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Insecticidal bioefficacy screening of some chalcone and acetophenone hydrazone derivatives on *Spodopetra Frugiperda* (Lepidoptera: Noctuidae)

Mohamed Sayed A. El-Gaby[a], Yousry A. Ammar[b], Ali M. Drar[c] and Mohamed A. Gad[c*]

[a]*Department of Chemistry, Faculty of Science, Al-Azhar University at Assiut, Assiut 71524, Egypt*
[b]*Department of Chemistry, Faculty of Science (Boys), Al-Azhar University, Nasr City, Cairo11884, Egypt*
[c]*Research Institute of Plant Protection, Agriculture Research Center, 12112 Giza, Egypt*

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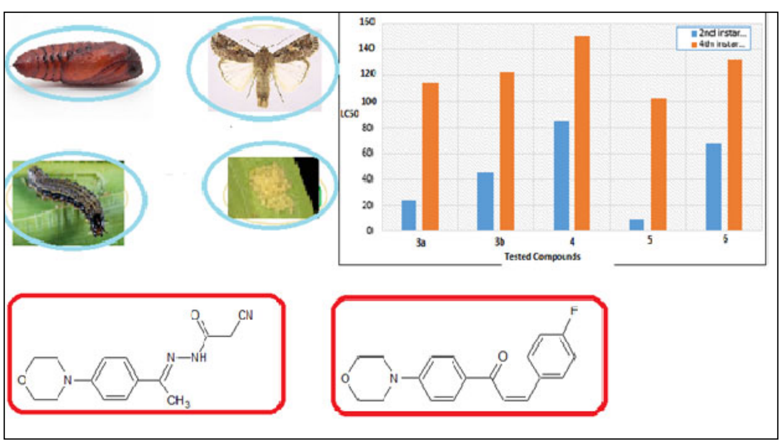
A B S T R A C T

Spodopetra Frugiperda is a highly polyphagous migratory lepidopteran pest species. It causes infestation in crops leading to severe crop losses. Being a new invasive parasite, its susceptibility to insecticides needs to be explored and therefore, there is an urgent need to develop the potent insecticides for the effective control of this insect pest. This is the first report on toxic effects produced by the prepared chalcone and hydrazone analogs, followed by structure relationships. Five compounds of chalcone and hydrazone derivatives have been synthesized in pure state as reported procedures, and their toxicity as potential insecticidal agent against *Spodopetra Frugiperda* was screened. Their characterizations by using spectroscopic analyses were performed. The toxicity data exhibited that compound **5** is more toxic about 2-fold than other synthesized compounds, the other screened compounds showed weak to strong toxicological activity against *Spodopetra Frugiperda* (lepidoptera: Noctuidae).



Graphical Abstract

* Corresponding author.
E-mail address: samy_adjey@yahoo.com (M. A. Gad)

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1. Introduction

The fall armyworm (*Spodopetra Frugiperda*) is a species in the order Lepidoptera and one of the species of the fall armyworm moths distinguished by their larval life stage.[1] The term "armyworm" can refer to several species, often describing the large-scale invasive behavior of the species' larval stage. It is regarded as a pest and can damage and destroy a wide variety of crops, which causes large economic damage.[2] Its scientific name derived from *frugiperda*, which is Latin for lost fruit, named because of the species' ability to destroy crops.[3] *Spodotera frugiperda* also known as the fall armyworm is a highly polyphagous migratory lepidopteran pest species.[4] It infects a wide range of crops including wheat, soybean, millets, peanut, sorghum, sugarcane and corn. It causes infestation in crops leading to severe crop losses. It is a destructive insect pest responsible for major problems in agricultural crop production, especially maize. The abilities of this insect pest to breed rapidly, migrate, and feed on a wide range of host plants, makes it very difficult to control.[5] Selection of proper insecticide is one of the important requirements for crop sustainability, which ensures the less toxicity to humans and more specific to key pests. The presently used pesticides for agricultural work to control insects and mealybugs are highly toxic in nature for human life. To achieve the ecofriendly, egalitarian and ethical pest management, there is a need of a mechanism which provides pest specific, less toxic and cost effective procedure.[6]

Chalcones are commonly known as α, β-unsaturated ketone consisting of two aromatic rings which are joined by three carbon chain. Chalcones are considered as one of the most significant type of natural products found in various plant species.[7] Chalcone compounds extensively is present in occurring in nature, is the natural organic-compound that a class is present in multiple medicinal plants such as Radix Glycyrrhizae, safflower, and its basic skeleton structure is 1,3-diphenylprop ketenes. Because the chalcone compounds molecule has bigger flexibility, can with different receptors bind, have many physiology and pharmacologically active, a lot of patents are reported their activity in medicine, as active anticancer.[8] Also, many hydrazone derivatives have been reported to possess broad spectrum insecticidal activity and are used as active ingredients for controlling agricultural and horticultural pests.[9-11]

Seeking pesticide activity from the natural resources treasure-house, utilize it to carry out the initiative of lead optimization and novel pesticide as model, is the focus of pesticide science research field in recent years.[12] Up till now for this reason, yet there are no chalcone compounds carries out control of plant disease and agricultural insect pests control as agricultural chemicals application. The objective of the invention is to expand the purposes of chalcone and acetophenone hydrazone derivatives, at pesticide field is provided.[13]

2. Materials and methods

2.1. Chemicals

Compounds **3a, 3b, 4, 5** and **6** were obtained according to the literature procedure.[14, 15] The larvae instar of *Spodopetra Frugiperda* (lepidoptera: Noctuidae insects were gathered from experimental farm fields of Agricultural research center, Sohag branch during 2021 season.

2.2. Laboratory Bioassay

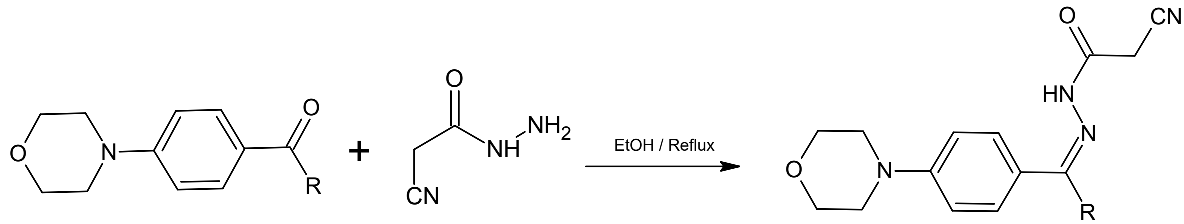
The insecticidal activity of all synthesized chalcone derivatives was measured via the leaf dip bioassay methods.[16-20] The results of laboratory testing are reported here for the target compounds to find out the required concentrations which are demanded to kill 50% (LC_{50}) of the 2nd instar larvae and 4th instar larvae of *Spodopetra Frugiperda* insects. In this work, five concentrations of each prepared of chalcone derivatives and 0.1% tween 80as surfactant were used. Nearly the same size 10 of 2nd instar larvae and 5 of 4th instar larvae insects where put in disks (9 cm diameter) of castor bean leaves which dipped in the tested concentration for 10 s, then left to dry and afforded to 2nd and 4th larvae, nearly of the same size. The larvae were placed in glass jars (5 pounds), and every treatment was replicated three times (10 larvae per each). Control disks were dunked in distilled water and Tween 80, then transferred to the untreated larvae, which were allowed to feed on castor beans for 48 h. Mortality percentage was recovered after 72 h for all insecticides. Mortality was redressed by Abbott's formula.[21] The measurements of the mortality relapse line were dissected by probit analysis.[22] Harmfulness index was determined by sun equations.[23]

3. Results and discussion

3.1. Chemistry

As following our project in prepared and toxicity evaluate the biological activity of chalcone analogues, here in five tested compounds where shown in (**Schemes 1, 2**) to determine their toxicity as insecticides. The five compounds, namely, cyanoacetic acid [4-4-(morpholin-4-yl)benzylidenyl]hydrazide (**3a**), 2-cyano-*N'*-(1-(4-morpholinophenyl) ethylidene)acetohydrazide (**3b**), 1-(4-morpholinophenyl)-3-phenylprop-2-en-1-one (**4**) , 3-(4-fluorophenyl)-1-(4-morpholinophenyl)prop-2-en-1-one (**5**), 1,3-bis (4-morpholino phenyl) prop-2-en-1-one (**6**).

Hydrazones **(3a)** and **(3b)** were prepared according to a reported procedure and spectral characteristics for our target synthesized compounds are registered and compared with data from literature.[14,15] A mixture of carbonyl compound **1** (0.01 mol) and cyanoacetic acid hydrazide **2** (0.01 mol) in absolute ethanol (20 mL) was refluxed for 1 h. After cooling the separated crystalline products were filtered, washed with a minimum amount of cold methanol, dried and recrystallized from the proper solvent. The synthesis of some hydrazones (**3a, b)** is illustrated and outlined in Scheme 1.



(1a, b) (2)

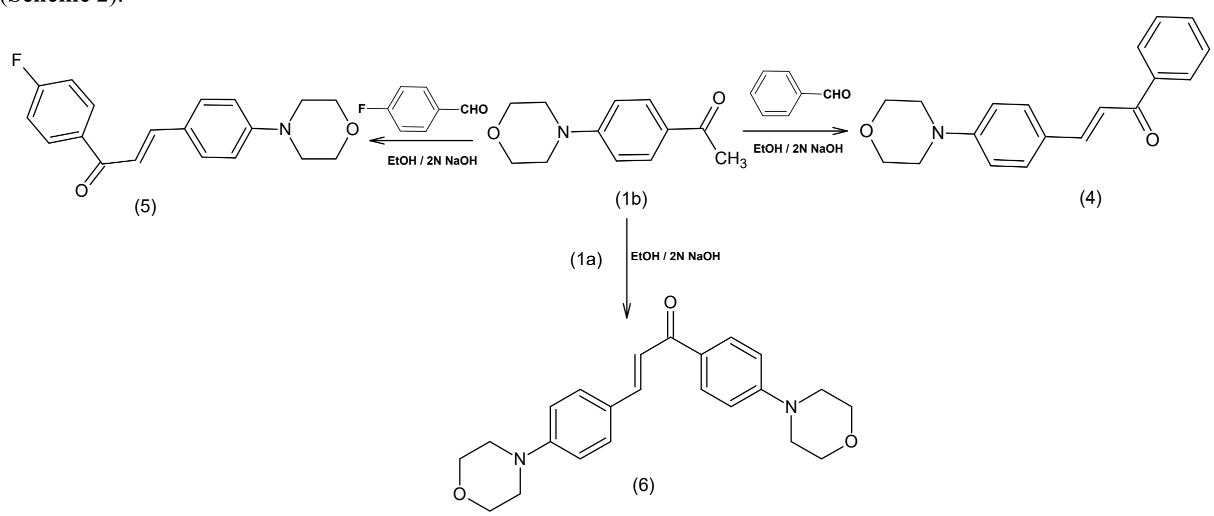
1a and 3a; R = H

1a and 3b; R= CH_3

.

Scheme 1. Synthesis of hydrazones **(3a)** and **(3b).**

Chalcones **4, 5** and **6**wereprepared as previously described *via Claisen-Schmidt* condensation of appropriate benzaldehyde with ethanone derivative **1b** at room temperature in the presence of 2*N* sodium hydroxide as a base catalyst (**Scheme 2**).[15]



Scheme 2. Synthesis of chalcones **4, 5** and **6.**

Table 1. Insecticidal activity of compounds **3a, 3b, 4, 5** and **6** against the 2nd instar larvae and 4th instar larvaeof *S. Frugiperda* after 72h of treatment.

2nd instar larvae				4th instar larvae		
Comp.	LC_{50} (ppm)	Slope	Toxic ratio	LC_{50} (ppm)	slope	Toxic ratio
3a	23.69	0.740 ± 0.248	0.417	114.01	1.213±0.297	0.900
3b	45.10	0.925 ± 0.255	0.219	122.48	1.188±0.298	0.836
4	85.67	1.417 ± 0.303	0.115	151.068	1.099±0.297	0.679
5	9.882	0.961 ± 0.261	1	102.66	1.281±0.298	1
6	67.56	1.410 ± 0.269	0.146	132.09	1.162± 0.299	0.777

3.2. Insecticidal bio-efficacy screening

All the target synthesized compounds have been screened for insecticidal bioefficacy as explained as following:

3.2.1. Toxicological activity test for nymphs of 2nd instar larvae of Spodopetra Frugiperda after 72h of treatment.

Results of compound **3a, 3b, 4, 5 and 6** were tested against 2nd instar larvae of *Spodopetra Frugiperda* are shown in **Table 1**. After 72 h of treatment, bioefficacy results of synthesized chalcone derivatives compounds exhibit high to low toxicological activity against *S. Frugiperda* after 72 hrs of the test with LC_{50} values vary from 23.69 to 85.67 in which for example LC_{50} value of compounds **3a, 3b, 4, 5** and **6** were 23.69, 45.10, 85.67, 9.88, and 67.56 ppm respectively. For this result, the toxicity of compound **5** was the most toxicological activity against 2nd instar larvae of *Spodopetra Frugiperda* after 72 hrs of treatment than the other synthesized chalcones derivatives compounds**.**

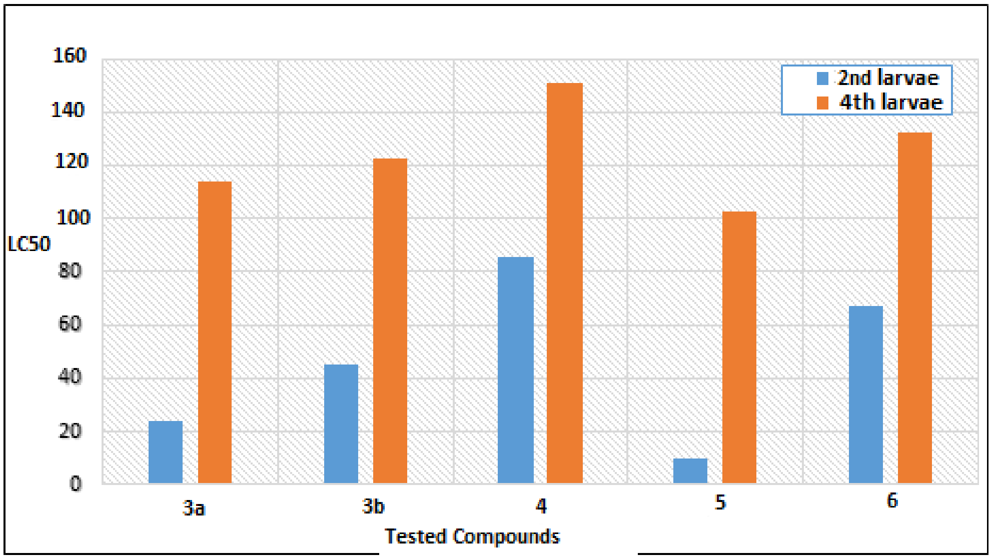


Fig1. Insecticidal activity of compounds **3a, 3b, 4, 5** and **6** against the 2nd and 4th larvae of *S. Frugiperda* after 72 h of treatment compared.

3.2.2. Toxicological activity test for 4th instar larvae of Spodopetra Frugiperda after 72h of treatment.

Results of compound compounds **3a, 3b, 4, 5** and **6** were tested against4th instar larvae of *Spodopetra Frugiperda* are shown in **Table 1**. After 72h of treatment, bioefficacy results of synthesized compounds exhibit high to low toxicological activity because the number of them were as nearly in activity than others after 72 hrs of the test with LC_{50} values vary from 102.66 to 151.068 in which LC_{50} value of compounds **3a, 3b, 4, 5** and **6** were 112.01, 122.48, 151.06, 102.66 and 132.09ppm respectively. For this result, the toxicity of compound **5** against 4th instar larvae of *S. Frugiperda* was the most active after 72 hrs of the test because LC_{50} value of compound **5** is 102.66 ppm.

4. Structure-action relationship

As a continuation of this work, a structure-action relationship was introduced here according to the toxicity value in the **Table 1** and **Fig. 1**. It is shown that the synthetic chalcone derivatives compound **5** are more active against 2nd instar larvae and 4th instar larvae of *Spodopetra Frugiperda* than the other chalcone and hydrazone synthesized derivatives. The high activity associated with compound **5**, **3a** and **3b** may be due to the presence of fluorophenyl, cyano group and methylidene acetohydrazide nucleus in their structure**.** Structure-relationship was introduced according to the toxicity value in the **Table 1** it appears from the general framework structure of chacone prepared compound **5** are more active against 2nd instar larvae and 4th instar larvae of *Spodopetra Frugiperda* than other tested compounds. The high activity associated with compound **5** may be due to the presence of fluorophenyl, and methylidene acetohydrazide nucleus in their structure. This work is considered important because it ensures importance of applied sciences as reported before.[24-41]

5. Conclusion

A series of chalcone and acetophenone hydrazone derivatives which are α,β-unsaturated ketone consisting of two aromatic rings were chemically prepared. The toxicity of these compounds was estimated against *Spodopetra Frugiperda* (Lepidoptera: Noctuidae) and showed that a number of the prepared compounds possess good toxicological activities as insecticides. Especially, compound **5** had the best insecticidal activity against 2nd instar larvae and 4th instar larvae of

Spodopetra Frugiperda than the other chalcone and hydrazone synthesized derivatives. The activity concerning compound **4** may be due to the existence of the fluorophenyl methylidene acetohydrazide nucleus attached to molecular structure. These results are hopeful and valuable for additional work on the improvement of new and other potent pesticides. Our research demonstrated that the chalcone and acetophenone hydrazone derivatives could effectively control against 2nd instar larvae and 4th instar larvae of *Spodopetra Frugiperda* and this emphasis our studies done before.[14, 15]

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