2023, 22(2): 238-263 Online: 2735-5098

EVALUATION OF CONVENTIONAL AND NANOEMULSION FORMULATIONS OF SOME INSECTICIDES AGAINST THE COTTON APHIDS, *APHIS GOSSYPII* GLOVER (HEMIPTERA: APHIDIDAE)

Moustafa A. Abbassy, Mona A. Abdel-Rasoul, Belal S. M. Soliman*, Atef M.K. Nassar

Department of Plant Protection, Faculty of Agriculture, Damanhour University, Egypt

*Corresponding author: bsoliman91@gmail.com

ABSTRACT

The effectiveness of three insecticides, imidacloprid, lambda-cyhalothrin, and cvantraniliprole, in their conventional and nanoemulsion formulations, were evaluated under laboratory and greenhouse conditions using the recommended dose against the cotton aphids, Aphis gossypii Glover (Hemiptera: Aphididae). The prepared insecticide nanoemulsions exhibited polydispersity indices ranging from 0.198 to 0.324 and zeta potentials between -27.2 and -38.1 mV. The most minor insecticide nanoemulsion droplet diameter was lambda-cyhalothrin (83.25 nm), and the largest was imidacloprid (112.32 nm). Preparing the imidacloprid, lambda-cyhalothrin, and cyantraniliprole as nanoemulsion formulations increased the toxicity by by1.41, 1.78, and 1.25fold after 24 hr., and 1.46, 1.7 and 1.45-fold after 48 hr., respectively against A. gossypii under the laboratory condition. Lambda-cyhalothrin showed the highest toxicity against A. gossypii, with fifty lethal concentration (LC50) values of 5.313 ppm and 1.808 ppm after 24 hrs. and 48 hr., respectively. Under the greenhouse conditions, the general means of reduction percentages of A. gossypii populations caused by conventional formulation of lambda-cyhalothrin, imidacloprid, and cyantraniliprole were 88.2, 81.74 and 78.91 %, respectively. While the means for the nanoemulsion formulations were 95.17, 86.2 and 85.91 %, respectively.

2023, 22(2): 238-263 Online: 2735-5098

After the second spray, these means recorded 87.72, 82.78, and 82.18% for conventional formulations and 91.22, 88.23, and 87.31% for nanoemulsion formulations, respectively.

DOI: https://doi.10.21608/jaesj.2023.239529.1114

Keywords: Nanoemulsion, Imidacloprid, Lambda-cyhalothrin, Aphis gossypii, Cyantraniliprole

INTRODUCTION

The cotton aphid, *Aphis gossypii* Glover (Hemiptera: Aphididae), is an important insect pest of many crops worldwide (Blackman and Eastop, 2000). The cotton aphid is highly polyphagous. Crop plants attacked by this aphid include cucurbits, cotton, citrus, coffee, eggplant, peppers, potato, okra, and many ornamental plants. This aphid has been regarded as a significant pest of cucurbits and may build up to large populations on these crops (Mendoza, 2001; Blackman and Eastop, 1984). *A. gossypii* can cause two types of injury to the plant. First, it can transmit viruses, and second, it can cause direct injury. (Blackman and Eastop 1984). Chemical control remains the most significant, reliable, and effective way to protect many crops from infestation with insect pests such as cotton aphids.

The neonicotinoid insecticide imidacloprid is considered one of the most promising insecticides in pest control. This insecticide's mode of action concerns insects' central nervous system, with low side effects on mammals. The action of this insecticide is caused by interfering with the transmission of stimuli in the insect nervous system. This mechanism leads to blockage of the nicotinergic neuronal pathway. By blocking nicotinic acetylcholine receptors, imidacloprid prevents acetylcholine from transmitting impulses between nerves, resulting in the insect's paralysis and eventual death (Yamamoto *et al.*, 1995; Matsuda *et al.*, 1999). Lambda-cyhalothrin is a synthetic pyrethroid insecticide that controls various pests on food/feed crops and livestock. Lambda-cyhalothrin is a member of the type II pyrethroids. The action of Lambda-cyhalothrin is caused by the slow depolarization of nerve membrane, which reduces the amplitude of the action potential leading

2023, 22(2): 238-263 Online: 2735-5098

to a loss of electrical excitability (Simon, 2008; Ahamad and Kumar, 2023).

Cyantraniliprole is a second-generation commercialized ryanodine receptor insecticide discovered after chlorantraniliprole by DuPont, which is produced from anthranilic diamides to control lepidopteran and sucking insects, including critical species of aphids, whiteflies, and thrips (Sattelle *et al.*, 2008). The mode of action of cyantraniliprole relies on the activation of the ryanodine receptors of insects, which are critical for muscle contraction (Lahm, *et al.*, 2007). This activation of the ryanodine receptors affects calcium homeostasis by unregulating the release of internal calcium in the cell, which leads to feeding cessation, lethargy, muscle paralysis, and ultimately, death of the insect (Cordova *et al.*, 2006; Jacobson and Kennedy, 2011).

Due to the intensive use of conventional formulations of pesticides, the hazard and residues of pesticides have increased for nontarget organisms such as humans and beneficial insects (insect predators, parasites, and pollinators). To reduce this hazard, nanotechnology has introduced a new trend in pesticide formulation. This trend is called nanoformulation of pesticides. These nanoformulations include nanocapsules, nanoparticles. nanosuspensions, and nanoemulsion pesticides. Nanoemulsion is a complex system that contains an oil phase with the presence of surfactant and water. This nanoemulsion form has become a topic for many studies, uses, and applications (Salim et al., 2011). Thus, keeping in view the information mentioned above, this study aims to evaluate the effect of conventional and nanoemulsion of imidacloprid, lambdacyhalothrin, and cyantraniliprole against the cotton aphids, A. gossypii under the greenhouse and laboratory conditions.

MATERIALS AND METHODS

The tested insecticides

Three insecticides in their conventional and nanoemulsion formulation have been chosen in this study as follows: Imidacloprid (ImiDOR 35%SC), which was obtained from Chema Trade Company,

J. Agric. & Env. Sci. (Damanhour University	7)
Print: ISSN 1687-1464	

2023, 22(2): 238-263 Online: 2735-5098

Nasr City, Cairo, Egypt, at a rate of 75 ml/100 liter of water, Lambdacyhalothrin (Lambda 5 % EC) which obtained from Kafr El- Zayat Company for Pesticides and Chemicals 61 Al- Hussein Street, Mohandessin – Giza – Egypt. At a rate of 95 cm3/100 liter, Cyantraniliprole (Benevia 10 % EC) at a rate of 60 cm3/100 liter of water was obtained from FMC Company, US.

Nanoemulsion preparation

The nanoemulsions of lambda-cyhalothrin, imidacloprid, and cyantraniliprole were prepared in two phases, including coarse emulsions prepared by stirring and further emulsification using highenergy ultrasonication. First, each insecticide dissolved in toluene and butanol (1:1) as a common solvent (oil phase). The oil phase was slowly added to the aquatic phase (water and tween 80) with stirring at 4000 rpm for 30 minutes. The formed emulsions were then sonicated for 30 minutes at a sonication power of 10 kHz and pulses nine cycles/sec using an Ultrasonic Homogenizer (HD 2070 with HF generator GM 2070, ultrasonic converter UW2070, booster horn SH 213 G, and probe microtip MS 73, \emptyset 3 mm). The temperature difference between the initial and final coagulant emulsion was not more than 25°C (Badawy *et al.*, 2017; Shahavi *et al.*, 2019).

Characterization of insecticide nanoemulsion

Centrifugation assay: This analysis of nanoemulsion was performed by centrifuging the samples for 30 minutes at 5000 rpm and noticing phase separation, creaming, and cracking. The nanoemulsion should have maximum stability, not a phase separation (creaming and cracking) (Shafiq and Shakeel, 2010).

Freeze-thaw cycle: In this test, the formulations were subjected to two different temperatures (-21°C and 21°C.) for each temperature test of at least 24 hr. (Kadhim and Abbas, 2015). The separation or creaming layer was examined.

J. Agric. & Env. Sci. (Damanhour	University)
Print: ISSN 1687-1464	

Heating cooling test: The prepared nanoemulsions were maintained at four °C and 40°C with storage for 48 h per temperature test. Nanoemulsions stable at this temperature were subjected to further study (Kadhim and Abbas, 2015).

Viscosity and pH measurements: The dynamic (absolute) viscosity (μ) of the insecticide nanoemulsions was measured by a Rotary Myr VR 3000 digital viscometer with L4 spindle at 200 rpm at 25°C without further dil. Each reading was taken after the equilibrium of the sample for two minutes. The samples were repeated four times, and the data expressed in mPa.s. The observation of pH value follows from the determination of stability of nanoemulsion due to alteration of pH of the presence of chemical reactions. The digital pH meter was used to determine the pH values of prepared nanoemulsions.

Particle size, zeta potential, and poly polydispersity index (PDI): The average volume of droplets (PDI) and diameters of nanoemulsions were measured by dynamic light dispersion method (DLS) using Zetasizer Nano ZS (Malvern Instruments, UK) at room temperature. Before measurements, samples were diluted to 10% with deionized water to avoid multiple dispersion effects. The size of emulsion droplets was estimated at an average of three measurements and was shown as an average diameter in nanometers. PDI values <0.25 indicated a narrow size distribution providing good stability of nanoemulsions (Sobhani *et al.*, 2015; Su *et al.*, 2017). The stability of the Nano imidacloprid was assessed in terms of zeta potential using a Zeta Plus tool (Malvern Zeta size Nano-zs90).

Scanning electron microscopy (SEM): SEM analysis used a JEOL JSM-5410 (Japan) electron microscope with a W-source operating at 80 kV. The sample was prepared on a glass slide $(1 \times 1 \text{ cm})$ after washing it with ethanol. A tiny drop of nanoemulsion was spread evenly over the glass slide and allowed to air dry. To make it conductive, gold coating with Jeol Quick Auto Coater was performed (JFC-1500). The nanoemulsions were then subjected to SEM analysis under ambient conditions.

2023, 22(2): 238-263 Online: 2735-5098

Laboratory insecticidal evaluation: Toxicity testing of imidacloprid, lambda-cyhalothrin, and cyantraniliprole and their nanoemulsion against laboratory strain of the cotton aphids (maintained under conditions of 25±2°C and 65±5 %RH on castor bean leaves) was conducted. The leaf-dip bioassay technique was adopted from (Moores et al., 1996; Aydin et al., 2005) with minor modifications. Cucumber leaf discs (55 mm diameter) were dipped in insecticide solutions for 10 s and dried on a paper towel. The Serial dilutions of formulated compounds were prepared in distilled water containing 500 ppm Tween 20 (as a non-ionic surfactant). Cucumber leaves were dipped for 10 s into deionized water containing 500 ppm Tween 20 for controls. In Petri plates (60 mm diameter), leaves were placed upside down on an agar bed. On the treated leaf, fifteen apterous adults of A. gossypii were inserted. Each insecticide had six concentrations, and each concentration had four replicates. These concentrations were 1, 5, 10, 15, 20, and 40 ppm for cyantraniliprole and imidacloprid and 0.5, 1, 5, 10, 15 and 20 ppm for lambda-cyhalothrin. After 24 and 48 hours, the mortality rate was calculated and corrected by Abott's formula (1925). Adults were considered dead if they showed no coordinated forward movement when prodded with a fine paintbrush. The LC₂₅, LC₅₀, and LC₉₀ values and 95% confidence limits were calculated according to Finnev (1971)using LdP-line. Ehab Software (http://www.ehabsoft.com/ldpline/).

Insecticidal evaluation under greenhouse conditions: The current experiment was planned to compare the efficiency of the conventional and nanoemulsion formulations of lambda-cyhalothrin, imidacloprid, and cyantraniliprole against the cotton aphids *A. gossypii*. This experiment was conducted in a commercial plastic greenhouse in the Damanhour district, El Behera Governorate, Egypt (9 X 40 m). The greenhouse was planted on the 10th of May 2023 with cucumber (*Cucumis et al.*) (variety, white wonder). Except for applying any pesticides, all the standard agricultural practices for greenhouse cucumber (fertilization, irrigation, light intensity pinching, photoperiod, and temperature) were carried out regularly (Raymond, 2003).

Statistical analysis

J. Agric. & Env. Sci. (Damanhour	University)
Print: ISSN 1687-1464	

The randomized complete block design (RCBD) was applied with four replicates/treatments and four for control in 28 plots. Every plot was separated from the other plot by one meter to reduce interference from another treatment drift. The sample size was many alive aphids found on four leaves from four different plants (16 leaves/plot). All treatments were applied during the cucumber fruiting stage. Counts of aphids were recorded immediately before treatment and at 1, 4, 7, and 14 days after treatment. An ordinary hand sprayer of 20 liters capacity with a bent-down Nozzle was used. Reduction percentages of *A. gossypii* numbers were calculated according to Henderson and Tilton equation (1955) and subjected to analysis of variance (ANOVA) (CoStat *et al.*, 1998).

RESULTS AND DISCUSSION

Characterization of lambda-cyhalothrin, imidacloprid, and cyantraniliprole nanoemulsions

The physical and chemical characteristics of the nanoemulsion of three insecticides (lambda-cyhalothrin, imidacloprid, and cyantraniliprole) are presented in Table 1.

Appearance and particle size (droplet diameter)

The particle (droplet) size and PDI results revealed that the three nanoemulsion preparations achieved the target nanometric size range. According to Sadurní *et al.* (2005), the average size of a drop of oil-inwater type nanoemulsions usually falls within the range of 20 to 200 nm. The lambda-cyhalothrin nanoemulsion had a minor droplet diameter (83.25 nm), while the imidacloprid nanoemulsion had the largest droplet diameter (112.32 nm) (Fig. 1). The lambda-cyhalothrin nanoemulsion also had the lowest PDI value (0.198). At the same time, the highest (0.324) was observed in the imidacloprid nanoemulsion. According to Baboota *et al.* (2007), the polydispersity index reflects particle size distribution in the formulation; a PDI of 0.3 indicates a system heterogeneity, while a PDI of <0.2 refers to a uniform droplet size distribution and homogeneous population.

J. Agric. & Env. Sci. (Damanhour U	niversity)
Print: ISSN 1687-1464	

2023, 22(2): 238-263 Online: 2735-5098

Viscosity and pH measurement

The viscosity of the lambda-cyhalothrin, imidacloprid, and cyantraniliprole nanoemulsions were 3.94, 4.33, and 4.55 mPa.s., respectively. The pH values were 6.12, 6.43, and 6.46, respectively.

Zeta potential

The measuring nanoemulsion zeta potential is helpful for understanding and predicting the interactions between nano-particles. The estimated zeta potential values (Fig. 1) were -38.1, -27.2, and -28.6 mV for imidacloprid, lambda-cyhalothrin, and cyantraniliprole insecticide nanoemulsions, respectively. Overall, zeta potentials more significant than 25 mV or lower than -25 mV typically indicate high degrees of stability (Shi *et al.*, 2017). The results of leaving the nanoemulsion of the three insecticides in different storage conditions and thermodynamic stability (centrifugation at 5000 rpm, storage at 25°C, heating cycle, and cooling and freeze-thaw cycle) are tabulated in Table 1. The results indicated that all nanoemulsions passed a centrifugation test at 5000 rpm. Also, the results showed that the heating-cooling test indicated that all samples were stable.

The morphology and shape of the nanoemulsions were studied using scanning electron microscopy (SEM), as shown in Fig. 2, and the shape of the droplets was spherical. The results of characterizations of nanoemulsion of the three insecticides are in harmony with the results of Badawy, *et al.* (2019) who determined the particle size of five insecticides i. e. chlorpyrifos, malathion, cypermethrin, deltamethrin and lambda-cyhalothrin by 18.35, 177.2, 84.99, 24.42 and 79.05 nm, respectively, while the PDI values ranged from 0.121 to 0.377.

 Table (1): The physical and chemical characteristics of the nano-formulations of lambda-cyhalothrin, imidacloprid, and cyantraniliprole

2023, 22(2): 238-263 Online: 2735-5098

Pesticides	А	В	С	D	$pH\pm SD$	Е	PDI (nm)	Zeta potential
Imidacloprid			\checkmark	4.33±0.16	<mark>6.43</mark> ±0.051	112.32	0.324	-38.1
Lambda cyhalothrin		\checkmark	\checkmark	3.94 ± 0.2	6.12±0.036	83.25	0.198	-27.2
Cyantraniliprole			\checkmark	4.55±0.18	6.46 ± 0.035	89.17	0.255	-28.6

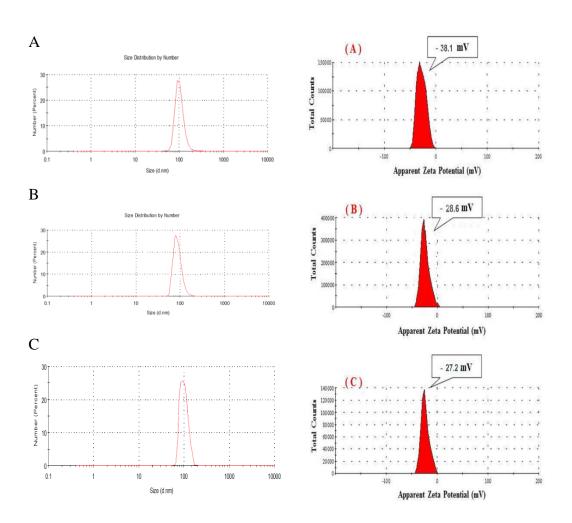
 $\sqrt{1}$ = the nanoemulsion formulation is stable without phase separation

A: Stability after centrifugation at 5000 rpm, B: Freeze thaw cycles, c: Heating-cooling cycle, D: Viscosity (mPa.s.) \pm SD, and E: Droplet size (nm)

Laboratory evaluation of tested insecticides and their nanoemulsions against *A. gossypii*

Results in Table (2) showed that, after 24 hr. of the exposure time, lambda-cyhalothrin nanoemulsion was the most effective insecticide against A. gossypii adults, followed by imidacloprid nanoemulsion and then cyantraniliprole nanoemulsion. The LC₅₀ values for the tested insecticides arranged in ascending order were as follows: lambda-cyhalothrin nanoemulsion (2.989 ppm with a confidence interval of 2.176- 3.964 ppm), imidacloprid nanoemulsion (4.758 ppm with a confidence interval of 1.807 - 7.737 ppm) and cyantraniliprole nanoemulsion (7.254 ppm with a confidence interval of 3.369 - 12.108 ppm). The toxicity index was employed to compare the efficiency of all tested insecticides at a fixed level (LC50) to their most effective insecticide. The toxicity index values were 62.82 and 41.2 %, as toxic as lambda-cyhalothrin nanoemulsion for imidacloprid nanoemulsion and cyantraniliprole nanoemulsion, respectively. The slop values for the nanoemulsion of imidacloprid, lambda-cyhalothrin, and cyantraniliprole were 1.658, 1.162, and 1.635, respectively.

The synthetic forms of lambda-cyhalothrin were the most effective insecticide against the adults of *A. gossypii* with lC_{50} of 5.313 ppm, compared with the synthetic forms imidacloprid (6.731 ppm) and finally synthetic forms of cyantraniliprole (9.103 ppm). Also, the toxicity index was employed to compare the efficiency of three tested compounds in their synthetic forms at a fixed level (LC₅₀) to their most effective insecticide. The values of the toxicity index were 78.93 % and 58.37 % as toxic as a synthetic form of lambda-cyhalothrin for imidacloprid and cyantraniliprole, respectively.

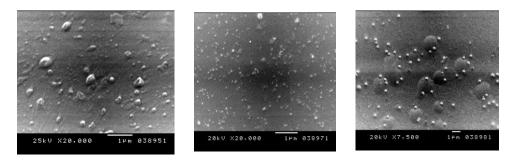


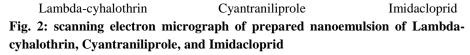
J. Agric. & Env. Sci. (Damanhour University) Print: ISSN 1687-1464

2023, 22(2): 238-263 Online: 2735-5098

Fig. 1. A typical particle size distribution by a dynamic light scattering (DLS) and Zeta potential distribution of the nanoemulsion formulations of A) Imidacloprid B) Cyantraniliprole, and C) Lambda-cyhalothrin

2023, 22(2): 238-263 Online: 2735-5098





Nanoemulsion formulations of lambda-cyhalothrin, imidacloprid, and cyantraniliprole were more toxic than conventional formulations, with relative efficiency of 0.437, 0.293, and 0.203 for imidacloprid lambda-cyhalothrin for and cyantraniliprole, respectively. After 48 hr. of the exposure time, based on data in Table (4), the results of tested insecticides against A. gosspii adults showed the lambda-cyhalothrin nanoemulsion was the most effective at the LC_{50} level (1.066 ppm with a confidence interval of 0.674-1.509 ppm), followed by Imidacloprid nanoemulsion (2.866 ppm with a confidence interval of 1.382-5.745 ppm) and cyantraniliprole nanoemulsion (4.466 ppm with a confidence interval of 1.57-7.289 ppm). Nano-lambdacyhalothrin was used as a standard in calculating the toxicity index due to its highest effect. Data in Table (4) illustrated that nano imidacloprid was 37.19%, followed by cyantraniliprole with 23.86% compared to Nano-lambda-cyhalothrin.

The LC₅₀ values of the synthetic formulation of lambdacyhalothrin, imidacloprid, and cyantraniliprole were 1.808 ppm, 3.744 ppm, and 6.512 ppm, respectively. After 48 hr. exposure time, the nanoemulsion formulations of lambda-cyhalothrin, imidacloprid, and cyantraniliprole were more toxic than that of the usual formulation with the relative efficiency of 0.41, 0.23, and 0.31 for lambda-cyhalothrin, imidacloprid and cyantraniliprole, respectively. After 48 hr. exposure time, nanoemulsion had the steepest slope with 1.136, 1.54, and 1.632 compared with 1.014, 1.581, and 1.784 for lambda-cyhalothrin,

2023, 22(2): 238-263 Online: 2735-5098

imidacloprid, and cyantraniliprole, respectively. The steepest slope indicates that the slightest change in the concentration of the insecticides caused high mortality compared with the least steep slope, which needed more concentration to give the same effect (percent mortality).

The current results are in harmony with the results of Barakat et al. (2023), who found that the LC_{50} values of acetamiprid, dinotefuran, and their nanoform were recorded at 3.566, 4.068, 0.590, and 0.705 ppm against A. craccivora, respectively. Also, the toxicity of acetamiprid against soybean Aphid, Aphis glycines was estimated as an LC50 value of 6.742 mg a.i./L with a confidence interval of 5.133-8.629 mg a.i./L (Zhang et al., 2022). In our results, the insecticide lambda-cyhalothrin was the most effective insecticide against the cotton aphids, A. gossypii, in agreement with the results of Kandil et al. (2022), who found lambdacyhalothrin (with an LC₅₀ of 0.021 μ g/mL) was the most effective insecticide against cowpea aphids, followed by cypermethrin, (0.056 µg/mL) dinotefuran (0.89 µg/mL), acetamiprid (0.95 µg/mL), and thiamethoxam (3.82 µg/mL). Also, in this respect, Assemi et al. (2014) found that the nano-pesticide of imidacloprid (0.512 ppm) was much more effective, around five times higher than synthetic (2.56 ppm) after 24 hours of application against *Myzus persicae* in laboratory bioassay. The current results are in harmony with the results of Zeng et al. (2016), who studied the toxicity of imidacloprid and cyantraniliprole against Myzus persicae and estimated the fifty lethal concentrations by 12.726 and 1.691 ppm for cyantraniliprole and imidacloprid, respectively.

J. Agric. & Env. Sci. (Damanhour University)	2023, 22(2): 238-263
Print: ISSN 1687-1464	Online: 2735-5098

			Convention	al formulation	l				Na	anoemulsior	l			
Treatments	I.C.	0 Values	Confide	ence limits	Slope	X ²	TI	LC ₅₀	Confider	nce limits	Slope	X ²	TI	RE
	LCS	0 Values	Lower	Upper	Slope	Λ	11	LC50	Lower	Upper	Slope	Λ		
	1C ₂₅	1.517	0.364	2.386				0.785	0.451	1.166				
Lambda- cyhalothrin	1C ₅₀	5.313	2.417	11.591	1.239 ±0.133	8.627	100	2.989	2.176	3.964	1.162 ±0.125	9.336	100	0.437
	LC90	57.515	41.065	502.206				37.884	23.978	73.238				
	1C ₂₅	2.618	0.916	3.744				1.864	0.399	2.454				
Imidacloprid	1C50	6.731	3.596	10.397	1.645 ±0.173	9.874	78.93	4.758	1.807	7.737	1.658 ±0.164	8.671	62.82	0.293
	LC90	40.494	28.333	123.576				28.224	20.275	107.96				
	1C ₂₅	3.942	2.812	5.04				2.806	0.746	3.813				
Cyantraniliprole	1C ₅₀	9.103	7.443	10.937	1.856 ±0.196	8.237	58.37	7.254	3.369	12.108	1.635 ±0.173	8.234	41.2	0.203
	LC90	44.641	33.34	67.66				44.079	33.358	192.043				

Table (2): Acute contact toxicity of Lambda-cyhalothrin, Imidacloprid, Cyantraniliprole against cotton aphids, *Aphis gossypii* after 24 h from treatment under laboratory conditions.

Toxicity index (TI) = (LC₅₀ of the most efficient compound/ LC₅₀ of the tested compound) \times 100

2023, 22(2): 238-263 Online: 2735-5098

Relative efficiency of nanonization (RE)= (LC₅₀ of conventional formulation – LC₅₀ of Nanoemulsion)/LC₅₀ of normal formulation

 Table (3): Acute contact toxicity of Lambda-cyhalothrin, Imidacloprid, Cyantraniliprole against cotton aphids, Aphis gossypii after 48

 hr. from treatment under laboratory conditions.

		C	Conventior	al formulat	ion			Nano formulation						
Treatments		0 Values	Confide	nce limits	Slope	X ²	TI	LC50	Confider	nce limits	Slope	X^2	TI	RE
	LCS	o values	Lower	Upper	Slope	Λ		LC50	Lower	Upper	Slope	Λ		
Lambda- cyhalothrin	1C ₂₅	0.391	0.169	0.662				0.272	0.119	0.462				
5	1C ₅₀	1.808	1.183	2.561	1.014 ±0.138	3.82	100	1.066	0.674	1.509	1.136 ±0.145	6.137	100	0.41
	LC90	33.227	18.31 6	87.137	10.150			14.315	9.035	28.705	±0.145			

Imidacloprid	1C ₂₅	1.402	0.341	1.972				1.046	0.371	3.217				
	1C50	3.744	1.498	5.858	1.581 ±0.154	8.973	54.71	2.866	1.382	5.745	1.54 ±0.182	6.052	37.19	0.23
	LC90	24.199	16.26 2	71.2		154	-	19.479	13.462	32.452				
Cyantranilipro le	1C ₂₅	2.726	0.659	3.533				1.724	0.328	2.218				
	1C50	6.512	2.757	10.76	1.784 ±0.181	5.966	42.81	4.466	1.57	7.289	1.632 ±0.16	6.703	23.86	0.31
	LC90	34.056	26.33 2	142.383				27.253	19.519	110.083				

J. Agric. & Env. Sci. (Damanhour University) 2023, 22(2): 238-263 Print: ISSN 1687-1464 Online: 2735-5098

Toxicity index (TI) = $(LC_{50} \text{ of the most efficient compound}/ LC_{50} \text{ of the tested compound}) \times 100$

Relative efficiency of nanonization (RE)= (LC₅₀ of normal formulation – LC₅₀ of Nanoemulsion)/LC₅₀ of normal formulation

J. Agric. & Env. Sci. (Damanhour University)	2023, 22(2): 238-263
Print: ISSN 1687-1464	Online: 2735-5098

Evaluation of the efficacy of the tested insecticides and their nanoemulsions to control A. gossypii

After the 1st spray

Based on the initial effect values (Table 4), the results indicated that lambda-cyhalothrin insecticide was the most effective insecticide (with reduction percentages of 86.33 and 96.62 % for usual and nanoemulsion formulation, respectively) followed by imidacloprid (88.74 and 90.18% for regular and nanoemulsion, respectively) and cyantraniliprole (78.19 and 83.62 %). At four days post-treatment, lambda-cyhalothrin showed the highest effective insecticide (with reduction percentages of 93.2 and 97.5 for normal and nanoemulsion, respectively), followed by imidacloprid (83.94 and 88.48 %), then cyantraniliprole (80.47 and 88.16%). At seven days post-treatment, the application of lambda-cyhalothrin resulted in a significant reduction in A. gossypii, which recorded 90 and 94.82 % reduction percentages for usual and nanoemulsion formulation, respectively. The insecticide imidacloprid came in the second rank with reduction percentages of 80.13 and 88.23 % for usual and nanoemulsion formulation, respectively. The insecticide cyantraniliprole was in last place, with and 87.59 % reduction percentages for regular and 80.11 nanoemulsions, respectively. The general means of reduction percentages of A. gossypii populations caused by the usual formulation of lambda-cyhalothrin, imidacloprid, and cyantraniliprole were 88.2, 81.74 and 78.91 %, respectively. While these means for the nanoemulsion formulations were 95.17, 86.2 and 85.91%, respectively.

After the 2nd spray

Data presented in Table (5) clarified those differences between the effects of lambda-cyhalothrin, imidacloprid, and cyantraniliprole against the total population of *A. gossypii* at 1, 4, 7, and 14 days after the second spray. After the second spray, all the treatments were significantly superior to the untreated control. Lambda 5 % EC formulation decreased *A. gossypii* population compared to other treatments at 1, 4, 7, and 14 days after the second spray. It gave the

2023, 22(2): 238-263 Online: 2735-5098

highest percent reduction one day after the second spray (86.95 and 90.71% for synthetic and nanoemulsion formulation, respectively) compared with ImiDOR 35%SC (89.95 and 92.2 %) and Benevia 10 % EC (85.43 and 88.34%).

Concerning the general means of the reduction percentages after the second spray, the results emphasize that the highest population reduction was recorded in lambda-cyhalothrin (with reduction percentages of 87.72 and 91.22% for synthetic and nanoemulsion formulation, respectively). The second-best treatment was imidacloprid (82.78 and 88.23 %) and cyantraniliprole (82.18 and 87.31%). The current results emphasize using these insecticides to suppress the A. gossypii population under economic threshold levels and recommend concentration to enhance the profitability of the crop and ensure quality. Therefore, our results align with the results of Ane et al. (2016), who investigated the impact of lambda-cyhalothrin and imidacloprid on aphids in strawberry fields in Pakistan. Their study demonstrated that both insecticides effectively lower aphid density in strawberries; lambda-cyhalothrin vielded however. better results than imidacloprid. Also, the current results are in accordance with the results of Fountain (2013) who reported control of potato aphid on strawberry. He found that cyhalothrin suppressed the number of aphids in field. The current results support the results of Lasheen, et al., (2020) who investigate the efficiency of four pesticides i. e. lambda cyhalothrin, thiamethoxam, azadirachtin and Beauveria bassiana against the white fly, Bemisia tabaci, infesting squash plants during the two growing season, 2017 and 2018, under field conditions. They found that the efficiency of the tested pesticides on white fly could be arranged according to their percentages of reduction in infestation in the follows order: lambda cyhalothrin > B. bassiana > thiamethoxam > azadirachtin, respectively.

J. Agric. & Env. Sci. (Damanhour University)	
Print: ISSN 1687-1464	

2023, 22(2): 238-263 Online: 2735-5098

Table (4): Efficiency of three insecticides in control of cotton aphid, *Aphis gossypii* populations at 1, 4, 7 and 14 days after treatment under greenhouse conditions after the 1st spray (means of reduction percentages % and mean numbers aphids/4 cucumber leaves between brackets):

Treatr	ments	Pre spray		General			
1 reatments		110 spray	1	4	7	14	mean
Cont	trol	(65.31±1.64)	(75.31±1.47)	(80.69±1.78)	(115.19±4.45)	(107.44±1.25)	
	Conventional		86.33±1.72 ^c	93.2±1.09 ^b	90±2.39 ^{bc}	83.28±2.3 ^b	88.2±4.24 ^b
Lambda-	formulation	(70.75±2.65)	(11.19±1.76)	(5.94±0.94)	(12.38±2.35)	(19.5±3.03)	
cyhalothrin	Nanoemulsion	(50.54.0.41)	96.62±0.51ª	97.5±1.73ª	94.82±1.63 ^a	91.74±2.26 ^a	95.17±2.72 ^a
	formulation	(73.56±2.41)	(2.88±0.52)	(2.25±1.58)	(6.63±1.79)	(9.94±2.38)	
	Conventional		88.74±2.94 ^{bc}	83.94±2.62 ^d	80.13±2.69 ^d	74.16±1.9°	81.74±5.97°
Tari Jan Januari J	formulation	(72.56±2.3)	(9.38±2.14)	(14.38±2.21)	(25.38±3.81)	(30.88±2.75)	
Imidacloprid	Nanoemulsion		90.18±2.48 ^b	88.48±1.89°	88.23±1.6°	77.88±2.4°	86.2±5.36 ^b
	formulation	(68.25±2.35)	(7.75±2.01)	(9.69±1.36)	(14.19±2.13)	(24.88±3.28)	

J. Agric. & Env. Sci. (Damanhour University)	
Print: ISSN 1687-1464	

2023, 22(2): 238-263 Online: 2735-5098

Cyantraniliprole	Conventional formulation	(66.25±1.74)	78.19±1.78 ^d	80.47±1.26 ^d	80.11±1.92 ^d	76.87±2.98°	78.91±2.4°
			(16.69±1.77)	(16±1.34)	(23.31±3.27)	(25.13±2.31)	
		(71.38±1.69)	83.62±4.18°	88.16±1.46°	87.59±1.23°	84.27±1.67 ^b	85.91±3 ^b
	Nanoemulsion formulation		(13.44±3.25)	(10.43±1.25)	(15.63±1.74)	(18.5±2.27)	
F- values			24.186	49.214	34.151	31.101	28.911
L. S. D.			3.7699	2.60005	2.933	3.40265	2.93145

Means followed by the same letter(s) within the same column are non significantly different ($P \le 0.05$)

Table (5): Efficiency of three insecticides in control of cotton aphid, *Aphis gossypii* populations at 1, 4, 7 and 14 days after treatment under greenhouse conditions after the 2nd spray (means of reduction percentages % and mean numbers aphids/4 cucumber leaves between brackets)

Treatr	nents	Pre spray	Post spray (days)				General
		110 spruy	1	4	7	14	mean
Cont	trol	(107.44±1.25)	(104±1.66)	(93.63±2.06)	(81.81±2.38)	(78.56 ±1.52)	
Lambda-	Conventional	(19.5±3.03)	86.95±2.21 ^{bc}	88.05±2.83 ^b	87.31±1.66 ^b	88.58±2.71a ^b	87.72±1.93 ^b
cyhalothrin formulation		(2.44±0.43)	(2±0.41)	(1.88±20.32)	(1.63±0.25)		

2023, 22(2): 238-263 Online: 2735-5098

	Nanoemulsion	(0.04.2.20)	90.71±3.04 ^{ab}	91.49±2.49 ^a	91.35±2.58ª	91.34±3.64 ^a	91.22±2.67 ^a
	formulation Conventional formulation	(9.94±2.38) (30.87±2.75)	(0.89±0.33)	(0.75±0.35)	(0.69±0.31)	(0.63±0.32)	
			89.95±0.53 ^{ab}	84.41±2.3°	79.51±1.45°	77.26±2.48°	82.78±5.31°
Imidacloprid			(1.88±0.78)	(4.19±0.69)	(4.81±0.52)	(5.13±0.66)	
	N7 1 1		92.2±1.71ª	88.56±2.2 ^{ab}	86.29±2.97 ^b	85.88±1.91 ^b	88.23±3.28 ^{ab}
Cyantraniliprole	Nanoemulsion formulation	ntional (25.13±2.31)	(3.0 ±0.29)	(2.44±0.24)	(2.56±0.47)	(2.56±0.47)	
	Conventional		85.43±3.19°	82.03±2.83°	89.82±4.32 ^{ab}	71.43±2.26 ^d	82.18±7.59°
	formulation		(1.88±0.48)	(3.94±0.77)	(1.94±0.83)	(5.25±0.65)	
	Nanoemulsion (18 5+2 2		88.34±2.12 ^{bc}	90.61±1.82 ^{ab}	91.57±1.28 ^a	78.71±0.9°	87.31±5.46 ^b
	formulation	(18.5±2.27)	(2.06±0.24)	(8.25±0.29)	(1.19±0.24)	(2.88±0.32)	
F- values			4.713	6.201	11.920	46.773	8.382
L. S. D.			3.42995	3.33265	3.68095	3.31095	3.3591

Means followed by the same letter(s) within the same column are nonsignificantly different ($P \le 0.05$)

2023, 22(2): 238-263 Online: 2735-5098

CONCLUSIONS

It was concluded the nanoemulsion formulations of imidacloprid, lambda-cyhalothrin and cyantraniliprole can be used as an effective alternatives of conventional formulations to control *A. gossypii* due to its physical characteristics. Prepared of nanoemulsion formulation increased the toxicity of imidacloprid, lambda-cyhalothrin and cyantraniliprole against *A. gossypii* by using the leaf-dip bioassay under laboratory conditions by1.41, 1.78 and 1.25-fold, after 24 hr. and 1.46, 1.7 and 1.45-fold after 48 hr., respectively. Lambda-cyhalothrin insecticides showed the highest toxicity against *A. gossypii* compared with imidacloprid and cyantraniliprole whether in the laboratory or in the greenhouses conditions.

REFERENCES

- Abott, W. S. (1925) A method of computing effectiveness of an insecticides.J.Econ.Entomol.18:265-267.
- Ahamad A., Kumar J. (2023) Pyrethroid pesticides: An overview on classification, toxicological assessment and monitoring. Journal of Hazardous Materials Advances. Journal of Hazardous Materials Advances. 10, 100284. https://doi.org/10.1016/j.hazadv.2023.100284
- Ane Noor U. I., Hussain M., Zainab T., Fatima S. (2016) Effect of Lambda Cyhalothrin and Imidacloprid on the population density of aphid attacking strawberry. International Journal of Fauna and Biological Studies; 3(1): 121-12
- Assemi, H., Sajjadi A. and Naghizadeh, F. (2014) Investigation of Different Values of Nano imidacloprid for Control of Tobacco Aphids *Myzus persicae* nicotianae in Laboratory. Agrotechnology, 3(1): DOI: 10.4172/2168-9881.1000128
- Aydin, H., Oktay, M, and Rkan, G. (2005) The efficacy of spinosad on different strains of *Spodoptera littoralis* (Boisduval) (Lepidoptera: Noctuidae). Turk. J. Biol. 30: 5-9.

- **Baboota, S., Shakeel, F., Ahuja, A., Ali J. and Shafiq, S. (2007)** Design, development and evaluation of novel nanoemulsion formulations for transdermal potential of celecoxib. Acta Pharmaceutica. 57:315-332.
- Badawy, M. E., Saad, A. S., Tayeb, E. H., Mohammed S. A., Abd-Elnabi, A. D. (2017) Optimization and characterization of the formation of oil-in-water diazinon nano-emulsions: Modeling and influence of the oil phase, surfactant and sonication. J Environ Sci Health Part B. 1–16. <u>https://doi.org/10.1080/03601234.2-017.1362941</u>.
- Badawy, M. E. I., Saad, A. S. A., Tayeb E. H. M., Mohammed S. A. and Abd-Elnabi, A. D. (2019) Development and characterization of nanoemulsions of some insecticides by high energy technique for targeting delivery. J Agric. Res., 2019, Vol. 57(1):15-23
- Barakat, D. A., Ibrahim, E. S. and Salama, M. R. (2023) Effectiveness and Persistence of Some Synthetic Insecticides and their Nanoformulation against Whitefly (*Bemisia tabaci*) and Aphids (*Aphis craccivora*) on Fennel Plants and Soil. Egypt. J. Chem. Vol. 66, No. 6 pp. 235 – 246. Doi:10.21608/EJCHEM.2022.155631.6717
- Blackman, R. L., and Eastop, V. F. (2000) Aphids on the world's crops: an identification and information guide, 2nd edition. Wiley, Chinchester, U.K.
- Blackman, R. L., and Eastop, V.F. (1984) "Aphids on the world's crops: An identification and information Guide". Wiley, New York.
- Cordova, D., Benner, E. A., Sacher, M. D., Rauh, J. J., Sopa, J. S. and Lahm, G. P. (2006) Anthranilic diamides: A new class of insecticides with a novel mode of action, ryanodine receptor activation. Pesticide Biochemistry Physiology 84:196-214. <u>https://doi.org/10.1016/j.pestbp.2005.07.005</u>
- **CoStat Statistical Software (1998)** Microcomputer program analysis version 6.400, CoHort Software, Berkeley, CA.

- Finney, S. J. (1971) Probit analysis. Astatistical treatment of the sigmoid response curve.7th Ed. Campridge Univ. Press, Cambridge, England.
- Fountain, M. (2013) Control of potato Aphid (Macrosiphum euphorbiae), in spring on strawberry. Agriculture and Horticulture Development board.
- Hamlen, R. A. (1977) Insecticides and insect growth regulators control of green peach aphid, banded greenhouse, thrips and a foliar mealybug on aphelandra. Proc. Fla. State. Hort. Soc., 90: 321-323
- Henderson, C. F. and Tilton, E. W. (1955) Tests with acaricides against the brown wheat mite. J Econ Entomol 48:157-161. https://doi.org/10.1093/jee/48.2.157
- Jacobson, H., and Kennedy, G. (2012) Residual suppression of tomato fruitworm damage to fresh market tomato by early season application of Cyazypyr. Arthropod Management Tests 37:E71. doi: 10.4182/amt.2012.E71
- Kadhim, D. H. and Abbas, H. A. (2015) Formulation and characterization of carvedilol nanoemulsion oral liquid dosage form. Int. J. Pharm. Pharmac. Sci. 7:209-216.
- Kandil, M. A., Fouad, E. A. and Mokbel, E. M. S. (2022) Efficiency of Certain Neonicotinoid Mixtures Against the Cowpea Aphid, *Aphis craccivora* (Koch) Egypt. Acad. J. Biolog. Sci., 14(2):91-99. DOI:<u>10.21608/eajbsf.2022.259585</u>
- Lahm, G. P., Stevenson T. M., Selby, T. P., Freudenberger, J. H., Cordova, D., Flexner, L., Bellin, C. A., Dubas, C. M., Smith, B. K., Hughes, K. A., Hollingshaus, J. G., Clark, C. E., Benner, E. A. (2007) Rynaxypyr: A new insecticidal anthanilic diamide that actsas a potent and selective ryanodine receptor activator. Bioorganic & Medicinal Chemistry Letters, 17, 6274–6279. https://doi.org/10.1016/j.bmcl.2007.09.012
- Lasheen, S.G., Sherif, R.M., Youssif, M.A.I. and Sallem H.M. (2020) Effectiveness of some insecticides against Bemisia tabaci (Genn) infesting squash plants. Zagazig J. Agric. Res., Vol. 47 No. (1); 79-86. Doi:10.21608/ZJAR.2020.70122

- Matsuda, K., Buckingham. S. D., Freeman, J. C., Squire, M. D., Baylis, H. A., Sattelle, D.B. (1998) Effects of the alpha subunit on imidacloprid sensitivity of recombinant nicotinic acetylcholine receptors. Br J Pharmacol123:518-524. <u>DOI:</u> 10.1038/sj.bjp.0701618
- Mendoza, A. H. D., Belliure, B., Carbonell, E. A. and Real. V. (2001) Economic thres-hold for *Aphis gossypii* (Hemiptera: Aphididae) on Citrus clementina. J. Econ. Entomol. 94(2): 439-444. <u>https://doi.org/10.1603/0022-0493-94.2.439</u>
- Moores, G. D., Gao, X., Denholm, I., Devonshire, A. L., (1996) Characterisation of insensitive acetylcholinesterase in insecticideresistant cotton aphids, *Aphis gossypii* glover (homoptera: Aphididae). Pesticide Biochemstiry Physiology, 56, 102–110. https://doi.org/10.1006/pest.1996.0064
- **Raymond, J. K. (2003)** Chrysanthemum, commercial greenhouse production. Auburn Univ.
- Sadurní, N., Solans, C., Azemar N. and García- Celma M. J. (2005) Studies on the formation of O/W nano-emulsions, by low-energy emulsification methods, suitable for pharmaceutical applications. Eur. J. Pharm. Sci. 26:438-445. DOI: <u>10.1016/j.ejps.2005.08.001</u>
- Salim, N., Basri, M., Rahman, M. A., Abdullah, D., Basri H., and Salleh, A. (2011) Phase behaviour, formation and characterization of palm-based esters nanoemulsion formulation containing ibuprofen. Nanomedic nanotechnol, 2(4): 1-5. DOI: 10.4172/2157-7439.1000113
- Sattelle, D. B., Cordova, D. and Cheek. T. R. (2008) Insect ryanodine receptors: molecular targets for novel pest control chemicals. Invertebr. Neurosci. 8: 107Đ119 DOI: <u>10.1007/s10158-008-0076-</u> <u>4</u>
- Shafiq S. and Shakeel F. (2010) Stability and self-nanoemulsification efficiency of ramipril nanoemulsion containing labrasol and plurol oleique. Clinical Research and Regulatory Affairs, 27(1):7-12. https://doi.org/10.3109/10601330903571691

- Shahavi, M. H., Hosseini, M., Jahanshahi, M., Meyer, R. L., and Darzi, G. N. (2019) Evaluation of critical parameters for preparation of stable clove oil nanoemulsion. Arabian journal of chemistry, 12(8), 3225-3230. https://doi.org/10.1016/j.arabjc.2015.08.024
- Shi B., Wang, Z., Wen, H. (2017) Research on the strengths of electrostatic and van der Waals interactions in ionic liquids. J Mol Liq. 241:486–488. https://doi. org/10.1016/j.ejpb.2006.10.014.
- Simon, J. Y. (2008) The Toxicology and Biochemistry of Insecticides. CRC Press, Boca Raton, London, New York, 276.
- Sobhani, H., Tarighi, P., Ostad, S.N., Shafaati, A., Nafissi-Varcheh N., and Aboofazeli, R. (2015) Formulation development and toxicity assessment of triacetin mediated nanoemulsions as novel delivery systems for rapamycin. Iran J. Pharm. Res. 14:3-21. PMID: <u>26185501</u>
- Su, R., Yang, L., Wang, Y., Yu, S., Guo, Y., Deng, J., Zhao, Q. and Jin, X. (2017) Formulation, development, and optimization of a novel octyldodecanol-based nanoemulsion for transdermal delivery of ceramide IIIB. Int. J. Nanomed. 12:5203-5221. DOI <u>https://doi.org/10.2147/IJN.S139975</u>
- Yamamoto, I., Yabuta, G., Tomizawa, M., Saito, T., Miyamoto, T., Kagabu, S. (1995) Molecular mechanism for selective toxi- city of nicotinoids and neonicotinoids. J Pestic Sci 20:33–40. Doi:10.1584/jpestics.20.33
- Zhang, A., Xu, L., Liu, Z., Zhang, J., Zhao, K. and Han, L. (2022) Effects of Acetamiprid at Low and Median Lethal Concentrations on the Development and Reproduction of the Soybean Aphid Aphis glycines. Insects, 13(1): 87. https://doi.org/10.3390/insects13010087
- Zeng X., He, Y., Wu, J., Tang, Y., Gu, J., Ding, W. and Zhang, Y. (2016) Sublethal Effects of Cyantraniliprole and Imidacloprid on Feeding Behavior and Life Table Parameters of *Myzus persicae* (Hemiptera: Aphididae). Journal of Economic Entomology, 109(4), 1595–1602. <u>doi: 10.1093/jee/tow104. Epub 2016 May 31.</u>

2023, 22(2): 238-263 Online: 2735-5098

تقييم التجهيزات التقليدية والمستحلبات النانوية لبعض المبيدات الحشرية ضد حشرة من القطن :Aphis gossypii Glover (Hemiptera Aphididae)

مصطفي عبداللطيف عباسي ، مني عبدالنبي عبدالرسول ، بلال سليمان محمد سليمان ، عاطف محمد خضر نصار

قسم وقاية النبات – كلية الزراعة – جامعة دمنهور – جمهورية مصر العربية *Corresponding author: bsoliman91@gmail.com

تم تقييم كفاءة بعض التجهيزات التقليدية والمستحلبات النانوية لثلاث مبيدات حشرية هي imidacloprid ، لامبادا-سيهالوثرين imidacloprid ايميداكلو بريد والسيانترانيليبرول cyantraniliprole تحت الظروف المعملية والصوبة الزراعية بالجرعات الموصى بها ضد حشرة من القطن A. gossypii. اظهرت المستحلبات النانوية التي تم تحضير ها دلائل تشتت متعدد تر اوحت بين 0,198 – 0,324 وجهد زيتا بين -27,2 و -38,25 مللي فولت. اصغر قطر اقطرات المستحلبات النانوية تم تسجيلها لمبيد lambda cyhalothrin (83,25 نانومتر) واكبر قطر كان لمبيد imidacloprid (112,32 نانومتر). اوضحت النتائج ان استعمال المستحلبات النانوية لمبيدات ايميداكلوبريد ولامبادا-سيهالوثرين ومبيد سيانتر انيليبرول ادي الى زيادة السمية عن التجهيزات التقليدية لحشرة من القطن بمقدار 1,41 و 1,78 و 1,25 ضعف معمليا بعد وقت تعرض 24 ساعة، على الترتيب. بعد مرور 48 ساعه كانت كان مقدار الزيادة في السمية 1,46 و 1,7 و 1,45 ضعف، على الترتيب. اعلى سمية تم تسجيلها لمبيد لامبادا-سيهالوثرين lambda-cyhalothrin وكانت قيم LC50 لهذا المبيد هي 5,313 و 1,808 جزء في المليون بعد 24 و 48 ساعة مدد تعرض، على الترتيب. تحت ظروف الصوبة، كانت المتوسطات العامة لخفض عشيرة حشرة من القطن بواسطة التجهيزات التقليدية لـ lambda-cyhalothrin و imidacloprid و cyantraniliprole هي 88,2 و 81,74 و 78,91 %، على الترتيب. وكانت هذه المتوسطات للمستحلبات النانوية 95,17 و 86,2 و 85,91 %، على الترتيب. بعد المعاملة الثانية في الصوبة كانت هذه المتوسطات 95,17 و86,29 و85,91 %، على الترتيب للتجهيزات التقليدية و91,22 و 88,23 و 87.31 % للمستحلبات النانوية، على الترتيب.