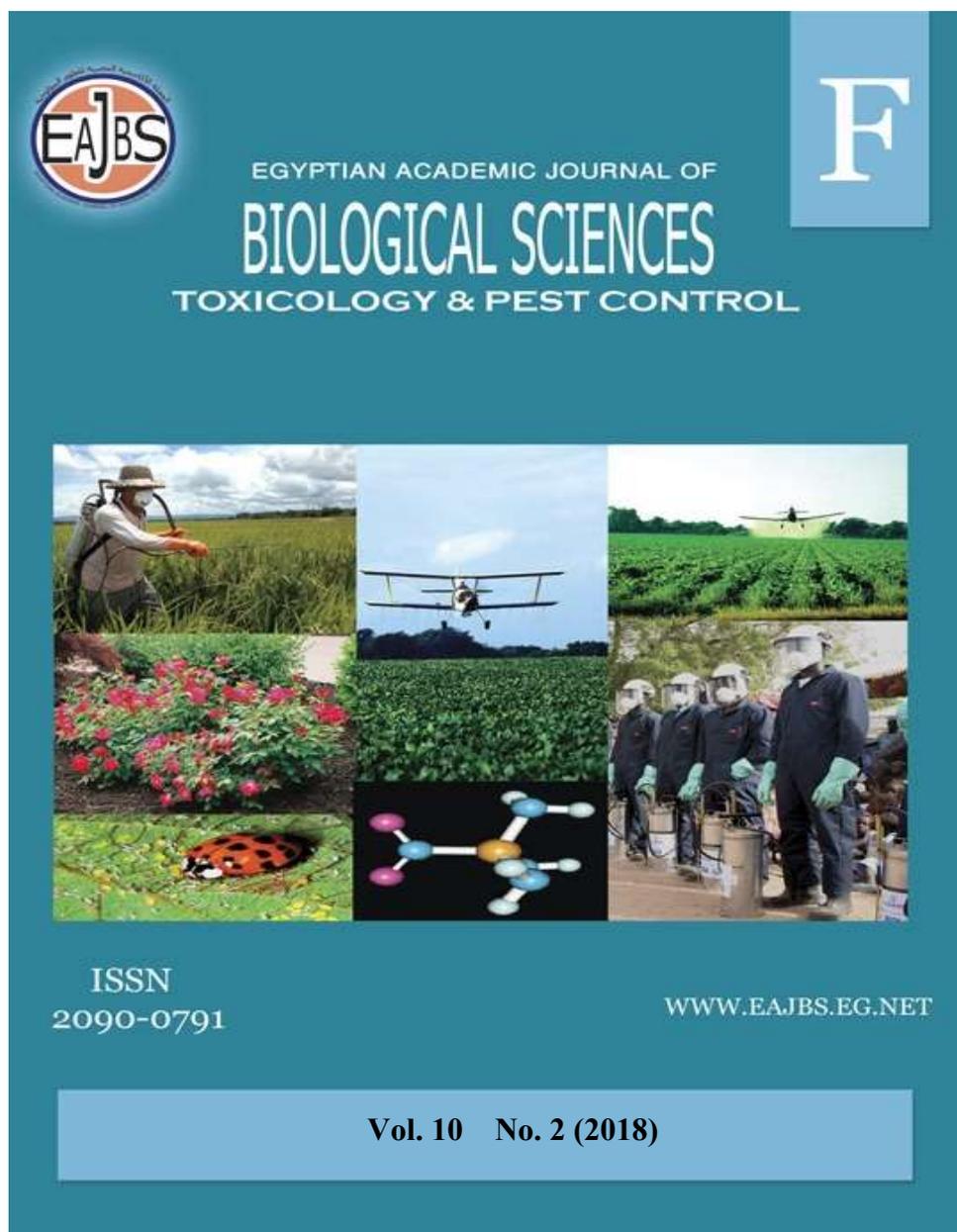


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Efficacy of *Artemisia judaica* Extract and Certain Insecticides against Cotton Leafworm, *Spodoptera littoralis* (Lepidoptera: Noctuidae)

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ARTICLE INFO

Article History

Received:15/8/2018

Accepted:22/9/2018

Keywords:

Artemisia judaica, fipronil, chromafenozide, pyridalyl, feeding indices, *Spodoptera littoralis*

ABSTRACT

Toxicity, feeding indices and residual efficacies of ethanol extract of *Artemisia judaica* and some synthetic insecticides were investigated alone and in combinations against 2nd instar larvae of *Spodoptera littoralis*. The LC₅₀s of chromafenozide, fipronyl, and pyridalyl were (0.025 and 0.016 mg L⁻¹), (0.140 and 0.065 mg L⁻¹), and (0.127 and 0.012 mg L⁻¹) at 72 and 96 hrs, respectively. *A. judaica* extracts had LC₅₀s (9482.99 and 2839.30 mg L⁻¹), LC₂₅s (3011.15 and 706.82 mg L⁻¹), and LC₁₀s (1072.27 and 202.19 mg L⁻¹) at 72 and 96 hrs, respectively. GC-MS analysis of this extract included fatty acids (51.5 %), eucalyptol (3.64 %), tetraeurin-A (2.84 %), coumarin (2.08 %) and flavone (0.81 %). All treatments submitted tests of anti-feedant activity, relative consumption rate, relative growth rate, and efficiency of conversion of ingested food were more affected at 96 hrs of exposure. In the laboratory, potentiation effects appeared in binary mixtures of for chromafenozide (LC₅₀) with *A. judaica* (LC₁₀) with CTFs (21.74 and 20.83) and with *A. judaica* (LC₂₅) (42.11 and 23.33) after 72 and 96 hrs of exposure, respectively. Fipronyl (LC₅₀) + *A. judaica* (LC₂₅) had additive effects with CTFs of -17.24 and 6.67 after 72 and 96 hrs of exposure, respectively. Oppositely, fipronyl (LC₅₀) + *A. judaica* (LC₁₀) and all mixtures of pyridalyl + *A. judaica* had antagonistic effects. The highest overall mean mortality over 16 days in semi-field experiments of the 2nd instar larvae exposed 96 hrs to *A. judaica* (706.82 mg L⁻¹) tank mixed with 0.5 FRs of fibronil and chromafenozide were (77.00 and 64.00 %) and (75.50 and 68.50 %) in seasons of 2017 and 2018, respectively. The residual efficacies of these mixtures possessed prolonged times and higher toxicity compared to their FR alone. The insecticidal roles related to phyto-components of this extract were discussed.

INTRODUCTION

Cotton leafworm (CLW), *Spodoptera littoralis* is the most injurious polyphagous insect to cotton in the Middle East, infesting about 87 host plants including important field crops and various fruits and ornamental trees. Therefore, several chemical applications have been required due to the existence of this pest during the whole cycle of the crop (Hatem *et al.*, 2009; Tiessen, 2012; Reda *et al.*,

2015). In respect of choosing organic natural pesticides over synthetic insecticides, policy and public opinion emphasized that natural products are uniformly safer and friendlier to the environment than synthetic pesticides (Bahlai *et al.*, 2010).

Many researches have been directed to study the comparative efficacy of plant extracts against various insect pests versus to conventional synthetic insecticides besides the co-potency factor of binary mixtures of insecticides with plant extracts. These trials were directed to curb the increases of insecticide resistance (Sinzogan, 2006; Mohan *et al.*, 2007; Tavares *et al.*, 2010; Shalaby *et al.*, 2013; Zayed, 2014; Adesina and Rajashekar, 2018). Economic studies, declared that binary mixtures considered to be more reasonable in cost than using synthetic insecticide alone. Therefore, the additions of plant extracts to insecticides enhance their efficacy and insure environmental safety (Shalaby *et al.*, 2013; Osman and Abou-zeid, 2015). So far, the combination between indoxacarb and *Pongamia glabra* extract oil effectively was used to control *Spodoptera litura* (*S. litura*) and *Heliothis armigera* under field condition and safe to adult predators of *Coccinella septempunctata* (Loganathan, 2004). Genus of *Artemisia* (Anthemideae tribe; Asteraceae family) in Egypt region included different species of *A. monosperma* Delile, *A. scoparia* Waldst., *A. judaica* L., *A. verlotiorum* Lamotte and *A. vulgaris* L. (Boulos, 2002). Phytochemical studies on many *Artemisia* species were characterized by some constitutes of polyacetylenes, lignans, sesquiterpene, lactones and flavonoids (Valant-Vetchera *et al.*, 2003). Particularly, *A. judaica* "Beitheran" was selected in this research as it is one of the perennial fragrant shrubs and distinguished by medical uses besides a very common anthelmintic drug in many Middle-Eastern and North African countries (Batanouny, 1999; Wyk and Wink, 2005). The populations of *A. judaica* colonized in dry flat areas of the south of Suez, Wadi Feran in Sinai and the high mountains of Saint Catherine in South Sinai. The morphological characters of *A. judaica* in these different locations showed variation in shoot length, number of leaf lobes, shape and length of capitulum, number of female flowers, number of bisexual flowers and number of seeds (Badr *et al.*, 2012).

Synthetic insecticides of chromafenozide, pyridalyl and fipronil share some specific criteria of lateness in initial killing action on target pests, relative short half-life time and safety on natural enemies. Chromafenozide termed as an insect growth regulator belongs to the dibenzoylhydrazine group of insecticides, which can be used to control lepidopteran pests on various crops. Development of chromafenozide was carried out through the collaborative work of Nippon Kayaku Co., Ltd. and Sankyo Co., Ltd. (Tomlin, 2009). Histopathological effects of the ecdysone agonist, Virtu® (chromafenozide) showed that ovarioles growth was stunted and vitellogenesis and chorion formation were inhibited in treated 4th instar larvae of F1 female of *S. littoralis* (Ahmed *et al.*, 2015). Dissipation of chromafenozide residues followed first order kinetics. The usage of chromafenozide at recommended dose does not pose any hazards to consumers and it can be utilized in formulating spray schedules and safety evaluation in strawberry (Malhat *et al.*, 2014). Chromafenozide considered being one of the lowest toxicity compared to IGRs of lufenuron, teflubenzuron, flufenoxuron, chlorfluazuron, methoxyfenozide on *Coccinellidae* spp, *Chrysoperla carnea* and true spider predators of *S. littoralis* (boisd) in cotton crop under field conditions (El-Sayed *et al.*, 2015). Moreover, pyridalyl is an insecticide of novel unclassified chemical insecticides. It exhibited high selectivity in cytotoxicity between the insect and mammalian cell line as well as insecticidal activity among insect species e.g *S. litura*, *Frankliniella occidentalis*. No acute toxicity of this product was monitored on non-target insects of *Orius stringicollis* and a pollinator *Bombus terrestris*. Thus,

pyridalyl may be useful for IPM programs of greenhouse cultivation system (Isayama *et al.*, 2005). Finally, fipronil belongs to the second insecticides generation of a new class called phenylpyrazoles, which acts through a different mechanism compared to other conventional insecticides. Fipronil or its metabolite non-competitively inhibit γ -aminobutyric acid (GABA)-induced ion influx, leading to neural hyper-excitation, and at sufficient concentrations, paralysis, and death (Cole *et al.*, 1993; Bobe *et al.*, 1998; Anadon and Gupta, 2012). The investigation on fipronil 20 % suspension concentrate (SC), which submitted to bio-efficacy and safety aspect tests showed that this formulated insecticide considered as a good option in sucking pests management in Chili ecosystem of Tamil Nadu. Although killing action of fipronil treatments were not observed immediately, it appeared to be potent on subsequent days post-treatment and safe to the natural enemies (Indhumathi *et al.*, 2017).

Therefore, this research was carried out to investigate the comparative studies on feeding indices for ethanol extract *A. judaica* of flowering parts alone versus to selected synthetic insecticides of chromafenozide, pyridalyl and fibronil. The feeding indices tests included some parameters of anti-feedant activity, relative consumption rate (RCR), relative growth rate (RGR) and efficiency of conversion of ingested food (ECI). Furthermore, these work also targeted the influence of this plant extract in binary mixtures with the selected synthetic insecticides against cotton leafworm, *S. littoralis* in laboratory and under field condition on cotton crop.

MATERIALS AND METHODS

Insect Rearing:

A laboratory susceptible strain of CLW, *S. Littoralis*, larvae were obtained from Integrated Protection Laboratory, Agriculture Research Center, Alexandria. Feeding was conducted on fresh castor leaves in laboratory under constant conditions (El-Defrawi *et al.*, 1964).

Plant Material, Extraction, and Formulation:

Samples of flowering parts of *A. judaica* herbs were collected from Saint Catherine, Sharm Al-Sheikh, South Sinai governorate. The collected flowering parts were left for drying at room temperature for about 10 days, and then milled to a powder form. The obtained powder was extracted in soxhlet with ethanol at 45 ± 5 °C for 12 hrs. Ethanol was discarded by rotary evaporator. The crude extract was then stored in a sealed glass bottle below 0 °C. The *A. judaica* crude extract was formulated by dissolving it in dimethyl sulfoxide (DMSO) containing 5 % Tween-20, freshly prepared before application.

Gas Chromatography–Mass Spectrometry (GC-MS) Analysis:

The chemical composition of *A. judaica* crude extract was performed using Trace GC Ultra-ISQ mass spectrometer (Thermo Scientific, Austin, TX, USA) with a direct capillary column TG–5MS (30 m x 0.25 mm x 0.25 μ m film thickness). The column oven temperature was initially held at 60 °C and then increased by 5 °C / min to 220 °C withhold 2 min then increased to 300 °C, 12 °C / min. The injector and MS transfer line temperatures were kept at 270 °C. Helium was used as a carrier gas at a constant flow rate of 1 ml / min. The solvent delay was 3 min and diluted samples of 1 μ l were injected automatically using Auto-sampler AS3000 coupled with GC in the split mode. EI mass spectra were collected at 70 eV ionization voltages over the range of m / z 40 – 650 in full scan mode. The ion source was set at 200 °C. The components were identified by comparison of their retention times and

mass spectra with those of WILEY 09 and NIST 11 mass spectral database.

Insecticides:

The used insecticides were chromafenozide (Vertu 5 % SC), Japan- Nippon Kayaku Co. Ltd., field dosage of 400 cm³ per fed. (4200m²), fipronil (Orbit 20 % SC), Indian- Sharda worldwide Export Pvt. Ltd., field dosage of 80 cm³ per fed., and pyridalyl (Pelio 50 % EC), Japan – Sumitomo chemical Ltd., field dosage of 100 cm³ per fed.

Laboratory Studies:

Toxicity of chromafenozide, fipronil and pyridalyl as well as the *A. judaica* extract were carried out against 2nd instars larvae of *S. littoralis* at 72 and 96 hrs of exposure using castor oil leaf discs dipping technique according to El-defrawi *et al.*, (1964). Leaf discs were dipped in a series of diluted concentrations for each tested insecticide solution for 20 seconds. The treated discs were allowed to dry well for 30 min at room temperature. Each treatment was assigned for seven serial dilutions with four replicates for each concentration. Ten numbers of Pre-starved (24 hrs) 2nd instars larvae were allowed to feed to sufficient treated leaf discs in each plastic cup for 72 and 96 hrs post-treatment. Mortalities of all treatments were checked. Mortality percentages were calculated and corrected in relative to control treatment according to the equation of Abbott, (1925) and then, submitted to probit analysis (Finney, 1971).

Assay of Feeding Indices:

A castor oil leaf-dip bioassay was used to check the feed indices of each of the plant extract and tested insecticides against the 2nd instar larvae of *S. littoralis* after 72 and 96 hrs of exposures. The castor leaves were dipped in sub-lethal concentrations of LC₅₀ and LC₂₅ for each insecticide and LC₅₀, LC₂₅ and LC₁₀ for *A. judaica* extract. Sub-lethal concentrations of all treatments were compared to control treatments for feeding parameters of anti-feedant activity, relative consumption rate (RCR), relative growth rate (RGR) and efficiency of conversion of ingested food (ECI). One leaf disc was placed in each cup for the pre-starved (24 hrs) 2nd instars larvae of *S. littoralis*. Larvae were allowed to feed on the treated leaf for 72 and 96 hrs. The leaf discs were replaced by new one at 48 hrs. Each concentration had 4 replicates and each replicate had five larvae. Weights of leaves and survival 2nd instar larvae were converted from fresh to dry weight ratios by using an oven (at 50°C for 24 hrs). Fresh and dry weights of these materials were recorded for each replicate to estimate the feeding indices at 72 and 96 hrs of exposure. A high precision balance (± 0.1 mg) was used to weight all materials. The anti-feedant activity percentages were calculated by weighting the leaf disc according to Saleh *et al.*, (1986). Furthermore, feeding indices parameters were calculated by the formulas of Waldbauer, (1968).

$$\text{Anti-feedant activity \%} = \left[1 - \frac{\% \text{ of treated leaf eaten}}{\% \text{ of untreated leaf eaten}} \right] \times 100$$

Feeding indices equations:

Relative consumption rate (RCR) = E / T A,

Relative growth rate (RGR) = P / T A,

Efficiency of conversion of ingested food (ECI) = 100P / E

A = mean of dry weight of larvae during T, E = dry weight of food eaten, P = dry weight gain of insect, T = duration of experimental period.

Co-toxicity Factor of Plant extract and Insecticides Binary Mixtures:

Equivalent concentrations to each of LC₂₅ and LC₁₀ of *A. judaica* extract were alternatively mixed to LC₅₀s of each of chromafenozide, fipronil, and pyridalyl. The

toxic effects of these binary mixtures were tested against 2nd instars larvae of CLW after 72 and 96 hrs of exposures. The joint actions of these binary mixtures were expressed by the equation of co-toxicity factor (CTF) performed by (Mansour *et al.*, 1966).

$$CTF = \frac{\text{Observed \% mortality} - \text{expected \% mortality}}{\text{expected \% mortality}} \times 100$$

Where:

CTFs $\geq +20$ indicate potentiation; < -20 indicate antagonism; and for the range from -20 up to +20 indicate additive effect.

Expected (%) mortality = Sum of % mortalities of each insecticide and plant extract alone at the same concentration levels used in binary mixture.

Observed (%) mortality = % mortality of binary mixture.

Field Trails:

The semi-field experiments were achieved at 16th and 19th of June in Ezbit Al-Bahr, El-Behira governorate on cotton variety (Giza 86) during two growing successive seasons of 2017 and 2018, respectively. All treatments were assigned to micro-plots (35 m²) in a randomized complete block design. Foliar spray of all treatments was carried out by Knapsack sprayer equipment (CP3) at a fixed volume of spray solution of 3.5 liters / micro-plot. Only tested insecticides that passed the combination tests in laboratory were submitted to the field experiments at half field (0.5 FR) and field rates (FR) alone and in combination with formulated *A. judaica* extract at rate of 0.5 gm of extract / 1 liter (equivalent to its value of LC₂₅ at 96 hrs post-treatment). Control plots were sprayed with water. Each treatment had four replicates. Samples of treated and untreated cotton leaves in designed micro-plots were collected at 0, 4, 8, 12, 16 days post-treatments. Samples of cotton leaves were preserved in perforated bags till transferred to the laboratory. One cotton leaf was placed in plastic cups containing five (24 hrs pre-starving) larvae of the 2nd instar. Four replicates were used in each treatment. The experiment was maintained under 27 °C and 65 % RH. Mortality was recorded after 96 hrs of exposure and corrected according to Abbott, (1925) equation.

Statistical Analysis:

Data of feed indices of the tested insecticides and *A. judaica* extract in laboratory experiments and efficacy of residual toxicity of these tested insecticides alone and in combine with *A. judaica* extract in semi-field trials were subjected to analysis of variance (ANOVA) using statistical software (SAS, 2002) at LSD between treatments (P = 0.05 %).

RESULTS

Toxicity of The Tested Insecticides and *Artemisia judaica* Extract:

The LC₅₀s of chromafenozide, fipronyl and pyridalyl against the 2nd instar larvae were 0.025, 0.140, and 0.127 mg L⁻¹ at 72 hrs, respectively. Meantime, the LC₅₀s of chromafenozide, fipronyl, and pyridalyl were 0.016, 0.065, and 0.012 mg L⁻¹ at 96 hrs, respectively (Table 1). On the other hand, the toxicity of *A. judaica* extracts at LC₅₀, LC₂₅, and LC₁₀ against the 2nd instar larvae were 9482.99, 3011.15, and 1072.27 mg L⁻¹ at 72 hrs, respectively. Meantime, the LC₅₀, LC₂₅, and LC₁₀ at 96 hrs were 2839.30, 706.82, and 202.19 mg L⁻¹, respectively (Table 2).

Table 1: Toxicity of the selected insecticides against 2nd instar larvae of *Spodotera littoralis* at 72 and 96 hrs of exposures:

Insecticides	Exposure time (hrs)	LC ₅₀ (mg L ⁻¹)	Confidence limits (mg L ⁻¹)	Slope ± SE	X ²	df
Chromafenozide	72	0.025	0.023-0.028	2.158 ± 0.163	9.13	5
	96	0.016	0.015-0.018	2.392 ± 0.174	5.064	5
Fipronyl	72	0.140	0.127-0.155	2.578 ± 0.179	7.649	5
	96	0.065	0.058-0.072	2.179 ± 0.163	4.576	5
Pyridalyl	72	0.127	0.116-0.138	2.851 ± 0.195	10.297	5
	96	0.012	0.011-0.014	2.136 ± 0.155	5.442	5

Table 2: Toxicity of *Artemisia judaica* extract against 2nd instar larvae of *Spodoptera littoralis* at 72 and 96 hrs of exposures:

Exposure time (hrs)	Toxicity (mg L ⁻¹)	Confidence limits (mg L ⁻¹)	Slope ± SE	X ²	df	
72	LC ₅₀	9482.99	7875.05 - 11710.19	1.35 ± 0.11	6.179	5
	LC ₂₅	3011.15	2396.39-3656.61			
	LC ₁₀	1072.27	737.67-1428.01			
96	LC ₅₀	2839.30	2272.35-3491.986	1.117 ± 0.093	5.044	5
	LC ₂₅	706.82	474.74-959.214			
	LC ₁₀	202.19	109.10-318.72			

GC-MS Chemical Profile of Ethanol Extract of *Artemisia judaica* (flower parts):

The results of GC-MS analysis of ethanol extract of *A. judaica* (flower parts) were represented in table (3). The major constituents of the *A. judaica* ethanol extract were fatty acids (FAs) and its esters (51.5 %). FAs comprised of linoleic acid (13.48 %) oleic acid (27.57 %), lauric acid (17.98 %), and palmitoleic acid (26.75 %). Besides, FAs esters comprised of palmitic acid methyl ester (6.01 %), α linoleic methyl ester (15.33 %), and palmitoleic acid hydroxyl propyl ester (28.89 %). In addition, other components were found in the extract represented by monoterpenes (eucalyptol 3.64 %), sesquiterpenes (tetraeurin-A 2.84 %), coumarins (coumarin 2.08 %), flavonoids (flavone 0.81 %).

Table 3: GC-MS Chemical Profile of Ethanol Extract of *Artemisia judaica* Flowering Parts:

No.	Identified compounds	Retention times	Area	Molecular weight
1	Palmitic acid methyl ester	6.01	0.62	374
2	13-Heptadecyn-1-ol	6.39	0.49	252
3	Benzo[b]thiophen-2-amine, n,n-dimethyl-3-phenyl-	7.44	0.91	253
4	Eucalyptol	9.31	3.64	154
5	1a,2,5,5a,6,9,10,10a-octahydro-5,5a,6-trihydroxy-1,4-bis(hydroxyl methyl)-1,7,9-trimethyl-1h-2,8a-methanocyclopenta(a)cyclopropa (e)cyclodecen-11-one	10.76	0.62	364
6	13,16-Octadecadienoic acid, methyl ester	11.69	2.42	290
7	2(3h)-Benzofuranone, hexahydro-4,4,7a-trimethyl-	12.66	1.14	182
8	Linoleic acid	13.48	2.07	280
9	α Linoleic acid methyl ester	15.33	11.86	496
10	Hi-oleic safflower oil	16.16	1.05	450
11	Lauric acid	17.98	2.04	216
12	Palmitoleic acid	26.75	0.42	254
13	Laureth-2	26.85	2.24	460
14	1h-Benzofuro[3,2-e]isoindole-4-carboxylic acid, 2,3,3a,4,5,10c-hexahydro-5-hydroperoxy-5-methyl-1,3-dioxo-2-phenyl-, methyl ester,(3aà,4á,5á,10cà)-	27.14	9.90	421
15	Oleic acid	27.57	17.07	282
16	Coumarin	28.76	2.08	344
17	Palmitoleic acid hydroxyl propyl ester	28.89	13.06	330
18	Tetraeurin - a - diol	29.21	0.60	280
19	Ethyl iso-allocholate	32.25	26.96	436
20	Flavone 4'-oh,5-oh,7-di-o-glucoside	32.31	0.81	594

Feeding Indices of The Treated 2nd Instar Larvae of *Spodoptera littoralis*:

Data of feeding indices of the 2nd instar larvae of *S. littoralis* treated with the tested insecticides at LC₅₀ and LC₂₅ after 72 and 96 hrs of exposures were represented in table (4 and 5). The most potent insecticides at LC₅₀ affected the overall feeding indices after 72 hrs of exposures were found in both treatments of fipronil, pyridalyl and lasted with chromafenozide. Fipronil was one of the most tested insecticides having the highest on RCR (0.1516 mg / mg / day) as well as its effectiveness on ECI (10.25 %) were relatively higher compared to control values. Pyridalyl had the same highest degree of effectiveness of fipronily on RCR (0.1049 mg / mg / day). In addition, pyridalyl had a relative higher anti-feedant activity of 24.28 % and had the second rank in effectiveness after fipronil on ECI (16.08 %). Chromafenozide had the second and third rank in its effectiveness on RCR (0.3361 mg / mg / day) and ECI (16.75 %), respectively. Likewise, the most potent insecticides at LC₅₀ affected the overall feed indices after 96 hrs of exposures were found in both treatments of fipronil, chromafenozide and lasted with pyridalyl. Fipronil, chromafenozide and pyridalyl had the same highest effectiveness on RCR (0.0451 mg / mg / day), (0.0327 mg / mg / day) and (0.0361 mg / mg / day), respectively with no significant differences between them in compare to control treatment. Chromafenozide had a relative high anti-feedant activity of 66.02 %. The highest anti-feedant activities with no significant differences were recorded for both treatments of fipronil and pyridalyl with percentages of 69.89 % and 69.46 %, respectively. All insecticides treatments at LC₅₀ had the same degree of effectiveness on the values of RGR of 2nd instar larvae at 72 and 96 hrs of exposure but all these insecticides had significant difference compared to control treatments.

The data of significant values of feeding parameters at 72 and 96 hrs of exposures showed that the most effective time of all tested insecticides treatments at LC₅₀ on anti-feedant activity absolutely settled at the time of 96 hrs of exposure. Whereas, the most effective time of chromafenozide and pyridalyl on RCR confirmed at 96 hrs of exposure and at 72 hrs of exposure in fipronil only. Both times of 72 and 96 hrs of exposure had the same effects on RGR. On the contrary, the most effective time of all tested insecticides treatments on ECI confirmed at the time of 72 hrs of exposure. Eventually, the majority of the most effective times settled at 96 hrs of exposure for most of the feeding parameters.

Table 4: Feeding indices of the 2nd instar larvae of *Spodoptera littoralis* treated with the selected insecticides at LC₅₀ at 72 and 96 hrs of exposure:

Treatments	Exposure time(hrs)	Anti-feedant Activity %	RCR (mg/mg/day)	RGR (mg/mg/day)	ECI %
Fipronil	72	10.54 ^d	0.1516 ^d	0.0158 ^c	10.25 ^{ed}
	96	69.89 ^a	0.0451 ^d	0.0154 ^c	36.91 ^{cb}
Chromafenozide	72	11.93 ^d	0.3361 ^c	0.0177 ^c	16.75 ^e
	96	66.02 ^b	0.0327 ^d	0.0116 ^c	35.68 ^{cbd}
Pyridalyl	72	24.28 ^c	0.1049 ^d	0.0170 ^c	16.08 ^{ced}
	96	69.46 ^a	0.0361 ^d	0.0163 ^c	48.28 ^b
Control	72	00.00 ^e	1.6603 ^a	2.2850 ^a	125.43 ^a
	96	00.00 ^e	1.0729 ^b	0.5197 ^b	48.68 ^b

Means for each column with the same letter are not significantly different for anti-feedant activity, RCR, RGR and ECI values according to LSD_{0.05} of interactions between treatments and exposure times =1.69, 0.128, 0.346, and 25.62, respectively.

The results showed that, the most potent insecticides at LC₂₅ affected the overall feeding indices of the 2nd instar larvae of CLW after 72 hrs of exposures

compared to the corresponding values of control treatments were revealed in both treatments of fipronil, pyridalyl and lasted with chromafenozide. Fipronil had the highest effect on ECI (17.42 %) as well as chromafenozide had a relatively high effect on ECI (29.98 %) compared to pyridalyl (60.48 %) and control (125.42 %). Pyridalyl had a relative high anti-feedant activity of 21.31 % compared to the full feeding activity in 2nd instar larvae treated with the other treatments. All insecticides treatments at LC₂₅ had the same degree of effectiveness on the values of RGR and significantly decreased compared to control treatment. Meantime, the most potent treatments at LC₂₅ affected the overall feeding indices of the 2nd instar larvae of CLW at 96 hrs of exposures were existed in both treatments of fipronil, chromafenozide and lasted with pyridalyl. Fipronil had the highest anti-feedant activity of 38.83 % compared to pyridalyl and chromafenozide, which considered having the second rank on anti-feedant activities of 22.64 and 16.63 %, respectively. Fipronil, chromafenozide and pyridalyl had the same degree of effectiveness on RCR at values of 0.2288, 0.2595 and 0.2814 mg / mg / day, respectively. Chromafenozide had the highest effect on ECI at percentage value of 21.06 % at 96 hrs of exposure compared to fipronil (37.07 %) and pyridalyl (54.48 %). All insecticides treatments at LC₂₅ had the same degree of effectiveness compared to control treatment on the values of RGR.

The data of significant values of feeding parameters at 72 and 96 hrs of exposures showed that the most effective time of all the tested insecticides treatments at LC₂₅ on anti-feedant activity and RCR parameters absolutely settled at the time of 96 hrs of exposure. Both times of 72 and 96 hrs of exposure had the same effects on RGR without significant differences between the insecticides treatments. Meanwhile, the most effective time of the tested insecticides treatments on ECI confirmed at the time of 96 hrs of exposure for chromafenozide and pyridalyl versus to 72 hrs of exposure in fipronil. Eventually, the majority of the most effective times of the tested insecticides treatments settled at 96 hrs of exposure for most of the feeding parameters.

Table 5: Feeding indices of the 2nd instar larvae of *Spodoptera littoralis* treated with the selected insecticides at LC₂₅ at 72 and 96 hrs of exposure:

Treatments	Exposure time(hrs)	Anti-feedant Activity %	RCR (mg/mg/day)	RGR (mg/mg/day)	ECI %
Fipronil	72	00.00 ^c	1.0546 ^b	0.1523 ^b	17.42 ^e
	96	38.82 ^a	0.2288 ^c	0.0751 ^b	37.07 ^{cebd}
Chromafenozide	72	00.00 ^c	0.7688 ^b	0.1523 ^b	29.98 ^{ced}
	96	16.63 ^b	0.2595 ^c	0.0601 ^b	21.06 ^{ed}
Pyridalyl	72	21.31 ^b	0.3006 ^c	0.1825 ^b	60.48 ^b
	96	22.64 ^b	0.2814 ^c	0.5226 ^b	54.48 ^{cb}
Control	72	00.00 ^c	1.6603 ^a	2.0850 ^a	125.42 ^a
	96	00.00 ^c	1.0729 ^b	0.5197 ^b	48.68 ^{cbd}

Means for each column with the same letter are not significantly different for anti-feedant activity, RCR, RGR and ECI values according to LSD_{0.05} of interactions between treatments and exposure times = 11.308, 0.3978, 0.5252, and 140.24, respectively.

The results of feeding indices of 2nd instar larvae of CLW treated with *A. judaica* extract at sub-lethal concentrations of LC₅₀, LC₂₅ and LC₁₀ at 72 and 96 hrs of exposures were represented in table (6). Data of feeding indices of 2nd instar larvae of CLW treated with *A. judaica* extract at 72 hrs of exposures compared to control treatments showed no anti-feedant activity at all of the sub-lethal concentrations. While RCR values of treated 2nd instar larvae with LC₅₀, LC₂₅ and LC₁₀ had

significant lower values of 0.1590, 1.0331 and 0.5548 mg / mg / day, compared to the corresponding value in control treatment of 1.6088 mg / mg / day at 72 hrs of exposure. RGR values of treated 2nd instar larvae with LC₅₀, LC₂₅ and LC₁₀ had significantly lower values of 0.0168, 0.1523 and 0.6881 mg / mg / day, respectively compared to the corresponding value in control treatment of 2.0643 mg / mg / day at 72 hrs of exposure. ECI values of treated 2nd instar larvae with LC₅₀ and LC₂₅ at 72 hrs of exposure had significant low values of 10.35 and 15.36 %, respectively but ECI at LC₁₀ had a value of 123.88 % compared to the corresponding value of control treatment (128.15 %). Data of feeding indices of 2nd instar larvae of CLW treated with LC₅₀ and LC₁₀ of *A. judaica* extract at 96 hrs of exposures compared to control treatments showed a highest anti-feedant activity of 51.55 and 30.67 % against 2nd instar larvae of CLW, respectively. RCR values of the treated 2nd instar larvae with LC₅₀, LC₂₅ and LC₁₀ of the extract had significant lower values of 0.0352, 0.2287 and 0.3585 mg / mg / day, respectively compared to the corresponding value of 1.0397 mg / mg / day in control treatment at 96 hrs of exposure. Concentrations of LC₅₀ and LC₂₅ at 96 hrs of exposure had the same highest significant degree of effectiveness on RGR values of 0.0135 and 0.0604 mg / mg / day, respectively and followed by a significant RGR value of LC₁₀ treatment (0.1575 mg / mg / day) compared to the corresponding value of 0.5041 mg / mg / day in control treatment. Concentrations of LC₅₀ and LC₂₅ at 96 hrs of exposures had the same highest significant degree of effectiveness on ECI values of 37.42 and 40.63 %, respectively and followed by a significant ECI value of 44.16 % at LC₁₀ treatment compared to control treatment (48.72 %).

Eventually, the data of feeding indices of 2nd instar larvae of *S. littoralis* showed that the most potent sub-lethal concentrations of *A. judaica* extract on feeding indices were dominantly found at LC₅₀ and LC₂₅. The data of the significant values of feeding parameters at 72 and 96 hrs of exposures showed that the most effective time of *A. judaica* extract at different sub-lethal concentrations on anti-feedant activity, RCR and RGR parameters absolutely settled at the time of 96 hrs of exposures versus to 72 hrs of exposure for the parameter of ECI only.

Table 6: Feeding indices of the 2nd instar larvae of *Spodoptera littoralis* treated with different sub-lethal concentrations of *Artemisia judaica* extract at 72 and 96 hrs of exposure:

Sub-lethal concentrations	Exposure time (hrs)	Anti-feedant Activity %	RCR (mg/mg/day)	RGR (mg/mg/day)	ECI %
LC50	72	00.00 ^c	0.1590 ^{de}	0.0168 ^d	10.35 ^d
	96	51.55 ^a	0.0352 ^e	0.0135 ^d	37.42 ^{cbd}
LC25	72	00.00 ^c	1.0331 ^b	0.1523 ^{cd}	15.36 ^{cd}
	96	30.67 ^b	0.2287 ^{de}	0.0604 ^d	40.63 ^{cbd}
LC10	72	00.00 ^c	0.5548 ^c	0.6881 ^b	123.88 ^a
	96	00.00 ^c	0.3585 ^{dc}	0.1575 ^{cd}	44.16 ^{cb}
Control	72	00.00 ^c	1.6088 ^a	2.0643 ^a	128.15 ^a
	96	00.00 ^c	1.0397 ^b	0.5041 ^{cb}	48.72 ^b

Means for each column with the same letter are not significantly different for anti-feedant activity, RCR, RGR and ECI values according to LSD_{0.05} of interactions between concentrations and exposure times = 6.061, 0.2054, 0.365, and 31.567, respectively.

Binary Mixtures Evaluation (Lab. Experiment):

The data of *A. judaica* extract effectiveness on the toxicity of the tested insecticides against 2nd instar larvae of *S. littoralis* after 72 and 96 hrs of exposure were manifested in table (7). All the results of CTF of binary mixtures of chromafenozide + *A. judaica* had potentiation effect. Mixtures of chromafenozide +

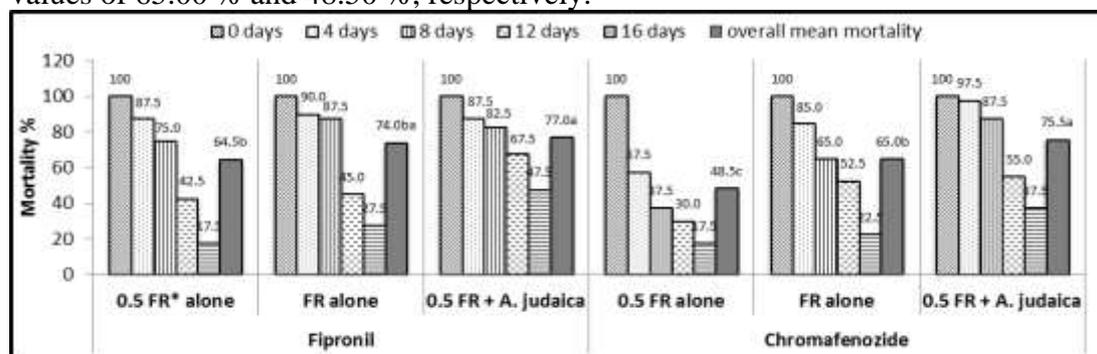
A. judaica at LC₅₀+LC₁₀ had CTF of 21.74 and 20.83 while mixtures of LC₅₀+LC₂₅ had 42.11 and 23.33 after 72 and 96 hrs of exposure, respectively. Furthermore, binary mixtures of fipronyl + *A. judaica* at LC₅₀+LC₂₅ had additive effect expressed by CTF of -17.24 and 6.67 after 72 and 96 hrs of exposure, respectively. On the contrary, mixtures of fipronyl + *A. judaica* at LC₅₀+LC₁₀ had antagonistic effect expressed by CTF of -25.00 after both of 72 and 96 hrs of exposure. The antagonistic effect revealed in all mixtures of pyridalyl + *A. judaica*.

Table 7: Effect of ethanol extract of *Artemisia judaica* on the toxicity of the selected insecticides against 2nd instar larvae of *S. littoralis* after different time of exposure:

Insecticides	Exposure time (hrs)	Concentrations of mixture	Expected % mortality	Observed % mortality	Co-toxicity factor	Action type
Chromafenozide + <i>A. judaica</i>	72	LC ₅₀ +LC ₁₀	47.50	67.50	21.74	Potentialiation
		LC ₅₀ +LC ₂₅	57.50	70.00	42.11	Potentialiation
	96	LC ₅₀ +LC ₁₀	60.00	72.50	20.83	Potentialiation
		LC ₅₀ +LC ₂₅	75.00	77.50	23.33	Potentialiation
Fipronyl + <i>A. judaica</i>	72	LC ₅₀ +LC ₁₀	70.00	52.50	-25.00	Antagonism
		LC ₅₀ +LC ₂₅	72.50	60.00	-17.24	Addition
	96	LC ₅₀ +LC ₁₀	65.00	50.00	-23.08	Antagonism
		LC ₅₀ +LC ₂₅	75.00	80.00	6.67	Addition
Pyridalyl + <i>A. judaica</i>	72	LC ₅₀ +LC ₁₀	50.00	37.50	-25.00	Antagonism
		LC ₅₀ +LC ₂₅	77.50	60.00	-22.58	Antagonism
	96	LC ₅₀ +LC ₁₀	57.50	42.50	-26.09	Antagonism
		LC ₅₀ +LC ₂₅	70.00	52.50	-25.00	Antagonism

Influences of *Artemisia judaica* Extract on Residual Efficacy of the Selected Insecticides (semi-field trials):

The data of *A. judaica* extract in the combine to each of fipronil and chromafenozide (passed the binary mixture tests in the laboratory) were tested in semi-field trials for their relative residual toxicity against 2nd instar larvae of *S. littoralis* after 96 hrs of exposure, during seasons of 2017 and 2018 (Fig. 1 and 2). Sufficient amount of formulated *A. judaica* extract was freshly prepared to apply in combination with the tested insecticides at the rate of 2.47gm / 3.5 liters (equivalent to LC₂₅) / micro-plot. In the season of 2017, the results of the joint action of *A. judaica* extract with fipronil 0.5 FR had an overall mean mortality percentage of 77.00 %, which was significantly higher than fipronil alone at FR and 0.5 FR with percentages values of 74.00 % and 64.50 %, respectively. On the other hand, overall mean mortality percentage of mixture of *A. judaica* extract with chromafenozide at 0.5 FR was 75.50 %. The overall mean mortality percentage of this mixture was significantly higher than chromafenozide alone at FR and 0.5 FR with percentage values of 65.00 % and 48.50 %, respectively.



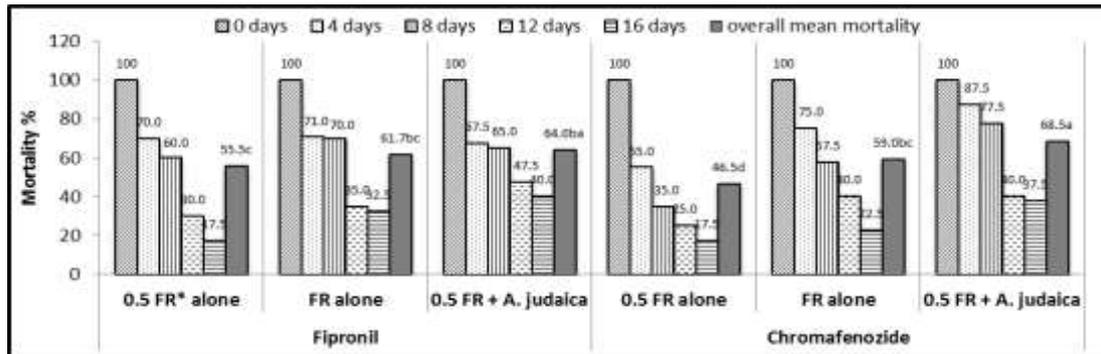
FR*: Field rate

Means with the same letter are not significantly different according to the LSD_{0.05} = 8.47.

Fig. 1. Effect of *Artemisia judaica* extract at LC₂₅ on the residual efficacy of fipronil and

chromafenozide against the 2nd instar larvae of *Spodoptera littoralis* at 96 hrs of exposure, season of 2017.

During the season of 2018, the results of the joint action of *A. judaica* extract with fipronil at 0.5 FR had an overall mean mortality percentage of 64.00 %, which was significantly higher than fipronil alone at FR and 0.5 FR with percentage values of 61.70 % and 55.50 %, respectively. Meanwhile, the overall mean mortality percentage of *A. judaica* extract with chromafenozide at 0.5 FR was 68.50 %. The overall mean mortality percentage of this mixture was significantly higher than chromafenozide alone at FR and 0.5 FR with percentages values of 59.00 % and 46.50 %, respectively.



FR*: Field rate

Means with the same letter are not significantly different according to the $LSD_{0.05} = 6.72$.

Fig. 2. Effect of *Artemisia judaica* extract at LC_{25} on the residual efficacy of fipronil and chromafenozide against the 2nd instar larvae of *Spodoptera littoralis* at 96 hrs of exposure, season of 2018.

Finally, the results of the season 2017 showed that the highest values of overall mean mortality had no significant differences in the mixtures of *A. judaica* extract with each of fibronil and chromafenozide at their 0.5 FR against 2nd instar larvae of CLW after 96 hrs of exposure. On the contrary, results of season of 2018 showed that *A. judaica* extract with chromafenozide at 0.5 FR was significantly higher than this extract with fibronil at 0.5 FR.

DISCUSSION

Herbal extract is natural products, environmentally friendly and cheap. The need for alternative non-chemical control strategies in crop protection systems has increased in the last decade due to problems associated with synthetic insecticides usages (Isman, 2006; El-Sharabasy, 2010). GC-MS analysis of ethanol extract of *A. judaica* flowering parts included main components of FAs (linoleic acid, oleic acid, lauric acid, and palmitoleic acid) as well as FAs esters (palmitic acid methyl ester, α linoleic methyl ester, and palmitoleic acid hydroxyl propyl ester). In addition, *A. judaica* extract had monoterpenes (eucalyptol), sesquiterpenes (tetraeurin-A), coumarins (coumarin), flavonoids (flavone). These determined compounds had some variances compared to the phytochemical components determined in the essential oil of *A. judaica* from the whole plant (stems, leaves, and flowers). Essential oil of *A. judaica* included major compounds of oxygenated monoterpenes (peritone, camphor and ethyl cinnamate). The prominent components in *A. judaica* oil included b-eudesmol, hexadecanoic acid, spathulenol, eudesma-4 (15),7-dien-1-b-ol, carvacrol and thymol which account for 67.3 % of the total essential oils composition (96.7 %)

(Lamya *et al.*, 2018). These significant variations in the chemical compositions of *A. judaica* essential oils were attributed to their locations that they developed. Compounds of α -bisabolol, *cis*-carvyl acetate, hexahydrofarnesyl acetone, α -cedrene epoxide, *iso*-amylphenylacetate, 2-methylbutyl-3-phenylpropanoate, hexadecanoic acid and aristolone were isolated first time from the oil of *A. judaica* (Al-Wahaibi *et al.*, 2018).

The data of toxicity and feeding indices of ethanol extract of *A. judaica* flowering parts could be justified for the presences of one or even more of the main phytochemical components in this extract that may have an important role as larvacidal and anti-feedant activity against the 2nd instar larvae of CLW. This justification meets the several studies on the structure–activity relationships of these semi-synthetic compounds for its insecticidal activity. The presence of the main components of FAs (linoleic acid, oleic acid, lauric acid, and palmitoleic acid) and FAs esters (palmitic acid methyl ester, α linoleic methyl ester, and palmitoleic acid hydroxyl propyl ester) in ethanol extract of *A. judaica* expected to give insecticidal activity against the larvae of *S. littoralis*. This was confirmed by the obtained results that showed toxic effects of linoleic acid methyl ester and oleic acid against the 2nd and 4th instar larvae of *S. littoralis* at 48 hrs of exposure. Unsaturated FAs of linoleic acid methyl ester (C18:2) and oleic acid (C18:1) showed relative significant effects compared to palmitic acid (C16:0) (Khamis *et al.*, 2016). Increasing in double bond numbers in 18:3 FAs were more toxic to growth than 18:2 FAs, and especially not toxic for those having 18:1 against ruminal bacterium *Butyrivibrio fibrisolvens* (Maia *et al.*, 2010). Moreover, the larval viability fifty (LV₅₀) values of linolenic and linoleic acid were 0.849×10^3 and 0.857×10^3 ppm, respectively. Thus, both FAs evaluated were found to have the insectistatic and insecticidal activities against *S. frugiperda* (Ramos-López *et al.*, 2012). In addition, a study on binary mixtures of the different compounds of natural coumarins had phago-depression effects against *S. frugiperda*. Coumarins derivatives transcend the predictable additive responses and could act synergistically against the larvae of *S. frugiperda*. In the chronic feeding bioassay, the addition of 50 $\mu\text{g/g}$ of synthesized coumarin in the form of benzopyran derivative to the diet of the larval gave rise to 80 % mortality in pupal stage (Vera *et al.* 2006). The determined sesquiterpene was Tetraneurin-A, belong to lactones compounds and possess C-14 and/or C-15 in oxygenated form, had inhibitory effects against *Heliothis zea* than those in unsubstituted analogues. The addition of 3.0 mM / kg of isolated tetraneurin-A to dietary of *H. zea* larvae declined the growth rate by 88 % compared to the control treatments (Isman and Rodriguez, 1983). Different polymethylated flavones and authentic analogues isolated from *Gnaphalium affine* had anti-feedant activity against *S. litura*. These flavonoids possessing 2-phenyl group known by B-ring form. The hypothesis of the B-ring effects in flavonoids was evaluated in the presence of chromone substitutions that eliminate the B-ring from. In spite of the phenyl flavonoids, some tested compounds did not show any insect anti-feedant activity against *S. litura* due to the deficiency in the 6-substituent group on the A-ring of the flavonoid. The tested flavonoids having substitutions with hydroxyl group as a hydrophilic group (water-hating group) on any of the 6-positions caused rises in anti-feedant activity of *S. litura* while hydrophilic substituents (water-loving groups) had a reduction effect on anti-feedant activity (Morimoto *et al.* 2003). Furthermore, eucalyptus oil a as common monoterpen in *A. judaica* extract possesses toxic and anti-feedant effects against 2nd and 4th instar larvae of *S. littoralis*. Thus, eucalyptus oil exhibited inhibitory effects that may not be able to detoxify by the detoxification enzymes of esterase, phosphatase, and GST in the larvae of *S.*

littoralis. Therefore, eucalyptus could be used in control applications as a part of integrated pest management (Ibrahim and Abd El-Kareem, 2018). High concentrations of ethanolic extract of *A. judaica* showed an inhibition of AchE activity in treated aphids but observed activation in GST was detected (Acheuk *et al.*, 2017).

In the laboratory experiments, binary mixtures of chromafenozide at LC₅₀ with *A. judaica* extract at LC₂₅ against 2nd instar larvae of CLW at 96 hrs of exposure had potent effects. Whereas, fipronil at LC₅₀ with *A. judaica* extract at LC₂₅ had additive effects at 96 hrs of exposure. We found that the more concentrations addition of extract (reach to LC₂₅) in these mixtures and more exposure time (at 96 hrs), the highest potentiation effects occurred. On the contrary, all mixtures of pyridalyl with *A. judaica* extract had antagonist action on larvae of CLW. These findings were agreed with the results of binary mixtures of conventional synthetic insecticides at 0.5 FR with three local plant extracts (*Azadirachta indica*, *Khaya senegalensis*, and *Hyptis suaveolens*) provided better protection of cotton against the bollworm, *Helicoverpa armigera*, than these insecticides or the plant extracts alone (Sinzogan *et al.*, 2006). In addition, acetone and petroleum ether plant extracts namely lupine, clove, dill, spearmint and their mixtures with the LC₅₀ of pirimiphos-methyl were evaluated against *Sitophilus oryzae* in wheat grains. The four plant extract mixtures with pirimiphos-methyl produced the pronounced additive effect at all concentrations (Zayed, 2014). Synergistic effects were found when neem seed kernel extract combined with the juvenile hormone mimic methoprene against some arthropods of medical and veterinary importance (Mulla and Su, 1999). Moreover, synergistic actions were revealed at all ratios especially at 1:1 ratio between cypermethrin and petroleum ether extract of *Solanum xanthocarpum* root against the larvae of malaria vector, *Anopheles stephensi* (Mohan *et al.*, 2007).

The data of semi-field experiments of *A. judaica* extract at LC₂₅ in combine with fipronil and chromafenozide had significant positive effects on the efficacy and residual toxicity of these insecticides alone against 2nd instar larvae of *S. littoralis* after 96 hrs of exposure. The efficacies of these mixtures were significantly higher than the field rate and half field rate of these insecticides alone during the seasons of 2017 and 2018. The rapid dissipations of using fipronil and chromafenozide alone could be explained by the residual dynamics experiments in cotton plants matrix. The main degradation had been latent in oxidation and photolysis pathways. The dissipation of fipronil in cotton plants expressed by half-life values had variations from 1.2 to 3.5 days after application (Wu *et al.*, 2017). In addition, the residues of chromafenozide dissipation expressed by half-life had ranged from 3.53 to 4.07 days. Therefore, the formulated chromafenozide could be included in the foliar spray schedules and safety evaluation for insecticide in strawberry (Malhat *et al.*, 2014). In this respect, *A. judaica* extract possesses some main components that might act as anti-oxidant agent to the tested synthetic insecticides that lead in preventing the rapid dissipation caused by the oxidative pathway. Thus, field experiments showed that the residual toxicity of these sprayed mixtures of the plant extract with the 0.5 FRs of the tested insecticides expected to be prolonged more than the FRs of the sprayed insecticides alone. Moreover, significant efficacies of these mixtures revealed against the larvae of CLW more than FRs of these insecticides alone. These findings were supported with many studies that had been carried out on the main components responsible for anti-oxidant and radical scavenging activities of in various *Artemisia* species (Kordali *et al.*, 2005; DaíseLopes-Lutz and Kolodziejczyk, 2008; Mohamed *et al.*, 2010).

CONCLUSION

The results of semi field experiments of binary mixtures of *A. judaica* extract at low sub-lethal concentrations with the half field rate of each of fipronil and chromafenozide had significant higher efficacies and residual toxicity than the field and half field rate of these insecticides alone against of *S. littoralis* larvae during the two seasons. The addition of this plant extract could help in eliminating the field rate of the tested insecticides and at the same time keep on their stable efficacies. In addition, the additions of this plant extract decreasing environmental hazard and curbing the propagation of insect resistance to words these insecticides. Eventually, these results considered as starting point for more prospect studies on the insecticidal structure–activity relationships of the main semi-synthetic compounds of *A. judaica* alone and with synthetic conventional insecticides against insect pests.

REFERENCES

- Abbott, W.S. 1925. A method for computing the effectiveness of an insecticide. *Journal of Economic Entomology*. 18: 265-267.
- Acheuk, F.; Lakhdari W.; Abdellaoui K.; Belaid M.; Allouane R. and Halouane F. 2017. Phytochemical study and bioinsecticidal effect of the crude ethonolic extract of the Algerian plant *Artemisia judaica*. (Asteraceae) against the black bean aphid, *aphis fabae* scop. *Agriculture and Forestry*. 63 (1): 95-104. DOI: <https://doi.org/10.17707/agricultforest.63.1.11>.
- Adesina, J.M. and Rajashekar Y. 2018. Phytochemical composition and insecticidal potentials of some plant aqueous extracts in suppressing *Podagraca* spp. (Coleoptera: Chrysomelidae) infestation on Okra (*Abelmoschus esculentus* L. Moench). *Advances in Horticulture Science*. 32 (1): 71-78.
- Ahmed, K.S.; Elbermawy S.M.; Ismael E.H.; Abdel-Gawwad R.M. and Saleh T.A. 2015. Histopathological effects of Diflubenzuron and chromafenozide (IGRs) on egg development in the cotton leafworm *Spodoptera littoralis* (Boisd) (Lepidoptera, Noctuidae). *Journal of Biological Science and Applied Research*. 1 (4):147-153.
- Al-Wahaibi, L.H.N.; Mahmood A.; Khan M. and Alkathlan H.Z. (2018). Comparative study on the essential oils of *Artemisia judaica* and *A. herba-alba* from Saudi Arabia. *Arabian Journal of Chemistry*. DOI: <https://doi.org/10.1016/j.arabjc.2018.03.004>.
- Anadon, A. and Gupta R.C. 2012. *Veterinary Toxicology*; Chapter 50: Fipronil. Oxford: ISBN: 978-0-12-385926-6 Copyright © 2012 Elsevier Inc., Academic Press. p. 604-608.
- Badr, A.; Morsy W.; Abdelfattah S.; Shams S. and Shehab A. 2012. Genetic diversity in *Artemisia monosperma* and *Artemisia judaica* populations in Egypt based on morphological, karyological and molecular variations. *Journal of Medicinal Plants Research*. 6 (1): 66-78_9.
- Bahlai, C.A.; Xue Y.; McCreary C.M.; Schaafsma A.W. and Hallett R.H. 2010. Choosing organic pesticides over synthetic pesticides may not effectively mitigate environmental risk in soybeans. *Public Library of Science*. 5 (6): e11250. Doi: <https://doi.org/10.1371/journal.Pone.0011250>.
- Batanouny, K.H. 1999. *Wild Medicinal Plants in Egypt; An inventory to support conservation and sustainable use*. With contributions by Aboutabl E.; Shabana M. and Soliman F. Academy of Scientific Research and Technology, Egypt and IUCN, Switzerland. p. 208.

- Bobe, A.; Meallier P.; Copper J. and Coste C.M. 1998. Kinetics and mechanisms of abiotic degradation of fipronil. *Journal of Agriculture and Food Chemistry*. 46: 2834–2839.
- Boulos, L. 2002. *Flora of Egypt*. Al-Hadara Publications, Cairo, Egypt. 3: 258-260.
- Cole, L.M.; Nicholson R. and Casida J.E. 1993. Action of phenylpyrazole insecticides at the GABA-gated chloride channel. *Pesticides Biochemical and Physiology*. 46: 47–54.
- DaiseLopes-Lutz and Kolodziejczyk P.P. 2008. *Screening of chemical composition, antimicrobial and antioxidant activities of Artemisia essential oils*. *Phytochemistry*. 69 (8): 1732-1738.
- El-Defrawi, M.E.; Topozada A.; Mansour N. and Zeid M. 1964. Toxicological studies on Egyptian cotton leaf worm *Prodenia litura* (F.), I: Susceptibility of different larval instars to insecticides. *Journal of Economic Entomology*. 57: 591 – 593.
- El-Sayed, A.A.A.; Amer E.A.A. and Desuky W.M. 2015. Side effect of some insect growth regulators (IGRs) on the associated predators of cotton leaf worm, *Spodoptera littoralis* (Boisd) (Lepidoptera, Noctuidae) in cotton fields. *Egyptian Academic Journal of Biological Science.(A.Entomology)*Vol. 8 (3): 79-86.
- El-Sharabasy, H.M. 2010. Acaricidal activities of *Artemisia judaica* L. extracts against *Tetranychus urticae* Koch and its predator *Phytoseiulus persimilis* Athias Henriot (Tetranychidae : Phytoseiidae). *Journal of Biopesticides*. 3 (2): 514 – 519.
- Finney, D.J. 1971. *Probit analysis: a statistical treatment of sigmoid response curve*. Cambridge University Press, London. 333pp.
- Hatem, A.E.; Homam H.B.; Amer R.A.M.; Abdel-Samad S.S.M.; Saleh H.A. and Hussien A.I. 2009. Synergistic activity of several acids in binary mixtures with synthetic insecticides on *Spodoptera littoralis* (Boisduval). *Boletin de Sanidad vegetal plagas*. 35: 533-542.
- Ibrahim, S.A. and Abd El-Kareem S.M.I. 2018. Enzymatic changes and toxic effect of some aromatic plant oils on the cotton leafworm, *Spodoptera littoralis* (Boisd.). *Egyptian Academic Journal of Biological Science (F-Toxicology and Pest control)*,Vol. 10 (1): 13- 24.
- Indhumathi, V.S.; Durairaj C., Nakkeeran S. and Shanmugaprema M. 2017. Safety of newer insecticide fipronil 200 SC to natural enemies in the Chilli ecosystem of Tamil Nadu. *Journal of Entomology and Zoology Studies*. 5 (6): 1975-1977.
- Isayama, S.; Saito S.; Kuroda K.; Umeda K. and Kasamatsu K. 2005. Pyridalyl, a novel insecticide: Potency and insecticidal selectivity. *Achieves of Insect Biochemistry and Physiology*. 58: 226-233.
- Isman, M.B. and Rodriguez E. 1983. Larval growth inhibitors from species of *Parhenium* (Asteraceae). *Phytochemistry*. 22 (12): 2709–2713.
- Isman, M.B. 2006. Botanical insecticides, deterrents, and repellents in modern agriculture and an increasingly regulated world. *Annual Review of Entomology*. 51: 45 – 66.
- Khamis, W.M.; El-Desouky S.E. and Gad A.A. 2016. Toxicity and antifeedant effects of apricot kernel extract and its main components against cotton leaf worm, *Spodoptera littoralis* (Lepidoptera: Noctuidae) larvae with reference to some physiological effects. 37: 3637-646.

- Kordali, S.; Kotan R.; Mavi A.; Cakir A.; Ala A. and Yildirim A. 2005. Determination of the chemical composition and antioxidant activity of the essential oil of *Artemisia dracuncululus* and of the antifungal and antibacterial activities of Turkish *Artemisia absinthium*, *A. dracuncululus*, *Artemisia santonicum*, and *Artemisia spicigera* essential oils. *Journal of Agricultural and Food Chemistry*. 53, (24): 9452-9458.
- Lamya, H.; Al-Wahaibi N.; Mahmood A.; Khan M. and Alkhatlan H.Z. 2018. Comparative study on the essential oils of *Artemisia judaica* and *A. herba-alba* from Saudi Arabia. *Arabian Journal of Chemistry*. 1878-5352. DOI: <https://doi.org/10.1016/j.arabjc.2018.03.004>.
- Loganathan, L. 2004. Efficacy of some botanicals alone and their combinations with synthetic insecticides against *Spodoptera litura* (FABRICUS). A thesis submitted to the faculty of post graduate school, Indian agricultural research institute, new Delhi in partial fulfillment of the requirements for the award of the degree of doctor of philosophy, Division of entomology Indian agricultural research institute, new delhi-110012.
- Maia, M. R.G.; Chaudhary L.C.; Bestwick C.S.; Richardson A.J; McKain N.; Larson T.R.; Graham I.A. and Wallace R.J. 2010. Toxicity of unsaturated fatty acids to the biohydrogenating ruminal bacterium, *Butyrivibrio fibrisolvens*. *Bio Medicine Central Microbiology*. 10(52): 10pp. DOI: <https://doi.org/10.1186/1471-2180-10-52>.
- Malhat, F.; Badawy H.M.A.; Barakat D.A. and Saber A.N. 2014. Residues, dissipation and safety evaluation of chromafenozide in strawberry under open field conditions. *Food Chemistry*. 152: 18–22.
- Mansour, N.A.; Eldefrawi M.E.; Topozada A. and Zeid M. 1966. Toxicological studies on the Egyptian cotton leafworm, *Prodenia litura*. VI. Potentiation and antagonism for organophosphorus and carbamate insecticides. *Journal of Economic Entomology*. 59: 307-311.
- Mohamed, A.H.; El-Sayed M.A.; Hegazy M.E.; Helaly S.E.; Esmail A.M. and Mohamed N.S. 2010. Chemical constituents and biological activities of *Artemisia herba-alba*. *Records of Natural Products*. 4 (1): 1-25.
- Mohan, L.; Sharma P. and Srivastava C.N. 2007. Comparative efficacy of *Solanum xanthocarpum* extracts alone and in combination with a synthetic pyrethroid, cypermethrin, against malaria vector, *Anopheles stephensi*. *Southeast Asian Journal of Tropical Medicine Public Health*. 38 (2): 156-260.
- Morimoto, M.; Tanimoto K.; Nakano S.; Ozaki T.; Nakano A. and Komi K. 2003. Insect anti-feedant activity of flavones and chromones against *Spodoptera litura*. *Journal of Agriculture Food and Chemistry*. 15, 51 (2): 389-93.
- Mulla, M.S. and Su T. 1999. Activity and biological effects of neem products against arthropods of medical and veterinary importance. *Journal of American Mosquito Control Association*. 15: 133-152.
- Vera, N.; Popich S.; Luna L.; Cravero R.; Gonzalez-sierra M. and Bardon A. 2006. Toxicity and synergism in the feeding deterrence of some coumarins on *Spodoptera frugiperda* SMITH (Lepidoptera: Noctuidae). *Chemistry and Biodiversity*. 3 (1): 21-6. DOI: <https://doi.org/10.1002/cbdv.200690002>.
- Osman, H.; Abou-zeid N. 2015. Bio-Efficiency component of *Capsicum* extract, Profenofos and their mixture on Some Biochemical and Histological aspects of *S. littoralis*. *Australian Journal of Basic and Applied Sciences*. 9 (20): 70-77.

- Ramos-López, M.A.; González-Chávez M.M.; Cárdenas-Ortega N.C.; Zavala-Sánchez M.A. and Pérez S.G. 2012. Activity of the main fatty acid components of the hexane leaf extract of *Ricinus communis* against *Spodoptera frugiperda*. African Journal of Biotechnology. 11(18): 4274-4278.
- Reda, B.F.A.; Al Bandari F.; Al Yousef and Hassan S.H. 2015. Toxicological effect of the botanical extract castor oil seeds *Ricinus communis* and their biochemical activity on the cotton leafworm, *Spodoptera littoralis* (Boisduval) (Lepidoptera: Noctuidae). Egyptian Academic Journal of biological Science Science (F-Toxicology and Pest control), Vol. 7 (1): 99-111.
- Saleh, M.A.; El-Bolok M.M.; Abdel Salam K.A. and Ibrahim N.A. 1986. Plant extracts affecting insect feeding growth and metamorphosis. Bulletin of Faculty of Agriculture, Cairo University. 37 (1): 526-539.
- SAS Institute, Inc. 2002. PC-SAS user guide, version 8. North Carolina statistical analysis system institute, Inc.
- Shalaby, F.F.; Hafez A.A.; Mohamed S.A. and Abd El-Aziz M.F. 2013. Toxicological and Environmental Chemistry. 95 (5): 778-789.
- Sinzogan, A.A.C.; Kossou D.K.; Atachi P. and VanHuis A. 2006. Participatory evaluation of synthetic and botanical pesticide mixtures for cotton bollworm control. International Journal of Tropical Insects Science. 26:246–255
- Tavares, W.S.; Costa M.A.; Cruz I.; Silveria R.D.; Serrao J.E. and Zanuncio J.C. 2010. Selective effects of natural and synthetic insecticides on mortality of *Spodoptera frugiperda* (Lepidoptera: Noctuidae) and its predator *Eriopis connexa* (coleopteran: coccinellidae). Journal of Environmental Science and Health, Part B. 45 (6): 557-61. DOI: <https://doi.org/10.1080/03601234.2010.493493>.
- Tiessen, S. 2012. Indiana's most unwanted invasive agricultural pest surveys program (CAPS), Egyptian cotton leafworm, *Spodoptera littoralis* (Boisduval). [<http://www.extension.entm.purdue.edu/CAPS/pestInfo/egyptleafworm.htm>]. Accessed: 2.7.
- Tomlin, C.D.S. 2009. The pesticide manual: A world compendium (15th ed.). Alton, U.K, British Crop Protection council. 1480 pp.
- Valant-Vetchera, K.M.; Fischer R.; Wollenweber E. 2003. Exudate flavonoids in species of *Artemisia* (Asteraceae-Anthemideae): new results and chemosystematic interpretation. Biochemical Systematic and Ecology. 31 (5): 487-498. DOI: [https://doi.org/10.1016/S0305-1978\(02\)00178-3](https://doi.org/10.1016/S0305-1978(02)00178-3).
- Wyk, B.V. and Wink M. 2005. Medicinal plants of the world. Briza Publications: Pretoria, South Africa ISBN 9781875093441 / ISBN 978-1-875093-44-1, Hardcover, 17 x 24 cm, 480 pp.
- Waldbauer, G. 1968. The consumption and utilization of food by insects. Advances in insect physiology. 5, 229–288.
- Wu, X.; Yu Y.; Xu J., Dong F.; Liu X.; Du P.; Wei D. and Zheng Y. 2017. Residue analysis and persistence evaluation of fipronil and its metabolites in cotton using high-performance liquid chromatography tandem mass spectrometry. Public Library of Science. 12 (3): e0173690. Doi: <https://doi.org/10.1371/journal.pone.0173690>.
- Zayed, G.M.M. 2014. Effect of some botanical products and their combinations with pirimiphos methyl against rice weevil, *Sitophilus oryzae* adult. Egyptian journal of plant protection research. 2 (2): 59-78.

ARABIC SUMMARY

الكفاءة الابدائية لمستخلص نبات البعيثران وبعض المبيدات الحشرية المختارة على دودة ورق القطن

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تمت دراسة السمية، مؤشرات التغذية والآثر الإبادى للمستخلص الإيثانولى لأزهار نبات البعيثران وبعض المبيدات الحشرية كلا على حدى فضلا عن خلطهم ضد الطور اليرقى الثانى لدودة ورق القطن. وكانت نتائج السمية للتركيزات المحققة لنسب الموت ٥٠% لمبيدات الكرومافينوزيد (٥٠،٢٥ و ٥٠،١٦ مجم \ لتر)، الفبيرونيل (٥٠،١٤٠ و ٥٠،٦٥ مجم \ لتر) والبيريداليل (٥٠،١٢٧ و ٥٠،١٢ مجم \ لتر) عند ٧٢ و ٩٦ ساعة على التوالي. هذا فإن التركيزات النصف قاتلة لمستخلص أزهار البعيثران (٩٤٨٢،٩٩ و ٢٨٣٩،٣٠ مجم \ لتر)، التركيزات القاتلة بنسبة ٢٥% (٣٠١١،١٥ و ٧٠٦،٨٢ مجم \ لتر) والتركيزات القاتلة بنسبة ١٠% (١٠٧٢،٢٧ و ٢٠٢،١٩ مجم \ لتر) عند ٧٢ و ٩٦ ساعة على التوالي. تحليل الكروماتوجرافى الغازى لهذا المستخلص يتضمن مركبات الاحماض الدهنية بنسبة ٥١،٥٠%، زيت الأوكالينول بنسبة ٣،٦٤%، التيترانويين-أ بنسبة ٢،٨٤%، الكومارين بنسبة ٢،٠٨% والفلافون بنسبة ٠،٨١%. وقد تبين أن اليرقات الخاضعة لجميع المعاملات كانت أكثر تأثرا عند ٩٦ ساعة من التعرض وفقا لإختبارات نشاط مانعات التغذية، معدل الاستهلاك النسبى، معدل النمو النسبى وكفاءة تحول الغذاء المهضوم. ومن خلال التجارب المعملية، أتضح التأثير التحفيزى للخلائط الثنائية لمبيد الكرومافينوزيد بتركيز نصف قاتل مع المستخلص عند تركيز قاتل لـ ١٠% من يرقات العمر الثانى لدودة ورق القطن وذلك من خلال قيم معامل السمية المشترك ٢١،٧٤ و ٢٠،٨٣ بعد ٧٢ و ٩٦ ساعة من التعرض للمعاملة على التوالي وكذلك مع المستخلص عند التركيز الربع قاتل ٤٢،١١ و ٢٣،٣٣ بعد ٧٢ و ٩٦ ساعة من التعرض للمعاملة على التوالي. وظهرت تأثيرات الاضافة لخلائط الفبيرونيل بتركيز نصف قاتل مع المستخلص بالتركيز القاتل لـ ٢٥% من خلال قيم معامل السمية المشترك ١٧،٢٤- ٦،٦٧ بعد ٧٢ و ٩٦ ساعة من التعرض على التوالي. وعلى النقيض، ظهر التأثير التثبيطى لخلائط الفبيرونيل بالتركيز النصف قاتل مع المستخلص بالتركيز القاتل لـ ١٠% فضلا عن جميع خلائط مبيد بايريداليل مع المستخلص. أظهرت التجارب النصف حقلية على يرقات العمر الثانى لدودة ورق القطن المعرضة للمعاملة لمدة ٩٦ ساعة أن متوسط نسبة الموت الكلى على مدى ١٦ يوم من المعاملة بالخليط المكون من مستخلص البعيثران بتركيز ٧٠٦،٨٢ مجم \ لتر مع نصف المعدل الحقلى لمبيد الفبيرونيل والكرومافينوزيد كانت (٧٧،٤٠٠ و ٦٤،٤٠٠%) و(٧٥،٥٠٠ و ٦٨،٥٠٠%) فى الموسمين الزراعيين ٢٠١٧ و ٢٠١٨ على التوالي. أمتد الأثر الإبادى لهذه الخلائط لفترات أطول وزادت سمية هذه الخلائط عن الاستخدام المنفرد لهذه المبيدات بمعدلاتهم الحقلية الكاملة. وأخيرا تم مناقشة النشاط الإبادى للحشرات لتلك المكونات التى تم تعريفها فى هذا المستخلص.