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Influence of some essential oils, chemical compounds and their mixtures against *Ceroplastes rusci* L. and *Asterolcanium pustolans* Cock on fig trees

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Abstract: Laboratory bioassay experiments were carried out to evaluate the relative efficacy of Nimbecidine EC**,** essential oils of *Brassica nigra* Koch., *Boswellia carterii* Birdw. and *Nigella sativa* L., mineral oils viz. KZ oil 95%, Super Royal oil 85.7 % and Malathion 57% E.C. against *Ceroplastes rusci* L. and *Asterolcanium pustolans* Cock. Considering the LC_{50} value, the Nimbecidine EC showed the most effect (0.0048 and 0.0042) whereas that of Super Royal oil was (0.295 and 0.275) in this concern. All tested compound gave satisfactory control against *C. rusci*. The reduction of infestation values, however, varied greatly with the time elapsed after spraying and according to the nature of each tested compound. Thus, Nimbecidine EC was the superior compound, after the chemicals insecticides Malathion, giving an average of 73.02 % reduction throughout the whole experimental period (2 months). Two month post-treatment, Nimbecidine EC, became more efficient and had almost similar activity as the chemical insecticides Malathion displaying 69.03 and 64.9% reduction in infestation of *Asterolcanium pustolans* insect pest. Joint action of certain mixtures against the tow insect populations was also investigated.
Key words: Nimbecidine. Essential oils. *Ceroplastes rusci*. *Asterolcanium pustolans*. Fig.

Introduction

Fig tree (*Ficus carica* L.) is a typical species of Mediterranean countries. It has been considered as a potential alternative fruit crop to common fruit trees such as peach, apple or apricot for several agricultural areas in the world. The world production of figs is about one million tons. The fig fruit is a highly perishable climacteric fruit and oldest species of the fruit tree having been cultivated by humans for over 5000 years[1, 2]. Syconia and leaves have provided valuable food for people and animals. Figs have been used for human consumption, fresh and dray for centuries and recently their nutritive and pharmacological values have been investigated[3-5]. Fig trees are infested with several insect pests mainly stem borers, scale insects, mealy bugs, and fruit flies,[6-7]. The fig waxy scale insect *Ceroplastes rusci* (order: Homoptera, family: Coccidae) and pustule scale insect *Asterolcanium pustolans* Cock (Homoptera, family: Asterolecanidae) are serious pests in Egyptian fig orchards. These insects cause so many damages to the fig trees. They encrust the twigs: often accompanied by shooty moulds and ants feeding on the honeydew excreted. Some cells of the cortex, particularly in *A. pustolans* infestation, getting lignified causing the epidermal layer to swell up and forming cup shaped around the insect [8]. They also cause branch dieback and green branches are sharply elbowed at the nodes with pits[9]. Thus, the fig fruits are considerably affected by reducing quality and diminishing their commercial value.

The chemical insecticides are often associated with residues that are dangerous for the consumer and the environment and at certain doses are toxic to humans and other animals, and some insecticides are suspected to be carcinogens[10-11]. The number of insect species with confirmed resistance to synthetic pesticides has continued to rise, apart from the risks associated with the use of these chemicals[12-13]. Furthermore, for the possibility of producing quality foodstuffs, it is necessary, among other things, to reduce the risks associated with excessive application of high pesticide doses in primary agricultural production. The current trend is the search for and use of alternative methods to manage pests, which, in the economic context, are effective without presenting the risks associated with the use of conventional pesticides.

An interest in natural products from plants has been increased due to the disruption of natural biological control systems, undesirable effects on non-target organisms, environmental hazards, and the development of resistance to synthetic insecticides, which are applied in order to reduce the populations of insects. Essential oils (EOs) from plants may be an alternative source of insect control agents, since they constitute a rich source of bioactive compounds that are biodegradable into nontoxic products and potentially suitable for use in integrated management programs. These materials may be applied to food crops shortly before harvest without leaving excessive residues. Furthermore, medically safe of these plant derivatives has emphasized also. For these reasons, much effort has been focused on plant EOs and their constituents as potential sources of insect control agents.[14-17]**.** As the result that found by [18], there has been an increased interest in developing potential alternative or additional control methods or materials that are effective against the target vector species, environmentally safe, biodegradable, with low cost, and can be used by individuals and communities in specific situation.

The objective of the present work was to evaluate the influence of some essential oils as well as chemical compound and some petroleum oils and their mixture against *Ceroplastes rusci* L. and *Asterolcanium pustolans* Cock on fig trees.

Experimental

Chemicals used:

1. Nimbecidine EC:

0.03% Azadirachtin is a neem-oil-based botanical insecticide containing Azadirachtin and other limonoids including Meliantriol, Salanin, Nimbin and a host of other terpinoids in the ratio as it occurs naturally in Neem. Formulated by T. Stanes & Company Limited, India was used at concentration 4ml/L.

2. Essential oils:

Three herbs belonging to three plant family, from Fam. Cruciferae, Black mustard, *Brassica nigra* Koch; from Fam. Burseracae Olibanum, *Boswellia carterii* Birdw. and from Fam. Ranunculaceae, Black cumin, *Nigella sativa* L. were purchased from a local market in Cairo, then blended to a fine powder using electric blender and preserved for extraction. Oils extraction of the plant was prepared using different methods; using (70%) ethanol and (70%) methanol (alcoholic extract). Extraction was made by placing each extract in a soxhelt reflux apparatus and completely evaporated by rotary evaporator [19]. The dried extracts were then weighed and stored in 40^{o}C for further tests. Essential oils were used with 2.5% concentration.

3. Mineral oils:

Two types of oils, specifically formulated for pest control were used:

a. **KZ oil 95%** the miscible type formulated by Kafr El-Zayat Co.
b. **Super Royal oil 85.7 %** El-Gameia El-Taawnia for petrol Company.

4. Malathion 57% E.C. Organophosphorus compound.

1- Comparative toxicity of different treatment to the fig scale insects.

Laboratory bioassay experiments were carried out to evaluate the relative efficacy of seven treatments, Nimbecidine EC**,** Essential oils, (*B. nigra*, *B. carterii* and *N. sativa*), KZ oil 95%, Super Royal oil 85.7 % and Malathion 57% E.C. against the *Ceroplastes rusci* L. and *Asterolcanium pustolans* Cock. Dipping method was used under laboratory conditions (30±1 ^{o}C and 62±5% R.H.). A serial of concentrations for each of the tested treatments were prepared by diluting the EC end product of each tested compound in distilled water. Infested fig branches with *Ceroplastes rusci* and *Asterolcanium pustolans* were transferred to the laboratory, where they were dipped into the insecticide solution for 10 seconds. Fifty branches (5 cm long) were used for each concentration (10 branches per replicate).

The control branches were dipped in distilled water only. Both treated and untreated branches were left for dryness, and then kept in polyethylene bags. Counts of the alive and dead adults were carried out after three days from treatment. Corrected mortality counts for adults according to [20] were statistically analyzed by [21].

2- Efficiency of the tested compounds against scale insects:

An orchard of fig trees, *Ficus carica* L. naturally infested with the aforementioned insects and showing different degrees of shriveling, was chosen at El-Kharouba village, North Sinai Governorate. The tree was 12 years' old and about 2.5 m in height. For the individual compounds, 12 trees were arranged in 3 replicates, 4 trees each. As for the combined effect study, anther 2 treatments (Nimbecidine + Super Royal oil and Nimbecidine + *B. nigra* oil) were set up in the same manner. Rows of fig trees were left, as borders among the treatments, to avoid any spry drift. All spray applications were made once on December, 2014 using knapsack sprayer 20 L capacity, to cover stems, branches and twigs; where most of the leaves were fallen. The concentrations of the test compounds are cited in tables (3 and 4).

In the meantime, the combined effect against both test insects was evaluated, using low concentration of Nimbecidine EC (2ml/L) mixed with either Super Royal oil or *B. nigra* oil (table 5).

Five of 2nd year growth green twigs, were randomly selected from each tree and cut off at the point of origin from the main branch. Each twig was trimmed to 20 cm in length and the small side branches were removed. The twigs were kept in plastic bags and transferred to the laboratory where they were examined. The number of living adult females per twig both before and after application was recorded in each treatment, (including control plots) and used as an index for the population density of the scales. Pretreatment counts were taken immediately before spraying application, whereas post treatment counts were taken 0.5, 1, 1.5 and 2 months after application.

Evaluation of all treatments was based on the reduction of the population density of alive individuals per replicate according to [22], data were statistically analyzed using[23].

Results and Discussion

1- Comparative toxicity of different treatment to the scale insects.

Seven compounds, Nimbecidine EC, Black mustard, *B. nigra,* Olibanum, *B. carterii* and Black cumin, *N. sativa*, Malathion, Kz- oil and Super royal were evaluated versus the fig waxy scale, *Ceroplastes rusci* and pustule scale insect *Asterolcanium pustolans* following the dipping technique [24-26]. Results in table (1) show the potency of the control agents against the *C. rusci*. Considering the LC_{50} value, the Nimbecidine EC showed the most effect whereas that of Super Royal oil was the least in this concern. The toxic effect of Nimbecidine EC compound was 61.5 folds as toxic as that of super royal oil at LC_{50} level (table 1). Regarding the toxicity index referring to Nimbecidine EC compound that level for Malathion compound was 15.00%. The toxicity index for the KZ oil and Super Royal recorded 2.47, 1.63%, respectively.

Table (1): Comparative toxicity of different treatment to the adult stage of *Ceroplastes rusci*

Treatment	LC 50	LC90	Slope	Confidence limit		*Index	** No of folds
				Lower	Upper		
Nimbecidine EC	0.0048	0.031	1.5	0.0032	0.0064	100	61.5
Brassica nigra oil	0.013	0.074	1.76	0.021	0.0052	36.90	22.7
Boswellia carterii oil	0.175	0.844	2.10	0.08	0.27	2.74	1.69
Nigella sativa oil	0.275	0.910	3.02	0.06	0.49	1.75	1.07
Malathion	0.032	0.311	1.03	0.0032	0.061	15.00	9.21
Kz- oil	0.194	0.886	2.19	0.158	0.23	2.47	1.52
Super royal	0.295	1.17	2.52	0.233	0.357	1.63	1

*= Index compared with Nimbecidine EC

**= Number of folds compared with Super Royal

Results in table (2) show the same manner of the potency of the control agents against the *A. pustolans*. Considering the LC_{50} value, the Nimbecidine EC showed the most effect whereas that of Super Royal oil was the least in this concern.

Table (2): Comparative toxicity of different treatment to the adult stage of *Asterolcanium pustolans*

Treatment	LC 50	LC90	Slope	Confidence limit		*Index	** No of folds
				Lower	Upper		
Nimbecidine EC	0.0042	0.021	2.00	0.0028	0.0056	100	65.5
Brassica nigra oil	0.084	0.820	1.02	0.072	0.096	5.0	3.3
Boswellia carterii oil	0.145	0.944	1.50	0.12	0.17	2.9	1.9
Nigella sativa oil	0.255	0.980	2.60	0.04	0.47	1.7	1.1
Malathion	0.031	0.211	1.50	0.02	0.042	13.5	8.9
Kz- oil	0.184	0.786	2.30	0.163	0.205	2.3	1.50
Super royal	0.275	1.070	2.60	0.206	0.344	1.5	1

*= Index compared with Nimbecidine EC

**= Number of folds compared with Super Royal

Starting with Nimbecidine EC, which gave the highest effect of *A. pustolans* with LC_{50} of 0.0042 followed descendingly by Malathion with 0.031, *B. nigra* oil with 0.084, *B. carterii* oil with 0.145, Kz-oil with 0.184, *N. sativa* oil 0.255 and Super royal 0.275. Many author's studies and found the similar results, [26] stated that laboratory studied showed highly superior susceptibility to neemix by different stage of *Ceroplastes floridensis* Com. on orange followed by other treatments. Abd El-Salam [27] found that different formulation of neem gave the highest effect against the adult stage of *Lasioderma serricorne* (F.) compared with Ethyl oleate oil.

The toxic effect of Nimbecidine EC compound was 65.5 folds as toxic as that of super royal oil at LC_{50} level (Table 2). Regarding the toxicity index referring to Nimbecidine EC compound that level for Malathion compound was 13.5%. The toxicity index for the KZ oil and Super Royal recorded 2.3, 1.5%, respectively; similar results were reported by [6].

2- Efficiency of the tested compounds against the fig waxy scale insect.

Data presented in table (3) indicated that, all tested compound gave satisfactory control against *C. rusci*. The reduction of infestation values, however, varied greatly with the time elapsed after spraying and according to the nature of each tested compound. The actual numbers of the females, in Nimbecidine EC treatment, for instance, were 13.6, 10.2, 9.4 and 6.6 corresponding to 30.3, 34.2, 33.8 and 34.3 in the respective control values 15 days , 1, 1.5 and 2 months after spraying, respectively. Thus, Nimbecidine EC was the superior compound, after the chemicals insecticides Malathion, giving an average of 73.02 % reduction throughout the whole experimental period (2 months). A schedule of sprays consisting of nimbecidine, garlic chilli kerosene extract followed by insecticide was found effective against chilli fruit borer, *Helicoverpa armigera* in recording least larval infestation and fruit damage, reported by[28].

The summer application of Actara, Dursban and Patron gave sufficient control of Fig Wax Scale. Fig Wax Scale fecundity was significantly affected by the different treatments[29]. Both Nimbecidine and Neemazal may have a significant effect on the reproduction of the cotton pest, *Earias vittella*. Therefore, less expensive natural biopesticide could be an alternative for chemical pesticides[30].

Application of Super Royal and Kz oils sharply decreased the population density in the first post-treatment count. The population, however, restored itself in the next successive inspections, giving an average of 67.6 or 66.4% reduction, respectively, throughout the whole experimental period table3. Similar results stated that KZ oil was the most effective oil at concentration of 2.0% and the nymphs were the most sensitive stage during a period of 1-3 months after application [31]**.** Sensitivity of some scale insects to insecticides; Basudin, Reldan, Sumithion, Oleoekaluk, Sumi oil and KZ oil tested **b**y [32]. They found that the nymphal stage was more susceptible, followed by adult females, while ovipositing females were less responsive. Simithion 50% (fenitrothion) (organophosphorus compounds), Admiral 10% EC (pyriproxyfen) (IGR), Super Masrona (mineral oil 94% EC), Admiral + oil and Jojoba extract on *P. oleae* in Ismailia. Obtained results revealed that oil alone or mixed with other compounds held superior category all over the time especially after three months of application[33].

The results in table 3 also cleared that Black mustard oil treatment caused pronounced effect where the number of insect dropped from 26.9 to 17.3 in the first post – treatment count. Afterwards, they remained constant (8.2 / 5 twigs) in the 4th post –treatment counts.

3- Efficiency of the tested compounds against the pustule scale insect:

Results in table (4) show that, the mean numbers of *A. pustolans* found on the fig trees before treatments, ranged from 42.3 to 56.2, indicating a relatively uniform distribution of insect infestation.

One month after spraying, the treatments suppressed the levels of infestation to different degrees compared to that of untreated control. Nimbecidine EC, Super royal and KZ oils significantly lowerd the percentage of infestation to 65.4, 69.8 and 73.7%, respectively, although they did not reach Malathion activity (79.1%). Two month post-treatment, Nimbecidine EC, became more efficient and had almost similar activity as the chemical insecticides Malathion displaying 69.03 and 64.9% reduction in infestation, respectively, table 4. Mineral oils were considered a promising control agent against wide varieties of pests. The petroleum oils proved to have considerable protection where 41.6 and 56.4 reduction was obtained for Super Royal and Kz oil, respectively, table 4.

The same manner was found when used kz, super royal and supermisrona at 1.5% at February in compared with the recommended spray. Alboleum 2% + malathion 0.15%[34-35]. Result indicated that's super misrona 1.5% gave superior effect, followed by kz oil and super royal oil. Similar results were reported by [36] *Earias vittella* was found to be very sensitive to the oil of *Eupatorium capillifolium* and *Callistemon lanceolatus* and the effect was time dependent. It should be mentioned that Nimbecidine EC exhibits multiple modes of action. It acts as an antifeedant repellent, ovi-position deterrent, insecticidal, insect growth regulator and sterilant.

The results in table 4 also showed that Black mustard oil among other essential oils, presented moderate activity; giving an average of 56.4% reduction, throughout the whole experimental interval. This was in accordance with[37,6] .

4- Joint action of certain mixtures against the tow insect populations

The possibility of mixing low concentration of Nimbecidine (2ml/L) with Super Royal or Black mustard oil was also investigated. The data showed that all mixtures proved to be promising to control the population of both species. For the waxy scale insect, the average reduction throughout the whole experimental period was recorded 76.4% when Super Royal was mixed with Nimbecidine table 5, while application alone average recorded 66.4 and 73.02% for Super Royal and Nimbecidine, respectively (table 3). The increased may reflect the potentiation effect between Nimbecidine and petroleum oil. Such potentiation was observed in most other mixtures. The mixture (Nimbecidine+ Black mustard oil) also displayed pronounced activity against the pustule scale insect, where the reduction value reached to 61.8% while it was 68.5% of the Malathion insecticides (table 5).

It should be noted that *C. rusci* was more susceptible, to all treatments than *A. pustolans*. This observation was demonstrated particularly in case of Nimbecidine applied either individually (tables 3, 4) or mixed with other tested oils (table, 5). Mixing mineral oils with conventional pesticides gave the highest % reduction in infestation with whitefly, *Bemisia tabaci* Genn. attacking tomato crop, while mineral oils or pesticides alone gave lowest reduction % in infestation[34-35]. Adding 1.5 % super royal oil to the lower concentration of neemix (0.025) displayed potential effect to the *Ceroplastes floridensis* Com. pest population on orange trees[26]. The miscible oils bio-dux, cabl-2 and citrole recorded highly reduction percent on *Kilifia acuminata* (Signoret) (Hemipera: Coccidae) infesting mango trees [38].

Table (3) Efficiency of different treatments applied against *Ceroplastes rusci* L. infesting fig trees

Treatment	Conc.	Mean number on insects /replicate and % reductions										
		Before treatment	15 day		1 month		1.5 month		2 month		Average	
			Mean No.	% R.	Mean No.	% R.	Mean No.	% R.	Mean No.	% R.	Mean No.	% R.
Nimbecidine EC	4ml/l	31.4a	13.6b	59.4	10.2c	73.0	9.4c	74.8	6.6c	82.6	9.9c	73.02
Brassica nigra oil	2.5	26.9a	17.3b	39.7	13.9b	57.1	12.3b	61.6	8.2c	74.8	12.9b	58.97
Boswellia carterii oil	2.5	27.7a	23.2a	21.5	18.3b	45.1	13.7b	58.4	10.2c	69.5	16.4b	49.4
Nigella sativa oil	2.5	28.0a	22.1a	26.02	17.1b	49.3	15.2b	54.4	10.7c	68.4	16.3b	50.2
KZ oil	1.5	28.0a	11.2b	62.5	8.5c	74.8	7.4c	77.8	15.3b	54.8	10.6c	67.6
Super royal	1.5	28.8a	12.1b	60.6	9.4c	72.9	9.3c	72.9	14.2b	59.2	11.3b	66.4
Malathion	1.5	28.7a	9.8c	67.9	8.6c	75.1	7.1c	79.2	7.8c	77.5	8.3c	75.3
Control	0.0	28.4a	30.3a	-	34.2a	-	33.8a	-	34.3a	-	33.2a	-

Mean within a column followed by the same letter are not significantly different at 5% level.

Table (4) Efficiency of different treatments applied against *Asterolecanium pustolans* Cock. infesting fig trees.

Treatment	Conc.	Mean number on insects /replicate and % reductions										
		Before treatment	15 day		1 month		1.5 month		2 month		Average	
			Mean No.	% R.	Mean No.	% R.	Mean No.	% R.	Mean No.	% R.	Mean No.	% R.
Nimbecidine EC	4ml/l	48.2a	27.9b	56.4	22.3b	65.4	17.8cd	73.4	25.6c	69.03	23.4c	66.3
Brassica nigra oil	2.5	52.3a	41.3b	40.5	30.2b	56.8	25.6c	64.7	34.6b	61.4	32.9c	56.4
Boswellia carterii oil	2.5	47.9a	38.3b	39.7	27.8b	56.6	26.2c	60.6	33.2c	59.6	31.4c	54.5
Nigella sativa oil	2.5	42.3a	34.3b	38.9	23.5b	58.5	23.6c	59.8	37.2a	48.7	29.7c	51.3
KZ oil	1.5	56.2a	28.6b	61.6	19.8b	73.7	36.4c	53.4	56.3a	41.6	35.3c	56.4
Super royal	1.5	55.1a	26.7b	63.5	22.3b	69.8	34.5c	54.9	41.2b	56.4	31.3c	60.6
Malathion	1.5	52.1a	20.9b	69.8	14.6c	79.1	22.3c	69.2	31.3c	64.9	22.3c	70.3
Control	0.0	43.5a	57.7a	-	58.2a	-	60.4a	-	74.6a	-	62.7a	-

Mean within a column followed by the same letter are not significantly different at 5% level.

Table (5). Efficiency of certain mixtures applied against *C. rusci* L. and *A. pustolans* Cock. infesting fig trees.

Treatment	**Conc. %**	**Mean number/5 twigs and % reductions in infestation after spraying**										
		***Ceroplastes rusci* L.**										
		Before Treatment	**15th day**		**1 Month**		**1.5 Month**		**2 Month**		**Average**	
			M.	**%R**	**M.**	**%R**	**M.**	**%R**	**M.**	**%R**	**M.**	**%R**
Nimbecidine + Super royal	2ml/l+1.5	31.5a	3.2c	91.9	7.5b	78.7	13.2b	64.03	10.8c	70.14	8.7b	76.4
Nimbecidine + *Brassica nigra*	2ml/l+2.5	32.0a	4.9c	87.8	8.3b	76.8	15.2b	59.2	11.5c	68.7	10b	73.3
Malathion	1.5	33.2a	10.8b	74.03	9.1b	75.5	8.5c	78.02	7.9c	79.3	9.1b	76.6
Control	0.0	29.7a	37.2a	-	33.2a	-	34.6a	-	34.1a	-	34.8a	-
Treatment	Conc. %	Mean number/5 twigs and % reductions in infestation after spraying										
		Asterolecanium pustolans Cock.										
		Before Treatment	15th day		1 Month		1.5 Month		2 Month		Average	
			M.	%R	M.	%R	M.	%R	M.	%R	M.	%R
Nimbecidine + Super royal	2ml/l+1.5	58.2a	30.1b	50.4	22.4b	61.1	19.2b	65.4	17.5b	72.0	22.3c	62.3
Nimbecidine + *Brassica nigra*	2ml/l+2.5	55.4a	27.9b	51.7	22.5b	59.0	18.9b	64.2	16.5b	71.4	21.5c	61.8
Malathion	1.5	60.0a	25.1b	59.9	20.1b	66.2	15.1b	73.6	16.3b	73.9	19.2c	68.5
Control	0.0	57.9a	60.4a	-	57.3a	-	55.2a	-	62.1a		58.8a	-

Mean within a column followed by the same letter are not significantly different at 5% level.

The present results concluded that Nimbecidine, the petroleum oils and Black mustard oil could be used successfully for controlling both insects on fig trees. The combined effect of Nimbecidine with some oils may prolong the activity as an additional privilege for protecting fig trees from insect attack.

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