Field Evaluation of the Bioactivity of Flonicamid and Flubendiamide and Their Mixtures with the Lemongrass, *Cymbopogon citratus,* Essential Oil on Fall Armyworm (Lepidoptera: Noctuidae) Infesting Sweet Corn and Dissipation of Chemicals in Seeds and Husks[1]

Moataz A.M. Moustafa, Nourhan A. El-Said, Nawal AbdulAziz Alfuhaid[3],
Dalia E. El Hefny[4], Rania M.A. Helmy[4], Adrien Fónagy[2,5], and
EL-Desoky S. Ibrahim

Department of Economic Entomology and Pesticides, Faculty of Agriculture, Cairo University,
12613 Giza, Egypt

J. Entomol. Sci. 60(4): 000–000 (Month 2024)
DOI: 10.18474/JES24-61

Abstract Chemical insecticides are currently the major management means used against *Spodoptera frugiperda* (J.E. Smith) larvae on sweet corn (*Zea mays* L. var. *saccharata*) in Egypt. However, essential oils-based pesticides (EOs) and new insecticides might also be used. As a first report, this study aimed to assess the effectiveness and biochemical impact of lemongrass (*Cymbopogon citratus* Stapf) EO, flonicamid, and flubendiamide insecticides alone or in combination for managing *S. frugiperda* on sweet corn under field conditions. In addition, the dissipation of these compounds was determined in corn seeds and corn husks using the QuEChERS method combined with high-performance liquid chromatography (HPLC-DAD). The field efficacy trials showed that flubendiamide alone or in combination with lemongrass EO was more effective than either lemongrass EO or flonicamid alone or combined. Additionally, biochemical analysis revealed that detoxification enzymes may play an important role in *S. frugiperda* adaptation to flonicamid and flubendiamide. The residues of flonicamid and flubendiamide in corn seeds were undetectable in all treatments. Conversely, corn husks contained high levels of flubendiamide and flonicamid residues after application at high dosages. Interestingly, the dissipation rates of both tested insecticides increased when combined with lemongrass. The half-life values for flonicamid following the applications on corn husks alone or in combination with lemongrass EO were 4.44 and 2.45 d, respectively, while the half-life values for flubendiamide were 1.25 and 2.72 d, respectively. Our results show the potential use of flubendiamide alone or with lemongrass EO for managing *S. frugiperda* on sweet corn crops.

Key Words *Spodoptera frugiperda*, sweet corn, lemongrass, flonicamid, flubendiamide

---

[1] Received 15 June 2024; accepted for publication 31 July 2024.

[2] Corresponding author (email: fonagy.adrien@atk.hu).

[3] Department of Biology, College of Science and Humanities, Prince Sattam Bin Abdulaziz University, Al-Kharj 11942, Saudi Arabia.

[4] Pesticide Residues and Environmental Pollution Department, Central Agricultural Pesticide Laboratory, Agricultural Research Center, Dokki, 12618 Giza, Egypt.

[5] Plant Protection Institute, Centre for Agricultural Research, Eötvös Lóránd Research Network (ELKH), 1022 Budapest, Hungary.

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Sweet corn (*Zea mays* L. var. *saccharata*) is a large source of calories in the human diet (Dagla et al. 2014) and contains high nutritional value. Due to its high market potential and commercial value, demand for sweet corn is gradually rising in the peri-urban areas (Ratnakala et al. 2023). Unfortunately, this crop is often infested with insect pests that can damage the different parts of the plant and hinder its development. One of these destructive and polyphagous insects is the fall armyworm, *Spodoptera frugiperda* (J.E. Smith) (Lepidoptera: Noctuidae) (Sunari et al. 2022). In Africa, *S. frugiperda* reportedly has caused losses with a monetary value of approximately US$13 billion per annum in maize alone (Day et al. 2017). Moreover, *S. frugiperda* has become a serious threat to maize production in Africa due to the availability of a diverse range of host plants throughout the year and favorable climatic conditions for its growth and development (Montezano et al. 2018). The control of lepidopteran pests, including *S. frugiperda*, on sweet corn relies heavily on the use of chemical insecticides belonging to the conventional and neonicotinoid classes. Nevertheless, fall armyworm management appears challenging due to its short life cycle, wide host range, rapid multiplication, ability to rapidly spread across large geographical areas (Day et al. 2017, Li et al. 2021, Nboyine et al. 2022, Prasanna et al. 2018), and development of insecticide resistance (Li et al. 2023). Therefore, new compounds with green chemistries could offer great opportunities for managing crop insect pests, as they maintain a high level of efficacy to the target pests, low toxicity to the non-target organisms, and less persistence in comparison to the generic group of insecticides (Kodandaram et al. 2017).

In recent years, essential oils (EOs) based-pesticides and their bioactive compounds have been preferred as safer alternatives to synthetic pesticides (El-Shourbagy et al. 2023, Smith et al. 2018) because of their negligible persistence in the environment and minimum chances of resistance development (Kiran et al. 2017). Species from the genus *Cymbopogon* (Poaceae) are widely known for producing lemongrass EO and for their insecticidal properties (Jovanović et al. 2020, Moustafa et al. 2021). Oils from the lemongrasses (*Cymbopogon* spp.) are one of about 400–500 commercially produced EOs (Tisserand and Young 2013). The insecticidal property of lemongrass EO is attributed to the various secondary metabolites, such as bioactive cyclic and acyclic terpenes (Eden et al. 2020), which disrupt neurotransmission in insects (Zibaee 2015). Other secondary metabolites, such as alkaloids, flavonoids, and carotenoids (Avoseh et al. 2015) also have been found in lemongrass extract, indicating its potential as a botanical insecticide. In addition, tannin compounds may inhibit the digestion enzymic activities in insects (Rahayu et al. 2018). On the other hand, citral, a mixture of geranial and neral, is considered for the insecticidal activity of lemongrass EO (Eden et al. 2020, Moustafa et al. 2023a, Solomon et al. 2012) due to its interaction with oxidative stress and intracellular oxygen radicals (Kapur et al. 2016, Sanches et al. 2017).

The selective insecticide flonicamid shows insecticidal activities against piercing-sucking insects (Li et al. 2018, Liu et al. 2014, Xu et al. 2021); whereas, its effects on lepidopteran insect pests remain largely unknown. Recently, the inward rectifier potassium (Kir) channel has been verified to be a target of flonicamid. Although functional characterization of lepidopteran Kir genes is lacking (Meng et al. 2021), the main insecticidal mechanism of flonicamid against piecing-sucking insects is starvation due to the inhibition of stylet penetration into plant tissues (Morita et al. 2007, 2014).

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

The diamide group is one of the promising groups of insecticides (Moustafa et al. 2024a) that have been used against wide range of insects. Flubendiamide, a member of the diamide group, is one of the effective insecticides against many insect orders, that is, Lepidoptera, Diptera, and Coleoptera (Li et al. 2019, Kadala et al. 2020). It has a novel mode of action targeting the ryanodine receptors (RyR) and causing massive release of calcium ions from muscle cells by activating calcium channels existing on RyR and resulting in insect death (Cordova et al. 2006, Uesugi et al. 2021).

The current work presents the first information on the effectiveness and biochemical impact of lemongrass (*C. citratus*) EO, flonicamid, and flubendiamide insecticides, alone or in combination, as alternatives to conventional insecticides for managing fall armyworm on sweet corn under field conditions. In addition, the dissipation of these compounds in corn seed and corn husks was determined for the first time using the QuEChERS and HPLC-DAD methods.

Materials and Methods

Insecticides and chemicals. The lemongrass EO formulation used in this study was obtained from the Medicinal and Aromatic Plants Research Department farms, Agricultural Research Centre, Giza, Egypt. The commercial insecticide formulations (flonicamid and flubendiamide) used in this study are shown in Table 1. Flonicamid and flubendiamide (98%) reference materials were purchased from Dr. Ehrenstorfer GmbH (Augsburg, Germany). The stock solutions of both insecticides (0.1 mg/mL) in acetonitrile (ACN) were prepared according to El-Hefny et al. (2024) and Moustafa et al. (2024b, c). All chemicals and reagents used in the extraction and clean up were obtained from Merck Company (Darmstadt, Germany).

Field testing. To evaluate the efficacy of the lemongrass alone or in combination with flonicamid and flubendiamide insecticides against *S. frugiperda* larvae, field experiments on sweet corn variety 3020 (Hytech Seeds Company, Egypt) were conducted at the farm of the Faculty of Agriculture, Cairo University, Giza, Egypt over 2 consecutive seasons (2022 and 2023). In each hill, 2 kernels were planted by hand at 25 cm. Plants were thinned to only per hill before first irrigation. All other agronomic practices were appropriately followed and applied. In both seasons, each plot area was 21 m^2 (21 m^2 = 1/100 Fadden, where one Fadden = 4,200 m^2 = 2.4 ha) and consisted of 10 rows (3.0 m long and 70 cm wide). Six treatments were used: 3 each for lemongrass EO, flonicamid, and flubendiamide alone, 2 each for the 2 mixtures (lemongrass + flonicamid and lemongrass + flubendiamide), and 1 for the control. Experiments were conducted under a randomized complete block design (RCBD) with 4 replicates per treatment (Moustafa et al. 2022, 2023b). The tested insecticides and their mixtures were diluted in 20 L of water for each before being applied to 84 m^2 of plant area. The control area received 20 L of water only. The number of *S. frugiperda* larvae was counted before spraying (0 time) and 1, 3, 5, 7, 10 and 15 d post spraying. The percentage reduction in *S. frugiperda* population was calculated according to Henderson and Tilton (1955) as follows:

Reduction (%) = $[(A \times C)/(B \text{ x } D)] \times 100$, where A = number of individuals in treatment after application; B = number of individuals in treatment before application; C = number of individuals in control before application, and D = number of individuals in control after application.

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Table 1. Tested insecticides and their rate of application.

Common Name	Trade Name (a.i. %)	Chemical Group	Rate of Application (a.i./hectare)
Lemongrass oil	Lemongrass oil	Bioinsecticide	476.2 ml
Flonicamid	Teppeki 50% WG	Pyridine carboxamide	59.5 g
Lemongrass + Flonicamid	—	Combination	238.1 ml + 29.6 g
Flubendiamide	Takumi 20% WG	Anthranilic diamide	47.6 g
Lemongrass + Flubendiamide	—	Combination	238.1 ml + 23.8 g

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Preparation of enzyme samples. Fall armyworm larvae were collected after treatment and 100 mg larvae were homogenized in phosphate buffer (pH 0.7). The homogenate was centrifuged at 12,000 g for 15 min, and the supernatants were collected and used as enzyme suspension (Moustafa et al. 2023a). Total protein was quantified according to Bradford (1976), utilizing bovine serum albumin (BSA) as the standard.

Total esterase (EST) assay. EST assay was conducted using α-naphthyl acetate according to Van Asperen (1962) and Moustafa et al. (2023c). Thirty μl of enzyme suspension was mixed with α-naphthyl acetate (30 mM), and the mixture was incubated at 27°C for 15 min. The reaction was then stopped by adding 50 μl of fast blue b, and the absorbance was measured at 600 nm by spectrophotometry, utilizing α-naphthol as the standard.

Glutathione S-transferase (GST) assay. GST assay was conducted using 1-chloro-2,4-dinitrobenzene (CDNB) following the method of Habing et al. (1974) and Moustafa et al. (2023c). Ten μl of enzyme suspension was added to 25 μl of CDNB (30 mM), and 25 μl of GSH (50 mM) was then added after which the rate of change in absorbance during 5 min was recorded at 340 nm.

Acetylcholine esterase (AChE) assay. AChE assay was determined using acetylthiocholine iodide as described by Ellman et al. (1961). One hundred μl of enzyme suspension was added to 50 μl of acetylthiocholine iodide (0.075 M). Fifty μl of dithio-bis-nitro benzoic acid (0.01 M) was then added to produce a yellow color, and the rate of change was measured at 412 nm for 5 min.

Statistical analysis of efficacy and enzyme activity. Data of the efficacy and biochemical impact of the treatments were coded and entered using the statistical package SPSS (V.22). ANOVA analyses were conducted using MiniTab software (V14.0). The results were first tested for satisfying the assumptions of parametric tests while the continuous variables were subjected to Shapiro-Wilk and Kolmogorov-Smirnov test for normality. The reduction percentage data were standardized for normality using arcsine square root. The *post hoc* analysis used Tukey (HSD) pairwise comparison, where *P*-values were considered significant at <0.05. Finally, the data were visualized using R studio (V 2022.02.4.).

Residue analysis. To evaluate flonicamid and flubendiamide dissipation, corn seeds and husks samples were collected randomly at 0 (2 h after spraying), 1, 3, 5, 7, 10, and 15 d after applications. Purification and extraction were performed as described by Anastassiades et al. (2003). A 5-g sample of homogenized husks or seeds was weighed into a 50-ml Teflon centrifuge tube after which 5 ml of Milli-Q water followed by 10 ml acetonitrile were added and shaken vigorously with vortex mixer for 2 min (El-Hefny et al. 2024, Moustafa et al. 2024b). Anhydrous NaCl (1 g) and anhydrous $MgSO_4$ (4 g) were added to the mixture and mixed with the vortex shaker for 1 min. After centrifugation at 5,000 rpm for 5 min, 2 ml of the clarified supernatant was transferred into a Teflon centrifuge tube (10 ml) containing 50 mg PSA and 300 mg $MgSO_4$. The mixture was centrifuged, and the acetonitrile layer was then filtered through a filter membrane (0.22) μm and identity determined by HPLC.

Instrumentation of flonicamid and flubendiamide analysis. The Agilent HPLC 1260 infinite series (Agilent Technologies) was used to determine the dissipation rate of flonicamid and flubendiamide in seed and husk tissues (Kandil et al. 2023). The HPLC system included a quaternary pump, a variable wavelength diode array detector

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

(DAD), and an autosampler with an electric sample valve. It employed an ODS analytical column that measured 150 mm × 4.6 mm × 5 m. The mobile phase for flonicamid and flubendiamide was acetonitrile (65%) + water (35%) and acetonitrile (60%) + water (40%), respectively. For both insecticides, a flow rate of 1 ml/min, an injection volume of 20 μl, and a detection wavelength of 205 nm were used. The retention time was found to be 4.6 and 3.4 min for flonicamid and flubendiamide, respectively.

Statistical analysis of residues. The dissipation kinetics of flonicamid and flubendiamide residues in corn husks followed the first-order kinetic model, is described as the following equation (Hoskins et al. 1961): $C_t = C_0\ e^{-kt}$, where $C_0 =$ initial residue concentration (mg kg^{-1}); $C_t =$ residue level (mg kg^{-1}) at time t (day) after the pesticide application; and $k =$ the degradation rate constant (day^{-1}).

Method validation. The analysis was validated following the guidelines provided by Sante (2019). To verify the viability of the procedure, linearity, accuracy, precision, limit of detection (LOD), and limit of quantification (LOQ) were evaluated. To evaluate the precision and accuracy of the procedure, blank samples of maize seeds and husks were treated with 3 concentrations of flonicamid and flubendiamide with 5 replicates per treatment. To determine the most efficient combination of purifying agents, the recovery rate and relative standard deviation (RSD) were calculated for additional concentrations of 0.01, 0.1, and 1 mg/kg (Moustafa et al. 2024b). The spiked samples were left for 1 h to allow for insecticide absorption, and then extraction, cleanup, and analysis were performed as previously described. The method's sensitivity was tested using both LOQ and LOD. The recommended method for determining the LOQ was established using the lowest spiked concentration quantification. The precision, expressed as the relative standard deviation within laboratory repeatability analyses (% RSD), was calculated by dividing the standard deviation by the average concentration while the accuracy (average recovery) was calculated by dividing the recovered concentration by the spiking one. To assess linearity and compute pesticide content in samples, a calibration curve was constructed using 7 distinct concentrations of the insecticide standards stock solution (10, 5, 1, 0.5, 0.1, 0.05, and 0.01 mg/L) prepared by dilution with acetonitrile.

Results

Efficacy of the tested insecticides. The results in Fig. 1 show that the *S. frugiperda* larval infestation significantly decreased 3 ($F = 14.10$; df = 5; $P = 0.0001$) and 5 d ($F = 60.91$; df = 5; $P = 0.0001$) after the application of lemongrass and flonicamid, alone or combined, in 2022. However, the infestation increased again 7 d after application, but was remained below the control, until the end of the experiment. The same trend was observed in 2023 (Fig. 1). When flubendiamide was applied alone or combined with lemongrass, similar results were observed but with a consistent decline across all intervals during both seasons (Fig. 1). One day after the application of lemongrass, flonicamid, (lemongrass + flonicamid), flubendiamide and (lemongrass + flubendiamide) in 2022, the percentage reduction in *S. frugiperda* larvae at was 61.45, 35.48, 23.22, 76.73, and 54.45 for the respective treatments (Table 2; Fig. 2). With lemongrass, flonicamid, and (lemongrass + flonicamid), the reduction decreased to 25.91, 15.88, and 19.36% after 15 d for the respective treatments (Table 2; Fig. 2). In contrast, after application with

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

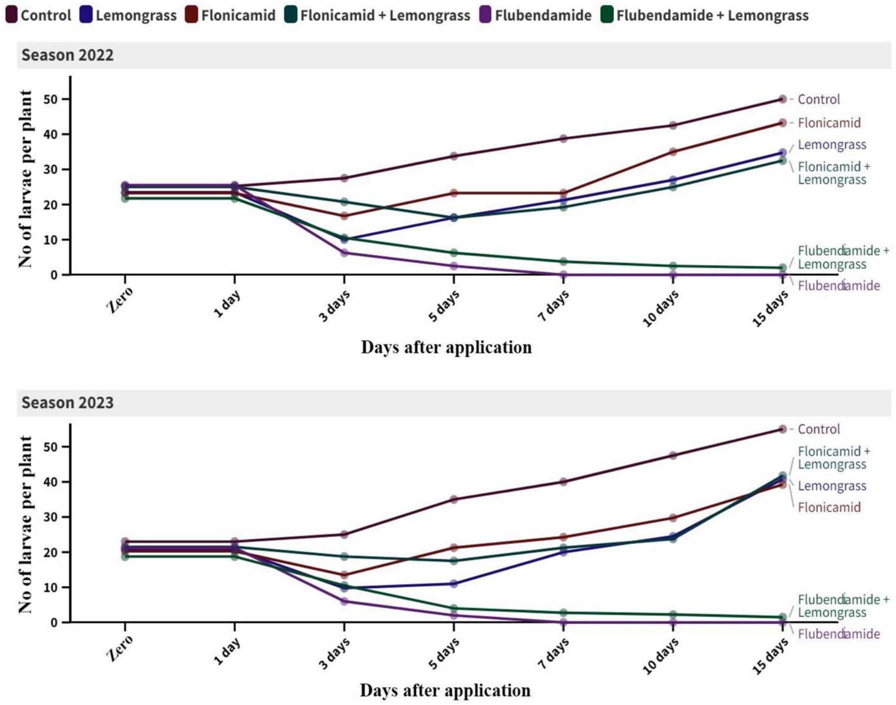

Fig. 1. Mean number (±SD) of *S. frugiperda* larvae on sweet corn plants after field application of flonicamid, flubendiamide alone or combined with lemongrass during the 2022 and 2023 seasons.

flubendiamide and (lemongrass + flubendiamide), the percentage of reduction consistently increased across all intervals. These trends were noticed in both seasons (Table 2; Fig. 2).

Effect of the tested insecticides on enzyme activity. The enzymatic activities of *a*-esterase, GST, and AChE were determined in *S. frugiperda* larvae 1, 3, 5, 7, 10, and 15 d after field application of lemongrass, flonicamid, and flubendiamide alone or as mixtures (Table 3). Data show that lemongrass significantly reduced the activity of *a*-esterase on day 1 and day 3 after field application (0.39 and 0.2 times, respectively). However, the activity insignificantly increased after 5, 7, and 15 d (2.13, 1.26, and 1.03 times, respectively). Additionally, flonicamid and its mixture with lemongrass significantly reduced the *a*-esterase activity on the first day (0.36 and 0.21 times, respectively) while they insignificantly increased it (1.29 and 1.18 times, respectively) on the third day after application (Table 3). On the other hand, flubendiamide significantly reduced the *a*-esterase activity (0.37 times) on the first day after application, while its mixture with lemongrass insignificantly reduced it (0.92 times). Concerning GST, lemongrass insignificantly reduced its activity at 1, 3, and 10 d (0.68, 0.91, and 0.48 times, respectively) and significantly increased it (2.9 times) at 7 d after application (Table 3). On the other hand, flonicamid and its mixture with lemongrass significantly increased the GST activity at

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Table 2. **Mean (±SD) percentage reductions in *S. frugiperda* larvae after field application of flonicamid and flubendiamide alone or combined with lemongrass during the 2022 and 2023 seasons.**

Season	Treatment	1 d	3 d	5 d	7 d	10 d	15 d
2022	Lemongrass	61.45 ± 4.76	81.6 ± 6.13	47.86 ± 4.9	43.42 ± 12.36	31.64 ± 5.96	25.91 ± 8.46
	Flonicamid	35.48 ± 6.71	33.93 ± 14.98	26.65 ± 4.96	26.21 ± 8.81	20.04 ± 5.27	15.88 ± 8.98
	Flonicamid + Lemongrass	23.22 ± 10.5	51.53 ± 5.09	50.69 ± 7.09	49.27 ± 8.86	40.04 ± 2.45	19.36 ± 5.9
	Flubendiamide	76.73 ± 5.34	86.97 ± 4.12	92.41 ± 3.55	100	100	100
	Flubendiamide + Lemongrass	54.45 ± 4.41	67.57 ± 13.84	76.91 ± 7.13	87.79 ± 4.68	91.84 ± 7.45	95.42 ± 3.4
F		29.71	14.41	61.71	44.08	160.71	136.50
P-value		0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
2023	Lemongrass	55.32 ± 11.74	79.85 ± 4.87	62.58 ± 15.56	44.13 ± 11.96	42.16 ± 8.22	20.36 ± 6.6
	Flonicamid	41.48 ± 8.56	46.87 ± 15.38	35.26 ± 13.67	37.21 ± 14.99	36.22 ± 10.18	21.19 ± 9.17
	Flonicamid + Lemongrass	48.69 ± 15.55	45.8 ± 17.53	44.07 ± 16.4	41.29 ± 13.27	45.94 ± 11.85	29.38 ± 5.7
	Flubendiamide	72.54 ± 6.56	94.05 ± 3.71	93.52 ± 4.13	100	100	100
	Flubendiamide + Lemongrass	47.18 ± 12.34	78.6 ± 7.43	84.86 ± 6.58	90.31 ± 5.05	93.97 ± 2.71	95.95 ± 3.66
F		3.31	10.98	12.51	23.73	44.26	144.21
P-value		0.039	0.0001	0.0001	0.0001	0.0001	0.0001

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

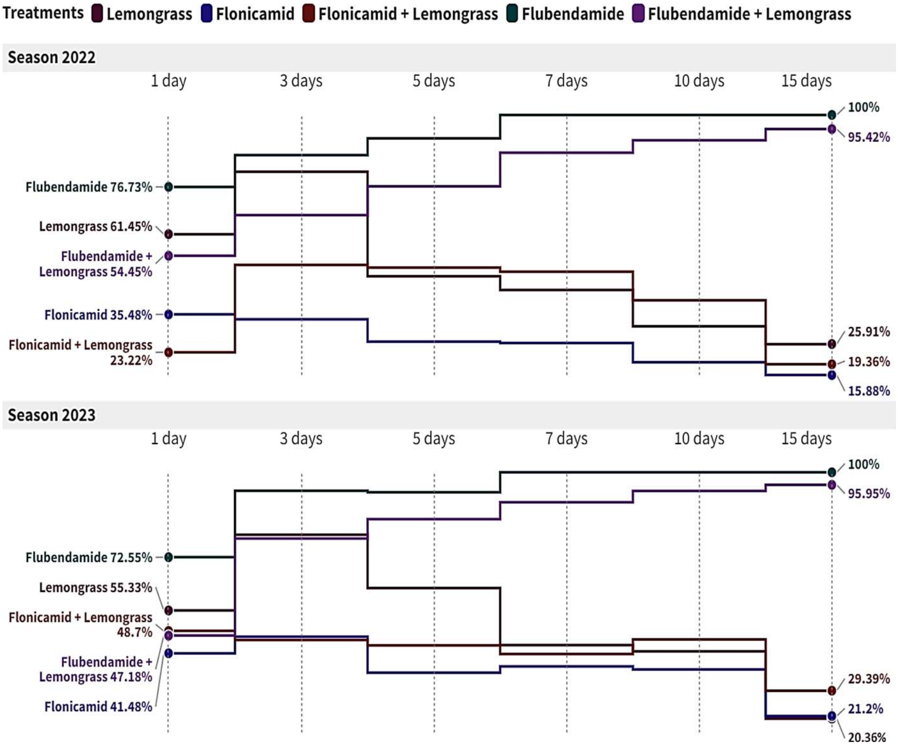

Fig. 2. Step slope chart representing the mean (±SD) percentage reductions in *S. frugiperda* larvae after field application of flonicamid and flubendiamide alone or combined with lemongrass during the 2022 and 2023 seasons.

5 d (4.59 and 4.44 times) and 15 d (2.52 and 2.59 times) after application (Table 3). In addition, flubendiamide alone significantly increased GST activity at 1 and 3 d after application (8.03 and 2.5 times, respectively) while its mixture with lemongrass significantly increased it at 7 and 15 d (2.45 and 3.22 times, respectively).

As to AChE, lemongrass insignificantly reduced its activity at 1, 5, 10, and 15 d (0.65, 0.86, 0.45, and 0.82 times, respectively) after application, while it significantly increased it at 7 d (2.35 times). On the contrary, flonicamid and its mixture with lemongrass significantly increased the AChE activity at 3 and 5 d after application (3.14 and 2.37 times and 3.73 and 4.34 times, respectively). Similarly, flubendiamide alone significantly increased AChE activity at 1 and 3 d (2.9 and 3.56 times, respectively) after application.

Method validation. To assess the linearity and matrix effect (ME), standard solution calibrations were created for flonicamid and flubendiamide in acetonitrile, corn seeds, and corn husks using 7 concentrations (10, 5, 1, 0.5, 0.1, 0.05, and 0.01 mg/L). Correlation coefficients (R^2) of $\geq$ 0.99 were obtained for flonicamid and flubendiamide in acetonitrile, and of 0.97–0.98 in corn seeds and corn husks,

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Table 3. Mean (±SD) enzymatic activity of EST (α-esterase), GST, and AChE in *S. frugiperda* larvae after field application of flonicamid and flubendiamide alone or combined with lemongrass during the 2022 and 2023 seasons.

Enzyme	Treatment	1 d	3 d
α-esterase (μmol/mg protein)	Control	1461.49 ± 267.86a	1047.92 ± 120.05ab
	Lemongrass	579.48 ± 165.86b	214.82 ± 59.13d
	Flonicamid	525.36 ± 50.03b	1361.28 ± 202.57a
	Flonicamid + Lemongrass	309.48 ± 160.9b	1234.39 ± 151.58a
	Flubendiamide	547.5 ± 75.66b	654.8 ± 92.78bc
	Flubendiamide + Lemongrass	1347.65 ± 50.19a	225.75 ± 86.66cd
GST (μmol/ml/mg protein)	Control	48.18 ± 5.2bc	51.02 ± 4.68b
	Lemongrass	32.72 ± 8.44c	46.41 ± 6.15b
	Flonicamid	65.77 ± 6.95bc	70.05 ± 5.84b
	Flonicamid + Lemongrass	20.77 ± 1.51c	125.8 ± 18.46a
	Flubendiamide	387.01 ± 35.5a	127.72 ± 30.8a
	Flubendiamide + Lemongrass	109.52 ± 26.2b	23.83 ± 4.8b
AChE (mmole/mg protein)	Control	5.7 ± 0.36bc	3.76 ± 0.67c
	Lemongrass	3.72 ± 0.79c	4.93 ± 0.32c
	Flonicamid	7.42 ± 0.96bc	11.82 ± 1.92ab
	Flonicamid + Lemongrass	2.98 ± 0.54c	8.9 ± 0.49b
	Flubendiamide	16.53 ± 2.68a	13.39 ± 1.61a
	Flubendiamide + Lemongrass	9.52 ± 1.96b	2.81 ± 0.38c

Means that do not share a letter in column are significantly different.

indicating a strong linear relationship. In addition, the LOQ of flonicamid and flubendiamide were 0.01 mg/kg.

The accuracy and precision of the method were verified using the recovery test and the relative standard deviation (RSD). Table 4 displays the flonicamid and flubendiamide recovery rates and the corresponding RSD for the 3 spike levels in corn seeds and corn husks. As shown, high recoveries of flonicamid and flubendiamide at the 3 spiking levels were obtained. In corn seeds and corn husks, the

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Table 3. Extended.

5 d	7 d	10 d	15 d
292.56 ± 54.54d	663.45 ± 269.54ab	734.27 ± 93.11c	386.88 ± 14.63c
624.3 ± 43.88cd	834.92 ± 110.9a	638.48 ± 39.2c	397.36 ± 51.91c
2369.57 ± 424.89b	410.99 ± 85.83ab	836.8 ± 39.9bc	1628.28 ± 199.23a
3445.53 ± 282.15a	302.81 ± 127.13b	1294.49 ± 125.21a	570.99 ± 228.37bc
—	—	—	—
1363.97 ± 46.65c	454.17 ± 111.55ab	1036.34 ± 61.29ab	1067.9 ± 168.71b
96.5 ± 34.97b	59.65 ± 14.97c	94.87 ± 8.77ab	48.02 ± 8.39b
145.96 ± 7.86b	173.11 ± 26.44a	45.47 ± 2.28b	48.4 ± 5.48b
442.63 ± 97.3a	112.02 ± 20.34bc	62.05 ± 11.8b	121.02 ± 18.19a
428.29 ± 53.75a	74.11 ± 10.55c	231.87 ± 98.27a	124.33 ± 19.36a
—	—	—	—
53.67 ± 11.08b	145.99 ± 12.61ab	89.33 ± 10.91ab	154.45 ± 16.42a
8.79 ± 1.63b	5.21 ± 1.39c	11.36 ± 2.58ab	8.07 ± 0.78c
7.52 ± 0.49b	12.24 ± 0.7ab	5.12 ± 0.54b	6.59 ± 0.59c
32.78 ± 4a	15.43 ± 4.02a	4.43 ± 0.36b	10.21 ± 0.94bc
38.17 ± 8.27a	6.43 ± 0.94bc	14.15 ± 4.6a	12.85 ± 1.83b
—	—	—	—
6.63 ± 1.16b	11.26 ± 1.16abc	12.59 ± 2.35ab	22.94 ± 1.2a

flonicamid recovery ranges were 95.97–106.0% and 81.32–89.72%, respectively, with RSD ranges of 1.57–2.65% and 1.68–2.95%, respectively. For the 3 spiking levels, the ranges of flubendiamide in corn seeds and corn husks were 83.29–94.70% and 91.97–99.24%, with RSD ranges of 4.66–8.03% and 1.40–5.80%, respectively.

Dissipation and terminal residue of flonicamid and flubendiamide in corn seeds and corn husks. Residues of flonicamid and flubendiamide alone or in combination with lemongrass EO in corn husks are displayed in Tables 5 and 6. In

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Table 4. Recovery of flonicamid and flubendiamide residues from corn seeds and corn husks.

Spiking Levels (μg/g)	Flonicamid				Flubendiamide			
	Corn Seeds		Corn Husks		Corn Seeds		Corn Husks	
	Recovery	RSD	Recovery	RSD	Recovery	RSD	Recovery	RSD
0.01	95.97	1.58	81.32	2.52	83.29	5.91	91.97	1.40
0.5	97.29	2.65	84.02	1.68	89.68	8.03	95.09	2.35
1	106.0	1.57	89.72	2.95	94.7	4.66	99.24	5.80

RSD, relative standard deviation.

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Table 5. Dissipation of flonicamid residues alone or combined with lemongrass oil in corn husks under open field conditions.

Days After Application	Flonicamid Alone			Flonicamid/Lemongrass Oil Mixture		
	Residues (mg/kg) n = 3	Dissipation Rate (%)	RSD (%)	Residues (mg/kg) n = 3	Dissipation Rate (%)	RSD (%)
0	3.45	0	0.51	1.52	0	0.59
1	2.34	32.18	0.22	1.21	20.4	0.17
3	2.09	39.43	0.03	0.52	65.8	0.10
5	1.51	56.24	0.27	0.44	71.7	0.08
7	1.4	59.43	0.1	0.19	87.5	0.06
10	0.56	84.2	0.11	0.07	95.4	0.03
15	ND	—	—	ND	—	—
$t_{1/2}$ (d)		4.44			2.45	

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Table 6. Dissipation of flubendiamide residues alone or combined with lemongrass oil in corn husks under open field conditions.

	Flubendiamide Alone			Flubendiamide/Lemongrass Oil Mixture		
Days After Application	Residues (mg/kg) n = 3	Dissipation Rate (%)	RSD (%)	Residues (mg/kg) n = 3	Dissipation Rate (%)	RSD (%)
0	9.35	0.00	1.22	5.03	0.00	1.14
1	5.61	40.00	1.53	3.07	38.96	1.25
3	2.67	71.44	1.39	2.26	55.06	1.55
5	1.73	81.49	1.87	1.77	64.81	1.01
7	1.24	86.73	0.99	1.06	78.92	0.78
10	0.88	90.58	1.42	0.23	95.42	1.15
15	ND	—	—	ND	—	—
$t_{1/2}$ (d)		1.25			2.72	

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

comparison, these residues in corn seeds were undetectable. Two hours after the application of flonicamid and flubendiamide alone, their residues were 3.45 and 9.35 mg/kg, respectively. One day after application, these levels dropped to 2.34 and 5.61 mg/kg with dissipation of 32.18 and 40.0%, respectively. On the 10th day, they reached 0.56 and 0.88 mg/kg with dissipation rates of 84.2 and 90.58%, respectively. Finally, they became undetectable on the 15th day. The half-lives for flonicamid and flubendiamide alone on corn husks were 4.44 and 1.25 d, respectively. On the other hand, the residues of flonicamid and flubendiamide mixtures with lemongrass oil in corn husks were 1.52 and 5.03 mg/kg, respectively, 2 h after application. One day after application, these residues declined to 1.21 and 3.07 mg/kg with dissipation rates of 20.4 and 38.96%, respectively. On the tenth day, the residues decreased to 0.07 and 0.23 mg/kg with dissipation rates of 95.40 and 95.42% for flonicamid and flubendiamide, respectively. Ultimately, on the 15th day, the residues of both insecticides became undetectable. The half-lives for flonicamid and flubendiamide mixtures with lemongrass on corn husks were 2.45 and 2.72 d, respectively (Tables 5 and 6).

Discussion

Essential oils (EOs) are produced by aromatic plants as secondary metabolites (Omotoso et al. 2020) and are used in the management of various insect insects and mites (Feroz 2020, Manh et al. 2020). However, their application can yield questionable outcomes due to several environmental factors, such as sunlight and UV (Moustafa et al. 2018) and potential phytotoxic effects (Chandler et al. 2011). To date, few studies have investigated the efficacy of EO-based biopesticides under conditions that reflect commercial practice. Therefore, the purpose of the current research was to assess the effectiveness of lemongrass oil, as a low-risk alternative to chemical pesticides, for managing pests (Radunz et al. 2024), alone or in combination with flonicamid and flubendiamide against *S. frugiperda* infestations in corn. In addition, this study assessed the biochemical impact of the tested compounds on enzymes' activities in *S. frugiperda* larvae and determined their persistence in sweet corn seeds and husks.

The overall results showed that flubendiamide and the flubendiamide/lemongrass mixture were the most effective in reducing *S. frugiperda* shortly after infestation (within a week after application). Flonicamid alone showed low efficacy against *S. frugiperda* larvae, while lemongrass alone and flonicamid/lemongrass mixture had an intermediate effect. These results agree with Cao et al. (2010) and Zhang et al. (2013) who reported that diamide insecticides, which selectively act on insect ryanodine receptors, display mortality rates, excellent fast actions, and long-term control of lepidopteran species. Additionally, diamide insecticides exhibit high bioactivity levels and excellent control of lepidopteran species (Razaq et al. 2007, Tohnishi et al. 2010, Yang et al. 2020, Zhang et al. 2020, Zhou et al. 2011). Also corroborating our results, Xing et al. (2013) noted that flubendiamide at 30 g ha^{-1} exhibited higher than 90% control efficacy against 4 lepidopteran pests 7 d after application; however, flonicamid had no insecticidal activity against rice stem borer, *Chilo suppressalis* Walker, larvae (Meng et al. 2021). The higher efficacy of flubendiamide could be explained by its unique mode of action in that it binds to

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

the lepidopteran ryanodine receptor (RyR), which is activated by the binding and releases Ca^{2+} that causes impairment of muscle regulation and results in rapid cessation of feeding and ultimately insect mortality (Cordova et al. 2006).

Insects often develop several strategies to overcome the potential toxicity of insecticides such as detoxification enzymes, which play an important part in the metabolism of pesticides in insects (Fouad et al. 2022), and developed resistance is usually associated with increased activity of these enzymes (Moustafa et al. 2023d). The insect detoxification enzyme system includes the metabolism and secretion of insecticides before reaching the target sites and producing their toxic effects (You et al. 2023). In the present study, *a*-esterase and GST activities in *S. frugiperda* larvae significantly declined immediately 1 d after the application of the tested compounds. However, these activities were restored or increased 15 d after application. The enzymatic activity of esterase and AChE in *S. frugiperda* larvae significantly decreased shortly after 1 d of the application of the tested compounds. However, the activity increased again after 10 and 15 d of application. Therefore, it can be suggested that the activities of these enzymes can serve as indicators of the adaptation of *S. frugiperda* to insecticides (Koirala et al. 2022). As a target for insecticides, GST is important for the detoxification of pesticides by converting their lipid metabolites (Korkina 2016).

The dissipation of pesticides in crops is affected by many factors such as the volatilization and photolysis of pesticides caused by light and high temperatures, scouring caused by rain, and the physical and chemical properties of pesticides (Chen et al. 2013, Subirats et al. 2005). In this study, the linearity, matrix effect (ME), limit of quantitation, precision, and accuracy of the suggested method were assessed. Accordingly, the method can be used for the quantitative analysis of flonicamid and flubendiamide in corn seeds and corn husks, which are essential food/feed for humans and livestock. The method has a high detection efficiency and is reasonably priced. The relative recoveries for most pesticides ranged from 70 to 120% in different matrices, as verified by García-Vara et al. (2023). Furthermore, the detection limits, which ranged from 0.01 to 20 ng/g, were lower than the highest amount of residue.

Regarding the dissipation of the tested compounds, the elevated levels of flonicamid and flubendiamide in corn husks may be a result of the direct spraying administration method. On the other hand, the pesticide residue levels in corn seeds were below the detection level. This can be attributed to the husks' ability to keep the applied pesticides away from reaching the seeds. As to flonicamid, it declined to 32.18% initially and then increased to 84.2% after 10 d, while flonicamid/lemongrass mixture declined to 20.4% initially and then heightened to 95.4% with corresponding half-lives of 4.44 and 2.45 d, respectively. The higher dissipation rate of the flonicamid/lemongrass mixture compared to flonicamid alone can be attributed to the addition of lemongrass. As flubendiamide alone, it also declined from 40% initially to 90.58% after 15 d, while the flubendiamide/lemongrass mixture dissipation rate was 95.4% after 15 d with corresponding half-lives of 1.25 and 2.72 d, respectively. Consistent with our present results are those of Kelageri et al. (2017) who found that the initial residue of flonicamid in greenhouse-grown tomatoes (*Solanum lycopersicum* L.) was 1.23 mg/kg, which became undetectable 10 d after application. In addition, flonicamid showed a high dissipation rate in 4 different crops including peach (*Prunus persica* L.), cucumber (*Cucumis sativus* L.),

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

cabbage (*Brassica oleracea* L.), and cottonseed (*Gossypium arboretum* L.), with half-lives ranging between 2.28 and 9.74 d (Zhang et al. 2022). Moreover, Liu et al. (2014) observed low residual levels of flonicamid, with corresponding half-life ranges of 10.3–14.2, 5.1–6.1, and 3.0–4.9 d in soil, apple, and cucumber, respectively, after different interval days (1 to 14 d) from spraying with 1.5 times higher of its recommended dose. In addition, the final residues of flonicamid ranged from 0.029 to 0.295 mg/kg in cucumbers, $<$0.01–0.174 mg/kg in apples, and $<$0.01–0.172 mg/kg in soil, respectively. The dissipation of total residues of flonicamid and its metabolites in soil and cabbage were well fitted by the first-order kinetics model with half-lives of 2.04–7.62 d and 1.97–4.99 d in soil and cabbage, respectively, after spraying flonicamid at recommended dose and 1.5-fold higher (Wang et al. 2018). In a similar study, Singh et al. (2023) reported that the initial residue of flubendiamide in tomato fruit grown under field and poly-house conditions rose to 94.75 and 73.85%, respectively, on the 10th day, with dissipation median time (DT_{50}) of 2.25 and 5.02 d, respectively.

In this context, a preharvest interval (PHI) of 1 d has been recommended for flubendiamide as its half-life ranged from 0.33 to 3.28 d after spraying of flubendiamide (480 SC) on tomato crop at 48 and 96 g active ingredient (a.i.)/ha (Sharma et al. 2014). In similar studies, the half-life of flubendiamide in tomato fruit was 1.64 and 1.98 d (Paramasivam and Banerjee 2012), while it was 3.9 and 4.45 d in cabbage (Mohapatra et al. 2010) after its application with 24 and 48 g a.i./ha, respectively.

In conclusion, it has been found that lemongrass oil had insecticidal effects on *S. frugiperda.* In addition, the dissipation rates of flonicamid and flubendiamide when applied alone were higher than those of their mixtures with lemongrass, i.e., lemongrass enhanced their dissipation rates in corn seeds. Therefore, flonicamid and flubendiamide residues were undetectable in corn seeds. This finding implies that harvesting the corn seeds shortly after the application of flonicamid and flubendiamide would be safe. On the other hand, the high residue levels of flonicamid and flubendiamide in corn husks can be attributed to the direct application method. Consequently, further research is needed to verify the safe utilization of corn husks treated with these materials as a raw material in animal feed.

Acknowledgments

We thank Prof. Hamed Khairullah Said (Associate Professor, Faculty of Agriculture, Cairo University, Egypt) for improving the manuscript, including the English language and grammar.

References Cited

Anastassiades, M., S.J. Lehotay, D. Tajnbaher and J.F. Schenck. 2003. Fast and easy multiresidues method employing acetonitrile extraction/partitioning and “dispersive solid-phase extraction” for the determination of pesticide residues in produce. J. AOAC Int. 86: 412–430. doi: 10.1093/jaoac/86.2.412.

Avoseh, O., O. Oyedeji, P. Rungqu, B. Nkeh-Chungag and A. Oyedeji. 2015. Cymbopogon species; ethnopharmacology, phytochemistry and the pharmacological importance. Molecules 20: 7438–7453. doi: 10.3390/molecules20057438.

Bradford, M.M. 1976. A rapid and sensitive method for the quantitation of microgram quantities of protein, utilizing the principle of protein-dye binding. Anal. Biochem. 72: 248248e254. doi: 10.1006/abio.1976.9999.

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Cao, G., Q. Lu, L. Zhang, F. Guo, G. Liang, K. Wu, K. Wyckhuys and Y. Guo. 2010. Toxicity of chlorantraniliprole to Cry1Ac-susceptible and resistant strains of *Helicoverpa armigera*. Pest Biochem. Physiol. 98: 99–103. doi: 10.1016/j.pestbp.2010.05.006.

Chandler, D., A.S. Bailey, G.M. Tatchell, G. Davidson, J. Greaves and W.P. Grant. 2011. The development, regulation, and use of biopesticides for integrated pest management. Phil. Trans. Roy. Soc. B. 366: 1987–1998. doi: 10.1098/rstb.2010.0390.

Chen, M., E.M. Collins, L. Tao and C.S. Lu. 2013. Simultaneous determination of residues in pollen and high-fructose corn syrup from eight neonicotinoid insecticides by liquid chromatography–tandem mass spectrometry. Anal. Bioanal. Chem. 405: 9251–9264. doi: 10.1007/s00216-013-7338-7.

Cordova, D., E.A. Benner, M.D. Sacher, J.J. Rauh, J.S. Sopa, G.P. Lahm and Y. Tao. 2006. Anthranilic diamides: a new class of insecticides with a novel mode of action, ryanodine receptor activation. Pestic. Biochem. Physiol. 84: 196–214. doi: 10.1016/j.pestbp.2005.07.005.

Dagla, M.C., R.N. Gadag, N. Kumar, B.C. Ajay and C. Ram. 2014. A potential scope of sweet corn for peri-urban farmers in India. Popul. Kheti. 2: 69–73.

Day, R., P. Abrahams, M. Bateman, T. Beale, V. Clottey, M. Cock and A. Witt. 2017. Fall armyworm: impacts and implications for Africa. Outlooks Pest Manag. 28: 196–201. doi: 10.1564/v28_oct_02.

Eden, W.T., D. Alighiri, K.I. Supardi and E. Cahyono. 2020. The mosquito repellent activity of the active component of air freshener gel from Java Citronella oil (*Cymbopogon winterianus*). J. Parasitol. Res. 2020: 9053741. doi: 10.1155/2020/9053741.

Ellman, G.L., K.D. Courtney, V. Andres Jr and R.M. Featherstone. 1961. A new and rapid colorimetric determination of acetylcholinesterase activity. Biochem. Pharmacol. 7: 88–95. doi: 10.1016/0006-2952(61)90145-9.

El-Hefny, D.E., E.S. Ibrahim, N.A. Alfuhaid, A. Fónagy and M.A.M. Moustafa. 2024. Field bioefficacy against *Aphis craccivora* and residue dynamics of the insecticides flonicamid and spiromesofen in faba bean (*Vicia faba* L.). J. Entomol. Sci. 59: 165–181. doi: 10.18474/JES23-39.

El-Shourbagy, N.M., S.M. Farag, M.A.M. Moustafa, L.A. Al-Shuraym, S. Sayed and O.H. Zyaan. 2023. Biochemical and insecticidal efficacy of clove and basil essential oils and two photosensitizers and their combinations on *Aphis gossypii* Glover (Hemiptera: Aphididae). Biosci. J. 39: e39100. doi: 10.14393/BJ-v39n0a2023-69129.

Feroz, A. 2020. Efficacy and cytotoxic potential of deltamethrin, essential oils of *Cymbopogon citratus* and *Cinnamonum camphora* and their synergistic combinations against stored product pest *Trogoderma granarium* (Everts). J. Stored Prod. Res. 87: 101614. doi: 10.1016/j.jspr.2020.101614.

Fouad, E.M., F.S. Ahmed and M.A.M. Moustafa. 2022. Monitoring and biochemical impact of insecticides resistance on field populations of *Spodoptera littoralis* (Boisd.) (Lepidoptera: Noctuidae) in Egypt. Polish J. Entomol. 91: 109–118. doi: 10.5604/01.3001.0015.9707.

García-Vara, M., C. Cristina Postigo, P. Palma and M.L. Alda. 2023. Development of QuEChERS-based multiresidue analytical methods to determine pesticides in corn, grapes, and alfalfa. Food Chem. 405: 134870. doi: 10.1016/j.foodchem.2022.13487.

Habig, W.H., M.J. Pabst and W.B. Jakoby. 1974. Glutathione S-Transferases: The first enzymatic step in mercapturic acid formation. J. Biol. Chem. 249: 7130–7139. doi: 10.1042/bj2150617.

Henderson, C.F. and E.W. Tilton. 1955. Tests with acaricides against the brown wheat mite. J. Econ. Entomol. 48: 157–161.

Hoskins, W.M. 1961. Mathematical treatment of the rate of loss of pesticide residues. FAO Plant Prot. Bull. 9: 163–168.

Jovanović, J., S. Krnjajić, J. Ćirković, A. Radojković, T. Popović, G. Branković and Z. Branković. 2020. Effect of encapsulated lemongrass (*Cymbopogon citratus* L.) essential oil against potato tuber moth *Phthorimaea operculella*. Crop Prot. 132: 105109. doi: 10.1016/j.cropro.2020.105109.

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Kadala, A., M. Charreton and C. Collet. 2020. Flubendiamide, the first phthalic acid diamide insecticide, impairs neuronal calcium signaling in the honey bees antennae. J. Insect Physiol. 125: 104086. doi: 10.1016/j.jinsphys.2020.104086.

Kandil, M.A., M.A.M. Moustafa, M.A. Saleh and I.R. Ateya. 2023. Dissipation kinetics and degradation products of cyantraniliprole in tomato plants and soil in the open field. Egyptian J. Chem. 66: 483–493. doi: 10.21608/ejchem.2023.195133.7625.

Kapur, A., M. Felder, L. Fass, J. Kaur, A. Czarnecki, K. Rathi and M.S. Patankar. 2016. Modulation of oxidative stress and subsequent induction of apoptosis and endoplasmic reticulum stress allows citral to decrease cancer cell proliferation. Scientific Rep. 6: 27530. doi: 10.1038/srep27530.

Kelageri, S.S., C.S. Rao, V.S. Bhushan, P.N. Reddy, S. Rani, M. Aruna, A.H. Reddy, D. Ravindranath, B. Ramesh and M. Hymavathy. 2017. Flubendiamide: residues and risk assessment in tomato *Solanum lycopersicum*. Int. J. Environ. Agric. Biotech. 10: 651–667. doi: 10.5958/2230-732X.2017.00081.X.

Kiran, S., A. Kujur, L. Patel, K. Ramalakshmi and B. Prakash. 2017. Assessment of toxicity and biochemical mechanisms underlying the insecticidal activity of chemically characterized *Boswellia carterii* essential oil against insect pest of legume seeds. Pestic. Biochem. Physiol. 139: 17–23. doi: 10.1016/j.pestbp.2017.04.004.

Kodandaram, M.H., Y.B. Kumar, K. Banerjee, S. Hingmire, A.B. Rai and B. Singh. 2017. Field bioefficacy, phytotoxicity and residue dynamics of the insecticide flonicamid (50 WG) in okra [*Abelmoschus esculenta* (L) Moench]. Crop Prot. 94: 13–19. doi: 10.1016/j.cropro.2016.12.003.

Koirala, B.K.S., T. Moural and F. Zhu. 2022. Functional and structural diversity of insect glutathione S-transferases in xenobiotic adaptation. Int. J. Biol. Sci. 18: 5713–5723. doi: 10.7150/ijbs.77141.

Korkina, L. 2016. Metabolic and redox barriers in the skin exposed to drugs and xenobiotics. Expert Opinion Drug Metabol. Toxicol. 12: 377–388. doi: 10.1517/17425255.2016.1149569.

Li, J.X., Y.J. Wang, J. Xue, P.S. Wang and S.M. Shi. 2018. Dietary exposure risk assessment of flonicamid and its effect on constituents after application in *Lonicerae japonicae* Flos. Chem. Pharm. Bull. 66: 608–611. doi: 10.1248/cpb.c17-00985.

Li, W., Y. Zhang, H. Jia, W. Zhou, B. Li and H. Huang. 2019. Residue analysis of tetraniliprole in rice and related environmental samples by HPLC/MS. Microchem. J. 150: 104168. doi: 10.1016/j.microc.2019.104168.

Li, X., H. Jiang, J. Wu, F. Zheng, K. Xu, Y. Lin and H. Xu. 2021. Drip application of chlorantraniliprole effectively controls invasive *Spodoptera frugiperda* (Lepidoptera: Noctuidae) and its distribution in maize in China. Crop Prot. 143: 105474. doi: 10.1016/j.cropro.2020.105474.

Li, X., Y. Liu, Z. Pei, G. Tong, J. Yue, J. Li and L. Ban. 2023. The efficiency of pest control options against two major sweet corn ear pests in China. Insects 14(12): 929. doi: 10.3390/insects14120929.

Liu, X.G., Y.L. Zhu, F.S. Dong, J. Xu and Y.Q. Zheng. 2014. Dissipation and residue of flonicamid in cucumber, apple, and soil under field conditions. Anal. Chem. 94: 652–660. doi:10.1080/03067319.2013.871714.

Manh, H.D., D.T. Hue, N.T.T. Hieu, D.T.T. Tuyen and O.T. Tuyet. 2020. The mosquito larvicidal activity of essential oils from *Cymbopogon* and *Eucalyptus* species in Vietnam. Insects 11: 128. doi: 10.3390/insects11020128.

Meng, X., Z. Wu, X. Yang, K. Qian, N. Zhang, H. Jiang and J. Wang. 2021. Flonicamid and knockdown of inward rectifier potassium channel gene CsKir2B adversely affect the feeding and development of *Chilo suppressalis*. Pest Manag. Sci. 77: 2045–2053. doi: 10.1002/ps.6232.

Mohapatra, S., A.K. Ahuja, M. Deepa, D. Sharma, G.K. Jagadish and N. Rashmi. 2010. Persistence and dissipation of flubendiamide and des-iodo flubendiamide in cabbage (*Brassica oleracea* Linn) and soil. Bull. Environ. Contamin. Toxicol. 85: 352–356. doi: 10.1007/s00128-010-0063-4.

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Montezano, D.G., D.R. Sosa-Gómez, A. Specht, V.F. Roque-Specht, J.C. Sousa-Silva, S.D. Paula-Moraes and T.E. Hunt. 2018. Host plants of *Spodoptera frugiperda* (Lepidoptera: Noctuidae) in the Americas. African Entomol. 26: 286–300. doi: 10.1007/s10709-015-9829-2.

Morita, M., T. Ueda, T. Yoneda, T. Koyanagi and T. Haga. 2007. Flonicamid, a novel insecticide with a rapid inhibitory effect on aphid feeding. Pest Manag Sci: formerly Pesticide Sci. 63(10): 969–973. doi: 10.1002/ps.1423.

Morita, M., T. Yoneda and N. Akiyoshi. 2014. Research and development of a novel insecticide, flonicamid. J. Pestic. Sci. 39: 179–180. doi: 10.1584/jpestics.J14-05.

Moustafa, M.A.M., M.A. Saleh, I.R. Ateya and M.A. Kandil. 2018. Influence of some environmental conditions on stability and activity of *Bacillus thuringiensis* formulations against the cotton leaf worm, *Spodoptera littoralis* (Boisd.) (Lepidoptera: Noctuidae). Egyptian J. Biol. Pest Contr. 28: 1–7. doi: 10.1186/s41938-018-0064-x.

Moustafa, M.A.M., M. Awad, A. Amer, N.N. Hassan, E.S. Ibrahim, H.M. Ali, M. Akrami and M.Z.M. Salem. 2021. Insecticidal activity of lemongrass essential oil as an eco-friendly agent against the black cutworm *Agrotis ipsilon* (Lepidoptera: Noctuidae). Insects 12: 737. doi: 10.3390/insects12080737.

Moustafa, M.A.M., A. Amer, L.A. Al-Shuraym, E.S. Ibrahim, D.E. El-Hefny, M.Z.M. Salem and S. Sayed. 2022. Efficacy of chemical and bio-pesticides on cowpea aphid, *Aphis craccivora*, and their residues on the productivity of fennel plants (*Foeniculum vulgare*). J. King Saud Univ.–Sci. 34: 101900. doi: 10.1016/j.jksus.2022.101900.

Moustafa, M.A.M., N.N. Hassan, N.A. Alfuhaid, A. Amer and M. Awad. 2023a. Insights into the toxicity, biochemical activity, and molecular docking of *Cymbopogon citratus* essential oils and citral on the cotton leafworm *Spodoptera littoralis.* J. Econ. Entomol. 116: 1185–1195. doi: 10.1093/jee/toad093.

Moustafa, M.A.M., D.E. El-Hefny, R.N. Abdel-kerim and M.A. Kandil. 2023b. Toxicity of bio and chemical insecticides against tomato leaf miner, *Tuta absoluta,* and its predators and determination of their residue dissipation in tomato fruits. J. Agric. Sci. Technol. 25: 1115–1128. doi: 10.22034/jast.25.5.1115.

Moustafa, M.A.M., E.A. Fouad, E. Ibrahim, A.L. Erdei, Z. Kárpáti and A. Fónagy. 2023c. The comparative toxicity, biochemical and physiological impacts of chlorantraniliprole and indoxacarb on *Mamestra brassicae* (Lepidoptera: Noctuidae). Toxics. 11: 212. doi: 10.3390/toxics11030212.

Moustafa, M.A.M., R.I.A. Moteleb, Y.F. Ghoneim, S.S. Hafez, R.E. Ali, E.E.A. Eweis and N.N. Hassan. 2023d. Monitoring resistance and biochemical studies of three Egyptian field strains of *Spodoptera littoralis* (Lepidoptera: Noctuidae) to six insecticides. Toxics. 11: 211. doi: 10.3390/toxics11030211.

Moustafa, M.A.M., E.A. Osman, E.M.S. Mokbel and E.A. Fouad. 2024a. Biochemical and molecular characterization of chlorantraniliprole resistance in *Spodoptera littoralis* (Lepidoptera: Noctuidae). Crop Prot. 177: 106533. doi: 10.1016/j.cropro.2023.106533.

Moustafa, M.A.M., H. EL-Gamal, N.A. Alfuhaid, A. Fónagy and E.S. Ibrahim. 2024b. Efficacy of the traditional and nano-forms of thiocyclam and chlorantraniliprole against *Spodoptera littoralis* and *Agrotis ipsilon* and analysis of their residues in tomato fruits. Int. J. Trop. Insect Sci. 44: 657–667. doi: 10.1007/s42690-024-01169-w.

Moustafa, M.A.M., D.E. El Hefny, N.A. Alfuhaid, R.M.A. Helmy, N.A. El-Said and E.S. Ibrahim. 2024c. Effectiveness and biochemical impact of flubendiamide and flonicamid insecticides against *Bemisia tabaci* (Hemiptera: Aleyrodidae) and residue dissipation in tomato plants and soil under greenhouse conditions. J. Entomol. Sci. 59: 289–310. doi: 10.18474/JES23-61.

Nboyine, J.A., E. Asamani, L.K. Agboyi, I. Yahaya, F. Kusi, G. Adazebra and B.K. Badii. 2022. Assessment of the optimal frequency of insecticide sprays required to manage fall armyworm (*Spodoptera frugiperda* JE Smith) in maize (*Zea mays* L.) in northern Ghana. CABI Agric. Biosci. 3: 3. doi: 10.1186/s43170-021-00070-7.

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Omotoso, S.E., B.A. Akinpelu and O.J. Soyelu. 2020. Insecticidal effect of lemongrass oil on behavioral responses and biochemical changes in cowpea weevil, *Callosobruchus maculatus* (Fabricius). J. Phytopathol. Pest Manag. 7: 14–30. https://www.ppmj.net/index.php/ppmj/article/view/189.

Paramasivam, M. and H. Banerjee. 2012. Persistence and dissipation of the insecticide flubendiamide and its metabolite desiodo flubendiamide residues in tomato fruit and soil. Bull. Environ. Contamin. Toxicol. 88: 344–348. doi: 10.1007/s00128-011-0461-2.

Prasanna, B.M., J.E. Huesing, R. Eddy and V.M. Peschke. 2018. Fall armyworm in Africa: a guide for integrated pest management. 109 pages: México CIMMYT USAID.

Radunz, A.L., M. Radunz, A.R. Bizollo, M.A. Tramontin, L.L. Radunz, M.P. Mariot, E.R. Tempel-Stumpf, J.F.F. Calisto, F. Zaniol, D. Albeny-Simoes, R.S. Rezende and J. Dal Magro. 2024. Insecticidal and repellent activity of native and exotic lemongrass on maize weevil. Brazilian J. Biol. 84: e252990. doi: 10.1590/1519-6984.252990.

Rahayu, R., J.R. Mairawita and R. Jannatan. 2018. Efficacy and residual activity of lemongrass essential oil (*Cymbopogon flexuosus*) against German cockroaches (*Blattella germanica*). J. Entomol. 15: 149–154. doi: 10.3923/je.2018.149.154.

Ratnakala, B., C.M. Kalleshwaraswamy, M. Rajkumar, S.S. Deshmukh, H.B. Mallikarjuna and L. Narasimhaiah. 2023. Field evaluation of whorl application of sand mixed entomopathogenic nematodes for the management of invasive fall armyworm, *Spodoptera frugiperda* (JE Smith) (Lepidoptera: Noctuidae) in sweet corn. Egyptian J. Biol. Pes.t Contr. 33: 58. doi: 10.1186/s41938-023-00706-y.

Razaq, M., A. Suhail, M.J. Arif, M. Aslam and A.H. Sayyed. 2007. Effect of rotational use of insecticides on pyrethroids resistance in *Helicoverpa armigera* (Lep.: Noctuidae). J. Appl. Entomol. 131: 460–465. doi: 10.1111/j.1439-0418.2007.01204.x.

Sanches, L.J., P.C. Marinello, C. Panis, T.R. Fagundes, J.A. Morgado-Díaz, J.C.M. de-Freitas-Junior and R.C. Luiz. 2017. Cytotoxicity of citral against melanoma cells: The involvement of oxidative stress generation and cell growth protein reduction. Tumor Biol. 39: 1010428317695914. doi: 10.1177/1010428317695914.

Sante. 2019. Main changes introduced in Document N° SANTE/11312/2021 with respect to the previous version (Document N° SANTE 12682/2019).

Sharma, K.K., I. Mukherjee, B. Singh, S.K. Sahoo, N.S. Parihar, B.N. Sharma, V.D. Kale, R.V. Nakat, A.R. Walunj, S. Mohapatra, A.K. Ahuja, D. Sharma, G. Singh, R. Noniwal and S. Devi. 2014. Residual behavior and risk assessment of flubendiamide on tomato at different agro-climatic conditions in India. Environ. Monit. Assess. 186: 7673–7682. doi: 10.1007/s10661-014-3958-4.

Singh, S., L.K. Saini, V.H. Solanki, R.V. Kansara, K.D. Gandhi and N. Patel. 2023. Dissipation kinetics and health risk assessment of certain insecticides applied in/on tomato under open field and poly-house conditions. Heliyon. 9: e14963. doi: 10.1007/s00003-021-01330-4.

Smith, G.H., J.M. Roberts and T.W. Pop. 2018. Terpene based biopesticides as potential alternatives to synthetic insecticides for control of aphid pests on protected ornamentals. Crop Prot. 110: 125–130. doi: 10.1016/j.cropro.2018.04.011.

Solomon, B., T. Gebre-Mariam and K. Asres. 2012. Mosquito repellent actions of the essential oils of *Cymbopogon citratus*, *Cymbopogon nardus,* and *Eucalyptus citriodora*: evaluation and formulation studies. J. Essent. Oil-Bear. Plants 15: 766–773. doi: 10.1080/0972060X.2012.10644118.

Subirats, X., S. Reinstadler, S. Porras, M. Raggi and E. Kenndler. 2005. Comparison of methanol and acetonitrile as solvents for the separation of sertindole and its major metabolites by capillary zone electrophoresis. Electrophor. 26: 3315–3324. doi: 10.1002/elps.200500056.

Sunari, A.S., I.K. Sumiartha, I.W. Supartha, I.F. Mahaputra, I.K.W. Yudha, I.W.E.K. Utama, P.A. Wiradana. 2022. Potential yield reduction of sweet and glutinous corn varieties damaged by the invasive pest *Spodoptera frugiperda* (JE Smith) (Lepidoptera:

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10

Noctuidae): Field trial scale. In 2nd Int. Conf. Educ. Technol. (ICETECH 2021), Atlantis Press. 14–19. doi: 10.2991/assehr.k.220103.003.

Tisserand, R. and R. Young. 2013. Essential oil safety: a guide for health care professionals. Elsevier Health Sciences. New York, NY.

Tohnishi, M., T. Nishimatsu, K. Motoba, T. Hirooka and A. Seo. 2010. Development of a novel insecticide, flubendiamide. J. Pest Sci. 35: 490–491. doi: 10.1584/jpestics.35.508.

Uesugi, R., A. Jouraku, N. Sukonthabhirom, S. Pattalung, N. Hinomoto, S. Kuwazaki, H. Kanamori and S. Sonoda. 2021. Origin, selection and spread of diamide insecticide resistance allele in field populations of diamondback moth in east and southeast Asia. Pest Manag. Sci. 77: 313–324. doi: 10.1002/ps.6020.

Van Asperen, K.A. 1962. Study of housefly esterases by means of a sensitive colorimetric method. J. Insect Physiol. 8: 401–416. doi: 10.1016/0022-1910(62)90074-4.

Wang, S., F. Jin, X. Cao, Y. Shao, J. Wang, Y. She, Y. Qi, C. Zhang, H. Li, M. Jin, J. Wang, H. Shao and L. Zheng. 2018. Residue behaviors and risk assessment of flonicamid and its metabolites in the cabbage field ecosystem. Ecotoxicol. Environ. Saf. 161: 420–429. doi: 10.1016/j.ecoenv.2018.05.074.

Xing, J., B. Zhu, J. Yuan, J. Yu, D. Dong, D. Zhou, H.U. Dongsong and C.H.E.N. Jie. 2013. Bioactivity and field efficacy of novel insecticide ZJ4042 against different lepidopterous pests. Chinese J. Pest Sci. 15: 159–164.

Xu, F., G.M. Du, D. Xu, L.Y. Chen, X.X. Zha and Z.Y. Guo. 2021. Residual behavior and dietary intake risk assessment of flonicamid, dinotefuran and its metabolites on peach trees. J. Sci. Food. Agric. 101: 5842–5850. doi: 10.1002/jsfa.11236.

Yang, S., W.J. Yan, Y.T. Tan, Y.Q. Wang, Q. Zheng, Z.X. Zhang and H.H. Yu. 2020. Evaluation of control effect of tetrachlorantraniliprole corn whole plant spray and flam opening application treatment on *Spodoptera frugiperda*. J. Environ. Entomol. 42: 76–81.

You, C.X., J. Liu, X. Li, W.J. Zhang, X.X. Yu, Q. He, N. Liu, Y.Y. Pan, K.D. Dai and C. Jiang. 2023. Cocktail effect and synergistic mechanism of two components of *Perilla frutescens* essential oil, perillaldehyde and carvone, against *Tribolium castaneum*. Ind. Crops Prod. 195: 116433. doi: 10.1016/j.indcrop.2023.116433.

Zhang, R.M., J.F. Dong, J.H. Chen, Q.E. Ji and J.J. Cui. 2013. The sublethal effects of chlorantraniliprole on *Helicoverpa armigera* (Lepidoptera: Noctuidae). J. Integr. Agric. 12: 457–466. doi: 10.1016/S2095-3119(13)60246-4.

Zhang, T., Y. Xu, X. Zhou, X. Liang, Y. Bai, F. Sun, W. Zhang, N. Wang, X. Pang and Y. Li. 2022. Dissipation kinetics and safety evaluation of flonicamid in four various types of crops. Molecules 27: 8615. doi: 10.3390/molecules27238615.

Zhang, X., F. Zhao, T. Bai, X. Mao and A. Zhang. 2020. Several insecticides to *Spodoptera exigua*: indoor toxicity and field efficacy. Chinese Agric. Sci. Bull. 36: 127–131.

Zhou, C., H. Wang, X. Li, W. Wang and W. Mu. 2011. Comparison of the toxicity of chlorantraniliprole and flubendiamide to different developmental stages of *Spodoptera exigua*. Acta Phytophy. Sin. 38: 344–350. doi: 10.1093/toxsci/kfy256.

Zibaee, I. 2015. Synergistic effect of some essential oils on toxicity and knockdown effects, against mosquitos, cockroaches, and houseflies. Arthropods 4: 107.

http://prime-pdf-watermark.prime-prod.pubfactory.com/ | 2025-02-10