

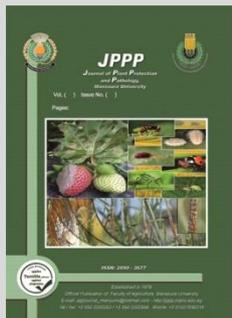
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### Toxicity and Biochemical Effect of Certain Insecticides against *Spodoptera littoralis* (Boisd.)

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#### ABSTRACT

The efficiency and biochemical effects of certain insecticides belonging to different groups namely: emamectin benzoate (Avermectin<sup>R</sup>), flubendiamide (Diamide), lufenuron (IGR) as pyridalyl (phenyl-pyridaloxo) were tested against 4<sup>th</sup> larval instar of *Spodoptera littoralis* (Boisd.) laboratory and field strains using dipping technique. Biochemical effects of the tested insecticides on acetylcholinesterase (AChE) and glutathione-S-transferases (GST) were determined in the treated larvae. The efficacy of the tested insecticides is arranged as follows to LC<sub>50</sub> values. The results showed that that flubendiamide was the superior toxicant insecticides (LC<sub>50</sub> values were 0.863 ppm and 2.73 ppm for lab and field strains respectively) followed by emamectin benzoate (LC<sub>50</sub> values were 1.8 ppm and 10.13 ppm for lab and field strains respectively) then pyridalyl (LC<sub>50</sub> values were 4.17 ppm and 38.2 ppm for lab and field strains respectively) and lufenuron (LC<sub>50</sub> values were 5.4 ppm and 76.92 ppm for lab and field strains respectively). The data revealed that the treatment with tested insecticides demonstrated inhibition in AChE and GST activity. Based on the results of the present study, it was confirmed the potency of these insecticides against *S. littoralis* therefore, should be use their use in control programmes.

**Keywords:** Acetylcholine esterase (AChE), glutathione-S-transferase (GST), *Spodoptera littoralis* (Boisd.)

#### INTRODUCTION

The Egyptian cotton leafworm, *Spodoptera littoralis* (Boisd.) is widely distributed in many countries and regions worldwide. *S. littoralis* is one of the most important lepidopteron pests which attack numerous economically important crops around the year causing economic losses. Conventional insecticides e-g organophosphates, pyrethroids and carbamates (Hilliou *et al.*, 2021) have played a major role in *S. littoralis* management. Unfortunately, their indiscriminate and continuous uses led to several problems as a result, this pest may develop resistance to those insecticides in many areas. Therefore, the continuous evaluation of the insecticides efficiency for controlling the insect in different areas became urgent. This will give the chance to replace the failed controlling agents by the effective alternatives. Moreover, the establishment of baseline susceptibility and the mechanism of resistance are necessary for effective resistance management strategies (Ismail *et al.*, 2020). The current trend in insect pests' controls is search for development of new control agents having novel biochemical targets to solve pest resistance problems ((Silva *et al.*, 2016; Sridhar *et al.*, 2016 and Ismail, 2020). In recent years, several new insecticidal groups having new chemistries like were commercially formulated and were traded in the egyptian market. Therefore, insecticides groups having new chemistries represent to different modes of action like; emamectin benzoate (Avermectin<sup>R</sup>), flubendiamide (Diamide<sup>R</sup>), lufenuron (IGR) and pyridalyl (phenyl-pyridaloxo) and estimate their biochemical effects on acetylcholinesterase (AChE) and glutathione-S-transferase (GST) as a results of treatment with the LC<sub>50</sub> values of these insecticides for 72hrs in the whole body tissues of the 4<sup>th</sup> instar larvae of *S. littoralis*.

#### MATERIALS AND METHODS

##### Experimental insect:

The laboratory strain of cotton leafworm, *Spodoptera littoralis* (Boisd.) was maintained under tightly conditions in the insect rearing room at 25±1 °C, with 65±5 % relative humidity avoiding any intentional chemical pressure in Central Agricultural Pesticides Laboratory, Agriculture (Capl) Research Center, (ARC) Egypt. Field strain of *S. littoralis* egg masses were collected from cotton fields of Kafr El-Sheikh, Governorate, Egypt.

##### Tested insecticides:

Emamectin benzoate (Radical<sup>®</sup> 1.9% EC) produced by Agromen Chemical Co., flubendiamide (Takumi<sup>®</sup> 20% WG) produced by Samtrade Co., lufenuron (Match<sup>®</sup> 5% EC) produced by Dow Agro Sciences and pyridalyl (Pleo<sup>®</sup> 50%EC) produced by sumitomo chemical. Co.

##### Bioassay studies:

Leaf-dipping technique was used to estimate the susceptibility of 4<sup>th</sup> larval instar of *S. littoralis* to the tested insecticides. The castor leaves (*Ricinus communis*) were dipped for 30 seconds in six serial concentrations of an aqueous solution to each insecticide. Three replicates were used each of which has 10 larvae of *S. littoralis*. The mortality was recorded after 3 days after treatment and corrected by Abbott's formula (1925) and subjected to probit-analysis method of Finney (1971).

##### Biochemical studies:

##### Sample preparation:

The survivor fourth larval instars were homogenizing in distilled water (1 gm/ml) using hard glass homogenizer on ice jacket then centrifuged at 8000 r. p. m for 15 min at 5 °C. The supernatant was kept at -20°C until used.

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**Enzyme activity:**

- a- Acetylcholinesterase (AChE) activity was determined according to the method described by Ellman *et al.* (1961).
- b- Glutathione-S-transferase (GST) activity was measured by the method of Habig *et al.* (1974).

**Statistical analysis:**

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

**RESULTS AND DISCUSSION**

**Toxicity of tested insecticides to 4<sup>th</sup> instar larvae of lab and field strains of *Spodoptera littoralis* (Boisd.):**

The potency of the four tested insecticides was evaluated against 4<sup>th</sup> larval instar of *Spodoptera littoralis* (Boisd.) as shown in Table (1). The results showed that flubendiamide was the superior toxicant insecticides (LC<sub>50</sub> values were 0.863 ppm and 2.73 ppm for lab and field strains respectively) followed by emamectin benzoate (LC<sub>50</sub> values were 1.8 ppm and 10.13 ppm for lab and field strains respectively) then pyridalyl (LC<sub>50</sub> values were 4.17 ppm and 38.2 ppm for lab and field strains respectively) and lufenuron (LC<sub>50</sub> values were 5.4 ppm and 76.92 ppm for lab and field strains respectively). The results confirmed that flubendiamide and emamectin benzoate were highly toxic against *S. littoralis* larvae than pyridalyl and lufenuron. Generally, field strain exerts tolerance ratios higher than lab strain. Field strain showed tolerance ratios of 3.17, 5.57, 9.16 and 14.24 to flubendiamide, emamectin benzoate, pyridalyl and lufenuron, respectively. In the present study, the tested different insecticides have different mode of action and showed low levels of tolerance, respectably for pyridalyl and lufenuron. Regarding emamectin benzoate and pyridalyl, these results are in agreement with Ismail and Sleem (2021), who reported that, the field strain of the *Agrotis ipsilon* have proved to be more susceptible to emamectin benzoate followed by pyridalyl than lab strain. In respect with flubendiamide and lufenuron, the results of this study are in accordance with Mushtaq and Sanobar (2017) who reported that, low resistance was recorded to flubendiamide and emamectin benzoate, lufenuron and methoxyfenozide in some populations of *S. litura*.

**Table 1. Median lethal concentrations, Confidence limits (CL) and Slope ± SE of tested insecticides on *Spodoptera littoralis* (Boisd.) larvae.**

| Insecticides       | Strains | LC <sub>50</sub> (ppm) | 95% CL       | Slope ± SE | *TR   |
|--------------------|---------|------------------------|--------------|------------|-------|
| Emamectin benzoate | Lab     | 1.8                    | (1.4-2.6)    | 2.4±0.25   | 5.57  |
|                    | Field   | 10.13                  | (6.57-12.44) | 1.97±0.23  |       |
| Flubendiamide      | Lab     | 0.863                  | (0.65-1.30)  | 0.98±0.43  | 3.17  |
|                    | Field   | 2.73                   | (2.47-3.12)  | 1.86±0.28  |       |
| Pyridalyl          | Lab     | 4.17                   | (3.04-5.34)  | 1.25±0.17  | 9.16  |
|                    | Field   | 38.2                   | (30.6-48.51) | 1.5±0.16   |       |
| Lufenuron          | Lab     | 5.4                    | (4.1-7.3)    | 1.9±0.21   | 14.24 |
|                    | Field   | 76.92                  | (6.57-12.44) | 1.59±0.41  |       |

\*TR = Tolerance ratio (LC<sub>50</sub> of field strain / LC<sub>50</sub> of lab strain).

**Activity of GST and AChE in the lab and field strains of *Spodoptera littoralis* (Boisd.):**

**GST:**

Data in Tables (2 and 3) indicated that treatments with four tested insecticides caused reduced in enzyme activity compared with control. The most reduced GST activity was noted with flubendiamide treatment (-53.65 and

-55.34% for lab and field strains respectively) followed pyridalyl (-34.08 and -39.56% for lab and field strains respectively) and emamectin benzoate -26.25 and 32.41%, respectively compared with control. The lowest effect on GST activity in case treatment with lufenuron.

**AChE:**

The data in Tables (2 and 3) clearly showed the activity of AChE it was clear significant decrease in treatment with flubendiamide by 0.474 (µmol/min/mg protein) and change% (29.36%) of lab strain while, field strain 0.664 (µmol/min/mg protein) and change% (32.86%) followed by emamectin benzoate and pyridalyl. While treatment with lufenuron cause lowest effect of AChE activity 0.613 µmol/min/mg protein) and change% (8.64%) of lab strain while, field strain 0.880 µmol/min/mg protein) and change% (12.39%).

In general, activity of AChE or GST could be strongly inhibited, *in vivo*, by different insecticides, depending on the insect species and enzyme inhibitors (Wu *et al.*, 2007).

These results similar to the results obtained by Ismail and Sleem (2021) who reported significantly decreased activity of GST in *A. ipsilon* larvae, lab and field strains after treatment with emamectin benzoate application while non-significantly inhibited following treatment with lufenuron. Furthermore, results agreed with Abdel-Aziz (2014) who aforementioned that the emamectin benzoate and pyridalyl were the most inhibitive of GST activity. Also, the results agreed with Abdel-Aziz and El-Gohary, (2013) reported a decrease in acetylcholine esterase (AChE) in 2nd larval instar of *S. littoralis* which treated with pyridalyl LC<sub>50</sub>.

**Table 2. Effect of tested insecticides on some enzymes activities in fourth larval instar of *Spodoptera littoralis* (Boisd.) lab strain.**

| Insecticides       | AChE activity         |           | GST activity          |           |
|--------------------|-----------------------|-----------|-----------------------|-----------|
|                    | (µmol/min/mg protein) |           | (nmol/min/mg protein) |           |
|                    | Mean ±SE              | Change%   | Mean ±SE              | Change%   |
| Control            | 0.671a±0.01           | -----     | 59.65a±3.9            | -----     |
| Emamectin benzoate | 0.552d±0.01           | (-) 17.73 | 43.99c±1.5            | (-) 26.25 |
| Flubendiamide      | 0.474e±0.01           | (-) 29.36 | 27.65e±2.2            | (-) 53.65 |
| Lufenuron          | 0.613b±0.01           | (-) 8.64  | 53.34b±1.4            | (-) 10.58 |
| Pyridalyl          | 0.583c±0.01           | (-) 13.11 | 39.32d±2.8            | (-) 34.08 |

**Table 3. Effect of tested insecticides on some enzymes activities in fourth larval instar of *Spodoptera littoralis* (Boisd.) field strain.**

| Insecticides       | AChE activity         |           | GST activity          |           |
|--------------------|-----------------------|-----------|-----------------------|-----------|
|                    | (µmol/min/mg protein) |           | (nmol/min/mg protein) |           |
|                    | Mean ±SE              | Change%   | Mean ±SE              | Change%   |
| Control            | 0.989a±0.01           | -----     | 67.11a±2.6            | -----     |
| Emamectin benzoate | 0.790cd±0.010         | (-) 20.12 | 45.36c±2.0            | (-) 32.41 |
| Flubendiamide      | 0.664e±0.01           | (-) 32.86 | 29.97e±1.9            | (-) 55.34 |
| Lufenuron          | 0.880b±0.01           | (-) 12.39 | 54.00b±3.3            | (-) 19.54 |
| Pyridalyl          | 0.810c±5.3            | (-) 18.11 | 40.56d±2.3            | (-) 39.56 |

**CONCLUSION**

In conclusion, this study indicated that the four tested insecticides had high toxicity on larvae of *S. littoralis*; the toxicities resulted mainly from different degree of effect on the activity of the tested enzymes. So, these insecticides are a useful tool in integrated pest management (IPM) programs of *S. littoralis*.

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### السمية والتأثير البيوكيميائي لبعض المبيدات الحشرية على دودة ورق القطن (*Spodoptera littoralis* (Boisd.))

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المعمل المركزي لمتبقيات المبيدات - مركز البحوث الزراعية - الجيزة - مصر

هدف الدراسة الحالية هو تقييم فاعلية بعض المبيدات الكيميائية من مجموعات مختلفة وهي: الايماميكثين بنزوات (مجموعه الأفيروماكتين)، فلوبيندياميد (مجموعه الدياميد)، البيريديليل (مجموعه فينووكسى- بيرادالوكسى)، ولوفينورون (مجموعه منظّمات النمو) ومدى سميتها على دودة ورق القطن عند المعاملة على العمر اليرقى الرابع للسلاطين المعملية والحقلية وكذلك متحدثه هذه المبيدات من تغيرات على النشاط الانزيمى للحشرة بعد تغذيتها على اوراق معاملة بالجرعة نصف المميّنة لكل مبيد وتقيم التغيرات فى نشاط بعض الإنزيمات مثل الأسيثيل كولين و الجلوتاتيون - اس - ترانسفيريز. وقد اظهرت النتائج المتحصل عليها بعد المعاملة بطريقه غمر الاوراق أن أكثر المبيدات سمية هو الفلوبيندياميد حيث سجل التركيز النصف المميّنه له (0.863 و 2.73 جزء فى المليون للسلاطين المعملية والحقلية على التوالي) يليه الايماميكثين بنزوات حيث سجل التركيز النصف المميّنه له (1.8 و 10.13 جزء فى المليون للسلاطين المعملية والحقلية على التوالي) ثم البيريديليل حيث سجل التركيز النصف المميّنه له (4.17 و 38.2 جزء فى المليون للسلاطين المعملية والحقلية على التوالي) بينما كانت نتيجة التركيز النصف المميّنه لمبيد الوفينورون (5.4 و 76.92 جزء فى المليون للسلاطين المعملية والحقلية على التوالي). وقد اسفرت النتائج عن ان المعاملة بهذه المركبات ادت إلى نقص فى نشاط كلا من الانزيمين. بناءً على نتائج الدراسة الحالية، وتم التأكد من فاعلية هذه المبيدات مما يجعلها احدي الطرق الفعالة فى نظام المكافحة المتكاملة لدودة ورق القطن.