

The concentration inhibition regression lines were produced by using Finney's method [38] as follows,

Toxicity index = EC_{50} of the most effective compound / EC_{50} of the tested compound x 100 .. (2)

and the inhibition percentages were corrected using Abbott's formula [39] according to the following equation,

Mortality % = The died number of TSSM counted after treatment / The died number of TSSM counted before treatment x 100 …………………. (3)

3. Results and Discussion

Preparation of jojoba oil as emulsifiable concentrate formulation (EC)

When selecting a pesticide formulation, several factors must be considered. These include the risks and benefits of the various possibilities, the feasibility of utilizing a certain formulation in a specific location to control the target pest, and if the formed product will provide effective control [40].

Physico-chemical properties of jojoba oil as active ingredient

The physico-chemical properties of jojoba oil as an active ingredient were shown in Table 1. It demonstrated no solubility in water or acetone, but complete solubility in xylene (100 %). On the other hand, it has a little amount of free acidity (0.02). These findings indicated that it may be formulated as emulsifiable concentrate (EC) [41]. Emulsifiable Concentrate (EC) formulations include oil, active ingredient (a.i), and surface active agents. A milky emulsion emerges spontaneously when the formulation is diluted in water. To be called an emulsifiable concentration, the formulation components must be in solution [42].

Table 1.Physico-chemical properties of jojoba oil as an active ingredient

Solubility % (W/V)			**Free acidity as % H_2SO_4**
Water	**Acetone**	**Xylene**	
N.S*	N.S*	100	0.02

N.S*:means insoluble

Physico-chemical properties of local jojoba oil 90 % EC formulation before and after hot storage

The effect of storage under normal and accelerated storage temperatures on the developed local formulation was shown in Table 2. There were no alterations in the formulations characteristics before and after storage. Before and after storage, it passed an emulsion stability test and maintained its free acidity. This means the formulation can maintain its features in both hot and ordinary storage conditions [43]. The new formula passed the cold test successfully, with no separation or sedimentation. This is in accordance with [44] and JMPS [45-46]. In some parts of Australia, night time temperatures often fall below 0 degrees Celsius. As a result, liquid formulations should be evaluated for seven days at 0 ±2 ˚C or lower. According to [35], the effect of low temperatures on stability should be tested and reported. Accelerated stability tests at high temperatures are used to accelerate the rate of chemical or physical changes in a product or both [47].

Table 2.Physico-chemical properties of local jojoba oil 90 % EC formulation before and after hot storage

Before storage			**After storage**		
Emulsion stability		Free acidity as H_2SO_4	Emulsion stability		Free acidity as H_2SO_4
Hard	Soft	0.02	Hard	Soft	0.02
Pass	pass		pass	pass	

Physico-chemical properties of the spray solution of the local formulation of jojoba oil at field dilution rate

The physical and chemical characteristics of a pesticide have a significant impact on its biological activity against the target pest species. The mode of action, dosage, mode of application, and subsequent environmental chemodynamics of pesticides are all determined by their physical features. Pesticides have a wide range of physical attributes depending on their chemical composition and formulation [48].

Adjuvants are commonly used to improve the efficacy of pesticides by changing the characteristics of the spray solution. Adjuvants are used to improve the biological activity of products by enhancing the penetration of active ingredients and their uptake by plants; increase product adhesion and rain fastness; modify the atomization process and thus reduce droplet driftability or improve the biological activity of products by enhancing the penetration of active ingredients and their uptake by plants [49]. Surfactants, which raise the surface tension between a liquid and a target, improve spray coverage and product retention on the target, and therefore improve spray deposition efficiency [50] are one type of dedicated adjuvant.

The physical properties of the spray solution at the field dilution rate (0.5 %) were displayed in Table 3. The spray solution has an acidic PH of 5.58, strong electrical conductivity (120 µhos), and viscosity (10 cm/poise), but low surface tension (33.06 dyne/cm). According to El-Sisi *et al*., the low pH value and high electrical conductivity of the spray solution would result in deionization of the insecticide, increased deposits and penetration in the treated surface, and expected improvement in pesticidal efficacy. Pereira stated that a low value of surface tension in the spray solution can cause an increase in spreading across the treated surface, resulting in an increase in pesticidal efficiency [52]. In contrast to surface tension, increased viscosity can improve pesticide efficacy by reducing drift and increasing stickiness [53].

Adjuvants impact the size of droplets formed by nozzles by influencing the physicochemical parameters of the spray liquid. The influence of spray liquid characteristics on drift risk was explored [54], and the authors found that emulsified oils increased droplet size and reduced the fraction of tiny droplets when atomized by flat fan, hollow cone, or twin fluid nozzles compared to water sprays. Vegetable oils, mineral oils, organosilicones, and water-insoluble surfactants all showed this effect. The use of pure water with esterified vegetable oil and organosilicone surfactants resulted in the formation of larger droplets. When an emulsifiable concentrate (EC) was used, the droplets had a smaller volume median diameter (VMD) and a higher drift potential (DP).

Table 3.Physico-chemical properties of spray solution at field dilution rate

Surface tension dyne/cm	Viscosity cm/poise	Conductivity µ mhos	PH
33.06	10	120	5.58

Table 4. showed the ovicidal effect of the three locally prepared formulations jojoba oil 90 % EC, Baraka oil 75 % EC and citric acid 90 % SP against *T. urticae* by serial concentrations. For the three formulations under study, each showed a gradual direct proportional relationship between the effect and the concentration. The lowest effect was the effect of the lowest concentration (10 ppm) and the highest effect was the effect of the highest concentration (100000 ppm) and the most obvious effect was for jojoba oil EC followed by Baraka oil EC formulations and then citric acid SP formulation. It appeared clearly in concentrations of 1000, 10,000 and 100,000 ppm. Essential oils have been shown to have insecticidal, miticidal, and broad spectrum activity against a variety of soft-bodied pests. *Thymus vulgeris, Lavendula officinalis, Eucalyptus camalduensis, Mentha longifolia*, *Salvia officinalis*, Rosemary, Garlic, Jojoba, Many studies have looked into the acaricidal effects of Myrtus communis and other essential oils. Antifeedant, repellent, moulting and respiration inhibition, growth and fecundity reduction, and lethal impact are just some of the effects of essential oils [55-56].

Table 4. Ovicidal Effect of jojoba oil 90 % EC, Baraka oil 75 % EC and citric acid 90 % SP against *T. urticae*

Local formulation	% of inhibition at 10 ppm	100 ppm	1000 ppm	10000 ppm	100000 ppm
Jojoba oil 90 % EC	10.8	32.9	63.7	87.4	94.5
Baraka oil 75 % EC	24.1	35.5	48.5	61.5	73.4
Citric acid 90 % SP	23	24.9	26.7	28.8	30.7

Table 5. showed EC_{50}, EC_{90} and slope values for the three locally prepared formulations. Jojoba oil 90 % EC showed the lowest EC_{50} value (376 ppm) followed by Baraka oil 75 % EC (1305 ppm) indicating that jojoba oil formulation was more effective than Baraka oil formulation although both of them are emulsifiable concentrates but the higher effect for jojoba oil may be attributed to the nature of active ingredient [56], or to the surfactants used for the preparation of each formula and its role in penetration and transport of the active ingredient (a.i) [57] , [49] or both. This was also confirmed by the slope values of both formulations whereas the value of the slope of jojoba oil (0.79) was higher than that of Baraka oil (0.33), which indicates a sharper line of toxicity and greater ovicidal activity.

Table 5. LCP lines of jojoba oil 90 % EC, Baraka oil 75 % EC and citric acid 90 % SP against eggs of *T. urticae*

Local formulation	EC_{50} ppm	EC_{90} ppm	Slope
Jojoba oil 90 % EC	376	15636	0.79
Baraka oil 75 % EC	1305	215922	0.33
Citric acid 90 % SP	NC*	NC*	NC*

NC*: Non calculated

Table 6. showed the effect of jojoba oil 90 % EC formulation on eggs of T. urticae under greenhouse conditions by two concentrations (1000 & 2000 ppm) after 1, 3, 5, 7 and 14 days from treatment. The obtained results declared that for both concentrations, the formulation succeeded to inhibit egg hatching with a gradual increase after 3, 5 and 7 days from

treatment by 55, 60, and 83 for 1000 ppm and 46, 73 and 95.4 % for 2000 ppm. Both concentrations completely inhibited egg hatching after 14 days from treatment, which means that jojoba oil 90 % EC formulation could inhibit *T. urticae* egg hatching even with the lowest concentration. Jojoba oil (0.25 percent) had a 97.6 percent mortality rate for *T. urticae* egg hatching when compared to other oils [58].

Table 6. Effect of jojoba oil 90 % EC formulation on eggs of *T. urticae* under greenhouse conditions

Concentration (ppm)	**% of inhibition after**				
	Day	**3 Days**	**5 Days**	**7 Days**	**14 Days**
1000	100	55	60	83	100
2000	100	46	73	95.4	100

Fig. 1. showed the relation between time and percentage of inhibition of egg hatching for each concentration of jojoba oil 90 % EC local formulation. It showed an inverse proportion between the percentage of inhibition and time (the highest effect for all tested concentrations was after two days from treatment and the lowest effect was after seven days from treatment), which means that, the effect was decreased with increasing exposure periods. For the three concentrations used, the effect was a delay of egg hatching; in addition all of them after seven days from exposure inhibited egg hatching by 35 – 50 %.

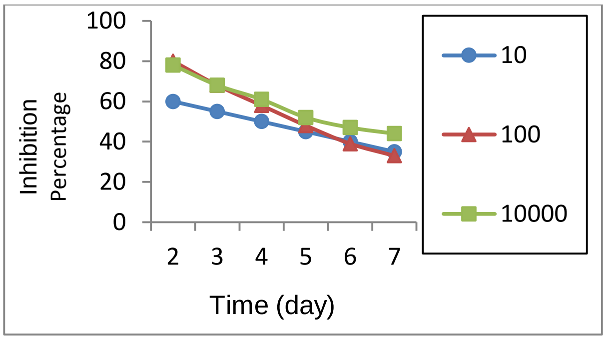

Figure 1.Relationship between time and % of inhibition of egg hatching for each concentration of jojoba oil 90 % EC local formulation

Fig. 2. showed the relation between time and percentage of inhibition of egg hatching for each concentration of Baraka oil 75 % EC local formulation. Three concentrations showed an inverse continuous proportion between percentage of inhibition and time (10, 100 and 10000 ppm), with these concentrations, the effect decreased with the increase in exposure periods, indicating that their effect was delay of egg hatching. On contrary the fourth concentration (1000 ppm) showed a direct proportion between the percentage of inhibition and time (the effect was increased gradually with increasing time). It inhibited egg hatching by 60.3 %.

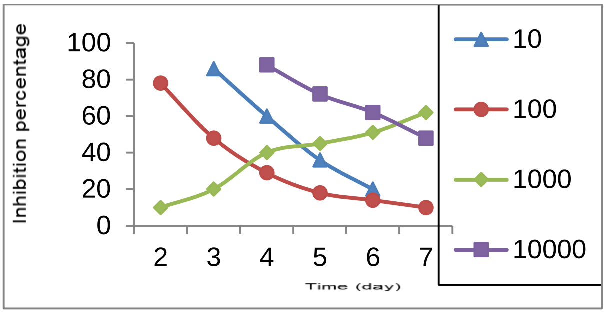

Figure 2.Relationship between time and % of inhibition of egg hatching of Baraka oil 75 % EC local formulation

Fig. 3. showed the relation between time and percentage of inhibition of egg hatching for each concentration of citric acid 90 % SP local formulation. 1000 and 100000 ppm showed a direct proportion between the percentage of inhibition and time, the effect that was considered as an inhibition of egg hatching. 100 ppm showed only a delay in egg hatching as the effect decreased with increasing time, while 10000 ppm showed constant effect without any change during the period of exposure.

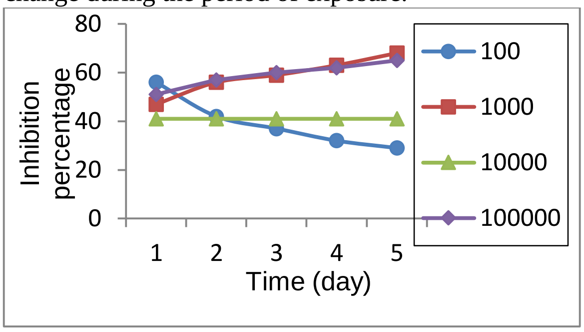

Figure 3.Relationship between time and % of inhibition of egg hatching for each concentration of citric acid 90 % SP local formulation

The three figures showed that jojoba oil local formulation is the only formula that revealed a regular relationship between percentage of inhibition for egg hatching and the period of treatment. The results showed also that the two other formulas showed fluctuations in the results as they showed an inverse correlation of percentage of inhibition with the period of the treatment in some concentrations and in some others showed a direct correlation.

Table 7. showed the effect of the newly prepared jojoba oil 90 % EC formulation by 1000 and 2000 ppm under greenhouse conditions on the individuals of *T. urticae* after 1, 3, 5, 7 and 14 days from treatment. Both concentrations showed fluctuated percentages of inhibition on individuals, for 1000 ppm the effect was decreased after three days from treatment and then it was increased after 5 days from treatment and returned back to decrease after 7 days from treatment. It was also the case with 2000 ppm as the percentage of inhibition decreased after 3 and 5 days from treatment and returned to increase after 7 days from treatment. But after 14 days from

treatment both concentrations inhibited markedly individuals by 84.4 and 91.4 % for 1000 and 2000 ppm respectively.

Table 7.Effect of jojoba oil 90 % EC formulation on individual of *T. urticae* under greenhouse conditions

Concentration (ppm)	Day	% of inhibition after 3 Days	5 Days	7 Days	14 Days
1000	50	33	60.1	34	84.4
2000	83.5	76.6	55.8	78.9	91.4

4. Conclusion

Jojoba oil was formulated as emulsifiable concentrate (EC) and passed all tests reported for (ECs). The ovicidal effect of jojoba oil 90 % EC, Baraka oil 75 % EC and citric acid 90 % SP was studied on eggs of *T. urticae* under laboratory conditions. Jojoba oil EC showed the highest effect followed by Baraka oil EC whereas citric acid SP showed the weakest effect. Jojoba oil 90 % EC was also tested on egg hatching and individuals of *T. urticae* under greenhouse conditions; it completely inhibited egg hatching and inhibited markedly individuals after 14 days from treatment. Depending on these results jojoba oil new formula can be used as acaricide after completion of all other necessary required laboratory and field experiments.

5. Conflicts of interest

The authors declare that they have no conflict of interest

6. References

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