

Efficacy of Certain Neonicotinoids Against Cotton Whitefly, *Bemisia tabaci* and Their Residues in Fruits and Leaves of Tomato Plants under Open Field Conditions

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ABSTRACT

Globally, neonicotinoids constitute a class of systemic insecticides that has become the most widely utilized group of insecticides. The efficiency of three neonicotinoid insecticides i.e. imidacloprid, thiamethoxam and dinotefuran at recommended dose were studied against the cotton whitefly, *Bemisia tabaci* (Gennadius) (Hemiptera: Aleyrodidae) on tomato plants under the open field conditions. The results showed that imidacloprid was the most efficient insecticide against the nymphs of *B. tabaci* followed by thiamethoxam and dinotefuran. The general means of reduction percentages of *B. tabaci* nymphs were 85.75, 83.24 and 75.11% after the 1st spray and 87.53, 85.03 and 73.96% after the 2nd spray for imidacloprid, thiamethoxam and dinotefuran, respectively. Residues of the tested pesticides in/on tomato fruits and leaves were determined using a QuEChERS method. Initial amount of the three insecticides were higher in tomato leaves compared with fruits. The half-life values ($t_{1/2}$) for the three insecticides in tomato fruits were 2.71, 2.95 and 1.87 days for imidacloprid, thiamethoxam and dinotefuran, respectively. While these values in tomato leaves were 2.91, 3.322 and 2.108 days for imidacloprid, thiamethoxam and dinotefuran, respectively. The levels of residues were above the maximum residue limits (MRLs) up to 3, 6 and 6 days after spray (DAS) for imidacloprid, thiamethoxam and dinotefuran, respectively in tomato fruits. The determined PHI for imidacloprid, thiamethoxam and dinotefuran were 6, 9 and 9 DAS, respectively. Washing of treated tomato fruits with tap water, 10% sodium bicarbonate, and 10% vinegar for 15 min were reported to be highly effective in reducing the level of the three insecticides. The residues of the three insecticides in tomato fruits pose low health risks to consumers.

Key words: Neonicotinoids, Cotton whitefly, Toxicity, Residues, QuEChERS.

INTRODUCTION

Similar to other plant species, tomato plants are subjected to infestation with several insect pests which usually cause serious injury and reduction to the final

yield (Ammar, 2007 and Mahmoud *et al.*, 2020). The cotton whitefly, *Bemisia tabaci* (Genn.) was an economically important pest on the tomato plants in different parts of the world. Worldwide, this polyphagous pest has the capability to attack over 600 different plant species in the open field and greenhouse conditions (Gelman *et al.*, 2007). *Bemisia tabaci* inflicts direct damage on its host plants by extracting sap from the plant foliage. Additionally, it causes indirect damage by transmitting plant pathogenic viruses and excreting honeydew which serves as an ideal medium for the growth of sooty mold (Henneberry *et al.*, 2000; Stansly *et al.*, 2004 and Hanafy *et al.*, 2014). Control of *B. tabaci* still depends on the application of insecticides (Ayad *et al.*, 2009; Shaoli Wang *et al.*, 2017 and Darwish *et al.*, 2021). Currently there are four generations of neonicotinoids. The first generation includes imidacloprid, acetamiprid, thiacloprid, and nitenpyram. The principal second-generation neonicotinoid is thiamethoxam, with dinotefuran serving as the primary third-generation neonicotinoid. Additionally, sulfoxaflor and cycloxyprid are classified as fourth-generation neonicotinoids (Simon-Delso *et al.*, 2015). Neonicotinoids act as agonists at nicotinic acetylcholine receptors in insects affecting initiation of the electric signal in the postsynaptic neuron. Unfortunately, the widespread use of synthetic insecticides to manage destructive insect pests like *B. tabaci* has led to various challenges. One significant issue is the presence of residues in vegetables and fruits, particularly with highly stable and persistent insecticides (AL-Eed, 2006). Therefore these residues must be regularly and widely monitored, removed by many methods such as peeling, washing, cooking, juicing, frying and freezing (Andrade *et al.*, 2015; El-Saeid & Selim, 2016; Ahlawat *et al.*, 2019 and Hassan *et al.*, 2019). The present investigation aims to study the susceptibility of the nymphs of white fly to imidacloprid, thiamethoxam and dinotefuran, to

determine their residues on and in tomato fruits and leaves, to determine the preharvest intervals, and to study the effect of different washing solution to remove there residues from the treated tomato fruits.

MATERIAL AND METHODS

Tested insecticides:

Imidacloprid (ImiDOR, 35% SC), thiamethoxam (Actara, 25% WG) and dinotefuran (Oshin, 20% SG) were obtained from Chema Industries Co., Syngenta Egypt Co. and Shoura Co., Egypt with application rates of 75 ml, 20 g and 125 g /100 litre of water, respectively.

Field studies

The current experiment was planned to compare the efficiency of three insecticides against the cotton whitefly, *B. tabaci*. The experiment was conducted on a private tomato farm located in the Nubaria district, Beheira Governorate, Egypt. Except the application of any pesticides, all the normal agricultural practices were carried out as usual. Randomized complete block design (RCBD) was applied with four replicates/ treatment and four replicates for control in a total of 16 plots. Every plot was separated from the other by 1 m to reduce interference from another treatment drift. Sample size was a number of *B. tabaci* nymphs found on ten leaves from five different plants of each plot (40 leaves / treatment). All treatments were applied during the tomato fruiting stage. Counts of nymphs of *B. tabaci* were recorded at first immediately before treatment and at 1, 4, 7 and 14 days after treatment. A standard 20-liter capacity hand sprayer, equipped with a downward-bent nozzle, was utilized. Reduction percentages of *B. tabaci* nymphs were calculated according to Henderson and Tilton equation (1955) and subjected to analysis of variance (ANOVA) (CoStat Statistical Software, 1998).

Henderson and Tilton equation (1955):

$$\% \text{ Reduction} = 100 \times 1 - \frac{T_a \times C_b}{T_b \times C_a}$$

Where:

C_b = mean number of nymphs in control plots before application

T_a = mean number of nymphs in treatment plots after application

C_a = mean number of nymphs in control plots after application

T_b = mean number of nymphs in treatment plots before application

Extraction, clean up procedures and residues determination

The extraction and clean-up of tomato samples were performed utilizing the QuEChERS method, following the procedure outlined by Anastassiades *et al.* (2003). Samples of 10 g of a homogenized tomato fruits and/or leaves were taken into a centrifuge tube (50-mL). Subsequently, 15 milliliters of acetonitrile containing 1.0% acetic acid were transferred to the centrifuge tube and vigorously shaken for 1 min. Subsequent to the addition of 4 g of anhydrous magnesium sulfate and 1 g of sodium acetate, the mixture was vigorously shaken for a duration of five minutes. Following this, the mixture underwent centrifugation at 3000 rpm for another five minutes. A volume of five milliliters from the supernatant was carefully transferred into a 15 ml centrifuge tube. Subsequently, it was shaken with 50 mg of primary secondary amine (PSA), 10 mg of graphitized carbon black, and 150 mg of magnesium sulfate. Following this, the centrifuge tube underwent centrifugation for a duration of 10 minutes at 6000 rpm. The supernatant containing imidacloprid, thiamethoxam, and dinotefuran was subjected to analysis using an Agilent 1260 HPLC system (USA), equipped with a quaternary pump, autosampler injector, thermostat compartment for the column, and a photodiode array detector. The chromatographic column employed was Zorbax C18 XDB (250 × 4.6 mm, 5 mm), maintained at room temperature. The mobile phase comprised acetonitrile and water in the ratios of (80:20), (90:10), and (60:40) for imidacloprid, thiamethoxam, and dinotefuran, respectively, at a flow rate of 1 ml/min.

Effect of different washing solutions on the residues in and on tomato fruits

To investigate the impact of a washing process aimed at eliminating residues of imidacloprid, thiamethoxam, and dinotefuran from treated tomato fruits, samples from the treated fruits (collected 2 hours after spraying) were immersed in separate jars, each containing one of the following solutions: tap water, 10% sodium bicarbonate, and 10% vinegar for 15 min (without shaking). The washed samples were allowed to dry in room temperature and then the analyzed as mentioned above (Pugliese *et al.* 2004; Zhang *et al.* 2007 and Andrade *et al.*, 2015).

Recovery rates and statistical analysis

To assess the recovery percentages, specified quantities of the active ingredients from the three tested insecticides were added to organic tomato fruits and leaves at concentrations of 2 and 4 mg/kg. The extraction and clean-up procedures followed the previously outlined methods. Table (3) provides the recovery rates for the three investigated insecticides

across various subsequent tomato fruits and leaves. All results obtained were corrected according to the recovery percentages. The degradation rates (k) and half-life periods of the three insecticides were determined following the methodology outlined by Gomaa and Belal (1975). Using the excel trend line; a straight line was fitted with intercept equal to logarithm of initial concentration. The slope of the line was calculated. Accordingly, the rate of degradation (k) of imidacloprid, thiamethoxam and dinotefuran and the half-life periods of the three insecticides were calculated as follows:

Rate of degradation (k) = 2.303×slope.

Half-life period (t½) = 0.693/k.

Estimated dietary exposure dose (EED) and risk quotient (RQ)

The exposure to the pesticide residues was evaluated via estimation of daily intake (EDI) and compared with acceptable daily intakes (ADIs)

Where:

EED (Estimated exposure dose) (mg / kg body weight / day) = Mean of insecticide residue (mg kg⁻¹) × amount food intake kgd⁻¹ per body weight (kg) (Kg/capita/day)

RQ (Risk Quotient) = EED/acceptable daily intake (ADI) (mg/kg bw)

The average estimated standard tomato intake for an Egyptian adult, with an average body weight of 60 kg, as reported by the Food and Agriculture Organization in 2012, was 200.93 g (0.20093 kg/capita/day. (WHO/GEMS/Food Cluster diets, 2012). The acceptable daily intake (ADI) for imidacloprid, thiamethoxam and dinotefuran was 0.06, 0.08, and 0.22 mg/kg/bw/day, respectively (Codex Alimentarius Commission for Pesticide Residues, 2016). An RQ value greater than 1 indicates that the risk of a pesticide for humans is considered unacceptable, whereas an RQ value less than 1 indicates minimal risk to humans (Zhang *et al.*, 2009).

RESULTS AND DISCUSSION

The efficiency of imidacloprid, thiamethoxam and dinotefuran against nymphs of *B. tabaci*

The data presented in Tables (1 and 2) illustrate the reduction percentages of *B. tabaci* nymphs in tomato plants for imidacloprid, thiamethoxam and dinotefuran after one, four, seven, and fourteen days post treatment. Imidacloprid was the most efficient insecticides against the nymphs of *B. tabaci* followed by thiamethoxam and dinotefuran. However, the tested insecticides exhibited different reduction percentages against *B. tabaci* as follows 80.49, 84.02 and 86.28% (after one day), 75.13, 86.47 and 89.39% (after four days), 73.51, 83.36 and 84.55 (after one week), 71.31, 80.8 and 82.78% (after two weeks) with general means of reduction percentages of 75.11, 83.24 and 85.75 for dinotefuran, thiamethoxam and imidacloprid, respectively after the 1st spray. After the 2nd spray, the general means of reduction percentages were 73.96, 85.03 and 87.53% for dinotefuran, thiamethoxam and imidacloprid, respectively. Our findings align with those of Li *et al.* (2021), whose study similarly reported that neonicotinoid insecticides were effective in the control of *Bemisia tabaci* on vegetables. El-Naggar and Zidan (2013) tested thiamethoxam and imidacloprid against sucking insects, and they found that both tested insecticides showed a moderate initial reduction in the population of jassids and the mature stages of whitefly. Also, it was found that imidacloprid demonstrated greater efficacy against sap-sucking pests compared to thiamethoxam. When applying thiamethoxam at rate of 100 g/ha, it was found most effective than imidacloprid (17.8 SL) at rate of 100 ml/ha in reducing the population of whitefly (Kumar *et al.*, 2017).

On the other hand, Smith *et al.* (2016) estimated the LC₅₀s for the field populations of *B. tabaci*. They observed that the LC₅₀ values varied within the following ranges: 0.901-24.952 for imidacloprid, 0.965-24.430 for thiamethoxam, 0.043-3.350 for dinotefuran, and 0.011-1.471 for flupyradifurone. These findings highlight the diverse susceptibility of *B. tabaci* field populations to various pesticides.

Table 1. Efficacy of three neonicotinoids insecticides on cotton whitefly, *Bemisia tabaci* nymphs under field conditions after the 1st spray

Insecticides	% of mortality post spray (days)*				General means
	1	4	7	14	
Dinotefuran	80.49±1.34 ^c	75.13±2.09 ^b	73.51±2.44 ^b	71.31±2.6 ^b	75.11±4.01 ^c
Thiamethoxam	84.02±1.51 ^b	86.47±2.01 ^a	83.36±1.11 ^a	80.8±.32 ^a	83.24±3.08 ^b
Imidacloprid	86.28±.98 ^a	89.39±1.69 ^a	84.55±1.4 ^a	82.78±.41 ^a	85.75±2.75 ^a
F values	20.318	60.56	48.207	42.736	44.918
L. S. D.	2.0742	3.09725	2.79025	2.8686	2.36445

No significant differences exist between means that share the same letter(s) within the same column (P ≤ 0.05) * Data were expressed as means ± SD

Table 2. Efficacy of three neonicotinoids insecticides on cotton whitefly, *Bemisia tabaci* nymphs under field conditions after the 2nd spray

Insecticides	% of mortality post spray (days)*				General means
	1	4	7	14	
Dinotefuran	79.2±2.35 ^c	75.38±1.59 ^c	72.89±1.83 ^c	68.38±1.72 ^b	73.96±4.4 ^b
Thiamethoxam	84.62±1.42 ^b	88.11±2.59 ^b	86.58±4.5 ^a	80.81±1.13 ^a	85.03±3.3 ^a
Imidacloprid	89.45±3.97 ^a	93.79±1.24 ^a	85.68±1.64 ^b	81.2±4.58 ^a	87.53±5.59 ^a
F values	13.534	99.301	66.025	25.247	40.73
L. S. D.	4.4574	3.0275	3.0158	4.6418	3.22285

No significant differences exist between means that share the same letter(s) within the same column ($P \leq 0.05$). *Data were expressed as means \pm SD

Experimental recovery

The efficiency of the analytical method was investigated through the implementation of recovery experiments. The procedures which were used for the determination of imidacloprid, thiamethoxam and dinotefuran residues in and on tomato fruits and leaves were applied for tomato samples were fortified with known amounts from each pesticide. Table (3) displays the average recoveries of imidacloprid, thiamethoxam, and dinotefuran in both tomato fruit and leaf samples. The obtained recoveries for imidacloprid in spiked tomato fruits and leaves varied between 101.34% and 103.45% for leaves and between 99.03% and 104.23% for fruits. As for thiamethoxam, the recoveries in spiked tomato leaves and fruits ranged from 97.28% to 99.48% for leaves and from 96.98% to 99.1% for fruits. Finally, the recoveries achieved for dinotefuran in spiked tomato leaves ranged from 101.79% to 104.3%, while in spiked tomato fruits, the recoveries ranged from 99.91% to 104.25%. According to Dg Sanco (2013), the recoveries obtained for imidacloprid, thiamethoxam, and dinotefuran fell within the 70-120% range, accompanied by a relative standard deviation (RSD %) value within the acceptable range of $\leq 20\%$. This suggests that the method exhibits good performance, sensitivity, and is suitable for determining these residues in both tomato leaves and fruits.

Determination of the residues of three neonicotinoid insecticides in tomato fruits and leaves

The obtained results in Table (4) show that the initial deposits of the imidacloprid insecticide in tomato fruits and leaves were 1.302 and 2.672 mg kg⁻¹, respectively. These residues decreased to 0.883 and 1.518 mg/kg after 1 day and to 0.553 and 0.962 mg/kg after three days post treatment. After 6 days post treatment the residues of imidacloprid were 0.337 and 0.611 mg/kg. In the fifteenth day of the treatment, the residues of imidacloprid reached to 0.029 and 0.047 in tomato fruits and leaves, respectively. The loss or dissipation percentages of imidacloprid were 32.18, 57.53, 74.12, 89.4, 96.39 and 97.78 % in tomato fruits and 43.19, 64, 77.13, 86.6, 93.49, and 98.24 in tomato leaves after 1, 3, 6, 9, 12 and 15 days, respectively.

The amount of imidacloprid remaining in tomato fruits was below the MRL of 0.5 mg/ kg (Codex Alimentarius Commission for Pesticide Residues, 2004) after 6 days of its application at the recommended rate. The half-life value ($t_{1/2}$) for dissipation of imidacloprid in tomato fruits was 2.7086 days, after the spraying with the recommended rate. The current results are in agreement with those of Sabry *et al.* (2016) who found that imidacloprid had a minimum preharvest interval (PHI) and was more toxic against the adults of *B. tabaci* on tomato plants. On the other hand, Hassanzadeh *et al.* (2012) found that imidacloprid residues dissipated below MRL of 1 (mg kg⁻¹) in 3 days in cucumber fruits, with half-life value of 2.91 days on tomato fruits (Sharma *et al.*, 2018).

Table 3. Averages of recovery percentages at different spiked levels

Sample	mg/kg	% of Recovery		
		Imidacloprid	Thiamethoxam	Dinotefuran
Leaves	1	103.45±2.71	98.89±2.19	101.79±1.86
	2	101.84±2.71	99.48±1.85	103.86±3.04
	4	101.34±1.8	97.28±1.2	104.3±1.25
Fruits	1	99.03±1.88	96.98±1.19	104.25±1.45
	2	104.23±3.3	97.74±1.45	99.91±2.35
	4	102.32±2.14	99.1±0.75	101.65±1.89

Data were expressed as means \pm SD

Table 4. Residue levels of imidacloprid, and % dissipation and persistence in fruits and leaves of tomato plants cultivated under field conditions

Days after treatment	Fruits			Leaves		
	Residues mg/kg*	Dissipation %	Persistence %	Residues mg/kg*	Dissipation %	Persistence %
0 (2hr.)	1.302±0.03	-	-	2.672±0.024	-	-
1	0.883±0.014	32.18	67.82	1.518±0.009	43.19	56.81
3	0.553±0.008	57.53	42.47	0.962±0.011	64	36
6	0.337±0.006	74.12	25.88	0.611±0.008	77.13	22.87
9	0.138±0.007	89.4	10.6	0.358±0.012	86.6	13.4
12	0.047±0.004	96.39	3.61	0.174±0.012	93.49	6.51
15	0.029±0.001	97.78	2.23	0.047±0.003	98.24	1.76
K	0.256			0.238		
t _{1/2}	2.7086			2.91		

K = Rate of degradation, t_{1/2}= Half-life values *Data were expressed as means±SD

The residue and % dissipation and persistence of thiamethoxam in tomato fruits and leaves are presented in Table (5), where the initial residue in fruits and leaves were 0.678 and 0.967 mg kg⁻¹ two hour after the spraying with the field recommended rate. The residues were degraded to 38.79 % and 51.09 after one day days showing residues of 0.415 and 0.473 mg/kg in tomato fruits and leaves, respectively. Furthermore, the residual amount of thiamethoxam dissipated calculated was 59.29, 70.21, 85.55, 94.54 and 97.64 % in tomato fruits and 74.46, 86.04, 90.59, 94.11 and 96.9 in tomato leaves after 3, 6, 9, 12 and 15 days, respectively. The amount of thiamethoxam remaining in tomato fruits was found to be under the established maximum residue limit (MRL) of 0.2 mg/kg, as defined by the Codex Alimentarius Commission for Pesticide Residues (2009), after 9 days of its application at the recommended dose. The half-life values (t_{1/2}) for the dissipation of thiamethoxam insecticide in tomato

leaves and fruits were determined to be 3.322 days and 2.9547 days, respectively. The present results are closely align with those reported by Rabea *et al.* (2018), who determined a half-life period (t_{1/2}) of 3.11 days for thiamethoxam on pepper fruits. Similarly, Abd-Alrahman (2014) observed a value of 2.92 days for the half-life of thiamethoxam in potato tubers following application at the recommended rate. On the other hand, Karmakar and Kulshrestha (2009) determined that when thiamethoxam was applied at a rate of 140 g a.i./ha, its half-life was 3.5 days. They recommended waiting for 8 days before safely consuming tomatoes treated with this pesticide. According to the recommended maximum residue limit, Abd El-Zaher *et al.* (2011) concluded that kidney bean horns were deemed safe for consumption after a period of 7 days, while Abd-Alrahman (2014) reported that potato tubers could be safely consumed after 6 days.

Table 5. Residue levels of thiamethoxam, and % dissipation and persistence in fruits and leaves of tomato plants cultivated under field conditions

Days after treatment	Fruits			Leaves		
	Residues mg/kg*	Dissipation %	Persistence %	Residues mg/kg*	Dissipation %	Persistence %
0 (2hr.)	0.678±0.015	-	-	0.967±0.011	-	-
1	0.415±0.007	38.79	61.21	0.473±0.014	51.09	48.91
3	0.276±0.008	59.29	40.71	0.247±0.003	74.46	25.54
6	0.202±0.006	70.21	29.79	0.135±0.008	86.04	13.96
9	0.098±0.005	85.55	14.45	0.091±0.004	90.59	9.41
12	0.037±0.002	94.54	5.46	0.057±0.005	94.11	5.89
15	0.016±0.002	97.64	2.36	0.03±0	96.9	3.1
K	0.235			0.2085		
t _{1/2}	2.9547			3.322		

K = Rate of degradation, t_{1/2}= Half-life values *Data were expressed as means±SD

Table (6) displays the residues and dissipation percentages of the insecticide dinotefuran in/on tomato leaves and fruits. Two hours post-treatment, the initial residue deposits on tomato fruits and leaves. The recorded amounts decreased to 1.083 and 2.132 mg kg⁻¹ one day after the application resulting degradation percentages of 30.8 and 36 %, respectively. Residues of dinotefuran in/on tomato (leaves and fruits) were gradually decreased to 0.562, 0.259, 0.092, 0.022, and 0.005 mg kg⁻¹ with corresponding degradation percentages of 64.09, 83.45, 94.12, 98.59 and 99.68 % in fruits, and 1.335, 0.596, 0.232, 0.072 and 0.02 mg kg⁻¹ with degradation percentages of 59.92, 82.11, 93.04 97.84 and 99.4 in leaves after 3, 6, 9, 12 and 15 days of application, respectively. Evaluation of the specified criteria, which include the established regression lines, such as slope, degradation constant (K), and half-life periods, revealed that dinotefuran degradation constant (K) values were 0.3704 and 0.3287 in/on tomato fruits and leaves, respectively. Regarding the half-life periods, dinotefuran exhibited values of 1.8712 and 2.108 days in/on tomato leaves and fruits, respectively. The maximum residue limit (MRL) of dinotefuran residues in/on tomato according to Codex Alimentarius Commission for Pesticide Residues (2013) was 0.5 mg/kg. The residue of dinotefuran in tomato fruits was below MRL of 0.5 mg kg⁻¹ after 6 days of its application at the recommended rate. Corroborating with the present findings, Rabea *et al.* (2018) observed that the half-life periods of dinotefuran and thiamethoxam on pepper fruits were 2 and 3.11 days, respectively. In accordance with the maximum residue levels (MRLs) (0.01 mg kg⁻¹ for dinotefuran and 0.7 mg/kg for thiamethoxam), the PHIs was determined to be 11 and 4 days, respectively. Similarly, Shams El Din *et al.* (2012) reported half-life values for dinotefuran in tomato and cucumber fruits as 1.72 and 3.18 days, respectively. In conclusion, the

levels of dinotefuran residues on/in tomato fruits were found to be below the maximum residue limits (MRL) one hour after application. This indicates that the tomato fruits can be safely used at any time after the application of dinotefuran.

Effect of different washing solutions on the removal of imidacloprid, thiamethoxam and dinotefuran residues from treated tomato fruits.

Data presented in Table (7) show the residues of imidacloprid, thiamethoxam, and dinotefuran in addition to the associated removal percentages, influenced by various washing solutions and processing treatments on tomato fruits. Results revealed that the residues of imidacloprid, thiamethoxam and dinotefuran on raw unwashed tomato fruits two hours after application were 1.302, 0.678, and 1.565 mg/kg. Washing the treated fruits with tap water resulted in a reduction of these amounts to 0.943, 0.573, and 1.012 mg/kg, accompanied by removal percentages of 27.57%, 15.49%, and 35.34%, respectively. Using the sodium bicarbonate the residues of the three insecticides were reduced to 0.759, 0.506 and 0.896 mg/kg for imidacloprid (41.71%), thiamethoxam (25.39%) and dinotefuran (42.75%), respectively. Finally, the use of 10% vinegar was the most effective solution causing 53.99, 35.99, and 60.77% removal percentages for the residues of imidacloprid, thiamethoxam and dinotefuran in tomato fruits, respectively.

Risk assessment

The health effects risk quotients (RQ values) for residues of imidacloprid, thiamethoxam, and dinotefuran in or on tomato fruits were calculated (Tables 8). Our findings indicated that the RQ values associated with imidacloprid, thiamethoxam, and dinotefuran residues in tomatoes were consistently lower than 1.

Table 6. Residues of dinotefuran, and % dissipation and persistence in fruits and leaves of tomato plants cultivated under field conditions

Days after treatment	Fruits			Leaves		
	Residues mg/kg*	Dissipation %	Persistence %	Residues mg/kg*	Dissipation %	Persistence %
0 (2hr.)	1.565±0.02	-	-	3.331±0.067	-	-
1	1.083±0.028	30.8	69.2	2.132±0.058	36	64
3	0.562±0.008	64.09	35.91	1.335±0.032	59.92	40.08
6	0.259±0.006	83.45	16.55	0.596±0.019	82.11	17.89
9	0.092±0.003	94.12	5.88	0.232±0.009	93.04	6.96
12	0.022±0.004	98.59	1.41	0.072±0.005	97.84	2.16
15	0.005±0	99.68	0.32	0.02±0.01	99.4	0.6
K	0.3704			0.3287		
t _{1/2}	1.8712			2.108		

K = Rate of degradation, t_{1/2}= Half-life values *Data were expressed as means±SD

Table 7. The efficacy of different washing processes for tomato fruits

Pesticides	Initial deposits mg/kg*	Tap water mg/kg	Removal %	10% sodium Bicarbonate mg/kg*	Removal %	10% vinegar mg/kg*	Removal %
Imidacloprid	1.302±0.03	0.943±0.038	27.57	0.759±0.017	41.71	0.599±0.031	53.99
Thiamethoxam	0.678±0.015	0.573±0.044	15.49	0.506±0.08	25.39	0.434±0.022	35.99
Dinotefuran	1.565±0.02	1.012±0.027	35.34	0.896±0.03	42.75	0.614±0.013	60.77

*Data were expressed as means±SD

Table 8. Residues means (mg/kg), estimated exposure dose (EED; mg/kg/bw/day), and risk quotient (RQ) of imidacloprid, thiamethoxam and dinotefuran in tomato fruits at various application time intervals

Pesticides	Days after treatment	Residues means mg/kg	EED (mg/kg/bw/day)	RQ	Health risk
Imidacloprid	0 (2hr.)	1.302	0.0044	0.0723	No
	1	0.883	0.003	0.049	No
	3	0.553	0.0019	0.031	No
	6	0.337	0.0011	0.0189	No
	9	0.138	0.0005	0.0077	No
	12	0.047	0.00016	0.0026	No
	15	0.029	9.7×10 ⁻⁵	0.0016	No
Thiamethoxam	0 (2hr.)	0.678	0.0023	0.0284	No
	1	0.415	0.0014	0.0174	No
	3	0.276	0.0009	0.0116	No
	6	0.202	0.00068	0.0085	No
	9	0.098	0.00033	0.004	No
	12	0.037	0.00012	0.0015	No
	15	0.016	5.36×10 ⁻⁵	0.00067	No
Dinotefuran	0 (2hr.)	1.565	0.0052	0.0238	No
	1	1.083	0.0036	0.0165	No
	3	0.562	0.0019	0.0086	No
	6	0.259	0.00087	0.0039	No
	9	0.092	0.00031	0.0014	No
	12	0.022	7.37×10 ⁻⁵	0.00033	No
	15	0.005	1.67×10 ⁻⁵	7.61×10 ⁻⁵	No

Consequently, the results imply that at the recommended dosage of these three tested insecticides, the potential risk to human health from their residues in tomatoes is negligible. These findings align with those of Abbassy *et al.* (2017), who observed that residues of chlorpyrifos-methyl and imidacloprid insecticides had no risk quotient, while fipronil presented a potential risk to humans depending on the consumption pattern of dates. Similarly, Sardar *et al.* (2023) reported that the relatively low values of cyantranilprole and indoxacarb residues in wild garlic pose a minimal risk to human health.

CONCLUSION

Based on the gained results and recommendations of EU for Maximum Residue Limits (MRL) of imidacloprid, thiamethoxam and dinotefuran we can concluded that imidacloprid was the most efficient against the nymphs of *B. tabaci*, thiamethoxam is moderate insecticide for control white fly in tomato crop. The half-life values ($t_{1/2}$) for the tested insecticides in tomato fruits were 2.71, 2.95 and 1.87 days for imidacloprid, thiamethoxam and dinotefuran, respectively. While these values in tomato leaves were 2.91, 3.32 and 2.11 days for imidacloprid, thiamethoxam and dinotefuran, respectively. The determined PHI for imidacloprid, thiamethoxam and dinotefuran were 6, 9 and 9 DAS, respectively. Washing

of treated tomato fruits with tap water, 10% sodium bicarbonate, and 10% vinegar for 15 min were reported to be highly effective in reducing the level of the three insecticides. The residues of the three insecticides in tomato fruits pose low health risks to consumers.

REFERENCES

- Abbassy M. A., Y. M. M. Salim, M.S. Shawir, A. M. K. Nassar. 2017. Disappearance and hazard quotient of chlorpyrifos-methyl, fipronil, and imidacloprid insecticides from dates. *J Consum Prot Food Saf*. DOI 10.1007/s00003-017-1111-3
- Abd El-Zaher, T.R., I.N. Nasr and H.A., Mahmoud. 2011. Behavior of some pesticide residues in and on tomato and kidney beans fruits grown in open field. *American-Eurasian Journal of Toxicological Sciences*, vol. 3, pp. 213-218. DOI: [10.13140/RG.2.2.16769.89442](https://doi.org/10.13140/RG.2.2.16769.89442)
- Abd-Alrahman, S.H. 2014. Residue and dissipation kinetics of thiamethoxam in a vegetable-field ecosystem using QuEChERS methodology combined with HPLC–DAD. *Food Chemistry*, vol. 159, pp. 1-4. <http://dx.doi.org/10.1016/j.foodchem.2014.02.124>. PMID:24767019
- Ahlatw, S., S. Gulia, K. Malik, S. Rani, R. Chauhan. 2019. Persistence and decontamination studies of chlorantraniliprole in *Capsicum annum* using GC-MS/MS. – *Journal of Food Science and Technology* 56: 2925-2931. DOI: [10.1007/s13197-019-03757-y](https://doi.org/10.1007/s13197-019-03757-y)
- AL-Eed, A.M. 2006. Determination of pirimiphosmethyl and chlorpyrifos-ethyl residues on tomato and pepperfruits grown in greenhouse. *J. Appl. Sci.*, 6 (4):979-982. DOI: [10.3923/jas.2006.979.982](https://doi.org/10.3923/jas.2006.979.982)
- Ammar, A. E. 2007. Conventional and developmental spraying techniques for controlling whitefly attacking tomato plants in greenhouses. *Egyptian Journal of Agricultural Research.*, 85, 1 (6): 67-75. DOI: [10.21608/ejar.2007.211061](https://doi.org/10.21608/ejar.2007.211061)
- Anastasiades, M., S. J. Lehotay, D. Stajnbaher and F. Schenck. 2003. Fast and easy multiresidue method employing acetonitrile extraction/partitioning and “dispersive solid-phase extraction” for the determination of pesticide residues in produce. *J. AOAC. Int.*, 86: 412–431. DOI: [10.1093/jaoac/86.2.412](https://doi.org/10.1093/jaoac/86.2.412)
- Andrade, GCRM, SHG. Monteiro, JA. Francisco, LA. Figueiredo, AL. Rocha, V. Tornisielo. 2015. Effects of types of washing and peeling in relation to pesticide residues in tomatoes. *J Braz Chem Soc* 26:1994–2002. <https://doi.org/10.5935/0103-5053.20150179>
- Ayad, F.A., H.S. Taha, A.F.E. Afsah, A.R.I. Hanafy, S.A.G. Metwally and S. Ayoub. 2009. Insecticides Rotation Strategy for Controlling *Bemisia Tabaci* (Genn.) on Tomato Crop. *Alex. Scie. Exch. J.* 30: 265-273.
- Codex Alimentarius Commission for Pesticide Residues (CAC/PR). 2004. <https://www.fao.org/3/y5764e/y5764e.pdf>
- Codex Alimentarius Commission for Pesticide Residues (CAC/PR) .2009. List of maximum residue limits for pesticides in food and animal feeds. <http://www.codexalimentarius.net>.
- Codex Alimentarius Commission for Pesticide Residues (CAC/PR). 2013. List of maximum residue limits for pesticides in food and animal feeds. <https://www.fao.org/fao-who-codexalimentarius/meetings/archives/en/?y=2013>
- Codex Alimentarius Commission for Pesticide Residues (CAC/PR). 2016. List of maximum residue limits for pesticides in food and animal feeds. <https://www.fao.org/fao-who-codexalimentarius/meetings/archives/en/?y=2016&mf=07>
- CoStat Statistical Software. 1998. Microcomputer program analysis version 6.400, CoHort Software, Berkeley, CA. <https://www.fao.org/fao-who-codexalimentarius/meetings/archives/en/?y=2013>
- Darwish A. A. E., M. M. R. Attia and A. M. H. Khozimy. 2021. Effect of Some Integrated Pest Management Elements on the Population Density of *Bemisia tabaci* (Gennadius) (Hemiptera: Aleyrodidae) on Tomato Plants (*Solanum Lycopersicum* L.). *Alex. Scie. Exch. J.* 6 (42): 157-68. DOI: [10.21608/ASEJAIQJSAE.2021.149092](https://doi.org/10.21608/ASEJAIQJSAE.2021.149092)
- Dg-sanco, E. C. 2013. Guidance document on analytical quality control and validation procedures for pesticide residues analysis in food and feed. SANCO/12571. http://www.eurlpesticides.eu/library/docs/allcrl/AqcGuidance_Sanco_2013_12571.pdf
- El-Naggar, J.B. and N.A. Zidan. 2013. Field evaluation of imidacloprid and thiamethoxam against sucking insects and their side effects on soil fauna. *Journal of Plant Protection Research* 53. (4):375-387. DOI: [10.2478/jppr-2013-0056](https://doi.org/10.2478/jppr-2013-0056)
- El-Saeid, M. and M. Selim. 2016. Effect of Food Processing on Reduction of Pesticide Residues in Vegetables. – *Journal of Applied Life Science* 8: 2. doi: [10.9734/JALSI/2016/26801](https://doi.org/10.9734/JALSI/2016/26801)
- Gelman, D.B., M.A. Pszczolkowski, M.B. Blackburn and S.B. Ramaswamy. 2007. Ecdysteroids and juvenile hormones of whiteflies, important insect vectors for plant viruses. *Insect Physiology J.* 53:274–284. DOI: [10.1016/j.jinsphys.2006.11.006](https://doi.org/10.1016/j.jinsphys.2006.11.006)
- Gomaa, E.A. and M.H. Belal. 1975. Determination of dimethoate residues in some vegetables and cotton plants. *Zagazig J. Agric. Res.*, 2 (2): 215-220. <https://link.springer.com/content/pdf/10.1007/BF02027014.pdf>
- Hanafy, A.R.I., F. Baiomy and T. A. Maha. 2014. Comparison between the infestation rate of certain pests on cucumber and kidney bean and its relation with abiotic factors and anatomical characters. *Egyptian Academic Journal, Biological Science. (A. Entomology)*, Vol. 7(2): 63 – 76. DOI: [10.21608/EAJBSA.2014.12947](https://doi.org/10.21608/EAJBSA.2014.12947)
- Hassan H., E. Elsayed, A. Abd El-Raouf, S. N. Salman. 2019. Method validation and evaluation of household processing on reduction of pesticide residues in tomato. *Journal of Consumer Protection and Food Safety* 14:31–39. <https://doi.org/10.1007/s00003-018-1197-2>

- Hassanzadeh, N., A.E. Sari, N. Bahramifar. 2012. Dissipation of Imidacloprid in Greenhouse Cucumbers at Single and Double Dosages Spraying. *J. Agric. Sci. Tech.*, 14:557–564. <http://jast.modares.ac.ir/article-23-10441-en.html>
- Henneberry, T. J., L. F. Jech and D. L. Hendrix. 2000. *Bemisia argentifolii* (Homoptera: Aleyrodidae) honeydew and honeydew sugar relationships to sticky cotton. *Southwestern Entomology*. 25:1-14. <https://doi.org/10.1093/ee/25.3.551>
- Henderson, C. F. and E. W. Tilton. 1955. Test with acaricides against the brown wheat mite. *J. Econ. Entomol.*, 48: 157-161.
- Karmakar, R. and G. Kulshrestha. 2009. Persistence, metabolism and safety evaluation of thiamethoxam in tomato crop. *Pest Management Science*, vol. 65, no. 8, pp. 931-937. <http://dx.doi.org/10.1002/ps.1776>. PMID:19459179.
- Kumar A., SK Sachan, S. Kumar and P. Kumar. 2017. Efficacy of some novel insecticides against white fly (*Bemisia tabaci* Gennadius) in brinjal. *Journal of Entomology and Zoology Studies*. 5(3): 424-427
- Li, Y., G.N. Mbata, S. Punnuri, A.M. Simmons, D. Shapiro-Ilan. 2021. *Bemisia tabaci* on Vegetables in the Southern United States: Incidence, Impact, and Management. *Insects*, 12, 198. DOI:10.3390/insects12030198
- Mahmoud, Y. A., I. M. A. Ebadah, W. Attwa, S. Moawad, N. Omar, T. E. Abd El-Wahab and H. Sadek. 2020. Susceptibility of different tomato, *Solanum lycopersicum* L., varieties to infestation with some insect pests in Egypt. *Bulletin of the National Research Centre*. 44(1): 1-5. DOI:10.1186/s42269-020-00300-4
- Pugliese, P., J.C. Molto, P. Damiani, R. Marin, L. Cossignani and J. Manes. 2004. Gas chromatographic evaluation of pesticide residue contents in nectarines after non-toxic washing treatments. *J Chromat-A*. 1050:185–191. <https://doi.org/10.1016/j.chroma.2004.08.03>
- Rabea, M.M., E.S. Ibrahim, D. Elhafny, and M.A. Bayoumi. 2018. Determination of dinotefuran and thiamethoxam residues in pepper fruits under greenhouse conditions using the QuEChERS method and HPLC/DAD. *Egyptian Journal of Chemistry*, vol. 61, no. 2, pp. 249-257. DOI:10.21608/ejchem.2018.2803.1227
- Sabry, A.H., S.M. Abolmaaty, T.A. Abd-El Rahman and A. Abd-El Rahman. 2016. Residue determination of some rational insecticides in tomato fruits and their efficacy on sweet potato whitefly, *Bemisia tabaci*. *Int. J. Curr. Sci.* 19: 37–46. <https://ijcspub.org/papers/IJCSP10A1473.pdf>
- Sardar, S. W., J. Y. Choi, Y. J. Jo, A. E. S. A. Ishag, M. W. Kim and H. J. Ham. 2023. Residues and Safety Assessment of Cyantraniliprole and Indoxacarb in Wild Garlic (*Allium vineale*). *Toxics*, 11: 219. <https://doi.org/10.3390/toxics11030219>
- Shams El Din, A. M., M.M. Azab, T. R. Abd El-Zaher, Z.H.A. Zidan and A. R. Morsy. 2012. Persistence of Acetamiprid and Dinotefuran in Cucumber and Tomato Fruits. *American-Eurasian Journal of Toxicological Sciences* 4 (2): 103-107. DOI: 10.5829/idosi.aejts.2012.4.2.1101
- Shaoli, W., Y. Zhang, X. Yang, W. Xie and Q. Wu. 2017. Resistance Monitoring for Eight Insecticides on the Sweetpotato Whitefly (Hemiptera: Aleyrodidae) in China. *Journal of Economic Entomology*, 110, 2: 660–666. <https://doi.org/10.1093/jee/tox040>
- Sharma, P.C., P. Chandresh and S. Sharma. 2018. Persistence of imidacloprid, indoxacarb and λ -cyhalothrin on tomato (*Solanum lycopersicum* L.) under protected cultivation. *Int. J. Curr. Microbiol. Appl. Sci.* 7:2783–2794. DOI: <https://doi.org/10.20546/ijemas.2018.7.707.325>
- Simon-Delso, N., V. Amaral-Rogers, L.P. Belzunces, J.M. Bonmatin, M. Chagnon, C. Downs, L. Furlan, D.W. Gibbons, C. Giorio, V. Girolami, D. Goulson, D.P. Kreuzweiser, C.H. Krupke, M. Liess, E. Long, M. McField, P. Mineau, E.A.D. Mitchell, C.A. Morrissey, D.A. Noome, L. Pisa, J. Settele, J.D. Stark, A. Tapparo, H. Van Dyck, J. Van Praagh, J.P. Van der Sluijs, P.R. Whitehorn, M. Wiemers. 2015. Systemic insecticides (neonicotinoids and fipronil): trends, uses, mode of action and metabolites. *Environ. Sci. Pollut. Res.* 22: 5–34. doi: 10.1007/s11356-014-3470-y.
- Smith, H.A., C.A. Nagle, C.A. MacVean, C.L. McKenzie. 2016. Susceptibility of *Bemisia tabaci* MEAM1 (Hemiptera: Aleyrodidae) to Imidacloprid, Thiamethoxam, Dinotefuran and Flupyradifurone in South Florida. *Insects*, 7, 57. <https://doi.org/10.3390/insects7040057>
- Stansly, P. A., P. A. Sanchez, J. M. Rodriguez, F. Canizares, A. Nieto, M. J. Lopez, M. Fajardo, V. Suarez and A. Urbaneja. 2004. Prospects for biological control of *Bemisia tabaci* (Homoptera, Aleyrodidae) in greenhouse tomatoes of southern Spain. *Crop Protection*, 23:701-712. <https://doi.org/10.1016/j.cropro.2003.11.016>
- WHO/GEMS/Food Cluster diets, 2012. https://extranet.who.int/sree/Reports?op=vs&path=/WHO_HQ_Reports/G7/PROD/EXT/GEMS_cluster_diets_2012&use_rid=G7_ro&password=inetsoft123
- Zhang, Z. Y., X. J. Liub and X. Y. Hong. 2007. Effects of home preparation on pesticide residues in cabbage. *Food Control*, 18 (12): 1484–1487. <https://doi.org/10.1016/j.foodcont.2006.11.002>
- Zhang, Z., H. Li, M. Wu, Y. Yuan, X. Hu, W. Zheng. 2009. Residue and risk assessment of chlorothalonil, myclobutanil and pyraclostrobin in greenhouse strawberry. *Chin J Pestic Sci* 11:449–455. <http://www.nyxb.cn/en/article/id/20090408>

الملخص العربي

سمية بعض المبيدات التابعة لمجموعة النيونيكوتينويد Neonicotinoid ضد حشرة الذبابة البيضاء *Bemisia tabaci* وتقدير متبقياتها في ثمار وأوراق الطماطم تحت الظروف الحقلية

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الطماطم كانت ٢,٧٠٨٦، ٢,٩٥٤٧ و ١,٨٧١٢ يوم لكل من إيميداكلوبريد، ثيامثوكسام ودينوتيفوران، على التوالي. بينما بلغت هذه القيم في أوراق الطماطم ٢,٩١، ٣,٣٢٢ و ١٠٨.٢ يوم لكل من إيميداكلوبريد، ثيامثوكسام ودينوتيفوران على التوالي. مستويات المتبقيات الحشرية كانت أعلى من قيم الحد الأقصى للمبيدات وفقاً لقوانين الاتحاد الأوروبي (EU)، منظمة الصحة العالمية/ منظمة الأغذية والزراعة (FAO/WHO) حتى ٣ و ٦ و ٦ أيام من الرش بالنسبة للإيميداكلوبريد والثيامثوكسام ودينوتيفوران، على التوالي في ثمار الطماطم. وكانت فترة ما قبل الحصاد PHI المحددة لإيميداكلوبريد وثيامثوكسام ودينوتيفوران ٦ و ٩ و ٩ يوم من الرش، على التوالي. أشارت النتائج أيضاً إلى أن غسيل ثمار الطماطم المعاملة بالمبيدات بماء الصنبور و ١٠% بيكربونات الصوديوم و ١٠% حمض الخليك لمدة ١٥ دقيقة كان فعالاً للغاية في تقليل مستوى متبقيات المبيدات الحشرية الثلاثة. ويمكن القول أن بقايا هذه المبيدات الموجودة في ثمار الطماطم تشكل مخاطر صحية منخفضة على المستهلكين.

تُعد مجموعة مبيدات نيونيكوتينويد Neonicotinoid من أكثر مجاميع المبيدات الحشرية الجهازية استخداماً على مستوى العالم. تمت دراسة كفاءة ثلاثة مبيدات حشرية من مجموعة نيونيكوتينويد وهي إيميداكلوبريد imidacloprid وثيامثوكسام thiamethoxam ودينوتيفوران dinotefuran بالجرعات الموصى بها ضد ذبابة القطن البيضاء *Bemisia tabaci* (Gennadius) (Hemiptera: Aleyrodidae) على نباتات الطماطم تحت الظروف الحقلية. أظهرت النتائج أن الإيميداكلوبريد هو أكثر المبيدات الحشرية فعالية ضد حوريات *B. tabaci* يليه مبيد الثيامثوكسام ثم الدينوتيفوران. كانت المتوسطات العامة لنسب الخفض في حوريات *B. tabaci* هي ٨٥,٧٥، ٨٣,٢٤ و ٧٥,١١% بعد الرش الأولى و ٨٧,٥٣، ٨٥,٠٣ و ٧٣,٩٦% بعد الرش الثانية لكل من إيميداكلوبريد، ثيامثوكسام ودينوتيفوران، على التوالي. تم تقدير متبقيات إيميداكلوبريد وثيامثوكسام ودينوتيفوران في وعلى ثمار وأوراق الطماطم باستخدام طريقة QuEChER. أوضحت النتائج أن المتبقيات الأولية بعد الرش (ساعتين) للمبيدات الثلاثة كانت أعلى في أوراق الطماطم مقارنة بالثمار، وأن قيم نصف العمر ($t_{1/2}$) للمبيدات الحشرية المختبرة في ثمار