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EFFICACY AND BIOCHEMICAL EFFECTS OF SOME INSECTICIDES AGAINST COTTON LEAFWORM, *Spodoptera littoralis* (BOISD.)

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Rasha A.A. Sleem[1*], El-Zemaity[2] M.E., Hussein[2] M.I. and Sherifa A.N. El-Sherif [1]

1- Bioassay Dept., Central Agric. Pesticides Lab. Agric. Res. Center, Dokki, Giza, Egypt
2- Plant Protection Dept., Fac. of Agric., Ain Shams Univ., P.O. Box 68, Hadayek Shubra 11241, Cairo, Egypt

*Corresponding author: rashasleem@yahoo.com

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ABSTRACT

The efficiency and biochemical effects of certain insecticides belonging to different groups namely: flubendiamide (diamide), pyridalyl (phenoxy-pyridaloxy), clothianidin (neonicotinoide), fipronil (phenylpyrazole) and spirotetramat (tetramic acid) and pirimiphos-methyl (Ops) were tested against 2nd larval instar of *Spodoptera littoralis* laboratory strain using dipping technique. The efficacy of the tested insecticides are arranged as follows to LC_{50} values. The results showed that flubendiamide was the superior toxicant insecticides (LC_{50} 1.03 ppm) followed by pyridalyl (2.13 ppm) then fipronil (7.42 ppm), clothianidin (26.87ppm), pirimiphos-methyl (76.31 ppm) and spirotetramat (431.91 ppm). Biochemical effects of the tested insecticides on acetylcholine esterase (AChE), glutathione-s-transferases (GST), adenosinetriphosphatase (ATPase), phenoloxidase, total calcium, and total protein were determined in the treated larvae. Data showed highest significant increase at AChE activity in treatment of fipronil by change% (70.59), while treatment with pirimiphos-methyl recorded highest significant decrease by change% (-56.41). Whereas, all treatments recorded increase in GST activity except pirimiphos-methyl recorded non significant decrease. All insecticides treatments were showed a decrease in phenoloxidase activity the highest decrease recorded by pirimiphos-methyl treatment by change % (- 53.06). Regarding change percentage of ATPase, activity data recorded significant increase with pirimiphos-methyl treatment by (72.91%). However, total protein in all treatments showed a significant decrease. On the other hand, treatment with pirimiphos-methyl recorded a significant decrease in total calcium and the corresponding change % was (- 6.0%). Previous data confirmed the mode of action of the novel tested insecticides.

Keywords: *Spodoptera littoralis*, Toxicology, Novel insecticides, Pirimiphos-methyl, Biochemical aspects.

1. INTRODUCTION

The Egyptian cotton leafworm, *Spodoptera littoralis* is a highly polyphagous defoliator of many cultivated crops. This pest one of the most important lepidopteron pests which attack numerous economically important crops around the year causing economic losses in infected crops in both open fields and greenhouses. At present strategy of agricultural pest control of *S. littoralis* using chemical control by conventional insecticides only became undesirable because insects often developing resistance for this insecticides after much successive application and this effect can cause a defect in the implementation of effective pest control programs in agriculture **(Knight and Norton, 1989)**. During the last five decades conventional insecticides, all neuroactive chemicals have played a major role in insect pests management in many crops. Unfortunately, their indiscriminate uses led to several problems like resistance, toxicity to non-target organisms and environmental long persistence. The current trend in insect pests controls is reduce the use of conventional insecticides and search for

development of new control agents having novel biochemical targets to solve pest problem, management resistance and safety use to human and the environment. In recent years, several new insecticide groups having new chemistries like neonicotinoids, diamide, phenyl-pyridaloxy, tetramic acid, oxidazine, keto-enol, and phenylpyrazole were commercially formulated and traded in many countries including Egypt. **(Kodandaram et al 2010)**.

The present study aimed to evaluate the efficacy of six insecticides; flubendiamide, pyridalyl, clothianidin, fipronil, spirotetramat and pirimiphos-methyl representative to different modes of action and estimate their biochemical effects on acetylcholinesterase, glutathione-s-transferases, phenoloxidase adenosinetriphosphatase, total calcium, and total soluble protein in 2nd larval instar of *S. littoralis*.

2. MATERIALS AND METHODS

2.1. Tested insect

The laboratory strain of cotton leafworm, *Spodoptera littoralis* was maintained under tightly conditions in the insect rearing room at 25±2ºC and 60±5 relative humidity avoiding any intentional chemical pressure in Central Agriculture Pesticides Laboratory (CAPL), Dokki, Giza was used. A standard rearing method described by **(EL-Defrawi et al 1964)** was followed**.**

2.2. Tested insecticides

Table 1. The tread name, common name, chemical group of tested insecticides in addition to their mode of action

	Trade name	Common name	Chemical group	IRAC MoA
1	**Takumi® 20% WG**	Flubendiamide	Diamide	Ryanodine receptor modulators
2	**Pleo® 50%EC**	Pyridalyl	Phenoxy-pyridaloxy	unknown
3	**Coach® 20%SC**	Fipronil	Phenylpyrazole	GABA-gated chloride channel antagonists
4	**Super Tox® 20%SC**	Clothianidin	Neonicotinoids	Nicotinic acetylcholine receptor (nAChR)competitive modulators
5	**Actellic 50%EC**	Pirimiphos-methyl	Organophosphate	Acetylcholinesterase inhibitors
6	**Movento® 10% SC**	Spirotetramat	Tetramic acid	Inhibitor of acetyl COA carboxylase

2.3. Leaf dipping technique

Leaf–dipping technique **(Shepared, 1958)** was used to estimate the susceptibility of 2nd larval instars of *S.littoralis* to the tested insecticides. Fresh and clean dried leaves of castor (*Ricinus communis*) were dipped for 30 seconds in serial concentrations (6-8) of an aqueous solution to each insecticide. Three replicates were used each of which has 10 healthy larvae of *S.littoralis*. The present mortality was recorded in the second day and corrected by **Abbott's formula (1925).**

Data analyzed by applying the Ldp line software **(Bakr, 2007).**The values of LC_{50} and slope in addition to toxicity index ,which calculated to **(Sun, 1950)** were illustrated in **Table (2).**

2.4. Biochemical analysis

The survivor second larval instars after exposure to median lethal concentration of each insecticides and the untreated larvae were cooled at –40ºC before homogenizing.

The activity of the total soluble protein, adenosinetriphosphatase (ATPase), glutathione -S-transferases (GST), phenoloxidase, acetylcholine esterase (AChE) and total calcium was determined spectrophotometrically in whole body tissue homogenate.

2.4.1. Preparation of insect samples for biochemical analysis

The frozen larvae were homogenized in a glass tissue grinder under an ice jacket in (1:7 w/ v) with homogenization buffer (pH 7.8) which prepared by dissolving 15 ml of glycerol into 75 ml distilled water and adding 606 mg Tris, 292 mg EDTA and 5mg phenylthiourea, the pH adjusted to 7.8 then the final volume was completed to 100 ml distilled water **(Salama, 1985).** Homogenates were centrifuged at 12000 rpm for 20 minutes at 4ºC and the resultant supernatants were held in clean Eppendorf for biochemical analysis.

2.4.2. Estimation methods of enzymatic activity

Acetylcholinesterase (AChE) activity was determined according to the method described by **Ellman et al (1961).** Glutathione –S-transferases (GST) activity was measured by the method of **Habig et al (1974).** Adenosinetriphosphatase (ATPase) estimated by the method of **Shiosaka et al (1971).** Phenoloxidase activity was determined using the method of **Ishayaa and Casida (1974).** Total protein was measured according to the method described by **Bradford, (1976).** Total calcium was determined using kit according to the method described by **Gindler, (1972)**.

Statistical analysis

Data statistically analyzed by using SPSS program V.16.0 and differences were considered significant at < 0.05 level.

3. RESULTS AND DISCUSSION

3.1. Toxicity of tested insecticides

The potency of the six tested commercial insecticides, pirimiphos-methyl, pyridalyl, flubendiamide, clothianidin, fipronil, and spirotetramat were evaluated against the new molted 2nd larval instar of *S. littoralis* as shown in **Table (2)**. Data indicated that the efficiency of tested insecticides varied according to the value of median lethal concentration (LC_{50}).

The main aim of this study was to evaluate six compounds representing different chemical groups against *S. littoralis* larvae in order to find alternative and supplements compounds to conventional chemical insecticides. The results in **Table (2)** show the efficiency of the tested insecticides; flubendiamide, pyridalyl, fipronil, clothianidin, pirimiphos-methyl and spirotetramat against second larval instar of cotton leaf worms. The candidate insecticides were arranged according to LC_{50} values in the following descending order; flubendiamide (1.03 ppm), pyridalyl (2.13 ppm), fipronil (7.42 ppm), clothianidin (26.87 ppm), pirimiphos-methyl (76.31 ppm) and then spirotetramat (431.91 ppm). Data indicate that flubendiamide was more effective than the other tested insecticides against *S. littoralis* larvae. On the contrary, spirotetramat had poor efficacy. The results confirmed that flubendiamide, pyridalyl and fipronil were highly toxic against *S. littoralis* larvae than the insecticides; clothianidin, pirimiphos-methyl and spirotetramat. The insecticides clothianidin and pirimiphos-methyl showed moderate activity toward *S. littoralis* larvae with LC_{50} 26.87 and 76.31ppm, respectively.

It always seems convenient to measure the efficiency or toxicity of different toxic insecticides by comparing them with a standard material. Accordingly, the toxicity index was employed. The toxicity index was obtained by comparing the efficiency of the tested compounds at a fixed level such as LC_{50} to their most effective compound. The toxicity indices of the tested insecticides revealed that pyridalyl and fipronil were 48.36 and 13.88% as toxic as flubendiamide. The toxicity index of remaining insecticides ranged between 0.24 and 3.83%.

The slope values of toxicity lines for flubendiamide, pyridalyl, fipronil, clothianidin, pirimiphos-methyl and spirotetramat were 2.166, 2.607, 1.723, 1.738, 1.346 and 3.781. Concerning the slope values, data in **Table (2)** indicate that spirotetramat showed the steepest toxicity line (slope = 3.781) followed by pyridalyl (2.607) and flubendiamide (2.166). The slope values of the other toxicity lines had approximately equal slope values and ranged between 1.346 and 1.738. Finally, data confirm that the new chemical groups of insecticides representative by flubendiamide, pyridalyl and fipronil have promising compounds, which showed high toxicity toward the second larval instar of *S. littoralis*.

Toxicity of the tested insecticides against different insects including *S.littoralis* was evaluated by many researchers such as **Argentine et al (2002)**, **Rastegari and Subrahmanyam (2003), Saito et al (2004) and Andric et al (2014).** Regarding the cotton leafworm, *S.littoralis*. **Abd-Allah (1998)** evaluated the efficacy of pirimiphos-methyl against *S.littoralis* in laboratory and field .The ob-

tained data founded that the LC_{50} value was recorded 1485.7 ppm after 24 h of treatment in laboratory using leaf dipping technique while pirimiphos-methyl led to reduction in *S.littoralis* field strain population by 80.11 % & 47.15% after 1 and 3 days of insecticides spraying . **Abdel-Rahim et al (2009)** reported that LC_{50} of pyridalyl was 1.8 ppm to 2nd larval instar larvae of *S.littoralis* laboratory strain. Also, **Abdel-Rahim (2011)** estimated pyridalyl bio-residual activity against 2nd larval instar of sensitive strain *S.littoralis* after feeding for 48 hrs on treated cotton leaves with the recommended rate of application. The mortality percentage reached to 100, 90, 62, 40% after 0, 3, 7, 12 day post application. **Dahi et al (2011)** noticed that LC_{50} of pyridalyl on *S. littoralis* 2nd instar larvae was 6.77 ppm after 24 hrs. **Abdel-Rahim and Zidan (2012)** evaluated the efficiency of flubendiamide against *S.littoralis* 2nd larval instar and found that the LC_{50} was 0.06 ppm. **Abdel-Hafez and Osman (2013)** reported that pyridalyl as a semi-synthetic insecticide was toxic against 2nd instar larvae of *S.littoralis* based on thier LC_{50} values which were ,2.56, 1.75, 0.58 and 0.63 ppm after 2, 3, 5 and 7 days. **Bhatti et al (2013)** evaluated flubendiamide efficiency using leaf dip bioassay against *S.littoralis* 2nd instar after 24, 48, 72 hrs. The values of LC_{50} were 2.81, 0.37 and 0.31 ul/ ml, respectively. Also, **Abd-El-Aziz (2014)** reported that the LC_{50} value of pyridalyl after 48 hrs from treatment of 4th larval instar *S.littoralis* was 3.94 ppm. **Andric et al (2014)** study the residual activity of pirimiphos-methyl against *Plodia interpunctella* (Hubner) larvae after 2, 7 and 14 days of insect exposure to treated concrete. The results that the pirimiphos-methyl achieved maximum efficacy (100%) in all treatment after 2-14 days of exposure. **Abou-Taleb et al (2015)** found that fipronil was toxic for the field strain of *S.littoralis* and recommended it can be a good alternative in controlling of cotton leafworm**. Saleh et al (2015)** recorded that LC_{50} value of pyridalyl was 18.679 ppm against the same instar after 24 hrs from treatment. **El-Dewy (2017)** found that the LC_{50} values of pyridalyl to 4th instar larvae to *S. littoralis* laboratory strain were 37.88, 13.01 and 10.16 ppm after 24, 48 and 72 hrs, respectively. **Hatem et al (2017)** evaluated the efficiency of clothianidin LC_{50} against *S. littoralis* 4th larval instar the LC_{50} value was 70.24 ppm after 24hrs.of treatment using leaf dipping techniques.

The present study considering and the previous investigations it could be concluded that flubendiamide, pyridalyl, clothianidin and fipronil can be use in IPM programs of *S. littoralis*.

Table 2. Susceptibility of 2nd larval instar of cotton leafworm, *S.littoralis* lab. strain to tested insecticides

	Insecticides	LC_{50} (ppm)	95% Fiducial Limits	Slope ± S.E.	Toxicity Index
1-	**Flubendiamide**	1.03	(0.794-1.332)	2.166±0.31	100
2-	**Pyridalyl**	2.13	(1.689-2.751)	2.607 ±0.43	48.36
3-	**Fipronil**	7.42	(5.565-9.938)	1.723±0.24	13.88
4-	**Clothianidin**	26.87	(19.598-42.541)	1.738±0.30	3.83
5-	**Pirimiphos-methyl**	76.31	(53.088-109.252)	1.346 ±0.19	1.35
6-	**Spirotetramat**	431.91	(379.546-490.681)	3.781± 0.58	0.24

3.2. Effects of tested insecticides on biochemical components of exposed insects

Effect of the tested insecticides at level of LC_{50} on some vital components of exposed insects namely: (AChE, ATPase, GST, phenoloxidase, total calcium, and total proteins) in the 2nd larval instar of *S.littoralis* was recorded in **Tables (3-4)**.

3.2.1. AChE

The data in **Table (3)** clearly showed the activity of AChE it was clear significant increase in treatment with fipronil by 0.178 (µmol/min/mg protein) and change% (70.59%) while, treatments with pirimiphos-methyl and pyridalyl recorded significant decrease by 0.045 and 0.067(µmol/min/mg protein) and change % (-56.41 and -34.96 %, respec-

tively). Treatments with flubendiamide and spirotetramat showed a non-significant decrease by 0.082 and 0.083 (µmol/min/mg protein) with change % values -21.36 &-19.82, respectively, also clothianidin showed non-significant increase by 0.132 (µmol/min/mg protein) with change% value 26.53% all treatments in comparison with untreated control which recorded enzymatic activity by 0.104 (µmol/min/mg protein).

3.2.2. GST

In case of GST activity, data in **Table (3)** indicate that treatments with flubendiamide , pyridalyl , clothianidin and spirotetramat caused various significant increase in enzyme activity only fipronil caused non-significant increase in enzyme activity. The difference in enzyme activity and change % were 3303.49 (149.53%), 3843(190.29%), 4702.46 (255.20%), 4262.12 (221.94%) and 1715.96 (29.61%) (mmol/min/mg protein) of flubendiamide, pyridalyl, clothianidin, spirotetramat and fipronil, respectively. on the other hand, treatment with pirimiphos-methyl recorded non-significant decrease in enzymatic activity by 994.91 (mmol/min/mg protein) and change %(- 24.85) all different treatments was compared with control 1323.89 (mmol/min/mg protein) .

3.2.3. Phenoloxidase

The obtained data tabulated in **Table (3)** indicated that the phenoloxidase activity achieved decrease in all insecticides treatments. Both pirimiphos-methyl, spirotetramat and fipronil recorded significant decrease in enzyme activity by 0.18, 0.31 and 0.33 (ΔOD/Min/mg protein) while , insecticides treatment with clothianidin, flubendiamide and pyridalyl recorded non-significant decrease in enzyme activity as shown 0.36, 0.38 and 0.38 (ΔOD/Min/mg protein) all tratments compared to control 0.39 (ΔOD/Min/mg protein). Changes % recorded (-53.06, -21.04,-16.20, -8.90, -4.31 and -2.60% for pirimiphos-methyl, spirotetramat, fipronil, clothianidin, flubendiamide and pyridalyl, respectively).

3.2.4. ATPase

The activity of ATPase matrix in **Table (4)**, treatments with insecticide pirimiphos-methyl recorded the highest significant increase level of enzyme activity by 31.62 (mmol/min/mg protein) with change % (72.91%). Both flubendiamide and clothianidin recorded non-significant increase in enzyme activity by 18.71 and 18.61 (mmol/min/mg protein) with change % (2.33 and 1.78 %, respectively). On the other hand treatments of fipronil, pyridalyl and spirotetramat also showed non- significant decrease in ATPase activity by 17.36, 17.42 and 17.67 (mmol/min/mg protein) with change % (-5.04, - 4.76 and -3.36%, respectively) in addition to enzyme activity in control by 18.29 (mmol/min/mg protein).

3.2.5. Total proteins

Data given in **Table (4)** indicated that all tested insecticides led to decrease in the total proteins content in all different treatments .The total protein content reached to 4.34, 4.78, 5.23, 4.91, 5.80 and 5.65 mg/ml homogenate for flubendiamide , pyridalyl , fipronil , clothianidin , pirimiphos-methyl and spirotetramat , respectively, in relevant to total protein content in control (7.02 mg/ml homogenate). The decreases in protein content expressed by change percentage. The highest decrease in activity observed in treatment with flubendiamide by change % (- 38.14 %), while the other treatments recorded change % value as follows (-31.82, - 29.97, -25.46, -19.43 and -17.32%) for treatments with pyridalyl, clothianidin ,fipronil, spirotetramat and pirimiphos-methyl, respectively.

3.2.6. Total calcium

Data in **Table (4**) recorded non-significant increase or decrease of calcium activity in all insecticides treatments expect treatment with pirimiphos-methyl recorded a significant decrease of calcium activity by 11.14 (mmol/L) with change % (- 6.0). The enzymatic activity and change % of different treatments were 11.71 (-1.16), 11.62 (-1.91%), 11.85 (0.0033%), 11.58 (-2.24%) and 11.92 (0.58%) (mmol/L) of flubendiamide, pyridalyl, fipronil, clothianidin and spirotetramat, respectively in comparison with activity in control 11.85 (mmol/L).

Organophosphates compounds are an inhibitor of acetylcholinesterase enzyme primarily by phosphorylation of the acetylcholinesterase enzyme (AChE) at nerve endings. Thus blocking the transmission of nervous impulse through the neuronal synapses thus, Ops plays a pivoted role in acetylcholinesterase inhibition **(Ishaya and Degheel, 1998, Thomson, 2001)** the obtained data confirmed this action same where pirimiphos-methyl inhibited the AChE by (-) 56.41% of the exposed insect.

Neonicotinoids are an agonist of nicotinic acetylcholinesterase receptors (nAChR) which mediate fast cholinergic synaptic transmission. Nicotine does not act as an acetylcholinesterase inhibitor **(Gour and Sridevi, 2012)**. This results in agreement with obtained results which showed an increase in the AChE activity of insect treated with clothianidin by (+) 26.53.

Fipronil is potent nerve poison that blocks the ɣ-aminobutyric acid (GABA) regulated chloride channel in neurons caused an excessive release of acetylcholine at presynaptic **(Gour and Sridevi, 2012),** it similar with our results which found an increase in AChE in treatment with fipronil.

Gour and Sridevi, 2012 reported that diamides, caused their toxicity effects by binding and stimulate muscular calcium channels, causing uncontrolled calcium release and resultant muscle contractions. On the other hand **(Bers, 2002; Fill & Copello, 2002; Fessenden et al 2004 and Ogawa, 2008)** evidence the flubendiamide mode of action when caused low concentration or inhibition of free calcium for ryanodine receptor (RyRs) contains two types of Ca^{2+} binding site which stimulatory high-affinity sites and inhibitory low-affinity sites. This result compatibility with the obtained result which recorded a decrease in flubendiamide treatment.

Pyridalyl is toxic for insect cells by causing inhibition of cell growth **(Saito et al 2004 & 2006)**. The insecticide activity and selectivity of pyridalyl may be the results of selection inhibition of cellular protein synthesis **(Moriya et al 2008)**. Conspicuous depletion in total protein content in 4th and 6th *S.littoralis* larval instar treated with pyridalyl recorded by **(Dahi et al 2011, Abdel-Aziz and El-Gohary, 2013)**. The previous data confirm the insecticides mode of action which in agreement with the obtained data of total protein of the exposed insect which decreased by (-) 31.82 % than control. **Nath et al (1997)** mentioned that, protein depletion in tissues may constitute a physiological mechanism and might play a role in compensatory mechanism under insecticidal stress to provide intermediates to the Krebs cycle by retaining free amino acid content in insect hemolymph.

These results similar to the results obtained by**(Gamil et al 2013; Abdel-Aziz and El-Gohary, 2013)** reported a decrease in acetylcholine esterase (AChE) and increased in glutathione -S-transferases (GST) in 2nd larval instar of *S.littoralis* which treated with pyridalyl LC_{50}. **Hatem et al (2017)** reported decrease in total protein content, increase in GST and slight increase in AChE activity in *S.littoralis* larvae which treated by clothianidin than untreated control.

Table 3. Enzymes activity in whole body homogenate of survivor second larval instars of *S.littoralis* after application with LC_{50} of each tested insecticides

Insecticides treatments	AChE activity (µmol/min/mg protein)		Glutathione S-transferases (mmol/min/mg protein)		Phenoloxidase (ΔOD/Min/mg protein)	
	Mean±SE	Change%	Mean±SE	Change%	Mean±SE	Change%
Flubendiamide	0.082±0.016	(-) 21.36	3303.49±398.34*	(+) 149.53	0.38±0.011	(-) 4.31
Pyridalyl	0.067±0.0113*	(-) 34.96	3843.12±26.90*	(+) 190.29	0.38±0.007	(-) 2.63
Fipronil	0.178±0.011*	(+) 70.59	1715.96±10.34	(+) 29.61	0.33±0.005*	(-) 16.20
Clothianidin	0.132±0.010	(+) 26.53	4702.46±80.22*	(+) 255.20	0.36±0.003	(-) 8.90
Pirimiphos-methyl	0.045±0.0048*	(-) 56.41	994.91±19.30	(-) 24.85	0.18±0.006*	(-) 53.06
Spirotetramat	0.083±0.009	(-) 19.82	4262.12±218.95*	(+) 221.94	0.31±0.007*	(-) 21.04
Control	0.104±0.0053	----	1323.89±13.70	----	0.39±0.009	----

Change % = mean activity of treated – mean activity of control / mean activity of control X 100 (according **Paul, 2008**).

* Significant at $p < 0.05$

Table 4. Protein concentrations and enzymes activity in whole body homogenate of survivor second larval instar of *S. littoralis* after application with LC_{50} of each tested insecticide

insecticides treatments	ATPase activity (mmol/min/mg protein)		Total soluble protein (mg/ml homogenate)		Ca++ activity (mmo/L)	
	Mean±SE	Change%	Mean±SE	Change%	Mean±SE	Change%
Flubendiamide	18.71±0.09	(+) 2.33	4.34±0.10*	(-) 38.14	11.71±0.054	(-)1.16
Pyridalyl	17.42±0.38	(-) 4.76	4.78±0.05*	(-) 31.82	11.62±0.014	(-)1.91
Fipronil	17.36±0.14	(-) 5.04	5.23±0.12*	(-) 25.46	11.85±0.19	(+) 0.0033
Clothianidin	18.61±0.11	(+) 1.78	4.91±0.04*	(-) 29.97	11.58±0.024	(-) 2.24
Pirimiphos-methyl	31.62±1.64*	(+) 72.91	5.80±0.02*	(-) 17.32	11.14±0.22*	(-) 6.0
Spirotetramat	17.67±0.62	(-) 3.36	5.65±0.07*	(-) 19.43	11.92±0.32	(+)0.58
Control	18.29±0.328	----	7.02±0.26	----	11.85±0.039	----

Change % = mean activity of treated – mean activity of control / mean activity of control X 100 (according **Paul, 2008**).

* Significant at $p < 0.05$

REFERENCES

Abbott W.S. 1925. A method for computing effectiveness of an insecticide. **J. Econ. Entomol., 18(1), 265-267.**

Abd-Allah S.A.A. 1998. Toxicological studies of some pesticides in relation to their side effects. M.Sc. Fac. Agric. Kafer-Sheikh, **Tanta Univ. pp. 126 -142.**

Abdel-Aziz H.S. and El-Gohary E.S. 2013. Biochemical studies of three new insecticides on cotton leafworm, *S. littoralis* (Boisd.). **Bull. Entomol. Soc. Egypt, Econ. Ser., 39, 77-93.**

Abd-El-Aziz S. Hanan 2014. Effect of some insecticides on certain enzymes of *Spodoptera littoralis* (bosid.). **J. Agric. Res., 92(2), 501-512.**

Abdel-Hafez H.F. and Osman H.H. 2013. Effects of pyridalyl and emamectin benzoate on some biological and biochemical parameters of *Spodoptera littoralis* (Boisd.) and Albino rat. **Egypt. Acad. J. Biolog. Sci., 6(3), 59-68.**

Abdel-Rahim E.F., Azab A.M., Ali M.M., Morsi G.A. and Ahmed M.A. 2009. Comparative toxicity of tree novel biotic compounds; spinosad, pyridalyl and radical in relative to a conventional insecticides, lannate against the field and laboratory strain of the second and fourth instar larvae of cotton leafworm *Spodoptera littoralis* (Boisd). **Egypt. J. Agric. Res., 87(2), 433-453.**

Abdel-Rahim F.M. Elham 2011. Comparative bioresidual activity of pyridalyl and methomy insecticides against larvae of the cotton leafworm, *Spodoptera littoralis* (Boisd.). **Egypt. J. Agric. Res. 89(1), 55-71.**

Abdel-Rahim F.M. Elham and Zidan E.W. 2012. Efficiency of two novel biotic compounds against the laboratory and field strain of the cotton leafworm, *Spodoptera littoralis* (Boisd.). **Egypt. J. Agric. Res. 90(1), 15-30.**

Abou-Taleb H., Barrania K. and Manal A. Attia 2015. A. Comparative Effectiveness of Fipronil and Other Insecticide Treatments against Cotton Leafworm and Role of Two Detoxification Enzymes. **Alex. Sci. Exch. J. 36(4), 342-349.**

Andric G., Kljajic P. and Prazic-Golic M. 2014. Residual efficacy of cypermethrin and pirimiphos-methyl against *Sitophilus granarius* (L.) and *Plodia interpunctella* (Hübner) on concrete surface. **J. Pesticides and Phytomedicine. (Belgrade), 29(4), 275–281.**

Argentine J.A., Jansson R.K., Halliday W.R., Rugg D. and Jany C.S. 2002. Potency, spectrum and residual activity of four new insecticides under glasshouse conditions. **Florida Entomologist, 85(4), 552-562.**

Bakr E. 2007. Ldp line software to calculate probit analysis (*http://www.ehabsoft.com/ldpline/*).

Bers D.M. 2002. Cardiac excitation-contraction coupling. **Nature. 415, 198-205.**

Bhatti, S.S., Ahmed M., Yousaf K. and Naeem M. 2013. Pyrethroids and new chemistry insecticides mixtures against *Spodoptera litura* (noctuidae: Lepidoptera) under laboratory conditions. **Asian J. Agri. Biol., 1(2), 45-50.**

Bradford M.M. 1976. A rapid and sensitive method for quantitation of microgram quantities of pro-

tein utilizing the principle of protein-dye binding. **Anal. Chem. *72*, 248-254.**

Dahi H.F., Aida S., Nehad M. and Abd-El Aziz M.F. 2011. Pyridalyl effectiveness on some biological and physiological parameters of cotton leafworm *Spodoptera littoralis* (Boisd.) (Lepidoptera: Noctuidae). **J. Am. Sci. 7(12), 855-863.**

El-Defrawi M.E., Toppozada A., Mansour N. and Zeid M. 1964. Toxicological studies on the Egyptian cotton leaf-worm, *Prodenialitura* F.I. Susceptibbility of different larval instars of *Prodenia* to insecticides. **J. Econ. Entomol., *57,* 591-593.**

El-Dewy E. Madeha 2017. Influence of Some Novel Insecticides on Physiological and Biological Aspects of *Spodoptera littoralis* (Boisduval). **Alex. Sci. Exch. J. 38(2), 250-258.**

Ellman G.L., Courtney K.D., Andres V. and Featherstone R.M. 1961. A new and rapid colorimetric determination of acetylcholinesterase activity. **Biochem. Pharmacol. *7,* 88-95.**

Fessenden J.D., Feng W., Pessah I.N. and Allen P.D. 2004. Mutational analysis of putative calcium binding motifs within the skeletal ryanodine receptor isoform, RyR1. **J. of Biological Chemistry, 279(51), 53028-53035.**

Fill M. and Copello J.A. 2002. Ryanodine receptor calcium release channels. **Physiological Reviews, 82(4), 893-922.**

Gamil, Walaa; Faiza M. Mariy and Sawsan M. Mohamed 2013. Physiological and histological response of certain larvae of cotton insect pests treated with some novel compound., Arab and Near East Plant Protection Newsletter. Special issue, November **pp. 33-34.**

Gindler M. 1972. Colorimetric method of calcium. **J. Am. Clin. Pathol. 58, 376-378.**

Gour T.B. and Sridevi D. 2012. Molecules with different chemistry in 1st Ed. Chemistry, toxicity and mode of action of insecticides, kalyani publishers, Ludhiana **pp. 191-214.**

Habig W.H., Pabst M.J. and Jakby W.B. 1974. Glutathion-S-transferase: The first step in mercapturic acid formation. **J. Biol. Chem., 249, 7130-7139.**

Hatem A.E., Sorour H.A. and Hassan A.T. 2017. Biochemical parameters, toxicological, development and reproductive effects of two novel insecticides on *Spodoptera littoralis* (Lepidoptera: Noctuidae). **Egypt. Acad. J. Biolog. Sci., 9(3), 135-147.**

Ishaaya I. and Casida J.E. 1974. Dietary TH 6040 alters composition and enzyme activity of housefly larval cuticle. **Pestic. Biochem. Physiol., 4, 484- 490.**

Ishaaya I. and Degheele D. 1998. Insecticide with novel mode of action mechanism and application. **Springer-Verlag Berlin Heidelberg, New York, pp. 263-270**.

IRAC, Insecticide Resistance Action Committee, 2018. *http://www.irac-online.org*

Knight A.L. and Norton G.W. 1989. Economics of agricultural pesticide resistance in arthropodes. **A Rev. Ent. 34, 293-313.**

Kodandaram, M.H., Rai B. and Haldar J. 2010. Novel insecticides for management of insect pest in vegetable crop: **A review. Veg. Sci., 37(2), 109-123.**

Moriya K., Hirakura S., Kobayashi J., Ozoe Y., Saito S. and Utsumi T. 2008. Pyridalyl inhibits cellular protein synthesis in insect, but not mammalian, **Celllines. Arch. Insect Biochem. Physiol. 69(1), 22-31.**

Nath B.S., Suresh A., Varma B.M. and Kumar R.S. 1997. Changes in protein metabolism in hemolymph and fat body of the silkworm, *Bombyx mori* (Lepidoptera: Bombycidae) in response to organophosphorus insecticides toxicity. **Ecotoxicology and Environmental Safety, 36(2), 169-173.**

Ogawa Y. 2008. Distinct mechanisms for dysfunctions of mutated ryanodine receptor isoforms. **Biochemical and Biophysical Research Communications. 369(1), 208-212.**

Paul P. 2008. How to calculate percentage *https://www.wcasa.org/file_open.php?id*=916

Rastegari N. and Subrahmanyam B. 2003. Sublethal Effects of Flufenoxuron, Fipronil and Methoxy-fenozide on *Spodoptera litura* (Fabricius)." **Pesticide Research J. 15(2), 168-172.**

Saito S., Isayama S., Sakamoto N. and Umeda K. 2004. Insecticidal activity of pyridalyl: acute and sub-acute symptoms in *Spodoptera litura* larvae. **J. Pest Sci. 29, 372–375.**

Saito S., Yoshioka T. and Umeda K. 2006. Ultrastructural effect of pyridalyl, an insecticidal agent, on epidermal cells of *Spodoptera litura* larvae and cultured insect cells Sf9. **J. Pestic. 31(3), 335-338.**

Salama H.S. 1985. Some biochemical changes in the larvae of the pink bollworm *Pectinophora gossypiella* (Saunders) in diapause and during termination of diapause. **M.Sc. Thesis, Fac. Sci., Ain Shams Univ., Cairo, Egypt. 155 p.**

Saleh A.A., Elgohary L.R., Watson W.M. and Elabassy A.S. 2015. Toxicity of some conventional and nonconventional insecticides against cotton lefworm, *Spodoptera littoralis* (Boisd.). **J. Plant Prot. and Path., Mansoura Univ., 6(4), 663-673.**

Shepard H.H. 1958. Methods of testing chemicals on insects, (1: 325, ed., Burgess Publishing Company).

Shiosaka T., Okuda H. and Fujii S. 1971. Mechanism of the phosphorylation of thymidine by the culture filtrates of *Clostridium perfringens* and rat liver extract. **J. Biochem. and Biophesis Acta., 246, 171-183.**

Sun Y.P. 1950. Toxicity index-An improved method of comparing the relative toxicity of insecticides. **J. Econ. Entomol., 43, 45-53.**

Thomson W.T. 2001. Agricultural Chemicals, Book I, Insecticides. Thomson Publications, Fresno, California, USA, **249 p.**

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كلية الزراعة، جامعة عين شمس، مارس 2019، القاهرة، مصر
مجلد(27)، عدد(1)، عدد خاص مارس، 837-846، 2019
Website: http://strategy-plan.asu.edu.eg/AUJASCI/

الفعالية والتأثيرات البيوكيميائية لبعض المبيدات تجاه دودة ورق القطن

[75]

رشا أحمد أحمد سليم[1*] – محمد السعيد الزميتى[2] – محمد ابراهيم حسين[2] –
شريفة عبد الحميد نصر الشريف[1]

1– قسم الإختبارات والبحوث الحيوية – المعمل المركزى للمبيدات– مركز البحوث الزراعية دقى– جيزة– مصـــــر
2– قسم وقاية النبات– كلية الزراعة– جامعة عين شمس– ص.ب 68– حدائق شبرا 11241 – القاهرة – مصر

*Corresponding author: rashasleem@yahoo.com

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الموجـــــــز

أجريت الدراسة الحالية لتقييم فعالية بعض المبيدات الكيميائية الحديثة من مجموعات مختلفة وهى: (فلوبينديميد (مجموعة الدياميد)، بريداليل (مجموعة فينيوكسى– بيرادالوكسى)، كلوثانيدين (مجموعة النيونكتينويدز)، فيبرونيل (مجموعة فينيل بيرازول) وسبيروتترامات (مجموعة تيتراميك اسيد) ومقارنة فعاليتها بالمبيد الفسفورى بيريميفوس– ميثيل (مجموعة الفوسفات العضوية) تجاه العمر اليرقى الثانى للسلالة المعملية لدودة ورق القطن. أظهرت النتائج المتحصل عليها بعد 48 ساعة من المعاملة بطريقة غمر الاوراق أن أكثر المبيدات سمية هو الفلوبينديميد حيث سجل التركيز النصفى المميت له (1.03 جزء فى المليون)، ثم مبيد البريداليل حيث سجل التركيز النصفى المميت له (2.13 جزء فى المليون)، ثم فيبرونيل حيث سجل التركيز النصفى المميت له (7.42 جزء فى المليون)، كلوثانيدين حيث سجل التركيز النصفى المميت له (26.87 جزء فى المليون) وسبيروتترامات حيث سجل التركيز النصفى المميت له (431.91. جزء فى المليون) وكانت نتيجة التركيز النصفى المميت لمبيد بيريميفوس– مثيل (76.31 جزء فى المليون) وتدل هذه النتائج أن جميع المبيدات الحديثة المختبرة كانت أكثر فعالية من البيريميفوس– ميثل ماعدا مبيد سبيروتترامات.

دلت نتائج تقدير المعايير البيوكيميائية (الأستيل كولين – الجلوتاثيون– اس– ترانسفيريز –أدينوزين تراى فوسفات– الفينول أوكسيديز– الكالسيوم الكلى والبروتين الكلى) وجود زيادة معنوية فى نشاط الاستيل كولين بقيمه (70.59%) سجلتها المعاملة بمبيد الفيبرونيل، بينما سجلت المعامله لمبيد بيريميفوس – ميثيل انخفاض معنوى بقيمة (–56.41 %). سجلت جميع المعاملات زيادة فى نشاط الجلوتاثيون–اس–ترانسفيريز ماعدا المعاملة بمبيد البيريميفوس– ميثيل، بينما سجلت جميع المعاملات بالمبيدات المختبرة انخفاض فى نشاط الفينول أوكسيديز وسجلت المعاملة لمبيد البيريميفوس– ميثيل اعلى انخفاض معنوى بقيمة (–53.06). سجلت المعاملة بمبيد بيريميفوس ميثيل أعلى نشاط معنوى فى انزيم الادينوزين تراى فوسفات بقيمة (72.91%). سجلت جميع المعاملات انخفاض معنوي فى المحتوى الكلى للبروتين. ومن الناحية الأخرى المعاملة بيريميفوس – ميثيل سجلت أقل إنخفاض معنوى فى نشاط الكالسيوم بقيمة (–6.0 %). وتؤكد النتائج السابقة على طريقة فعل المبيدات الحديثة المختبرة .

الكلمات الدالة: دودة ورق القطن، مبيدات كيميائية حديثة، دراسات توكسيكولوجية، بيوكيميائية

تحكيم: ا.د هانـــي محمود عاشـــور
ا.د سيد عبد اللطيف دحروج