Chemical Design, Synthesis and Bio-efficacy Screening of New Growth Inhibitors of *Spodoptera littoralis* (Boisd.)

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Abstract

The present work aimed to find new growth inhibitors agents *spodoptera littoralis* (Boisd.), several inhibitors structurally relevant to the insect growth regulator, Fenoxycarb and the naturally transpiring juvenile hormone of insects were chemically designed, prepared and evaluated as anti-proliferative agents. Epialohydrins derivatives have been synthesized and their agricultural bio-efficacy as insecticides against *spodoptera littoralis* (Boisd.). Insecticidal bio-efficacy data showed that that some compounds are very active against *spodoptera littoralis* (Boisd.).

Keywords: Insecticidal bio-efficacy; *Spodoptera littoralis* (Boisd.); Insect growth regulator; The toxicity ratio

Graphical Abstract

Introduction

Spodoptera littoralis (Boisduval) (Lepidoptera: Noctuidae) is considered one of the key pests that cause great damage to cotton plants as well as other plants in Egypt [1,2] larvae of this insect can feed on about ninety economically, monetarily and significant plant pertinence to forty families. The achieved control is not successful, when using synthetic insecticides [3] and some biorational agents for example *Bacillus thuringiensis* is Berliner to resist the pest, because of the insect's high ability to develop resistance toward most of traditional components [4]. Therefore, we need novel compounds that are efficient against this insect, safe to human and ecological well-disposed [5,6]. The substitutional control techniques that show presage as a potential tactic in *S. littoralis* the executive's projects is the utilization of biorational control operators for example synthetic insect growth regulators and those dependent on normally materials [7]. Insect growth regulators are development controllers are ventured to be more secure for useful living beings than regular mixes, and they have been effectively utilized in IPM programs against many tree and little natural product [8,9]. There is a need for different insecticides having different modes of action Juvenile hormones analogues [10-13] sesquiterpenoid moieties arranged and discharged by the corpora allata, are significant insect hormones that standardize a bulky diversity of processes during postembryonic growth and adult reproduction in insects [14]. Juvenile hormone analogues display powerful creepy crawlly adolescent hormone-mirror action, and consequently cause genuine aggravations in the advancement, multiplication, and conduct of a wide scope of bug [15-16]. This carbamate is utilized for pests' control in farming, ranger service, and put away items, and is additionally utilized as a general wellbeing bug-spray Juvenile hormone analogue is recorded for control of caterpillars, and scales in Europe, and for flame ants in the U.S. Like most IGRs, fenoxycarb has surprisingly low human toxic quality and is significantly more bug particular than the ordinary bug sprays [17]. The main objective of this study was to determinate the toxicity of new growth inhibitors agents against the cotton leafworm, *S. littoralis* larvae under laboratory conditions.

Results and Discussion

The start reagent can be prepared by the reaction of 2-naphthol

with epichlorohydrin in presence of catalytic amount of sodium hydroxide afforded of 2-((2naphthyloxy) methyl)-oxiran1a (Figure 1-3).

Figure 1: Synthesis of 2-((2naphthyloxy) methyl)-oxiran1a

Figure 2: The nucleophilic attack of compound 1a

Figure 3: Oxidation of compound 4

Experimental

A Fisher-Johns apparatus was exercised to register the MP. of every synthesized compound. Infra-red and elemental analyses (C, H, N, and S) were accomplished through a Pye Unicam SP3-100 spectrophotometer utilizing the KBr disk manner and a Vario EL C, H, N, S analyzer, separately. A Bruker 400MHz spectrometer was utilized to measure DEPT 135 spectra and the ^1H and ^{13}C NMR

spectra within the TMS as an interior standard. Chemical shifts were estimated in ppm. Reaction headway and perfection of the prepared sections were checked by thin layer chromatography. The insecticidal reference, fenoxycarb (insect growth regulators), was achieved from Sigma Aldrich (France), *spodopetra littoralis* (Boisd) were combined from cotton area of the exploratory farm of Agricultural research center. The insecticidal effective of the

reported fenoxycarb juvenile hormones as active ingredient and the prepared components was screened against larva of the *spodopetra littoralis* (Boisd).

Synthetic Procedure for 2-[(naphthalen-2-yloxy) methyl] oxirane(1a). According to the reported procedure, by reaction of 2-naphthol(0.04mol) with epichlorohydrin (0.12mol) and sodium hydroxide 25% in water was stirred in ice bath for 4 h, compound 1a was prepared, The formed precipitate was filtered off and recrystallized from methanol in which give white powder of 1a. Yield: 81%; MP:92-94 °C. IR (v) (KBr) cm^{-1} , 3049 (C-H aromatic), 2913, 2880 (C-H aliphatic); ^1H NMR (DMSO- d_6): δ 6.7 (m, 7H, Ar-H), 4.47, 3.91, 3.41, 2.77, 2.76 for (s, 1H, CH), respectively. Elemental analysis calculated for $\text{C}_{13}\text{H}_{12}\text{O}_2$ (%) Calcd. /found C, 77.98/77.97; H, 6.04/6.02.

General Procedure of Synthetic 3-Cyano-4, 6-distyrylpyridine-2(1H)-thione (1-4, 611). A mixture of compound 1a (1mmol), nucleophile (3mmol) and drops of tri ethylamine in ethanol was stirred and refluxed at ambient temperature for 5h to give a precipitate which filtered off and recrystallized from 1,4-Dioxan. 1-(naphthalen-2-yloxy)-3-(piperidin-1-yl) propan-2-ol (1) Pale red crystals. Yield: 91%; MP: 119–122 °C. IR (v) (KBr) cm^{-1} : 3362 (OH), 2934, 2915, 2824 (C-H aliphatic). ^1H NMR (DMSO- d_6): δ 7.19–7.89 (m, 7H Ar-H), 4.72 (s, 1H, OH), 4.11 (s, 1H, CH), 4.00 (m, 4H, 2CH₂), 1.5 (m, 10H, 5CH₂). ^{13}C NMR (DMSO- d_6): δ 157.20, 134.81, 129.69, 127.21, 126.77, 123.92, 119.2, 107.25, 71.78, 70, 28, 67.00, 55.32, 26.19, 24.64. Elemental analysis calculated for $\text{C}_{18}\text{H}_{23}\text{NO}_2$ (%) Calcd./found C, 75.76/75.77; H, 8.12/8.11; N, 4.91/4.89.

1-(morpholin-4-yl)-3-(naphthalen-2-yloxy) propan-2-ol (2)

Yellow crystals in 59% yield, M.P 112-115 °C. IR(v) (KBr) cm^{-1} : 3316 (OH), 3273(NH), 3080 (C-H aromatic), 2989, 2879 (C-H aliphatic). ^1H NMR (DMSO- d_6): δ 7.18–7.89 (m, 7H Ar-H), 4.72 (s, 1H, OH), 4.1 (s, 1H, CH), 4.00(s, 4H, 2CH₂), 1.5(m, 8H, 4CH₂). ^{13}C NMR (DMSO- d_6): δ 157.20, 134.81, 129.69, 127.94, 127.21, 126.03, 125.77, 107.25, 71.78, 67.00, 62.36, 26.19, 24.64. Elemental analysis calculated for $\text{C}_{17}\text{H}_{21}\text{NO}_3$ (%) Calcd. /found C, 71.06/71.05; H, 7.37/7.36; N, 4.87/4.86.

3,3'-(piperazine-1,4-diyl) bis(1-(naphthalen-2-yloxy) propan-2-ol) (3)

Yellowish white powder 87% yield, MP:188-190 °C. IR (v) (KBr) cm^{-1} : 3377 (OH), 2925, 2815 (C-H aliphatic), ^1H NMR (DMSO- d_6): 7.18–7.89 (m, 14H Ar-H). 4.84 (s, 2H, OH), 4.13(s, 2H, 2CH), 4.01 (s, 8H, 2CH₂), 2.3(s, 8H, 4CH₂). ^{13}C NMR (DMSO- d_6): δ 157.16, 134.78, 129.71, 127.94, 126.80, 123.96, 107.27, 71.61, 67.01, 61.61, 54.13. Elemental analysis calculated for $\text{C}_{30}\text{H}_{34}\text{N}_2\text{O}_4$ (%) Calcd. /found C, 74.05/74.03; H, 7.04/7.02; N, 5.76/5.77.

1,3-bis(naphthalen-2-yloxy) propan-2-ol (4)

White powder 79% yield, MP. 111-113 °C. IR (v) (KBr) cm^{-1} : 3293 (OH), 3056, 3026 (C-H aromatic), 2928, 2877 (C-H aliphatic), ^1H NMR (DMSO- d_6): δ 7.21-7.85 (m, 14H, Ar-H), 5.56 (s, 1H, OH), 4.42 (s, 1H, CH), 4.25 (s, 4H, 2CH₂). ^{13}C NMR (DMSO- d_6): δ

139.20, 136.21, 133.69, 132.35, 128.85, 128.77, 125.32, 37.3, 45.25. Elemental analysis calculated for $\text{C}_{23}\text{H}_{20}\text{O}_3$ (%) Calcd. /found C, 80.21/80.19; H, 5.8/5.7.

Synthetic procedure for 1,3-bis(naphthalen-2-yloxy) propan-2-one (5)

Solution of compound 4 (1g, 0.0029mmole) from ethanol absolute (10ml) added to sodium ethoxide solution (0.06 gram, 0.0025mmole) and benzoyl chloride (0.49gram, 0.0025mmole) then refluxed for 5 hours. The product was formed are filtered off and recrystallized from methanol to afford compounds 5. Yellowish white in 51% yield, MP. 162-164 °C. IR (v) (KBr) cm^{-1} : 2897 (C-H aliphatic), 1701 (C=O). ^1H NMR (DMSO- d_6): δ 7.21-7.76 (m, 14H Ar-H), 4.5 (s, 2H, CH₂), 4.4 (s, 2H, CH₂). ^{13}C NMR (DMSO- d_6): δ 174.06, 166.42, 136.30, 133.67, 132.35, 129.50, 129.04, 128.95, 128.73, 36.82, 46.35. Elemental analysis calculated for $\text{C}_{23}\text{H}_{18}\text{O}_3$ (%) Calcd. /found C, 80.68/80.67; H, 5.30/5.31.

3,3'-(ethane-1,2-diylbis(azanediyl))bis(1-(naphthalen-2-yloxy)propan-2-ol) (6)

Yellowish powder in 59 % yield, MP. 219-222 °C. IR (v) (KBr) cm^{-1} : 3341 (OH), 3325 (NH), 3033 (C-H aromatic), 2922 (C-H aliphatic). ^1H NMR (DMSO- d_6): δ 7.12–7.81 (m, 2H, 2NH and 14H, Ar-H), 5.05 (s, 2H, 2OH), 4.12 (m, 2H, 2CH and 8H, 4CH₂), 3.28 (s, 4H, 2CH₂). ^{13}C NMR (DMSO- d_6): δ 157.20, 133.90, 128.06, 126.90, 124.15, 123.14, 119.29, 71.39, 57.22, 31.73, 22.98. Elemental analysis calculated for $\text{C}_{26}\text{H}_{28}\text{N}_2\text{O}_4$ (%) Calcd./found C, 73.02/73.01; H, 7.0/7.02; N, 6.08/6.06.

1-(butylamino)-3-(naphthalen-2-yloxy) propan-2-ol (7)

White powder in 88% yield, MP. 233-236 °C. IR (v) (KBr) cm^{-1} : 3496 (OH), 3024 (C-H aromatic), 2920, 2819 (C-H aliphatic). ^1H NMR (DMSO): δ 7.12–7.71 (m, 1H, NH and 7H, Ar-H), 4.95 (s, 1H, OH), 4.12 (s, 4H, CH), 2.9 (s, 1H, CH), 4.82 (s, 3H, CH), 4.82 (s, 1H, CH), 4.82 (s, 1H, CH), 4.82 (s, 3H, CH₃). ^{13}C NMR (DMSO- d_6): δ 157.20, 134.70, 129.94, 128.96, 127.89, 127.05, 126.69, 123.89, 119.15, 107.2, 71.17, 67.86, 58.64, 55.78, 29.50, 20.54, 14.25. DEPT 135 (DMSO- d_6): δ 129.56 (CH), 127.90 (CH), 127.06 (CH), 126.7 (CH), 123.90 (CH), 119.18 (CH), 107.14 (CH), 106.9 (CH), 71.17 (CH), 70.97 (OCH), 14.33(OCH₃). Elemental analysis calculated for $\text{C}_{17}\text{H}_{21}\text{NO}_2$ (%) Calcd. /found C, 74.69/74.77; H, 8.48/8.47; N, 5.12/5.13.

1-(hexylamino)-3-(naphthalen-2-yloxy) propan-2-ol (8)

Pale white powder 73% yield, MP. 123–126 °C. IR (v) (KBr) cm^{-1} : 3313 (OH), 3024 (C-H aromatic), 3055, 3029 (C-H aromatic), 2920 (C-H aliphatic). ^1H NMR (DMSO- d_6): δ 7.12–7.81 (m, 7H, 7H, Ar-H), 4.25 (s, 1H, OH), 4.12 (s, 2H, CH), 2.8 (s, 1H, CH), 2.7 (s, 1H, CH), 2.6 (s, 2H, CH), 1.44 (s, 2H, CH), 1.42 (s, 6H, CH), 1.2 (s, 3H, CH₃). ^{13}C NMR(DMSO- d_6): δ 157.14, 134.80, 129.66, 128.96, 127.92, 127.11, 126.75, 123.92, 119.21, 107.26, 71.39, 68.55, 52.97, 49.96, 31.73, 29.98, 26.92, 22.55, 14.33. Elemental analysis calculated for $\text{C}_{19}\text{H}_{25}\text{NO}_2$ (%) Calcd. /found C, 75.71/75.70; H, 9.03/9.05; N, 4.65/4.63.

3,3'-(hydrazine-1,2-diyl) bis(1-(naphthalen-2-yloxy) propan-2-ol) (9)

Yellowish powder in 59 % yield, MP. 219-222 °C. IR (ν) (KBr) cm^{-1} : 3340, 3323 (OH, NH), 3039 (C-H aromatic), 2924 (C-H aliphatic). ^1H NMR (DMSO- d_6): δ 9.71 (s, 2H, 2NH), 7.18-7.81 (m, 28H, Ar-H), 5.11 (s, 2H, 2OH), 4.51 (m, 2H, 2CH), 4.15 (m, 8H, 4CH₂). ^{13}C NMR (DMSO- d_6): δ 157.11, 134.78, 129.69, 127.93, 126.78, 123.96, 107.28, 71.36, 68.80, 53.25. Elemental analysis calculated for $\text{C}_{26}\text{H}_{28}\text{N}_2\text{O}_4$ (%) Calcd. /found C, 72.20/72.18; H, 6.53/6.52; N, 6.48/6.49.

1-cyclopropyl-6-fluoro-7-(4-(2-hydroxy-3-(naphthalen-2-yloxy)propyl)piperazin-1-yl)-4-oxo-1,4-dihydroquinoline-3-carboxylic acid(10)

Pale white powder in 70% yield, MP. 210-212 °C. IR (ν) (KBr) cm^{-1} : 3344 (OH), 3018 (C-H aromatic), 2957, 2863 (C-H aliphatic), 1726 (C=O). ^1H NMR (DMSO- d_6): δ 14.60 (OH), 8.46 (NH), 7.12-7.71 (m, 10H, Ar-H), 4.95 (s, 1H, OH), 4.12-3.81 (m, 5H, CH), 2.6 (d, 8H, CH₂), 1.12 (m, 5H, CH). ^{13}C NMR (DMSO- d_6): δ 157.20, 134.70, 129.94, 128.96, 127.89, 127.05, 126.69, 123.89, 119.15, 107.2, 71.17, 67.86, 58.64, 55.78, 29.50, 20.54, 14.25. Elemental analysis calculated for $\text{C}_{30}\text{H}_{30}\text{FN}_3\text{O}_5$ (%) Calcd. /found C, 67.78/67.76; H, 5.69/5.68; N, 7.90/7.91.

3,3',3''-nitrilotris(1-(naphthalen-2-yloxy) propan-2-ol) (11)

Pale White powder in 92% yield, MP. 138-140 °C. IR (ν) (KBr)

cm^{-1} : 3221 (OH), 2919, 2813 (C-H aliphatic). ^1H NMR (DMSO- d_6): δ 7.12-7.81 (m, 21H, Ar-H), 5.04 (s, 3H, OH), 4.12 (m, 6H, CH₂ and 3H, CH), 2.9 (m, 6H, CH₂). ^{13}C NMR (DMSO- d_6): δ 157.11, 134.78, 129.70, 127.94, 123.97, 119.23, 107.28, 73.02, 69.80, and 53.2. Elemental analysis calculated for $\text{C}_{39}\text{H}_{39}\text{NO}_6$ (%) Calcd. /found; C, 75.83/75.82; H, 6.36/6.35; N, 2.27/2.28.

Laboratory Bioassay

The insecticidal bio-efficacy of all prepared Epihalohydrins analogues was assessed by the leaf dipping bioassay procedure. Research facility of tested results are accounted here for the used compounds to discover the appropriate concentration that are required to kill half 50% (LC₅₀) of the pests. In this search, five concentrations of each synthesized naphthalene derivatives and 0.1% Triton X-100 as surfactant were utilized. A number total of 10 larvae of *spodoptera littoralis* (Boisd.), almost of the 2nd instar larvae and 4th instar larvae size, Disks (9cm. diameter) of castor bean leaves were dipped in the tested concentrations for 10 seconds then left to dry and offered to larvae. Larvae were placed into glass jars (5 pounds), every treatment was recreated multiple times (10 larvae per each). Control disks were dunked in distilled water only. In which allowed the larvae to feed on castor bean leaves for 48hr [18]. then transferred to the untreated ones. Mortality percentages were recorded after 72hr. for all insecticides. Mortality was redressed by Abbott's formula [19]. The measurements mortality relapse lines were measurably dissected by probit analysis [20]. Harmfulness Index was determined by sun equations [21].

Insecticidal Bio-efficacy Examination

Table 1: Insecticidal activity of Compounds 1–11 and Fenoxycarb against the 2nd larvae of *spodoptera littoralis* (Boisd.), after 72h of Treatment.

Compound Fenoxycarb	LC ₅₀ (ppm) 2.73	Slope $\pm$ SE 0.238 $\pm$ SE	Toxic Ratio ^a 1
1	25.04	0.283 $\pm$ 0.083	0.109
2	17.56	0.238 $\pm$ 0.076	0.155
3	15.64	0.299 $\pm$ 0.083	0.174
4	38.69	0.256 $\pm$ 0.082	0.07
5	34.68	0.272 $\pm$ 0.083	0.078
6	6.07	0.262 $\pm$ 0.079	0.449
7	44.08	0.279 $\pm$ 0.084	0.0619
8	42.05	0.293 $\pm$ 0.0862	0.0651
9	10.06	0.256 $\pm$ 0.076	0.275
10	4.801	0.262 $\pm$ 0.079	0.568
11	3.710	0.197 $\pm$ 0.075	0.449

^aThe ratio of the LC₅₀ values of fenoxycarb for baseline toxicity and the compounds are defined as a toxic ratio.

The compounds which prepared have been tested for their insecticidal bio-efficacy, demonstrated as follows: compounds from 1-11 were tested against the 2nd instar larvae for their activity as insecticidal, and the outcomes are introduced in Table 1. After 72h of treatment, activity results demonstrated that these compounds

show high to low insecticidal activity against the 2nd instar larvae and the LC₅₀ regards stretched out from 3.71 to 44.08ppm, however the LC₅₀ estimation of fenoxycarb was 2.37ppm. This shows that nearly insecticidal bio-efficacy of fenoxycarb as insect growth regulators after 72h of test. In which compounds 6, 10 and 11 have

generally excellent insecticidal bio-efficacy against 2nd because that their LC₅₀ esteems were 6.07, 4.80 and 3.710, respectively, in which the LC₅₀ estimation value of fenoxycarb was 2.37ppm.

The results of insecticidal bio-efficacy after 72h for compounds 1-11 were tested against the 4th instar larvae shown in Table 2. Varied from strong to weak with LC₅₀ values assorted from 50.60

to 103.01ppm, while the LC₅₀ value of fenoxycarb was 42.231ppm. In which demonstrates that various them have tested compound near that of fenoxycarb as juvenile hormone after 72h of test. For instance, compounds 6, 10 and 11 have generally excellent insecticidal exercises activities against 4th instar larvae because their LC₅₀ values were 54.631, 57.622 and 50.606, respectively, and the LC₅₀ value of fenoxycarb was 43.231 ppm.

Table 2: Insecticidal activity of compounds 1-11 and Fenoxycarb against the 4th larvae of *spodoptera littoralis* (Boisd.), after 72h of Treatment.

Compound Fenoxycarb	LC ₅₀ (ppm) 43.231	Slope ± SE 0.251 ± 0.0820	Toxic ratio ^a 1
1	72.253	0.232 ± 0.0856	0.598
2	69.252	0.255 ± 0.0862	0.624
3	80.352	0.220 ± 0.0811	0.538
4	86.921	0.231 ± 0.0823	0.497
5	97.371	0.229 ± 0.0813	0.443
6	54.631	0.189 ± 0.0775	0.791
7	103.01	0.234 ± 0.0830	0.419
8	86.98	0.231 ± 0.0823	0.497
9	62.352	0.252 ± 0.0821	0.693
10	57.622	0.213 ± 0.0799	0.75
11	50.606	0.254 ± 0.0830	0.856

Structure Framework Relationship

It shows up from the general search structure of the prepared naphthalene derivatives compounds of 11 and 10 are more active against 2nd instar larvae and 4th instar larvae than the another's of tested compounds. It additionally demonstrated that exacerbates that contain an exceptionally ether group in the structure claim a high insecticidal bio-efficacy. Thus, compounds 6, 9, 10, and 11 more active than compounds 1, 2, 3, 4, 7 and 8 in-insecticidal bio-efficacy.

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