J. of Plant Protection and Pathology, Mansoura Univ., Vol. 16 (9):437 - 443 , 2025

Journal of Plant Protection and Pathology

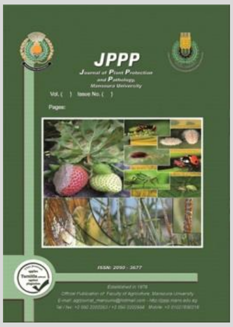
Journal homepage & Available online at: www.jppp.journals.ekb.eg

Effect of Formulated Petroleum oil solo and mixed with Hexamine as novel substituents of pesticides against black bean aphid *Aphis fabae* Scop. Infesting *Vicia faba* L. plants

Cross Mark

Eskander, M. A.[1]* and Raghda M. Hassan[2]

[1]Pesticides, Formulation Department, Central Agricultural Pesticide Laboratory (CAPL) Agricultural Research Center, 12618-Dokki-Giza, Egypt.
[2]Bioassay Department, Central Agricultural Pesticide Laboratory (CAPL) Agricultural Research Center, 12618-Dokki-Giza, Egypt.



Article Information
Received 3/ 8 /2025
Accepted 14/ 9/2025

ABSTRACT

Oils are among the safest and most effective substitutes for synthetic insecticides, and they have been used as pesticides for centuries. Hexamine is an organic compound used in various industrial applications, included as precursor for synthesis of other chemicals like plastics and pharmaceuticals. The main goal of this research paper is formulating mineral oil with hexamine mixture in suitable formulations and measures their toxicity against *aphis fabae* as safety alternatives of chemical insecticides. Mineral oil formulated in emulsifiable concentrates (EC), hexamine as soluble powder (SP) and mineral oil with hexamine mixture as oil in water emulsion (EW), they all passed the designated examinations. Toxicity of prepared formulations determined in laboratory against nymphs, adults and winged stages of *aphis fabae* and the results obtained indicated that mineral oil with hexamine mixture (EW) displayed the greatest toxicity against all aphids stages after 72 hr. of treatment; however, nymph stage showed the most susceptible to tested formulations followed by adults and winged stages. The toxicity was increased gradually by the time after treatment and with the increasing of the concentration used. Results of greenhouse experiment showed clearly that the mixture of mineral oil with hexamine (EW) was the best formulation for control *Aphis fabae* infested faba bean plants and increased the efficacy more than ten days followed by hexamine(SP)and mineral oil(EC). On the other hand, mineral oil (EC) formulation showed excellent efficacy five days then decreased. Finally, mineral oil with hexamine formulation could be used in control of *Aphis fabae* infested faba bean.

Keywords: Mineral oil, Hexamine, *Aphis fabae, Vicia faba*, formulation.

INTRODUCTION

Mineral oils have a number of advantages over traditional pesticides, including a shorter residual life, a lower potential for insect pests to become resistant to them, and a lower toxicity to natural enemies and other non-target animals, such as vertebrates. Because of these qualities, using oils is far more sustainable over the long run (Martin- Lopez, *et al.,* 2006; Sharma *et al.,* 2017). It is known that mineral oils can breakdown internal lipids, break through the insect's cuticle, and then reach the internal cellular structures (Najar-Rodriguez *et al.,* 2008). Nevertheless, there are certain disadvantages for the usage of mineral oils, which have reduced their acceptance in pest control initiatives. Due mostly to insufficient coverage of foliage and plant development between treatments, oils have relatively poor and variable efficacy in the field (Boiteau et al., 2009). It is recognized that higher concentrations of mineral oils are phytotoxic. (Hodgkinson, et al., 2002). Other than the treated leaves, mineral oils are not known to become systemic within plants (Fageria *et al.,* 2014b). As a result, several sprays are required to cover the newly emerging leaves. Spraying twice a week throughout the early season and later at weekly intervals is standard procedure. (Groves *et al.,* 2009). Aphid flying activity is a crucial aspect to be considered in addition to the fast crop growth. (Dupuis, *et al.,* 2017b)

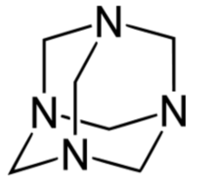
People in many parts of the world depend on the faba bean (*Vicia faba* L.) as a crop. In Egypt, it has become especially significant as a winter legume crop. It is regarded as one of the most popular foods in Egypt and has high protein content. (Saleh, *et al.,* 2021). Aphids in particular are sucking pests that attack bean crops, causing significant loses. (Abdel-Alim, 1994; Mahmoud *et al.,* 2017; El- Sarand *et al.,* 2019). Black bean aphid (*Aphis fabae* Scop.) is spread almost worldwide (Esmaeili- Vardanjani, *et al.,* 2013) It occurs throughout Europe, Western Asia, Africa and South America (Meradsi and Laamari, 2018). It targets every part of the plant, but it prefers to drop nymphs on the tops of newly emerging plants. (Stoddard, *et al.,* 2010) resulting in leaf deformation and growth suppression based on the duration of the infestation and the degree of colonization (Goszczynski, *et al.,* 2002). The purpose of this research is formulating and testing the toxicity of mineral oil and its mixture with hexamine against *Aphis fabae* infesting *Vicia faba* plants in greenhouse as a cheap, available and safety alternatives of chemical pesticides.

MATERIALS AND METHODS

1- Tested chemical.

a) Hexamine. It is a heterocyclic organic compound with the chemical formula $(CH_2)_6N_4$

* **Corresponding author.**
E-mail address: magdi.eskander25@gmail.com
DOI: 10.21608/jppp.2025.409322.1364



b) Mineral oil. It consists of aromatic oils, paraffins, napthenes, and refined hydrocarbons derived from petroleum. Cutting and grinding fluids use a broad range of mineral oil compositions. Additives are used to change the lubricating characteristics for certain uses.

c) Surface active agent: Tween 20, Span 80, Triton X100, Sodium dodecyl sulfate (SDS) and Sodium lignosulfonate.

d) Solvents: Acetone, ethanol, xylene, and dimethyl formamide (DMF). All previous material was provided by El- Gomhoria Co. Cairo, Egypt.

2- **Physicochemical characteristics** of suggested materials.

- **Solubility:** It was determined according to (Nelson & Fiero, 1954).
- **Free acidity or alkalinity:** it was determined by using the same methods outlined in the WHO recommendation (1979).

3. Prepare Hexamine as 90 % (SP).

The most straightforward, affordable, simple to make, solvent-free, and environmentally friendly formulations are soluble powders. Different weights of Hexamine and wetting agents were mixed in a number of trials, and the following tests were performed: solubility, surface tension, foam, pH, and free alkalinity or acidity before and after storage at 54±20C for 14 days, according to CIPAC, 2002) to verify that the successfully prepared recipe is appropriate for use and manufacturing. (Eskander and Emara, 2025).

1. Physico-chemical properties of Hexamine 90 %(SP)

Solubility, and free acidity/ alkalinity were determined as mentioned before.

- **Surface tension:** it determined using Du-Nouy tensiometer according to ASTMD-1331 (2001)
- **pH:** The Cole-Parmer pH conductivity meter 1484-44 was used to determine it in accordance with (Dobrat and Martijn, 1995).
- **Foam:** It was measured using the methodology outlined by (CIPAC 2002).

4- Prepare mineral oil 90 % (EC):

By combining varying weights of mineral oil with emulsifier or blending emulsifiers and stirring until homogeneity was reached, mineral oil was made into emulsifiable concentrate (EC). An emulsion stability test was performed on prepared formulations using the procedure described in CIPAC MT 36.1(2002) to determine which was suitable and efficient for use and production.

5- Formulating hexamine with mineral oil as 45 % (EW):

Because oil in water emulsion formulations is good for the environment and effective against the target pest, they can also be used to mix two active chemicals with different modes of action to boost the effectiveness of a newly produced formulation. The same technique outlined by was used to create mineral oil with hexamine (EW) formulations. Salvica, *et al.* (2012). Numerous specialized tests were performed on prepared formulas: Prior to and after storage at 54 ± 2 0C for 14 days, emulsion stability, foam, and free acidity or alkalinity were assessed using the technique described by CIPAC (2002) to verify that the recipe is effective and appropriate for usage and manufacturing.

5. Physicochemical properties of mineral oil (EC) and mineral oil with hexamine mixture as (EW):

a) Viscosity: The Brookfield viscometer model DVII+Pro was used to determine it, and the technique described by ASTMD-2196 (2005).

b) Surface tension, PH, Foam, and free acidity or alkalinity was determined as shown before.

c) Emulsion stability test: It was carried out in accordance with FAO/WHO MT 36.3. (2010).

6- Physico-chemical properties of locally prepared formulations spray solution at field dilution rate (0.5 %):

a) Surface tension, Viscosity, and pH, were ascertained as previously stated.

b) Electrical Conductivity and Salinity: Is measured in µmhos, and the Cole-Parmer PH/Conductivity meter 1484-44 was used to measure it, according to Dobrat and Martijn (1995).

7. Bioassay

Leaf-dipping bioassay

The leaf-dipping bioassay method as described by Moores *et al.* (1996) with slight modifications was used. Faba bean leaves were dipped in the aqueous solution of prepared formulations for about 10 seconds, and allowed to dry on a paper towel. Leaves were then placed upside down on an agar bed in small Petri dishes (60 mm diameter). Ten individuals of every stage nymph, adults and winged of *Aphis fabae*. were placed on the treated leaf surface, while leaves dipped in water served as controls. Three replicates batches of 10 individual of each stage were used for each formulation concentrations, five concentrations 2, 1, 0.5, 0.25, and 0.125% were used for each formulation. Petri dishes containing aphids were kept in the rearing chamber until mortality was recorded after 24, 48, and 72 hrs. The mortality data were corrected according to Abbott's formula (Abbott, 1925).

Corrected mortality%=1- (n t after treatment/n Co after treatment) x 100

n= insect population

t= treated population

Co = control

Data analysis:

The LC_{50} and LC_{90} values, 95% confidence limits and the slopes of the regression lines were estimated by LDP-Line statistical computer program. (Houndété *et al*., 2010 b and Shadmany *et al*., 2015).

Greenhouse experiment:

The prepared formulation scored excellent toxicity against different stages of *aphis fabae*, an experiment carried out in greenhouse to assess the insecticidal effect and determined the appropriate formula and concentration used also to determine the phytotoxicity of the tested prepared formulation on faba plants. Faba bean plants grew in pots 2 kg capacity, 5 replicates for each treatment, and five pots served as an infested control. One week after germination the artificial infestation done and let to adapt and production under greenhouse conditions, one week later, three concentrations of each formulation prepared: 1, 0.5 and 0.25% and sprayed on infested plants using hand sprayer. The populations of aphids inspected at 1, 3, 5, 7, and 10 days before and after treatments. The reduction percentages of aphids/ leaf corrected according to (Henderson and Tilton 1955)

RESULTS AND DISCUSSION

1.Physicochemical characteristics of the components of formulation:

Testing the toxicity of any active ingredient need to study their physicochemical characteristics and formulate them in appropriate form to facilitate handling, storage, transport and application on the target under study. Table (1) displayed the physicochemical characteristics of mineral oil and hexamine as active ingredients, the results revealed that mineral oil was insoluble in water and miscible with acetone, xylene, ethanol and dimethylformamide (DMF). On the other hand, hexamine showed good solubility in water and was insoluble with acetone, xylene, ethanol and dimethylformamide (DMF). Mineral oil displayed an acidic property and its acidity value as H_2SO_4 was 0.08, while hexamine showed alkaline property and its alkalinity value as NaOH was 0.25. The obtained results in table (1) concluded that, mineral oil could formulate as emulsifiable concentrate (EC) and hexamine formulate as soluble powder (SP) formulation, and its mixture formulate as oil in water emulsion (EW).

Table 1. Physicochemical properties of tested materials mineral oil and hexamine.

Compound	% Solubility (W/V)					Free alkalinity as % NaOH	Free acidity as % H_2SO_4
	Water	Acetone	DMF	Ethanol	Xylene		
Mineral oil	Insoluble	miscible	miscible	miscible	miscible	-	0.08
Hexamine	Soluble	insoluble	insoluble	insoluble	insoluble	0.25	-

From the previous results hexamine displayed good solubility in water so it formulated as soluble powder formulation (SP) and the tests which confirm the success of this formula was done and results in table (2) indicated that the prepared formulation showed hundred percent solubility in water and slightly foam formed before and after accelerated storage, however slightly decrease in pH value was noticed after accelerated storage where it was 7.7 before and became 7.6 after storage. While the (SP) formulation showed slightly increase in alkalinity value after accelerated storage where it was 0.22 before and became 0.25 after storage.

Table 2. Physicochemical properties of the prepared SP formulation before and after accelerated storage

Formulation	Before storage						After storage					
	Solubility		Foam		pH	Free alkalinity as NaOH	Solubility		Foam		pH	Free alkalinity as NaOH
	S. W	H. W	S. W	H. W			S. W	H. W	S. W	H. W		
Hexamine (SP)	100	100	0	2	7.7	0.22	100	100	0	3	7.6	0.24

S.W.= soft water H.W.= Hard water

Mineral oil displayed insoluble in water so it could be formulated as emulsifiable concentrate (EC) using emulsifier or blend of emulsifiers to facilitate its dilution with water during application against tested insect. Mineral oil mixture with hexamine prepared as oil in water emulsion (EW). Data in table (3) illustrate the physicochemical properties of prepared (EC) and (EW) formulations before accelerated storage at $54^0C\pm2$, and the results displayed that no oily or cream separation recorded or foam with soft and hard water. Mineral oil with hexamine mixture (EW) displayed viscosity value higher than mineral oil (EC) where their values were 24.34 and 18.13 centipoise respectively, also (EW) formulation revealed surface tension value 33.42 dyne/cm, while (EC) formulation revealed lower surface tension value 29.26 dyne/cm. mineral oil (EC) displayed pH value 5.33 and acidic property and its acidity value as H_2SO_4 was 0.142, while mineral oil with hexamine mixture indicated pH value 7.52 and alkaline property where its alkalinity value as NaOH was 0.152, these results slightly differ after accelerated storage at $54^0C\pm2$ for 14 days as illustrated in table (4) where the resulted indicated clearly that no any oily or cream separation recorded or any foam. On the other hand, little decrease in viscosity values with two prepared formulations, where it was 24.34 and 18.13 centipoise became 24.44 and 18.22 centipoise for (EW) and (EC) respectively. Surface tension revealed little decrease with (EW) and (EC) formulations, their values were 33.42 and 29.26 became 32.78 and 28.41 dyne/cm for (EW) and (EC) respectively. While slightly increase of pH values from 5.33 and 7.52 to 5.38 and 7.55 for (EC) and (EW) formulation respectively. Mineral oil (EC) revealed slightly decrease in acidity values from 0.142 before to 0.140 after storage, also mineral oil with hexamine mixture (EW) its alkalinity value changed from 0.152 to 0.149 after storage. The previous results from tables 2, 3, and 4 confirmed that the newly prepared formulations (SP), (EC) and (EW) could be successful and suitable for used in pest control under laboratory and open field conditions.

Table 3. Physicochemical properties of the prepared (EC) and (EW) formulations before accelerated storage.

Formulation	Viscosity centipoise	Surface tension dyne/cm	Emulsion stability ml cream separation		Foam		PH	Free acidity as H2SO4	Free alkalinity As NaOH
			H.W	S.W	H.W	S.W			
(EC)	18.13	29.26	0	0	0	0	5.33	0.142	-
(EW)	24.34	33.42	0	0	0	0	7.52	-	0.152

S.W.= soft water H.W.= Hard water

Table 4. physicochemical properties of the prepared EC and EW formulations after accelerated storage.

Formulation	Viscosity centipoise	Surface tension Dyne/cm	Emulsion stability ml cream separation		Foam		PH	Free acidity as H_2SO_4	Free alkalinity as NaOH
			H. W	S. W	H. W	S. W			
(EC)	18.22	28.41	0	0	0	0	5.38	0.140	-
(EW)	24.44	32.78	0	0	0	0	7.55	-	0.149

S.W. = soft water H.W. = hard water

2- Bioassay:

After prepared the tested materials in suitable formulation form and passed successfully of all specified tests that confirm the suitability for production and application. The toxicity of these prepared formulations against different stages of black bean *Aphis fabae* determined under laboratory conditions. Results illustrated in tables (5) and (6) and figures (1&2) revealed that all tested formulations displayed 100% mortality at concentrations 1 and 2% after 72 hrs. of treatment against nymph stages of *Aphis fabae*, while mineral oil with hexamine mixture (EW) and hexamine (SP) displayed 100% after 48 hrs. of treatment at 2% concentration followed by mineral oil (EC) which obtained 94.17 % mortality at the same concentration. Also, after 48 hrs. at 1% concentration mineral oil with hexamine mixture (EW) revealed the best mortality percentages followed by hexamine (SP) and mineral oil (EC), where their mortality values were: 98.78, 92.00 and 88.72% respectively.

Data in tables (7) and (8) and figures (3&4) obtained the results of tested formulations against adults of *A. fabae* in laboratory experiment. The prepared formulations (EC), (SP) and (EW) displayed great toxicity (100%) against adults of *A. fabae* with the highest tested concentration 2% after 72 hrs. of treatments, however mineral oil (EC) formulation indicated the highest toxicity value 100% with the second concentration 1% followed by mineral oil with hexamine (EW) formulation 97.46, and hexamine (SP) formulation 94.25%. These results have been explained the role of mixing mineral oil with hexamine (EW) formulation, where this formulation containing 35% mineral oil and 10% hexamine and gave results comparable with mineral oil 90% (EC) and Hexamine 90% (SP).

Table 5. The toxicity of prepared formulations against nymphs of *Aphis fabae* under laboratory conditions.

Materials	Conc. (%)	% Mortality of aphids after 24 hrs.	48 hrs.	72 hrs.
Mineral oil 90% (EC)	2	76.88	94.17	100
	1	71.46	88.72	100
	0.5	58.16	72.81	86.94
	0.25	43.52	54.18	61.23
	0.125	31.41	36.22	40.11
Hexamine 90% (SP)	2	78.67	100	100
	1	73.81	92.00	100
	0.5	62.33	76.65	88.75
	0.25	48.17	57.36	65.51
	0.125	32.15	38.44	42.27
Mineral oil + Hexamine45% (EW)	2	89.18	100	100
	1	84.46	98.78	100
	0.5	71.15	82.33	91.78
	0.25	60.11	70.45	79.33
	0.125	45.25	51.82	57.91

Table 6. LC_{50}, LC_{90}, and Slope for prepared formulation against nymphs of *Aphis fabae* under laboratory conditions.

materials	24 hrs.			48 hrs.			72 hrs.		
	LC_{50}	LC_{90}	Slope	LC_{50}	LC_{90}	Slope	LC_{50}	LC_{90}	Slope
Mineral oil 90% (EC)	0.32	5.85	1.02 ± 0.132	0.18	2.21	1.19 ± 0.16	0.16	0.49	2.64 ± 0.26
Hexamine 90% (SP)	0.29	4.58	1.07 ± 0.14	0.19	0.83	2.03 ± 0.19	0.16	0.49	2.65 ± 0.27
Mineral oil +Hexamine 45% (EW)	0.16	1.98	1.16 ± 0.15	0.13	0.56	2.04 ± 0.22	0.11	0.42	2.12 ± 0.28

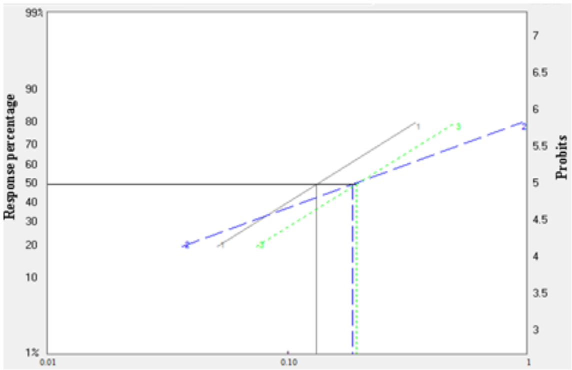


Figure 1. Ldp lines of tested formulations against nymph stage after 48 hr. (1= mineral oil + hexamine EW; 2= mineral oil EC and 3 = hexamine SP.

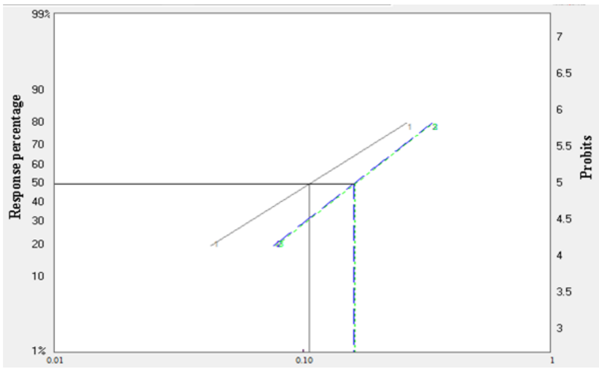


Figure 2. Ldp lines of tested formulations against nymph stage after 72 hr. (1= mineral oil + hexamine EW; 2= mineral oil EC and 3 = hexamine SP.

LC_{50} values displayed in table (8) revealed that the toxicity of all tested formulations increased by increasing the concentrations and time after treatment. On the other hand, decreasing the values of LC_{50} express about increasing the toxicity value, where the values of LC_{50} decreased by increased the time of treatment with all tested formulations, where the LC_{50} values for mineral oil (EC), hexamine (SP) and mineral oil with hexamine mixture (EW) were: 0.82, 0.79, and 0.62 ppm after 24 hrs. of treatment, while their values after 48 hrs. of treatments were: 0.29, and 0.31 and 0.25 ppm, however after 72 hrs. of treatment were: 0.19, 0.20 and 0.18 ppm respectively. These results agreeable with (Eskander, *et al.,* 2025) they reported that mineral oil with hexamine mixture (EW) formulation showed the strongest effectiveness against nymphs and adults of the mealybug *Paracoccus marginatus,* followed by the mineral oil (EC) formulation and the hexamine (SP) formulation.

Table 7. The toxicity of prepared formulations against adults of *Aphis fabe* under laboratory conditions.

Materials	Conc. (%)	% Mortality of aphids after 24 hr.	48 hr.	72 hr.
Mineral oil 90% (EC)	2	64.18	82.95	100
	1	52.93	78.83	100
	0.5	41.52	62.27	84.24
	0.25	32.62	44.45	55.11
	0.125	21.28	33.11	37.47
Hexamine 90% (SP)	2	68.77	87.84	100
	1	51.95	76.79	94.25
	0.5	40.33	60.44	81.33
	0.25	30.55	42.22	54.11
	0.125	19.20	30.64	34.34
Mineral oil + Hexamine 45% (EW)	2	71.46	92.87	100
	1	56.38	86.49	97.46
	0.5	44.88	64.59	88.14
	0.25	36.74	47.22	56.26
	0.125	23.11	34.21	38.55

Table 8. LC_{50}, LC_{90}, and Slope for prepared formulation against adults of *Aphis fabae* under laboratory conditions.

materials	24hr			48hr			72hr		
	LC_{50}	LC_{90}	Slope	LC_{50}	LC_{90}	Slope	LC_{50}	LC_{90}	Slope
Mineral oil 90% (EC)	0.82	18.83	0.94± 0.13	0.29	3.08	1.24 ± 0.15	0.19	0.58	2.66 ± 0.24
Hexamine 90% (SP)	0.79	12.01	1.09± 0.14	0.31	2.52	1.41 ± 0.15	0.20	0.76	2.24 ± 0.18
Mineral oil +Hexamine 45% (EW)	0.62	11.00	1.02± 0.14	0.25	1.56	1.68 ± 0.16	0.18	0.58	2.54 ± 0.24

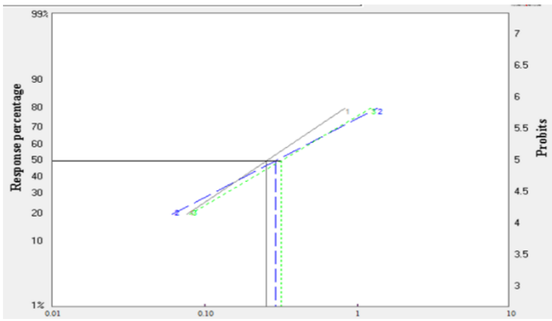


Figure 3. Ldp lines of tested formulations against adult stage after 48 hr. (1= mineral oil + hexamine EW; 2= mineral oil EC and 3 = hexamine SP.

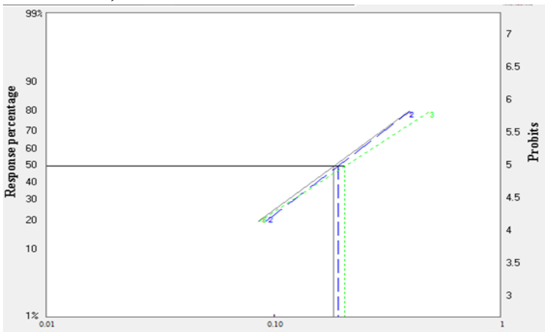


Figure 4. Ldp lines of tested formulations against adult stage after 72 hr. (1= mineral oil + hexamine EW; 2= mineral oil EC and 3 = hexamine SP.

For nymphs, their LC_{50} values were 0.0509, 0.0653, and 0.1015 ppm, whereas for adults, they were 0.0513, 0.0767, and 0.1224 ppm. Additionally, nymphs were more affected by these compositions' efficacy than adults were. Kraiss and Cullen (2008) observed nearly 100% soybean aphid (*Aphis glycines*) death 72 hours after mineral oil treatment in a lab setting.

An experiment conducted in laboratory to measure the toxicity of prepared formulations against winged aphis stage and data illustrated in table (9 and 10) and figures (5&6) revealed that all tested formulations showed good toxicity under laboratory conditions where mineral oil with hexamine mixture (EW) formulation displayed the highest mortality at the highest tested concentration 2% after 72 hr. of treatment followed by hexamine (SP) and mineral oil (EC), and its mortality values were: 92.59, 88.84 and 82.48% respectively. However, with 1% concentration its mortality values were: 80.45, 71.17 and 66.71% respectively. On the other hand, data in table (10) displayed the values of LC_{50} of tested formulation as follow: 0.91, 1.38 and 1.60% after 24 hr. and 0.41, 0.57 and 0.71% after 48 hrs. and 0.21, 0.33 and 0.4% after 72hrs of treatment for mineral oil with hexamine mixture (EW), hexamine (SP) and mineral oil (EC) respectively. That obtained clearly the toxicity increased by increasing the time after treatment and the concentration used.

Table 9. The toxicity of prepared formulations against winged of *Aphis fabae* under laboratory conditions.

Materials	Conc. (%)	% Mortality of aphids after 24hr.	48hr.	72hr.
Mineral oil 90% (EC)	2	51.18	70.93	82.48
	1	43.87	54.72	66.71
	0.5	35.23	40.14	55.63
	0.25	22.41	33.17	41.28
	0.125	14.35	22.46	25.19
Hexamine 90% (SP)	2	54.22	72.39	88.84
	1	45.78	58.27	71.17
	0.5	36.44	45.41	60.36
	0.25	26.14	37.71	41.82
	0.125	16.53	25.64	29.91
Mineral oil + Hexamine 45% (EW)	2	61.22	76.18	92.59
	1	52.66	67.27	80.45
	0.5	41.19	54.17	71.21
	0.25	30.37	41.11	56.44
	0.125	21.44	27.33	37.22

Table 10. LC_{50}, LC_{90}, and Slope for prepared formulation against winged of *Aphis fabae* under laboratory conditions.

materials	24hr			48hr			72hr		
	LC_{50}	LC_{90}	Slope	LC_{50}	LC_{90}	Slope	LC_{50}	LC_{90}	Slope
Mineral oil 90% (EC)	1.60	40.36	0.91 ± 0.14	0.71	11.62	1.05 ± 0.14	0.40	4.07	1.27 ± 0.14
Hexamine 90% (SP)	1.38	38.78	0.88 ± 0.14	0.57	10.93	1.00 ± 0.14	0.33	2.77	1.38 ± 0.15
Mineral oil +Hexamine 45% (EW)	0.91	23.54	0.91 ± 0.14	0.41	6.11	1.10 ± 0.14	0.21	1.72	1.39 ± 0.15

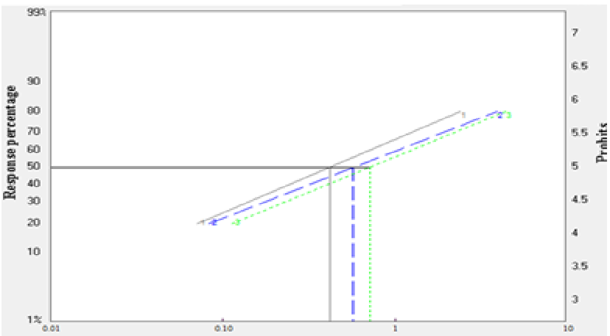


Figure 5. Ldp lines of tested formulations against winged stage after 48 hr. (1= mineral oil + hexamine EW; 2= hexamine SP and 3 = mineral oil EC.

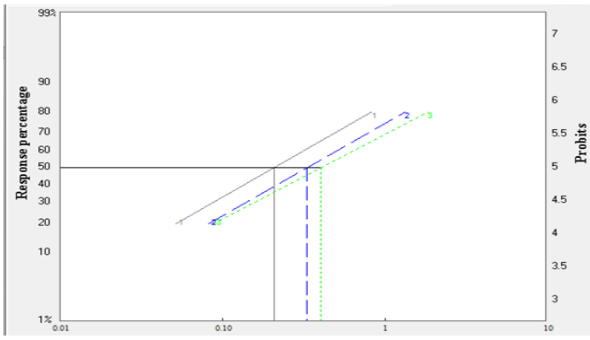


Figure 6. Ldp lines of tested formulations against winged stage after 72 hr. (1= mineral oil + hexamine EW; 2= hexamine SP and 3 = mineral oil EC.

Evaluation under greenhouse conditions:

Green house experiment carried out at winter season of 2025 to evaluate the efficacy of prepared formulations against *Aphis fabae* infested broad bean plants growing in pots to confirm the great efficacy displayed under laboratory conditions. All prepared formulations displayed good insecticidal efficacy against *A. fabae*, the results illustrated in table (11) revealed that mineral oil with hexamine mixture (EW) formulation showed the highest reduction percentages of aphids number/plant 24 hrs. post treatment at the highest tested concentration 1% followed by mineral oil (EC) formulation and hexamine (SP) formulation at the same concentrations, and their reduction % values were: 51.74, 45.72 and 42.44% respectively. However, five days of spraying the prepared formulations, the values of reduction percentages were: 94.84, 90.63 and 88.84% for (EW), (EC), and (SP) respectively. On the other hand, after 10 days of treatment the efficacy of tested formulations decreased by increasing the period after spraying, and decrease the tested concentrations where the % reduction were: 88.62, 72.43 and 54.48% for mineral oil with hexamine mixture (EW), Hexamine (SP), and mineral oil (EC) respectively. This agreed with Singh and Nagaich (1976) found that all of the peach-potato aphids (*Myzus persicae*) died after a few hours of 2% mineral oil being sprayed on potato plants, but that the aphids returned a week later. Also, (Yankova *et al.,* 2009) discovered that the population of *M. persicae* on pepper in a greenhouse decreased by more than 80% when the mineral oil Akarzin was applied at a rate of 0.4%.

Table 11. Insecticidal efficacy of locally formulated materials against *Aphis fabae* under greenhouse conditions.

Materials	Conc. (%)	% Reduction of aphids number after				
		1 day	3 days	5 days	7 days	10 days
Mineral oil 90% (EC)	1	45.72	67.41	90.63	68.82	54.48
	0.5	36.45	48.66	65.22	53.42	44.72
	0.25	26.55	37.49	46.73	40.82	36.33
Hexamine 90% (SP)	1	42.44	64.28	88.84	81.77	72.43
	0.5	34.22	45.88	63.76	61.57	60.59
	0.25	23.89	35.39	44.67	42.89	41.22
Mineral oil + Hexamine 45% (EW)	1	51.74	72.56	94.84	90.73	88.62
	0.5	38.92	53.22	68.52	66.54	64.39
	0.25	27.41	40.22	47.33	45.66	43.88

CONCLUSION

The locally prepared formulations, Mineral oil (EC), hexamine (SP) and mineral oil with hexamine mixture (EW) passed successfully of all specific tests that permits useable and production. Its toxicity assessed against nymphs, adults and winged stages of black bean aphids under laboratory conditions, all prepared formulations displayed excellent efficacy 100% against nymph stages after 72 hrs. of treatment with 1 and 2% concentrations. However, with adults the concentration 1% of mineral oil (EC) was the best followed by mineral oil with hexamine mixture (EW) and hexamine (SP). While winged aphis showed lower susceptible for the tested formulations where the concentration 2% after 72 hrs. of treatment displayed 92.59, 88.84 and 82.48% for mineral oil with hexamine mixture (EW), hexamine (SP) and mineral oil (EC). Greenhouse experiment was conducted and results concluded that, all prepared formulations were shown excellent efficacy till five days post treatment. While these results decreased at day 7 and day 10 post treatment. On the other hand, mineral oil with hexamine mixture (EW) formulation showed acceptable efficacy till ten days of treatment that's due to the two modes of action of the components of formulation.

REFERENCES

Abbot, W. S. A. (1925). A method of computing the effectiveness of an insecticide; J. Econ. Ent., 18(2), 265-267. https://doi.org/10.1093/jee/18.2.265a

Abdel-Alim, A.A. (1994): Ecological studies on certain insects infesting cowpea plants in Minia region. *Minia Journal Agriculture Research & Development,* 16(2): 261-273.

American Society of Testing Materials ASTM (2001). Standard Test Method for Surface and Interfacial Tension Solution D-1331.

American Society of Testing Materials ASTM (2005). Standard Test Method for Rheological Properties of Non – Newtonian Materials by Rotational (Brookfield type) Viscometer, D-2196 copyright ASTM, Bar Harbor Drive, West Conshohocken, PA 19248-2959, United States.

Boiteau G, Singh M, Lavoie J (2009) Crop border and mineral oil sprays used in combination as physical control methods of the aphid-transmitted potato virus Y in potato. Pest Manag. Sci. 65:255–259

CIPAC (2002). Collaborative International Pesticides Analytical Council Limits Hand book Vol. F. Physico- chemical Methods for technical and formulated pesticides.

Dobrat, W. &Martijn, A. (1995). Collaborative International Pesticides Analytical Council CIPAC hand book, F, Physicochemical Methods for Technical and Formulated Pesticides.

Dupuis B, Bragard C, Carnegie S, Kerr J, Glais L, Singh M, Nolte P, Rolot JL, Demeulemeester K, Lacomme C (2017b) Potato virus Y: control, management and seed certification programmes. In: Lacomme C, Glais L, Bellstedt D, Dupuis B, Karasev A, Jacquot E (eds) Potato virus Y: biodiversity, pathogenicity, epidemiology and management. Springer, Cham, pp 177–206

El-Sarand, E. A., Refaei, E. A. and El-Dewy, M. E. H.(2019): Population density of Empoasca Spp.; Liriomyza trifolii (Burgess) and the common natural enemies on faba bean plants in relation to sowing dates in Kafr El-Sheik Governorate. *Journal of Plant Protection and Pathology,* 10 (2): 147 – 153.

Eskander, M. A., and Emara, R. Azza (2025) Study the efficacy of formulated malic acid alone and its mixture with thiourea against *Fusarium oxysporium* in *Cucumis sativus* plants. J. of plant protection and pathology, Mansoura Univ., Vol. 16(2): 111- 118.

Eskander, M. A., Asmaa, A. Tharwat and Dalia Nabil (2025). Formulating hexamine, mineral oil, mixture and evaluation of its insecticidal activity against Papaya Mealybug *Paracoccus marginatus* (Hemiptera: Pseudococcidae) under laboratory conditions. I. J. Appl. & Nat. Sci. 17 (1), 285 -292 ISSN: 0974-9411 (Print), 2231-5209 (Online) journals.ansfoundation.org

Esmaeili-Vardanjani M., Askarianzadeh A., Saeidi Z., Hasanshahi G. H., Karimi, J., Nourbakhsh S. H. 2013. A study on common bean cultivars to identify sources of resistance against the black bean aphid, Aphis fabae Scopoli (Hemiptera: Aphididae). Archives of Phytopathology and Plant Protection, 46 (13): 1598–1608. https://doi.org/10.1080/03235408.2013.772351

Fageria MS, Boquel S, Leclair G, Pelletier Y (2014a) Quantification of mineral oil accumulation and movement in potato plants and its significance in potato virus Y management. Pest Manag Sci 70(8):1243–1248

FAO/WHO (2010). Manual on Development and Use of FAO and WHO Specifications for Pesticides, 1st Ed. 3rd Rev. FAO Plant Production and Protection, FAO, Rome, MT 36.3

Goszczyński W., Cichocka E., Leszczyński B. 2002. Beetroot damage due to the black bean aphid (Aphis fabae Scop) infestation. Electronic Journal of Polish Agricultural Universities, 5 (2): #02. http://www.ejpau.media.pl/volume5/issue2/horticulture/art-02.html

Groves R, Charkowski A, Crockford A, Coltman R, Hafner R, Bula K (2009) Integrated pest and disease management: reducing current season spread of Potato virus Y in potato. Phytopathol 99:S47

Henderson, C. F. and Tilton, E. W. (1955). Tests with acaricides against the brown wheat mite. J. Econ. Entomol., 48: 157- 161.

Hodgkinson MC, Johnson D, Smith G, Beattie GAC (2002) Causes of phytotoxicity induced by petroleum derived spray oil. In: Beattie GAC, Watson DM, Stevens ML, Rae DJ, Spooner-hart RN (eds) Spray oils beyond 2000- Sustainable pest and disease management: Proceedings of a Conference Held from 25 to 29October 1999 in Sydney, New South Wales, Australia. University of Western Sydney, Sydney, pp. 170–178.

Houndeté T. A., Kétoh G. K., Hema O. S., Brévault T., Glitho I. A. and Martin T. (2010 b): Insecticide resistance in field populations of *Bemisia tabaci* (Hemiptera: aleyrodidae) in West Africa. Pest Management Science; 66: 1181–1185.

Kraiss H, Cullen EM (2008) Efficacy and non-target effects of reduced-risk insecticides on *Aphis glycines* (Hemiptera: Aphididae) and its biological control agent Harmonia axyridis (Coleoptera: Coccinellidae). J Econ Entomol 101(2):391–398

Mahmoud, M. A.; K. A. El-Khawass; S. A. Hammad and M. I. Ali (2017): Effect of Temperature, Relative Humidity and Natural Enemies on some Insect Pests Infesting Faba Bean Plants at El-Monofia Governorate. *Mansoura Journal of Plant Protection and Pathology,* Vol.8 (9), 447 – 451.

Martin-Lopez B, Varela I, Marnotes S, Cabaleiro C (2006) Use of oils combined with low doses of insecticide for the control of Myzus persicae and PVY epidemics. Pest Manag Sci 62:372–378

Meradsi F., Laamari M. 2018. Behavioral and biological responses of black bean aphid (Aphis fabae, Scopoli, 1763) on seven Algerian local broad bean cultivars. Acta Agriculturae Slovenica, 111 (3): 535–543. https://doi.org/10.14720/aas.2018.111.3.02

Moores, G. D.; Gao, X.; Denholm, I. and Devonshire, A. L. (1996). Characterization of insensitive acetylcholinesterase in insecticide resistant cotton aphids, Aphis gossypii Glover (Homoptera: Aphididae). Pestic. Biochem. Physiol., 56: 102-110.

Najar-Rodriguez AJ, Lavidis NA, Mensah RK, Choy PT, Walter GH (2008) The toxicological effects of petroleum spray oils on insects–evidence for an alternative mode of action and possible new control options. Food Chem Toxicol 46(9):3003–3014

Nelson, F. C., &Fiero, G. W. (1954). Pesticide formulations, a selected aromatic fraction naturally occurring in petroleum as a pesticide solvent. Journal of Agricultural and Food Chemistry, 2(14), 735-737. https://doi.org/10.1021/jf60034a005.

Saleh H.A.; A.M. khorchid and Mona, I. Ammar (2021). Population fluctuations of two aphids and their main predators in broad bean plants in Qalyubiya Governorate. Egypt. Acad. J. Biolog. Sci., (A. Entomology) Vol. 14(1) pp.29-36 DOI: 10.21608/EAJBSA.2021.145808

Salvica G., Dragica B., Ljiljana R., & Andelka T. (2012). Development of water-based pesticides system. Journal Pesticides and Phytomedicine (Belgrade). 27 (1), 77-81. DOI: 10.2298/PIF1201077G

Shadmany M., Omar D. and Muhamad R. (2015): Biotype and insecticide resistance status of *Bemisia tabaci* populations from Peninsular Malaysia. Journal of Applied Entomology; 139 (1-2): 67-75.

Sharma PL, Verma SC, Chandel RS, Shah MA, Gavkare O (2017) Functional response of Harmonia dimidiata (Fab.) to melon aphid, Aphis gossypii Glover under laboratory conditions. Phytoparasitica 45(3):373–379.

Singh RA, Nagaich BB (1976) Effect of power oil on aphid transmission of potato virus Y. J Indian Potato Assoc 3:21–23

Stoddard F. L., Nicholas A. H., Rubiales D., Thomas J., Villegas-Fernández A. M. 2010. Integrated pest management in faba bean. Field Crops Research, 115 (3): 308–318. https://doi.org/10.1016/j.fcr.2009.07.002

World Health Organization, WHO (1979) Specification of Pesticides Used in Public Health, 5th Ed. Geneva.

Yankova V, Markova D, Todorova V, Velichkov G (2009) Biological activity of certain oils in control of green peach aphid (*Myzus persicae* Sulz.) on pepper. Acta Hortic 830:619–626

تاثير زيت البترول المجهز منفردا او مخلوطا مع الهكسامين كبدائل جديدة للمبيدات الحشرية ضد حشرة من الفاصوليا السوداء التي تصيب نباتات الفول البلدي

مجدي عدلي اسكندر[1] و رغدة محسن حسن[2]

[1]قسم بحوث مستحضرات المبيدات - المعمل المركزى للمبيدات- مركز البحوث الزراعية- الجيزة- مصر

[2]قسم التحاليل الحيوية - المعمل المركزى للمبيدات- مركز البحوث الزراعية- الجيزة- مصر

الملخص

تُعد الزيوت من أكثر البدائل أمانًا وفعاليةً للمبيدات الحشرية الصناعية، وقد استُخدمت كمبيدات حشرية لقرون. الهيكسامين مركب عضوي يُستخدم في تطبيقات صناعية متنوعة، بما في ذلك كمادة أولية لتصنيع مواد كيميائية أخرى كالبلاستيك والأدوية. الهدف الرئيسي من هذا البحث هو تجهيز الزيت المعدني مخلوطا مع الهيكسامين في صورة مناسبة، وقياس سميته ضد حشرة منّ الفول كبدائل آمنة للمبيدات الحشرية الكيميائية. زيت معدني مجهز في صورة مُركّزات قابلة للاستحلاب (EC)، وهيكسامين في صورة مسحوق قابل للذوبان في الماء (SP)، وزيت معدني مخلوطا مع هيكسامين في صورة مستحلب زيت في ماء (EW). وقد اجتازت جميعها الاختبارات اللازمة. حُدّدت سمية التركيبات المُحضّرة مختبريًا ضد الحوريات والبالغات والأطوار المُجنحة من حشرة المنّ، وأشارت النتائج إلى أن الزيت المعدني مع خليط الهكسامين (EW) أظهر أعلى سمية ضد جميع أطوار المنّ بعد ٧٢ ساعة من المعاملة؛ إلا أن طور الحوريات كان الأكثر تأثرًا بالتجهيزات المُختبرة، يليه طورا البالغين والأطوار المُجنحة.كما ازدادت السمية بزيادة مدة المعاملة والتركيز المستخدم. أظهرت نتائج تجربة الصوبة بوضوح أن خليط الزيت المعدني مع الهكسامين (EW) كان التركيبة الأمثل لمكافحة حشرة المن التي تصيب نباتات الفول البلدي ، وزادت فعاليته لأكثر من عشرة أيام، يليه الهكسامين (SP) والزيت المعدني (EC).من ناحية أخرى، أظهرت تركيبة الزيت المعدني (EC) فعالية ممتازة بعد خمسة أيام، ثم انخفضت. بناءً على هذه النتائج، يُمكن استخدام تجهيزة مخلوط الزيت المعدني مع الهكسامين في مكافحة حشرة المنّ على نبات الفول البلدي، وذلك بعد استكمال الدراسات اللازمة الأخرى.