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Evaluation of the Efficacy of the Newly Formulated Salt Mixture As 10 % Soluble Concentrate on Armored Scale Insect *Hemiberlesia lataniae* (Hemiptera: Diaspididae)

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ABSTRACT

Copper sulfate and alum were mixed and formulated after carrying out the necessary experiments as 10 % soluble concentrate (SL). The new formula passed all physico-chemical properties defined for soluble concentrates. It was then tested on the adults and nymphs of the armored scale insect *Hemiberlesia lataniae* (Hemiptera: Diaspididae) under laboratory conditions, it showed low efficacy on the adults while it showed high efficacy on the nymphs, as its EC_{50} value for the nymphs was 474 ppm after 72 hours from exposure, the results that were confirmed on experimentation under field conditions on both stages under study, fluctuated percentages of inhibition were obtained in case of adults while it showed high percentage of inhibition on the nymphs. The new formula proved good efficacy on the nymphs of *H. lataniae* and could be used as an insecticide after completing the other specified experiments in the future.

Keywords: Armored Scale Insects, *H. lataniae*, Soluble Concentrate and Formulation.

1. INTRODUCTION

Scale insects are a significant pest that has been found to harm a large variety of host plants all over the world **(Franco *et al.* 2009; Mazzeo *et al.* 2014)**. These insects can be found on the leaves, twigs, branches, and roots of their hosts, and some even dwell inside plant domatia **(Kondo *et al*. 2008)**. They feed mainly entirely on the phloem of their host plants, causing direct damage, but they can also produce indirect harm by spreading plant diseases by injection or honeydew buildup, increasing plant pathogen infection **(Ross *et al.* 2010)**. Some species, however, feed on parenchyma tissue directly by eating the contents of parenchyma cells **(Kondo *et al.* 2008)**.

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When a plant is attacked by a large number of these insects, it may have slowed growth, yellow blotches on its leaves, dead branches, and lose some or all of its leaves.

Fig, guava, pear, apple, grape vine, olive trees, and ornamental plants are all infested by *H. lataniae* (Hemiptera: Diaspididae) (Signoret) **(Radwan, 2014 and Attia *et al.*, 2017)**. When trees are highly infested with *H. latania,* the tree becomes weak and loses its yield, it suckers the plant sap, weakening the tree and producing deformations as a result of the toxic saliva's activity, As a result, abnormal color of some leaves, twigs, and berries, as well as pitting of the bark stems, can be used to determine its existence **(Daneel, 1998)**. *H. lataniae* can lead to problems during the fruit's production and packing. Despite their importance as pests, these insects have relationships with their host plants.

This armored scale insect also causes histological changes in fruits and tree branches **(Hernandez *et al.* 2013)**.

Ficus nitida is an evergreen woody plant with a spreading growth style that typically matures into dense shade trees. Ficus nitida is a fast-growing evergreen tree that is commonly employed as a street or yard tree. Apart from being a dense tree, it can also take the appearance of a dense spherical canopy with a globe-shaped top that appears dense and weighty due to its gorgeous leaves. It works well in large containers as a container plant **(Auslander *et al.*, 2003 and Abo-Shanab, 2011)**.

Copper fungicides are still quite inexpensive and effective when compared to many other options. Copper is frequently used in disease management programs due to the requirement to rotate these compounds with others with distinct mechanisms of action **(Michaud *et al.*, 2003)**. **Teran *et al.*, (1993)** concluded that copper sulphate fungicide was found to be harmless for two aphytis parasitoids (Hymenoptera: Aphelinidae) and also vital for citrus scale control. Furthermore, **Michaud *et al.*, 2003** exposed the larvae of three lady beetle species (Coleoptera: Coccinellidae) to field rates of copper sulphate combined with petroleum oil, a mixture routinely used in Florida lemons.

It was also shown that alum have an insecticidal efficacy, as gooseberry caterpillars would be killed by alum dissolved in tobacco liquor; it is strict and could have served as an insect repellant by altering plant flavor. Soft-bodied forms may be affected by the high osmotic pressure of a concentrated alum solution or alum in tobacco solution **(Smith & Secoy, 1976).**

It is required to employ novel chemistry insecticides due to the detrimental effects of insecticides, particularly conventional insecticides, which are less hazardous to the environment, safer to use, and more effective **(Korrat *et al.*, 2012)**. Toxicity against insect pests is increased when insecticides are mixed (insecticide mixtures) **(Warnock & Cloyd, 2005)**. Because insects do not acquire resistance to various modes of action at the same time, it is advised as a "resistance management program" **(Yu SJ, 2008)**. Pesticide blends affect plant translocation and absorption. For 14 generations, Sumithion and Fenvalerate mixtures slowed the development of resistance in *Myzus persicae*. Insecticide mixtures can control many pest species, save time, reduce application costs, reduce quantity, reduce the number of sprays, have synergistic combined action, and are environmentally friendly **(Regupathy & Ramasubramanian, 2004)**.

In most cases, pesticide compounds in their "raw" or unformulated state are unsuitable for pest control. These concentrated chemicals and active components could be challenging to handle and transport since they don't mix well with water, chemically unstable and concentrated. Manufacturers add inert chemicals like clays and solvents to increase application effectiveness, safety, handling, and storage for these reasons. Inert compounds have no pesticidal efficiency and are used to carry active ingredients. Mixing active and inert ingredients produces what is called pesticide formulation **(Fishel, 2010)**.

Hence, the present investigation was emphasized on the use of a formulation of a mixture of two salts (copper sulfate and alum) on armored scale insect *H. lataniae* (Hemiptera: Diaspididae) as a serious step in the preparation of a local pesticide to be used in the field of pest control after taking all other related tests into consideration in the future.

2. MATERIALS AND METHODS

2.1. Tested chemicals:

1) Copper sulphate (Copper (II) sulfate, molar mass 249.6 g·mol^{-1}), was supplied by EL-Gomhoria Co., Cairo, Egypt.

2) Alum (potassium aluminum sulfate , molar mass 474.3 g·mol^{-1}), was supplied by EL-Gomhoria Co., Cairo, Egypt.

2) Surface active agents: Sodium lauryl sulfate (SLS) and Triton X-100 were supplied by EL-Gomhoria Co., Cairo, Egypt.

2.2. The physico-chemical properties of basic formulation constituents:

2.2.1. Active ingredient:

The following physico-chemical properties of copper sulfate and alum mixture as active ingredient determined were:

a) Solubility: It was determined by measuring the volume of available solvents (distilled water, acetone and xylene) for complete solubility or miscibility of one gram of active ingredient at 20 ˚C **(Nelson and Fiero, 1954)**. Solubility percentage was calculated according to the following equation:

$$\% \text{ solubility} = W/V \times 100$$

[Where; W= active ingredient weight, V= volume of solvent required for complete solubility].

b) Free acidity or alkalinity: It was determined according to the method described by **WHO specification (1979)**.

c) Melting point: It was determined on an electric digital melting point (Gallenkamp) 9200 A apparatus.

2.2.2. Surface active agents:

a) Hydrophilic-lipophilic balance (HLB): The solubility of surfactant in water is considered as approximate guide to its hydrophilic-lipophilic balance (HLB) **(Lynch and Griffin, 1974)**.

b) Surface tension: It was determined by using Du-Nouy tensiometer for solutions containing 0.5 % (W/V) surfactant according to **ASTM D-1331 (2001)**.

c) Critical micelle concentration (CMC): The concentration in which the surface tension of solution doesn't decrease with further increase in surfactant concentration, (CMC) of the tested surfactants was determined according to the method described by **(Osipow, 1964)**.

d) Free acidity or alkalinity: It was determined by the same method described before.

2.2.3. Local prepared soluble concentrate formulation:

a) Surface tension: It was determined as shown before.

b) Free acidity or alkalinity: It was measured as mentioned before.

2.2.4. Spray solution at field dilution rate:

a) PH: It was determined by using Cole-Parmer PH conductivity meter 1484-44 according to (**Dobrat and Martijn, 1995)**.

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

c) Electrical Conductivity: It was determined by using Cole-Parmer PH / Conductivity meter 1484-44, where µmhos is the unit of electrical conductivity measurements according to (**Dobrat and Martijn, 1995)**.

d) Viscosity: It was determined by using Brookfield viscometer model DVII+Pro, where centipoise is the unit of measurement according to **ASTM D-2196 (2005)**.

2.3. Bioassay

The efficiency of the salt mixture 10 % soluble concentrate formulation on the adults and nymphs of *H. lataniae* under laboratory and field conditions was evaluated according to the following procedure:

2.3.1. Under laboratory conditions:

The leaf spraying technique was used as a toxicity test for the tested 10 % soluble concentrate formulation against (nymphs and adult female) stages of *H. lataniae* infest Ficus leaves. Infested Ficus leaves were transported to the laboratory and sprayed with three successive concentrations 300, 600 and 1200 ppm from the formulation. Leaves were left to dry at ambient temperature and put in polyethylene bags. Five replicates were conducted for each concentration of the tested formulation; each replicate consisted of five leaves. Control leaves were sprayed by water. After application, polyethylene bags (tested and control) were placed under laboratory conditions and examined for mortality and life after 24, 48 and 72 hrs.

2.3.2. Under field conditions:

Four rows of Ficus trees in quadratic form in Qalub, Qalubiya governorate were chosen to test by the new formulation, three rows sprayed with three different concentrations from the formulation and the fourth one was used as control. Each row consists of nine trees and divided to three replicates three trees/replicate, thirty leaves for each replicate were picked up to evaluate the treatment efficiency. These samples were kept in paper bags, and then were taken to laboratory for examination. Data of the pre-treatment, control and post-treatment samples were recorded for live stages (nymphal and adult female) to calculate reduction percentage in population. Two post-treatment counts were taken after 2 and 4 weeks from spraying.

2.4. Statistical analysis:

Under laboratory conditions, Inhibition percentages were corrected using **Abbott's formula (1925)**, and the concentration inhibition regression lines were drawn according to the method of **Finney (1952)**. Under field conditions, the percentage of reduction in each stage was estimated according to the equation of **Henderson and Tilton (1955)**.

3. RESULTS AND DISCUSSION

Some pesticide active ingredients dissolve easily in a liquid solvent, such as water or a petroleum-based derivative. On mixing, they form a solution that does not settle out or separate. These formulations usually contain active ingredient, solvent and one or more other ingredients. The determining factors on choosing the suitable kind of formulation for a definite active ingredient are its physico-chemical properties. Depending on these properties, the salt mixture was prepared as soluble concentrate formulation (SL).

3.1. Physico-chemical properties of copper sulfate and alum mixture as active ingredient:

Table (1) showed that, both salts were insoluble in acetone and xylene, but both recorded medium degree of solubility in water, 10 and 40 % for copper sulfate and alum respectively. In addition, both of them showed acidic property. The result obtained from solubility refers to the possibility of preparing this mixture as soluble concentrate (SL); in addition, their acidic property determines the kind of adjuvants used to be acidic.

Table (1): Physico-chemical properties of copper sulfate and alum mixture as active ingredients

Salt	Solubility % (W/V)			Free acidity as % H_2SO_4	Melting point ˚C
	Water	Acetone	Xylene		
$CuSO_4$	10	N.S*	N.S*	4.88	80
$KAl(SO_4)_2$	40	N.S*	N.S*	4.4	92-95

N.S*: means insoluble.

3.2. Physico-chemical properties of surface active agents:

Table (2) showed the physico-chemical properties of two surfactants, anionic sodium lauryl sulfate (SLS) and nonionic Triton X-100, the latter showed complete solubility in water and a good degree of solubility in acetone and xylene, While the former showed little degree of solubility in water, but no solubility at all in acetone and xylene. Both surfactants lowered surface tension of water from 72 to 27.8 and 29 dyne/cm for (SLS) and Triton X-100 respectively. Similarly, both of them showed HLB values greater than 13 indicating that both of them were considered as dispersing agents. In addition, both showed acidic properties from the free acidity or alkalinity point of view. The main idea for measuring the physico-chemical properties of surfactants was to choose which one of them was compatible with active ingredient to be used in the formulation. From the above results, both surfactants could be used in the formulation of the salt mixture under study as soluble concentrate (SL).

Table (2): Physico-chemical properties of surface active agents

Surface active agent	Solubility % (W/V)			Surface tension dyne/cm	HLB	CMC %	Free acidity as % H_2SO_4	Free alkalinity as % NaOH
	Water	Acetone	Xylene					
Sodium lauryl sulfate	27.8	N.S*	N.S*	27.8	>13	8	-	0.48
Triton X-100	100	52	52	29	>13	0.25	-	0.02

3.3. Physico-chemical properties of the 10 % soluble concentrate local formulation before and after accelerated storage:

According to data presented in Table (3), the formulation showed relatively the same values for PH, surface tension and solubility before and after accelerated storage conditions, free acidity is the only parameter that showed relatively different values although it was alkaline in both cases, which means that the new formula kept its physico-chemical properties under either normal or accelerated storage conditions.

Table (3): Physico-chemical properties of the 10 % soluble concentrate local formulation before and after accelerated storage.

Before storage					After storage				
PH	Free acidity as % H_2SO_4	Surface tension dyne/cm	Solubility	Sedimentation	PH	Free acidity as % H_2SO_4	Surface tension dyne/cm	Solubility	Sedimentation
5.09	12.75	36	soluble	Nil	5.03	10.29	37.3	soluble	Nil

3.4. Physico-chemical properties of spray solution at field dilution rate (0.5 %):

Measuring the physico-chemical properties of the spray solution declared an increase in its viscosity and electrical conductivity while surface tension was decreased with an acidic PH value. The wettability of a plant leaf is a desirable property in application; it depends on leaf contact, constituents of the leaf and characteristics of the pesticide **(Kissmann, 1998)**. Good wettability occurs when the attraction between the leaf surface and water is greater than the surface tension of the liquid. Thus, the surface tension of the agrochemical should not be very high (**Pereira *et al.*, 2016)**. Decreasing the pH value of the spray solution would lead to an increase in attraction between spray solution and treated plant with a consequence increase in deposition and penetration on the tested surface which will in turn increase the effectiveness **(Molin and Hirase, 2004)**. The increase in viscosity is a required property for pesticide spray solution as was stated by **Richardson (1974)**, Increasing viscosity of spray solution causes reduction drift, retention sticking of spray solution on the surface of plant leaf with an increase in the pesticidal efficiency. In addition, the increase of the electrical conductivity of spray solution is a desired property too as it may lead to deionization of pesticide formulation and consequently increasing its deposit and penetration through the surface of the tested plant, consequently the pesticide effectiveness of these formulation was increased **(El-Attal *et al.*, 1984)**.

Table (4): Physico-chemical properties of spray solution at field dilution rate

Surface tension dyne/cm	Viscosity cm/poise	Conductivity µ mhos	PH
34.8	8.48	660	5.56

Table (5) showed the Insecticidal activity of the 10 % soluble concentrate formulation against the adults of *H. lataniae* under laboratory conditions with serial concentrations (300, 600 and 1200 ppm) after 24, 48 and 72 hrs. from exposure. There was an inhibition effect after 72 hrs. (The effect was between 30.3 and 30.9 %, no significant differences between the tested concentrations observed).

Table (5): Insecticidal effect of the 10 % soluble concentrate local formulation against the adults of *H.lataniae* under laboratory conditions:

Concentration (ppm)	% of change in inhibition after			A.T*
	24 hrs.	48 hrs.	72 hrs.	
300	17.5	2	11.4	30.9
600	24.6	6.3	-0.6	30.3
1200	0.6	43.9	-0.13.6	30.9

A.T*: Accumulation toxicity: the total sum of toxicity in the end of the experiment for each concentration.

Table (6) showed the ET_{50} and EC_{50} of the 10 % soluble concentrate formulation against the nymphs of *H. lataniae* under laboratory conditions with the previously reported serial concentrations after 24, 48 and 72 hrs. Direct relationship between concentration and the percentage of inhibition was observed, the highest effect was found after 72 hrs. from exposure (474 ppm) with the increase in the slope value from 0.77 after 48 hrs. to 3.4 after 72 hrs. from treatment. The ET_{50} value for 600 ppm was (24.2 hrs.) lower than that for 1200 ppm (29.98 hrs.) indicating the ability of the first concentration to exhibit faster effect on the nymphs than the second concentration. These results could be explained on the basis of the temporary effect of the toxicant to control the nymphs in addition to the ability of the nymphs to dispose the toxicant.

Table (6): ET_{50} and EC_{50} of the 10 % soluble concentrate formulation against nymphs of *H. lataniae* under laboratory conditions

Concentration (ppm)	% of inhibition after			ET_{50}	Slope
	24 hrs.	48 hrs.	72 hrs.		
300	26.1	54	27.8	N.C*	N.C*
600	29.2	65.6	67	24.2	3.25
1200	36.5	72.6	92.6	29.98	
EC_{50} ppm	NC*	719	474	-	-
Slope	NC*	0.77	3.4	-	-

N.C*: Non calculated

The results obtained from treating *H. lataniae* adults and nymphs by the formulation under laboratory conditions declared low efficacy on the adults but high efficacy on the nymphs, and consequently the new formula was tested on the adults and nymphs under field conditions by serial concentrations (700, 1400 and 2800 ppm). Table (7) showed the effect on the adults. With all tested concentrations, fluctuations were observed in the percentages of inhibition, as the inhibition was increased with some concentrations and decreased with other concentrations. The results that may be explained on the basis of the instability of the population density under field conditions

Table (7): Effect of the 10 % soluble concentrate local formulation against the adults of *H. lataniae* under field conditions

Concentration (ppm)	% of inhibition after		
	24 hrs.	48 hrs.	2 weeks.
700	54	46	60
1400	45	46.5	34.8
2800	74.7	39	46.7

Table (8) showed the effect of the 10 % soluble concentrate formulation against the nymphs of *H. lataniae* under field conditions with the same previously used concentrations. It was also observed that there was a gradual direct increase in the percentage of inhibition with the increase in the concentration for each period of exposure, the highest percentage of inhibition 86.9 % was found for the highest concentration used (2800 ppm) after 2 weeks from exposure. The results that could be explained on the basis of the effect of the additives used in the formulation that facilitates the penetration of the pesticide with a consequence increase in the toxicant concentration **(Fishel, 2010)**. As another explanation, the nymphs was affected_greatly with the increase in concentration as a reason for the absence of the armored scale in crawlers and white cap stage in addition, to the thin armored scale of the first and second stages of *H. Latania* nymphal stage resulting in the ability of the toxicant concentration to increase inside the nymphs.

Table (8): Effect of the 10 % soluble concentrate local formulation against the nymphs of *H. lataniae* under field conditions

Concentration (ppm)	% of inhibition after		
	24 hrs.	72 hrs.	2 weeks.
700	24.1	35.9	72.6
1400	29.5	43.5	78.1
2800	40.9	44.7	86.9

The 10 % soluble concentrate formulation showed a gradual uniform inhibition effect on nymphs than on adults under both laboratory and field conditions, the results of the present study were in conformity with the findings of **(Helmy *et al.*, 2001)**.

CONCLUSION:

Copper sulfate and alum mixture was formulated as 10 % soluble concentrate. The new formula succeeded to pass all physical and chemical tests specified for this kind of formulation. On testing on the adults and nymphs of *H. lataniae* under laboratory conditions, it showed good inhibition effect on the nymphs, the results that were also confirmed on testing under field conditions. The newly prepared soluble concentrate formulation could be used in field of insect control after completing the other required studies in the future.

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