Effectiveness and Biochemical Impact of Flubendiamide and Flonicamid Insecticides against *Bemisia tabaci* (Hemiptera: Aleyrodidae) and Residue Dissipation in Cherry Tomato Plants and Soil under Greenhouse Conditions[1]

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Abstract We investigated the effectiveness and biochemical impact of the insecticides flubendiamide and flonicamid compared with azadirachtin and acetamiprid against the whitefly *Bemisia tabaci* (Grennadius) (Hemiptera: Aleyrodidae) infesting cherry tomato (*Solanum lycopersicum* var. *cerasiforme* L.) grown under greenhouse conditions. The dissipation of both insecticides in the plants and in the soil also was determined using the QuEChERS (quick, easy, cheap, effective, rugged, and safe) and liquid chromatography–electrospray ionization–mass spectrometry methods. Both insecticides were more effective for reducing *B. tabaci* populations than were either acetamiprid or azadirachtin. Biochemical analysis revealed that esterase may play an important role in whitefly adaptation to flubendiamide and flonicamid. The QuEChERS method was determined suitable for quickly detecting residues of flubendiamide and flonicamid in complex matrices. The recovery rates on tomato fruit samples were 92.8–106.0%, with a relative standard deviation (RSD) range of 0.46–2.65%. For soil samples, the recovery rates were 81.3–95.7% with RSDs of 1.20–3.86%. We further determined that flubendiamide had dissipation half-lives of 3.13, 3.63, and 3.68 d in tomato fruit, tomato leaves, and soil, respectively. Flonicamid had half-lives of 4.25, 3.54, and 2.60 d in fruit, leaves, and soil, respectively. These results suggest that preharvest intervals of 3 and 7 d are appropriate for flubendiamide and flonicamid, respectively, in cherry tomato production. The risk quotient was >1 by the day 5 after application; however, that value declined to <1 on day 7 after application, indicating little long-term risk to human health.

Key Words flubendiamide, flonicamid, *Bemisia tabaci*, enzymes activity, dissipation

Cherry tomato (*Solanum lycopersicum* var. *cerasiforme* L.) production in greenhouses is compromised by the whitefly *Bemisia tabaci* Gennadius (Hemiptera: Aleyrodidae), causing significant yield losses sometimes as high as 100%

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(McKenzie et al. 2014, Mugerwa et al. 2021, Ochilo et al. 2019). Moodley et al. (2019) estimated the losses as exceeding US$100 million each year. Therefore, conventional chemical insecticides are routinely used as a preferred tactic for preventing and managing whitefly attacks on tomatoes, thus ensuring tomato quality and yield (Ghosal and Chatterjee 2018). The performance of novel ecofriendly insecticides against *B. tabaci* and other whitefly pests is continually being evaluated (Kumar et al. 2019).

Synthetic amide insecticides are a class of novel insecticides that have excellent effectiveness against target insects and low toxicity for nontarget species (Lin et al. 2021). Flubendiamide is a diamide insecticide that was developed by Bayer Crop Science (Leverkusen, Germany) and has been used recently (Casida 2015, Sparks and Nauen 2015) against many lepidopteran, dipteran, and coleopteran insects (Kadala et al. 2020, Li et al. 2019). It has a novel mode of action, acting as an activator of the insect ryanodine receptor and causing massive release of calcium ions from muscle cells (Cordova et al. 2006, Uesugi et al. 2020). However, because of its environmental behavior and ecological toxicity, flubendiamide may be a danger to invertebrates and poses a significant risk to aquatic habitats (Lin et al. 2021).

Flonicamid (*N*-(cyanomethyl)-4-(trifluoromethyl)-3-pyridinecarbox-amide) is a new selective systemic insecticide with extremely potent insecticidal efficacy against whiteflies and other piercing-sucking insects (Morita et al. 2007). It works by obstructing type-A potassium channels, thus preventing insects from moving to and attacking tomato plants (Morita et al. 2000).

Because pesticide residues may adversely affect the quality and growth of tomato plants, the QuEChERS (quick, easy, cheap, effective, rugged, and safe) method is currently used for the rapid identification of pesticides in agricultural goods (Anastassiades et al. 2003). Standard QuEChERS methods also have been used for clean-up procedures with primary secondary amine (PSA), clean-up C18 bulk sorbent, and graphitized carbon black (GCB) as adsorbents (Anastassiades et al. 2003, El-Hefny et al. 2021). Those chemicals effectively absorb pigments and minimize matrix effects. The residual behavior, dissipation behavior, and dietary risk of tested pesticides in various crops also must be assessed to ensure safety and protect the environment (Zhang et al. 2022) by estimating the risk quotient, taking into consideration dissipation as an indicator for human health safety (El Hefny et al. 2021, Moustafa et al. 2023a).

In this study, the biological effectiveness and biochemical impact of flubendiamide and flonicamid insecticides, as alternatives to azadirachtin and acetamiprid, for managing whiteflies were assessed in developing a pest management spray schedule for cherry tomato crops. The dissipation of both insecticides also was determined in cherry tomato fruits and leaves and in soil using the QuEChERS and liquid chromatography–electrospray ionization tandem mass spectrometry (LC-ESI MS/MS) methods.

Materials and Methods

Insecticides and chemicals. Four commercial insecticide formulations (azadirachtin; Gaara Co., Cairo, Egypt; acetamiprid, Shoura Chemicals Co., Giza, Egypt; flubendiamide, Sama-Trade Co., Giza, Egypt; and flonicamid, Shoura Chemicals Co., Giza, Egypt) were used in this study (Table 1). The flubendiamide

Table 1. Tested insecticides and their application rates.

Common Name	Trade Name (% active ingredient)	Chemical Group	Application Rate (/100 L of water)
Azadirachtin	Ashouk (0.15% EC)	Biopesticide	350 ml
Acetamiprid	Mospilan (20% SP)	Neonicotinoid	25 g
Flubendiamide	Takumi (20% WG)	Diamide	50 g
Flonicamid	Tebekki (50% WG)	Pyridine carboxamide	50 g

and flonicamid reference materials (98%) were purchased from Dr. Ehrenstorfer GmbH (Augsburg, Germany). Extraction and clean-up chemicals were obtained from Merck Company (Darmstadt, Germany). Anhydrous magnesium sulfate ($MgSO_4$) fine powder and sodium chloride (NaCl) that had been dried in an oven at 250°C for 4 h were stored in desiccators prior to use. The PSA sorbent (Bondesil-PSA, 40 M) was obtained from Supelco Analytical Products (Sigma-Aldrich Company, Darmstadt, Germany). Stock solutions (1000 mg/L) of flubendiamide and flonicamid were prepared with acetonitrile. To prepare the sample matrices for high-performance liquid chromatography (HPLC) detection, calibrated matrix and solvent solutions at concentrations of 10, 5, 1, 0.5, 0.1, 0.05, and 0.01 mg/L were applied to the sample matrices. The resulting mixes were then passed through a 0.22-μm-pore size filter.

Greenhouse experiment. To evaluate the effectiveness of the tested insecticides against *B. tabaci* nymphs, greenhouse experiments were conducted at the Faculty of Agriculture (Giza, Egypt) over two consecutive seasons (2022 and 2023). Cherry tomato (cv. 'Katalina 522') seedlings with four or five true leaves were transplanted in a randomized complete block design with four replicates. The greenhouse (10 × 60 m) was divided into five beds. Each bed was 1.2 m wide. The in-row distance between plants was 50 cm. Each experimental unit consisted of 10 plants. This area was managed with recommended agricultural practices for the entire season. The insecticides were applied with a Cooper Pegler CP_3 knapsack sprayer with irrigation water used for dilutions (Moustafa et al. 2022). The chemical application began when the whitefly infestation reached four nymphs per leaf. After insecticide application, randomly selected tomato leaves were collected at 1, 3, 5, 7, 10, 15, and 21 d after application. One hundred leaves (25 leaves/replicate) were collected from each treatment for analysis (Moustafa et al. 2022, Kandil et al. 2023). The percentage of infestation reduction was calculated based on *B. tabaci* nymph infestation before and after application of each insecticide according to Henderson and Tilton (1955).

Enzyme sample preparation. Adults of *B. tabaci* were collected at 1, 7, 15, and 21 d after flubendiamide and flonicamid application at their recommended concentrations. Ten milligrams of adults from each treatment and control group were weighed and homogenized in phosphate buffer (0.1 M, pH 7). Three replicates were made for each treatment. The homogenates were centrifuged at 12,000 × *g* for 15 min, and the supernatant was stored at −20°C.

Total esterase activity. Total esterase activity was analyzed with α-naphthyl acetate (α-NA) as a substrate, according to Van Asperen (1962) with some modifications (Moustafa et al. 2023b, 2023c). Thirty microliters of supernatant was mixed with 30 mM α-NA for 15 min, after which Fast Blue B solution was added to stop the reaction. The absorbance was read at 600 nm, and the activity was assessed by comparison with the α-naphthol standard curve.

Glutathione *S*-transferase (GST) activity. GST activity was measured according to Habing et al. (1974) and Moustafa et al. (2023b, 2023c). Ten microliters of enzyme solution was added to 25 µl of a solution of 1-chloro-2,4-dinitrobenzene (30 mM) and 25 µl of glutathione (50 mM dissolved in pH 6.5). The enzyme kinetics readings were recorded at 340 nm for 5 min.

Cytochrome P-450 activity. Cytochrome P-450 (a mixed function oxidase) activity was determined as described by Hansen and Hodgson (1971) and Moustafa et al. (2023b, 2023c) using *p*-nitrophenol to generate the standard curve. The substrate of *p*-nitroanisole (2 mM) was mixed with 90 µl of enzyme solution for 2 min. Nicotinamide adenine dinucleotide phosphate (9.6 mM) was then added to the mixture. The absorbance was read at 405 nm for 15 min.

Statistical analysis of efficacy and enzyme activity. The effectiveness and biochemical activity of the tested insecticides were coded and entered using the statistical package SPSS (Statistical Package for the Social Sciences, v. 22, IBM, Armonk, NY). The results were tested for satisfying assumptions of parametric tests. The continuous variables were subjected to Shapiro-Wilk and Kolmogorov-Smirnov test for normality. The reduction percentage data were standardized for normality using the arcsine square root. Data are presented as mean ± standard deviation. Analyses of variance were conducted for effectiveness and enzyme activity using MiniTab software (v. 14.0). The post hoc analysis was performed with a Tukey pairwise honestly significant difference (HSD) test, and results were considered significant at $P < 0.05$. The data were visualized, when possible, using R studio (v. 2022.02.4.).

Residue analysis sample processing. Purification and extraction were performed according to the methods of Anastassiadis et al. (2003) and Moustafa et al. (2023a). Cherry tomato fruit and leaf samples were collected randomly 2 h after spraying (0 d) and 1, 3, 5, 7, 10, 15, and 21 d after treatment. A homogenized tomato sample (10 g) was weighed into a 50-ml Teflon centrifuge tube to which 10 ml of acetonitrile was added. Each tube and its contents were shaken for 2 min on a Vortex mixer to ensure that the solvent interacted with the entire sample. One gram of NaCl and 4 g of $MgSO_4$ were then added to the solution and shaken for 1 min. A 10-ml Teflon centrifuge tube containing 50 mg of PSA and 300 mg of $MgSO_4$ for fruits and 50 mg of GCB for leaves was filled with 2 ml of the cleared supernatant following centrifugation at 5,000 rpm for 5 min. The mixture was then shaken for 1 min and centrifuged in a microcentrifuge for 5 min at 5,000 rpm.

Soil (5 g) was placed in a 50-ml Teflon centrifuge tube. To ensure that the solvent interacted with the entire sample, 10 ml of acetonitrile and 5 ml of water were added. The tube was then shaken for 2 min on a Vortex mixer. Following the addition of 1 g of NaCl and 4 g of $MgSO_4$, the tube and its contents were again shaken for 1 min. A 10-ml Teflon centrifuge tube with 50 mg of PSA and 300 mg of $MgSO_4$ was filled with 2 ml of the cleared supernatant following centrifugation at 5,000

rpm for 5 min. After shaking for 1 min, the mixture was centrifuged with a microcentrifuge for 5 min at 5,000 rpm. The acetonitrile layer was passed through a 0.22-μm-pore-size filter for processing by HPLC.

An autosampler with an electric sample valve, a variable wavelength diode array detector, and a quaternary pump were part of the HPLC system (Agilent HPLC 1260 infinite series; Agilent Technologies, Santa Clara, CA). A 150 mm × 4.6 mm × 5 m octadecyl-silica analytical column was used in the HPLC system. The injection volume was 20 μl, the detection wavelengths were 210 and 220 nm, and the flow rates of the mobile phases (acetonitrile 90% + water 10% and acetonitrile 65% + water 35%) were 0.8 and 1 ml/min for flubendiamide and flonicamid, respectively. The retention times for flubendiamide and flonicamid were 4.6 and 4.7 min, respectively.

Statistical analysis of residues. The first-order kinetic model was used to describe how flonicamid and flubendiamide residues dissipated in tomato tissues and soil, as calculated with the formula of Hoskins et al. (1961): $C_t = C_0 e^{-kt}$, where C_0 is the initial concentration (mg/kg), C_t is the residue concentration (mg/kg) at time t (day) after the pesticide treatment, and k is the degradation rate constant (per day). The half-life ($t_{1/2}$) used to assess the dissipation rate was determined using the formula of Saber et al. (2016), $t_{1/2} = \ln 2/k$.

Dietary exposure risk assessment. Formulae used to calculate the risk quotient (RQ) for the long-term intake risk and the national estimated daily intake (NEDI, mg/kg/day) were those of Wang et al. (2016) and Qian et al. (2017), where NEDI = STMRi × Fi/bw, and RQ = NEDI/ADI. In these equations, STMRi is the median residue data from controlled trials (in our case, we used the mean because we had three replicates), Fi is food intake (kg/day), bw is body weight (kg), and ADI is the acceptable daily intake. In Egypt, the average body weight of an adult is 60 kg, which was used in our calculations. RQ values <1 are regarded by consumers as acceptable risks, whereas RQ values >1 are regarded as unacceptable risks (Oliva et al. 2017, Zhang et al. 2021).

Method validation. The guidelines for the validation of analytical methods and quality control procedures for pesticide residues in food and feed were published by the European Commission (2019). To determine the viability of the suggested procedure, linearity, matrix effect (ME), accuracy, precision, and limit of quantification (LOQ) were all evaluated. Three concentrations of flubendiamide and flonicamid standard solutions were applied to blank samples of the tomato fruits and soil to assess the method accuracy and precision. Each treatment was replicated five times. For additional concentrations of 0.1, 0.5, and 1 mg/kg, the recovery rate and relative standard deviation (RSD) were calculated to determine the most effective combination of purifying agents. The limit of detection and the LOQ were used to test the sensitivity of the method. The lowest spiked concentration quantification was used to define the LOQ of the suggested approach.

Because coextracts produced during pretreatment affect the accuracy of the results, the ME was calculated according to the formula of Hoff et al. (2015): ME% = [(k matrix – k solvent)/k solvent] × 100, where k solvent is the slope of the solvent calibration curve and k matrix is the slope of the matrix calibration curve. An ME value of >10% indicates a definite matrix strengthening or weakening effect.

Results

Effectiveness test. After the application of all four the tested insecticides, *B. tabaci* nymph infestation decreased significantly during both the 2022 season ($F = 43$; df $= 28$; $P = 0.0001$) and the 2023 season ($F = 48.57$; df $= 28$; $P = 0.0001$) (Fig. 1). The application of flubendiamide and flonicamid resulted in lower infestation than did application of acetamiprid and azadirachtin in both seasons (Tables 2, 3). One day after the application of azadirachtin, acetamiprid, flubendiamide, and flonicamid during the 2022 season, the reductions in *B. tabaci* nymph infestation were 18.3, 67.0, 81.5, and 86.9%, respectively, whereas 3 d after application reductions were 35.98, 72.5, 86.47, and 94.49%, respectively (Table 4; Fig. 2). Residual effects were noted 5–21 d after application of the four insecticides, with mean reductions of 90.95, 85.82, 60.11, and 43.91%, respectively. A similar trend was observed in 2023 when flubendiamide and flonicamid were more effective than acetamiprid and azadirachtin (Table 5; Fig. 2). The reductions in *B. tabaci* infestation 1 d after of application of flubendiamide and flonicamid were 78.61 and 84.14%, respectively, and reached 89.28 and 92.71%, respectively, 3 d after application. Residual effects of flubendiamide and flonicamid resulted in mean reductions of 87.75 and 90.37%, respectively.

Effect of flubendiamide and flonicamid on detoxifying enzymes. The activities of α-esterase, cytochrome P-450, and GST were determined in *B. tabaci* adults at 1, 7, 15, and 21 d following field application of flubendiamide and flonicamid. Flonicamid increased the α-esterase activity of *B. tabaci* at 7, 15, and 21 d after application by 1.36, 1.97 and 1.76 times, respectively (Table 6; Fig. 3), whereas flubendiamide increased α-esterase activity only 7 and 21 d after application by 1.56 and 1.68 times, respectively. In contrast, both insecticides did not significantly reduce the cytochrome P-450 activity (0.0154 ± 0.0019 and 0.0153 ± 0.004, respectively) at 21 d after application compared with the control (0.0184 ± 0.0016). In addition, flonicamid and flubendiamide did not significantly decrease the activity of GST at 7 d (8.7 ± 1.28 and 10.11 ± 4.61, respectively) and 21 d (6.58 ± 0.29 and 9.1 ± 2.46, respectively) after application compared with the control (Table 6; Fig. 3).

Method validation. ME, linearity, LOQ, accuracy, and precision were among the validation parameters assessed for the analytical method. The correlation coefficient (R^2) of the flubendiamide or flonicamid calibration curve with the matrix calibration curve was used to measure the linearity. The linear range complies with the requirement for R^2 (0.99). For tomato fruits, the matrix standard curve and the standard curves of the flubendiamide and flonicamid solutions yielded R^2 values of >0.98, indicating a strong linear relationship. According to Matuszewski et al. (2003), ME describes how matrices other than the tested compound might affect the target's response value. In cherry tomato fruits, the MEs of flonicamid and flubendiamide were ≥ 0.98. No measurable ME was present. As a result, the effect of other matrices on the target compound's response value was minimal. Flubendiamide and flonicamid in cherry tomatoes could be quantified only to a maximum of 0.01 mg/kg.

The recovery test and RSD were used to confirm the procedure's accuracy and precision. The recovery rates of flubendiamide and flonicamid from tomato obtained utilizing the ideal purifier combination are shown in Table 7. The recovery

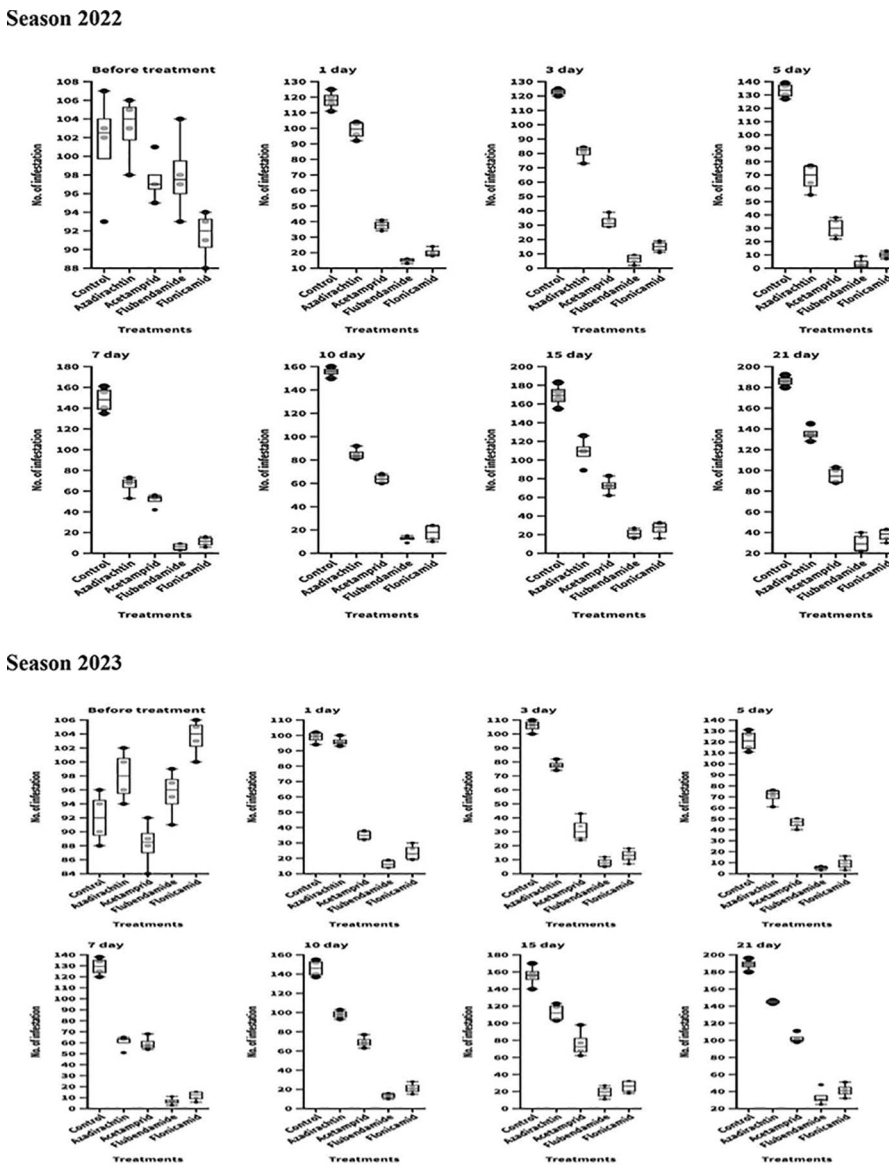

Fig. 1. Mean (±SD) number of *B. tabaci* nymphs on cherry tomato plants after field application of four insecticides during the 2022 and 2023 seasons.

rates and the matching RSD of flubendiamide and flonicamid are shown for the three spiking levels in fruits and soil. Thus, by using the suggested procedure, high recoveries of flubendiamide and flonicamid at the three spiking levels were obtained within the acceptance criterion of 80–110%, with a precision RSD of ≥20% (European Commission 2019).

Table 2. Mean (±SD) number of *B. tabaci* nymphs on cherry tomato plants before and after field application of four insecticides in the 2022 growing season.*

Treatment	Before Application	After Application						
		Day 1	Day 3	Day 5	Day 7	Day 10	Day 15	Day 21
Control	101.3 ± 5.11a	118 ± 5.14a	122.8 ± 1.92a	133.3 ± 4.91a	148 ± 10.79a	155.5 ± 3.64a	169 ± 10.29a	186 ± 4.47a
Azadirachtin	103 ± 3.08a	98.8 ± 4.96b	80.5 ± 4.5b	68 ± 9.08b	65.8 ± 7.66b	85 ± 4.3b	108.5 ± 13.12b	135.5 ± 6.1b
Acetamiprid	97.5 ± 2.17ab	37.5 ± 2.69c	32.5 ± 4.09c	30 ± 6.67c	51.3 ± 5.44b	63.8 ± 3.34c	72.5 ± 7.43c	95 ± 6.59c
Flubendiamide	98 ± 3.93ab	15 ± 1.22d	6.25 ± 2.94e	3.8 ± 3.26d	6.3 ± 2.77c	12.5 ± 2.17d	21.3 ± 4.43d	30 ± 7.71d
Flonicamid	91.5 ± 2.29b	20 ± 2.44d	15 ± 3.16d	10 ± 2.23d	11.3 ± 3.96c	17.5 ± 6.1d	26.3 ± 6.6d	37.5 ± 5.31d

* Within a column, means with different letters are significantly different (Tukey's HSD, $P = 0.05$).

Table 3. Mean (±SD) number of *B. tabaci* nymphs on cherry tomato plants before and after field application of four insecticides in 2023 growing season.*

Treatment	Before Application	After Application						
		Day 1	Day 3	Day 5	Day 7	Day 10	Day 15	Day 21
Control	92 ± 3.16bc	98.8 ± 3.11a	105.8 ± 3.76a	121 ± 8.24a	129.3 ± 7.11a	146 ± 7.64a	155.5 ± 10.64a	188.5 ± 5.72a
Azadirachtin	98 ± 3.16ab	96 ± 2.54a	77.8 ± 2.86b	70.8 ± 5.93b	60.5 ± 5.54b	98 ± 3.8b	112.5 ± 8.64b	145 ± 1.58b
Acetamiprid	88.3 ± 2.86c	35 ± 2.54b	31.8 ± 7.49c	46.3 ± 4.14c	59.3 ± 5.35b	69.3 ± 5.11c	76.3 ± 13.64c	102.5 ± 5.02c
Flubendiamide	95.5 ± 2.95bc	16.3 ± 2.27c	8 ± 2.73d	5.3 ± 1.47d	6.8 ± 2.86c	13.3 ± 2.38d	19.3 ± 6.17d	33.8 ± 8.58d
Flonicamid	103.5 ± 2.29a	23.8 ± 4.49c	12.8 ± 4.14d	9.3 ± 4.81d	11.5 ± 3.77c	21.3 ± 4.81d	25.3 ± 6.33d	41.3 ± 6.86d

* Within a column, means with different letters are significantly different (Tukey's HSD, $P = 0.05$).

Table 4. Mean (±SD) percent reduction in *B. tabaci* nymph infestation on cherry tomato plants after field application of four insecticides in the 2022 growing season.*

Insecticide	Day 1	Day 3	Day 5	Day 7	Day 10	Day 15	Day 21	Average Residual Effect**
Azadirachtin	18.3 ± 1.41a	36 ± 2.2a	50.2 ± 3.62c	56.7 ± 2.89b	46.7 ± 5.68c	37.1 ± 5.19c	29 ± 3.09c	43.9 ± 10.67
Acetamiprid	67 ± 2.12b	72.5 ± 3.2b	76.6 ± 3.44b	64.1 ± 2.98b	57.4 ± 3.72b	55.5 ± 3.73b	47± 4.31b	60.1 ± 10.55
Flubendiamide	81.5 ± 2.66c	86.5 ± 2.33c	91.7 ± 2.4a	89.9 ± 4.74a	87.3 ± 4.26a	82.6 ± 3.89a	77.7 ± 2.65a	85.8 ± 6.3
Flonicamid	86.9 ± 1.58d	94.5 ± 2.63d	97.1 ± 2.3a	95.6 ± 1.89a	91.7 ± 3.04a	87 ± 2.55a	83.4 ± 3.82a	90.9 ± 5.88

* Within a column, means with different letters are significantly different (Tukey's HSD, $P = 0.05$).

** Average residual effect was calculated at 5–21 d after application.

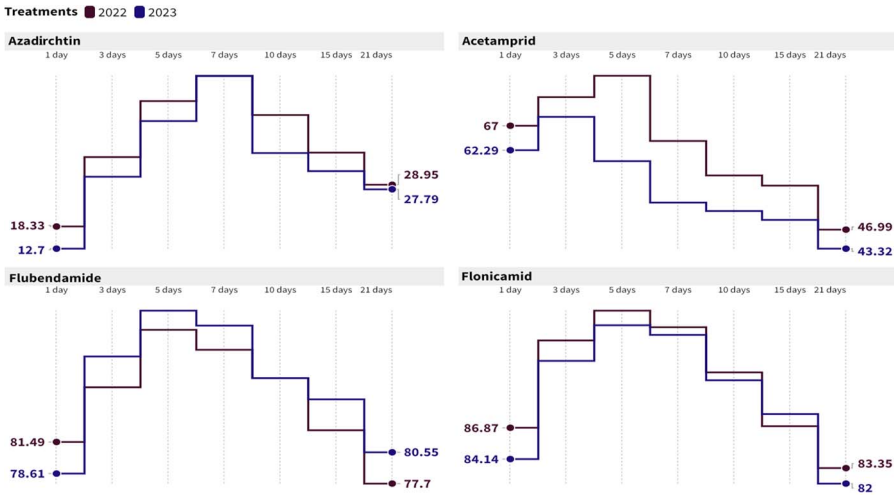

Fig. 2. Step slope chart representing the mean (±SD) percent reductions in *B. tabaci* nymphs after field application of four insecticides during the 2022 and 2023 seasons.

Dissipation and terminal residue of flubendiamide and flonicamid in fruits, leaves, and soil. The dissipation of flubendiamide and flonicamid in cherry tomato fruits and leaves and in soil was determined. The dissipation kinetics and half-life are displayed in Tables 8 and 9. In fruits, leaves, and soil, flubendiamide and flonicamid had half-lives of 4.25–2.60 d. The final residual amounts of flubendiamide and flonicamid in fruits, leaves, and soil are displayed in Tables 8 and 9. Flubendiamide had an initial residue of 3.80, 6.33, and 1.30 mg/kg in fruits, leaves, and soil, respectively (Table 8). After 1 d, the residue concentration dropped to 2.95, 5.84, and 0.99 mg/kg, respectively, with losses of 22.36, 7.74, and 23.84%, respectively. At 15 d following treatment, the concentrations in fruits, leaves, and soil decreased to 0.05, 0.1, and 0.07 mg/kg, respectively, with losses of 98.68, 98.42, and 94.61%, respectively. At 21 d after treatment, the residues in fruits and soil were below the detection limits. Thus, if the maximum residue level (MRL) for tomato were 2 mg/kg, the recommended preharvest interval (PHI) would be 3 d if the specified dosage and safety interval recommendations were followed. The residues of flonicamid in fruits, leaves, and soil were 4.08, 7.51, and 3.72 mg/kg, respectively, 2 h after treatment (Table 9). At 1 d, the residues dropped to 3.36, 5.57, and 2.17, respectively. At 3 d after application, rapid dissipation was noted in fruits, leaves, and soil, with flonicamid residues declining to 2.64, 4.33, and 1.58 mg/kg, respectively, and losses of 35.29, 42.34, and 57.52%, respectively. After 15 d, the residues fell to 0.05 and 0.16 mg/kg and not detectable, with 98.77, 97.86, and 100% loss, respectively. Flonicamid residues were no longer identifiable at 21 d following application. Accordingly, the PHI recommendation is 7 d when using the specified dosage and based on an MRL of 0.5 mg/kg for cherry tomato (Table 9).

Table 5. Mean (±SD) percent reduction in *B. tabaci* nymph infestation on cherry tomato plants after field application of four insecticides in the 2022 growing season.*

Insecticide	Day 1	Day 3	Day 5	Day 7	Day 10	Day 15	Day 21	Average Residual Effect**
Azadirachtin	12.7 ± 2.11c	31 ± 1.87c	45.1 ± 4.58c	56.6 ± 5.07b	37 ± 3.16c	32.4 ± 3.18c	27.8 ± 1.9c	39.8 ± 10.83
Acetamiprid	62.3 ± 3.29b	68.7 ± 6.18b	60.2 ± 2.84b	52.2 ± 3.16b	50.6 ± 3.17b	48.9 ± 5.37b	43.3 ± 2.43b	51.02 ± 6.51
Flubendiamide	78.6 ± 4.04a	89.3 ± 3.23a	93.5 ± 3.67a	92.1 ± 3.05a	87.3 ± 2.79a	85.4 ± 3.37a	80.6 ± 3.14a	87.8 ± 5.67
Flonicamid	84.1 ± 3.02a	92.7 ± 2.64a	95.8 ± 1.79a	95 ± 2.25a	91 ± 2.24a	88.1 ± 4.24a	82 ± 4.3a	90.4 ± 5.94

* Within a column, means with different letters are significantly different (Tukey's HSD, $P = 0.05$).
** Average residual effect was calculated at 5–21 d after application.

Table 6. Mean (±SD) enzymatic activity of α-esterase, cytochrome P-450, and GST in *B. tabaci* adults at 1, 7, 15, and 21 d following field application of flubendiamide and flonicamid.*

Enzyme	Treatment	Enzyme Activity (Mmole/mg protein)			
		Day 1	Day 7	Day 15	Day 21
α-esterase	Control	23.94 ± 9.91a	18.21 ± 6.008a	11.35 ± 0.67a	7.32 ± 1.42a
	Flubendiamide	20.15 ± 8.05a	28.57 ± 19.1a	8.9 ± 0.9a	12.31 ± 4.94a
	Flonicamid	17.72 ± 5.11a	24.82 ± 8.22a	22.42 ± 8.04a	12.95 ± 2.92a
P-450	Control	0.019 ± 0.0054a	0.0202 ± 0.0032a	0.0137 ± 0.0027a	0.0184 ± 0.0016a
	Flubendiamide	0.0209 ± 0.0043a	0.0268 ± 0.0161a	0.0141 ± 0.0014a	0.0153 ± 0.004a
	Flonicamid	0.0202 ± 0.0007a	0.0182 ± 0.0027a	0.0194 ± 0.0067a	0.0154 ± 0.0019a
GST	Control	8.76 ± 1.54a	10.27 ± 1.6a	9.29 ± 0.73a	9.58 ± 0.59a
	Flubendiamide	9.67 ± 1.06a	10.11 ± 4.61a	6.72 ± 0.9a	9.1 ± 2.46a
	Flonicamid	10.08 ± 0.2a	8.7 ± 1.28a	10 ± 3.71a	6.58 ± 0.29a

* Within a column, means with different letters are significantly different (Tukey's HSD, $P = 0.05$).

Risk assessment. The RQ is of crucial significance to human health safety; it is calculated by dividing the NEDI by the ADI. An RQ value of <1 indicates low potential risk to consumers, and a value of >1 means high potential risk. For long-term consumption, flubendiamide and flonicamid had a high risk for the first 5 d of use on tomatoes treated with the recommended concentrations. According to our findings, the RQ was >1 for 0, 1, 3, and 5 d following application. However, for days 7, 10, and 15, the RQ values were <1, which indicates a low risk for human health (Table 10).

Discussion

Chemical control is considered one of the preferred methods for quick relief from insect pests. However, pesticide application can yield questionable outcomes

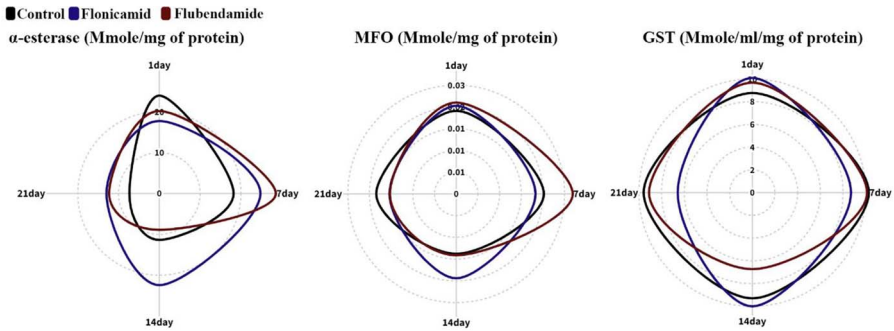

Fig. 3. Radar chart representing the activities of α-esterase, cytochrome P-450, and GST in *B. tabaci* adults at 1, 7, 15, and 21 d after field application of flubendiamide and flonicamid.

Table 7. Mean (±SD) percentage recovery of flubendiamide and flonicamid residues from cherry tomato fruits and soil.*

Spiked level (μg/g)	Flubendiamide				Flonicamid			
	Tomato fruits		Soil		Tomato fruits		Soil	
	Recovery ± RSD	RSD_r	Recovery ± RSD	RSD_r	Recovery ± RSD	RSD_r	Recovery ± RSD	RSD_r
0.1	92.76 ± 0.40	0.46	85.36 ± 3.3	3.86	95.97 ± 1.50	1.58	81.32 ± 2.10	2.52
0.5	95.10 ± 0.67	0.70	85.81 ± 1.03	1.20	97.29 ± 2.58	2.65	84.02 ± 1.42	1.68
1	98.24 ± 1.01	1.02	95.71 ± 1.53	1.60	106.0 ± 1.67	1.57	89.72 ± 2.65	2.95

* RSD = relative standard deviation; RSD_r = recovery repeatability (precision).

Table 8. Dissipation kinetics of flubendiamide residues (mean ± SD) in cherry tomato fruits and leaves and in soil under open field conditions.

	Tomato Fruits			Tomato Leaves			Soil		
Day After Application	Residue (mg/kg)	% Loss	% Persistence	Residue (mg/kg)	% Loss	% Persistence	Residue (mg/kg)	% Loss	% Persistence
0	3.80 ± 1.25	0.00	100.0	6.33 ± 1.05	0.00	100.0	1.30 ± 0.52	0.00	100.0
1	2.95 ± 1.04	22.36	77.64	5.84 ± 1.09	7.74	92.26	0.99 ± 0.63	23.84	76.16
3	1.82 ± 0.95	52.10	47.90	3.72 ± 0.92	41.23	58.77	0.77 ± 0.19	40.76	59.24
5	0.98 ± 2.01	74.21	25.79	2.90 ± 0.48	54.18	45.82	0.39 ± 0.47	70.00	30.00
7	0.45 ± 1.44	88.15	11.85	1.25 ± 0.94	80.25	19.75	0.24 ± 1.37	81.53	18.47
10	0.27 ± 0.98	92.89	7.11	0.57 ± 1.32	90.99	9.01	0.11 ± 1.26	91.53	8.47
15	0.05 ± 2.27	98.68	1.32	0.10 ± 1.29	98.42	1.58	0.07 ± 1.50	94.61	5.39
21	ND*			0.06 ± 2.00	99.05	0.95	ND		
$t_{½}$ (days)		3.13			3.63			3.68	
MRL** (mg/kg)		2							
PHI† (day)		3							

* ND = not detectable.
** MRL = maximum residue level.
† PHI = preharvest interval.

Table 9. Dissipation kinetics of flonicamid residues (mean ± SD) in cherry tomato fruits, leaves, and soil under open field conditions.

	Tomato fruits			Tomato leaves			Soil		
Day After Application	Residue (mg/kg)	% Loss	% Persistence	Residue (mg/kg)	% Loss	% Persistence	Residue (mg/kg)	% Loss	% Persistence
0	4.08 ± 0.98	0.00	100.0	7.51 ± 0.82	0.00	100.0	3.72 ± 052	0.00	100.0
1	3.36 ± 0.47	17.64	82.36	5.57 ± 0.48	25.83	74.17	2.17 ± 0.93	41.66	58.34
3	2.64 ± 1.20	35.29	64.71	4.33 ± 0.55	42.34	57.66	1.58 ± 0.22	57.52	42.48
5	1.09 ± 0.85	73.28	26.72	2.22 ± 0.21	70.43	29.57	0.84 ± 0.17	77.41	22.59
7	0.42 ± 0.56	89.7	10.30	1.31 ± 0.63	82.55	17.45	0.31 ± 0.82	91.66	8.34
10	0.13 ± 1.83	96.81	3.19	0.24 ± 0.85	96.80	3.20	0.11 ± 0.55	97.04	2.96
15	0.05 ± 1.22	98.77	1.23	0.16 ± 0.99	97.86	2.14	ND*		
21	ND			0.04 ± 1.36	99.46	0.54	ND		
$t_{½}$ (days)		4.25			3.54			2.60	
MRL** (mg/kg)		0.5							
PHI† (day)		7							

* ND = not detectable.
** MRL = maximum residue level.
† PHI = preharvest interval.

Table 10. National estimated daily intake (NEDI) and risk quotient (RQ) of flubendiamide and flonicamid in cherry tomato fruits.

Day After Application	Flubendiamide			Flonicamid		
	Residue (mg/kg)	NEDI	RQ	Residue (mg/kg)	NEDI	RQ
0	3.8	0.82	8.17	4.08	0.88	8.77
1	2.95	0.63	6.34	3.36	0.72	7.22
3	1.82	0.39	3.91	2.64	0.57	5.68
5	0.98	0.21	2.11	1.09	0.23	2.34
7	0.45	0.10	0.97	0.42	0.09	0.90
10	0.27	0.06	0.58	0.13	0.03	0.28
15	0.05	0.01	0.11	0.05	0.01	0.11
21	ND*	ND	ND	ND	ND	ND

* ND = not detectable.

due to several factors (Moustafa et al. 2018). Therefore, the residue dissipation pattern, half-life, and PHI must be evaluated for each pesticide. The purpose of the current study was to study the effectiveness of azadirachtin, acetamiprid, flubendiamide, and flonicamid against *B. tabaci* infestation and to determine the biochemical impact and persistence of flubendiamide and flonicamid residues in cherry tomato fruits and leaves and in soil samples.

Our results clearly indicate that the initial kill and residual effect on the whitefly (*B. tabaci*) nymphs in the 2022 season were the same as those obtained in 2023. Flonicamid and flubendiamide resulted in the highest reduction in *B. tabaci* infestation, followed by acetamiprid and azadirachtin. These results agree with those of Assadi et al. (2022), who reported significant insecticidal activity of flonicamid against eggs, nymphs, and adults of *B. tabaci,* indicating that this pesticide could be used effectively for *B. tabaci* management (Huded et al. 2019, Kodandaram et al. 2017, Roditakis et al. 2014, Sadhana et al. 2021). Flubendiamide achieved adequate control of whitefly nymphs (Dake and Bhamare 2019), but acetamiprid effectiveness was limited due to the resistance of field populations of piercing-sucking insects to acetamiprid and other insecticides (Koo et al. 2014, Ullah et al. 2020, Wang et al. 2007). Azadirachtin has was less effective against *B. tabaci,* as occurs with other neem (*Azadirachta indica* Juss) products. Azadirachtin contains triterpenoids, and these active ingredients undergo rapid degradation by ultraviolet radiation when used as a foliar application (Barnaby et al. 1989, Barrek et al. 2004, Johnson et al. 2003). Mortality of *B. tabaci* under field conditions were 55.4–67.0% with azadirachtin (Prijović et al. 2012).

Detoxification enzymes are the key factors in the metabolism of toxic compounds in insects (Moustafa et al. 2023d, Prasannakumar et al. 2023). Therefore, insect resistance is usually associated with increased activity of these enzymes (Fouad et al. 2022). In the current study, the enzymatic activity of esterase did not increase significantly in the nymphs after flubendiamide and flonicamid application.

Thus, esterase enzyme activity could serve as an indicator of whitefly adaptation to insecticides. In contrast, both insecticides did not decrease the activities of cytochrome P-450 and GST, which may be a major cause of whitefly nymph mortality.

Pesticide residues in plants are significantly influenced by a number of essential elements, including the photolysis and volatilization of pesticides brought on by high temperatures and light, rain, and the physical and chemical properties of pesticides (Subirats et al. 2005). With regards to residues, our results for both flubendiamide and flonicamid in cherry tomato fruits and leaves and in soil samples agreed with those of Kelageri et al. (2017), who reported 1.23 mg/kg in tomato samples taken from polyhouses; this residue diminished to below detectable limits by day 10, with a half-life of 6.18 d. Deposits of 0.90 mg/kg in open fields diffused to below detectable limits by day 7, with a half-life of 6.07 d, indicating that polyhouse dissipation was slower than that in the open fields for a variety of reasons.

According to the Organization for Economic Cooperation and Development calculator and an assessment of the chronic hazard exposure factoring in average body weight, national tomato consumption per capita, and the ADI of flubendiamide, MRLs of 3 mg/kg for polyhouse tomatoes and 2 mg/kg for open field tomatoes are advised. A 4% acetic acid solution (61.63%) was the least effective decontamination method (17.71%) for removing flubendiamide residues from cherry tomatoes, whereas a vegetable wash was the most effective procedure for removing flubendiamide residues (65.39%) and can be recommended as a risk mitigation method for food safety.

Our findings were also consistent with those of Wang et al. (2018), who investigated the persistence of flonicamid and its metabolites in soil and cabbage (*Brassica* sp.) after harvest and their dissipation behavior. The half-lives of flonicamid alone and total residues (the sum of flonicamid and its metabolites) were 1.49–4.59 and 1.97–4.99 d, respectively, in cabbage plants and 2.12–7.97 and 2.04–7.62 d, respectively, in soil. Flonicamid decomposed quickly. When 50% flonicamid WG was sprayed once or twice at the recommended dose and at 1.5 times the recommended dose, the highest residues of total flonicamid in cabbage and soil at various PHIs (3, 7, and 14 d) were 0.070 and 0.054 mg/kg, respectively.

According to consumption data from China, flonicamid's RQ was $<$16.84%, which suggests that using it is safe for people. These findings could assist the Chinese government in determining the MRL for flonicamid in cabbage and could provide guidance for the safe and responsible use of flonicamid in agriculture. Li et al. (2021) found that total and chlorfluazuron RQs were both $<$1. This finding suggests that there is very little dietary danger from chlorfluazuron in tea. However, compared with chlorfluazuron, total flonicamid ingestion carries a threefold greater risk. Consuming tea containing flonicamid and its metabolites poses a risk to human health. In contrast, Kelageri et al. (2017) found that flubendiamide in tomatoes has low risk, and the total dry matter intake was less than the ADI in open fields and polyhouses. Jankowska et al. (2022) investigated the long-term risk of pesticide residues on some vegetables. The RQ was $>$1 for flonicamid in Brussels sprouts (*Brassica* sp.), which indicated negative health effects on humans. This finding is not consistent with those of our study, where the RQ was high for only 5 d and then decreased. Evaluation of the dissipation and health risk for some insecticides in tomatoes under field and polyhouse conditions revealed that no health

risk for consumers for the tested pesticides, including flubendiamide (Singh et al. 2023). These results support the accuracy and precision of the proposed procedure, affirming its suitability for the quantitative analysis of flubendiamide and flonicamid in cherry tomato samples.

In conclusion, flubendiamide and flonicamid could be used as effective insecticides in the whitefly management program in Egypt, and this program should include various groups of insecticides. Flubendiamide and flonicamid concentrations in diverse matrices were determined quickly and effectively in our study with a QuEChERS technique. For flubendiamide and flonicamid, the PHI were 3 and 7 d, respectively. Estimations of food exposure become are crucial when investigating pesticide residues.

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