## Dissipation behavior and residues of some neonicotinoids compounds used against sucking insects on tomato fruits under open field conditions.

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**ABSTRACT:** The current study set out to provide light on how various neonicotinoid substances, such as acetamiprid, imidacloprid, thiacloprid, and thiamethoxam, used to deter sucking insects in tomato fruits, evaporate and persist in the environment after being utilised in an open field. Prior to HPLC-DAD analysis, the QuEChERS technology was used to find pesticide residues in fruits grown in Egypt's El-Behera Governorate. The method validation for extraction and quantitative determination of tested pesticide residues in tomato fruits using HPLC-DAD was applied at fortification points of 0.1, 0.5, and 1.0 mgkg-1 in tomato samples. The results showed that the ranges of the mean recoveries for the drugs acetamiprid, imidacloprid, thiacloprid, and thiamethoxam were 87.20 to 98.28%, 89.59 to 99.67%, 93.45 to 100.05%, and 82.13 to 99.84%, respectively.

**KEYWORDS:** Dissipation, Residues, insecticides, tomato, sucking insects, HPLC-DAD, QuEChERS.

### **1. INTRODUCTION:**

Neonicotinoid insecticides with novel modes of action, such as imidacloprid, acetamiprid, thiacloprid, thiamethoxam, clothianidin, and dinotefuran, are efficient pesticides. (Gupta et al., 2008). Neonicotinoid pesticides are frequently used on fruits and vegetables to ensure their quality, meet consumer demand, and for commercial purposes. Neonicotinoids block the feeding reflex of nuisance insects by acting on a particular protein (the nicotinic acetylcholine receptor) in their brains. The QuEChERS method was developed as a new sample preparation technique for pesticide multiresidue analysis between 2000 and 2002. (Anastassiades et al., 2003).

One of the most important ingredients in human meals worldwide is the tomato fruit (Lycopersicon esculentum), which is eaten either fresh or home-processed into juice or paste. Additionally, the antioxidant molecules found in tomatoes, including ascorbic acid, vitamin E, carotenoids, flavonoids, and phenolic acids, which support human health, make them an effective food. In Egypt, one of the most significant solanaceous vegetable crops is the tomato (Lycopersicon Esculentum Mill). There are numerous harmful bugs currently infesting the tomato plants. (**Palumbo and Natwick, 2010**).

The current investigation focused on the dissipation rate and residue levels of Neonicotinoid

insecticides including acetamiprid, imidacloprid, thiacloprid, and thiamethoxam in tomato fruits under field conditions in order to detect the Pre-Harvest Interval (PHI) of tomato treated with tested pesticides.

# 2.MATERIALS AND METHODS: 2.1.Pesticides used:

Reference standards for acetamiprid, imidacloprid, thiacloprid, and thiamethoxam with >97% purity was bought from Dr. Ehrenstorfer GmbH (Augsburg, Germany). All additional HPLC grade solvents and reagents were purchased from Sigma Aldrich. In acetonitrile, a stock solution of the pesticides under examination was made and stored at a temperature of 1 mg/ml. By serially diluting the stock solution, calibration standard and working solutions with concentrations ranging from 0.01 to 5.0 ug/ml were created. Agilent Technologies sold the QuEChERS salts: 4 g MgSO4, 1 g NaCl, 1 g trisodium citrate dihydrate, 0.5 g disodium hydrogen citrate sequihydrate, and d-SPE salts. (Wilmington, DE, USA). 2.2. Field trials and sample collection

The field investigations were completed in Egypt's El-Beheira Governorate, Itay El-Barud city. The tomato plants were spaced 0.5 m apart in rows that were 1 m long. The experimental area was divided into five plots, one plot for control samples and other plots for treatments by (Acetamiprid) Mospilan 20% SP with

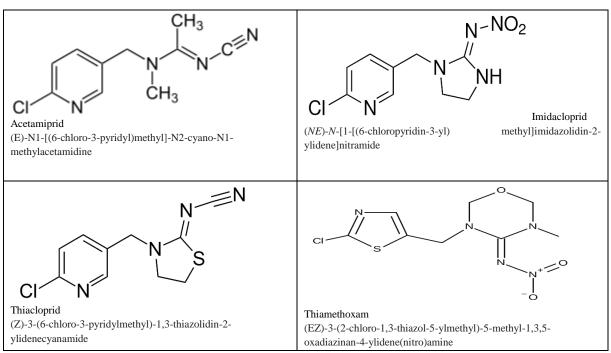


Fig. (1): Structures formula of tested insecticides

rate 25 g/100 L. water, another plot for treatment by (Imidacloprid) Pestidor 25 % WP with rate 100 g/100 L water, another plot for treatment by thiamethoxam (Actara 25% WG with rate 20 g./ 100 L water and the last plot for treatment with thiacloprid (Calypso 48% SC) with rate 120 cm<sup>3</sup> /feddan for each plot. Two-kilo grammes of tomato fruits were randomly taken from the control and treated plots one hour after application, one day, three days, seven days, and fifteen days afterwards. Fruit samples were kept until extraction in a freezer at 20°C.

#### 2.3. Sample processing

According to the official method designated by Anastassiades *et al.*, in 2003, extraction and purification were completed using a QuEChERS liquid extraction salt packet and dispersive cleanup kits. A 10 gramme homogenized sample was weighed into a 50 mL centrifuge tube. The steps of the analytical process were as follows: First, a 10 g sample was placed into a centrifuge tube. Next, 10 mL of acetonitrile and 10 mg of PSA and 150 mg of anhydrous MgSO<sub>4</sub> were added to each tube. Finally, the tubes were closed, and shaken vigorously by hand for 1 minute, and centrifuged at 3500 rpm for 5 minutes.

The tube was then vortexed for 1 minute and centrifuged at 3500 rpm for 5 minutes. The supernatants were filtered for HPLC-DAD analysis, using a 0.2 m PTFE filter (Millipore, Billerica, MA). By addition various standard solution concentrations to 10 g of tomato control samples at three levels of 0.1 to 1.0 mg/kg, fortified samples were created. Prior to

extraction, the fortified samples were allowed at room temperature for 30 min to allow the pesticide to permeate the matrix. Five copies of the identical analysis procedures were used to examine each fortification level.

#### 2.4. Instruments and apparatus

The chromatographic investigations were carried out using an Agilent 1260 series HPLC system with a quaternary pump, variable wavelength diode array detector (DAD), and an analytical column (Nucleosil C18) (30 mm x 4.6 mm id x 5 m). The flow rate of mobile phase's was 0.8 ml/min and the injection volume was 20  $\mu$ l and the detection wavelength was 270 nm and 246 nm, with mobile phase (acetonitrile 60% + water 40%), and (acetonitrile 30% + water 70%) for imidacloprid and acetamiprid, respectively. The retention period for imidacloprid was 3.8 min. and acetamiprid was 7.01 min.

On the other hand, the flow rate of the mobile phase (acetonitrile 90% + water 10%) and (acetonitrile 80% + water 20%) was 0.8 ml/min. with injection volume 20 µl and wavelength 254 nm and 240 nm and the retention time was 2.94 and 3.41 min. for thiamethoxam and thiacloprid, respectively.

#### 2.5. Method validation.

According to SANCO/1257(2013), laboratory technique validation was done to demonstrate that the method is suitable for the extraction and quantitative assessment of the tested pesticide in tomato. The approach was validated using a common validation

technique using the following parameters. For HPLC analysis, the assessed pesticide residues in tomatoes were either serially diluted with pure solvent at concentrations of 5, 1, 0.5, 0.1, 0.05, and 0.01, (matrix effect) contrasted with samples that were first extracted and then spiked with the assessed pesticide in the same solvent at the same concentration level, and (selectivity and sensitivity) determined the limit of quantification. Five repeats were utilised to examine the recovery (bias).

#### 2.6. Statistical analysis.

By analyzing the residue concentration with the amount of time that had passed after application, the dissipation kinetics of the tested pesticide residues in tomatoes were identified. The best curve fitting equations with the highest coefficients of determination (R2) were then identified. Exponential relationships that correlate to the general first-order kinetics equation were found to be relevant for the dissipation of tested pesticide residues in tomatoes:

#### $Ct=C0e^{-kt}$

Where C0 signifies the initial deposits after treatment, Ct is the pesticide residue concentration at the time of t, and k is the constant rate of pesticide dissipation every day. The examined insecticide's dissipation half-life periods (RL50=ln 2/k) were calculated using this equation.

### **3. RESULTS AND DISCUSSION:**

Tested pesticides were evaluated by using HPLC-DAD. The QuEChERS method were used to extract samples using a followed by a d-SPE cleanup step

#### 3.1. Validation study

The method was verified in accordance with [SANCO/12495/2013], and the matrix effect, linearity, LOD, LOQ, accuracy, and precision were evaluated as different validation criteria.

#### 3.2. Matrix effect (ME)

Blank samples were evaluated using the same apparatus to ensure that there are no peaks that interfere with the tested insecticides retention time. The following equation was used to compute the matrix effect: Matrix effect (%) =  $(S1/S2 \times 100) - 100$ , **Abdel Ghani and Hanafi (2016)**. While, S1 and S2 are, respectively, the slopes of the standard curves for the sample matrix and the pure solvent. The three categories that the percentage ME would fall into are no matrix effect (between -20% and 20%), medium matrix effect (between -50% and -20%), and severe

matrix effect (below -50% or over 50%). It might also be positive. The findings revealed that the matrix effect for the tested pesticides ranged from -12.58 to 14.74%, indicating that no interfering endogenous peak appeared and did not significantly suppress or increase the instrument's response. Saber *et al.*, 2016 and Ferrer *et al.*, 2011

#### **3.3. LOD and LOQ determination**

The LOD was established to be the analyzer's lowest concentration, which matched a signal-to-noise ratio of 3:1. The LOQ of the approach was obtained by identifying the pesticides at various concentrations at which the chromatographic peaks could be identified in samples [SANCO/12495/2013]. The signal to noise ratio of 10:1 was utilised as the LOO. The recommended method's low detection and quantification limitations allow for its use for the accurate assessment of pesticide residues in tested crops. LOD and LOQ were calculated and found to be, respectively, 0.01 and 0.1 mg/kg.

#### 3.4. Linearity

For each of the six tested pesticide concentrations, ranging from 0.01 to 5.0 mg kg1, calibration curves created by triple injection (n=3) were constructed to test the method's linearity. All of the pesticides that were examined displayed excellent linearity, with  $R^2$  values ranging from 0.97 to 0.99. A reasonable value for the relative standard deviation (RSD) is 15.2%. For tomato fruits, the correlation coefficient ( $R^2$ ) varied from 0.95 to 0.97.

#### 3.5. Accuracy and precision

Three levels of tested pesticide standards, ranging from 0.1 to 1.0 mg/kg, were applied to blank samples to assess the method's accuracy. The fortified samples underwent five replication studies (n=5). An assessment of the method's precision was made possible by calculating the recovery average at the measured values. The correlation between standard deviation and average concentration, known as the relative standard deviation (% RSD), has been recognized as an accuracy indicator. The accuracy has been calculated using the percentage difference between the discovered and known concentrations. In accordance with the listed criteria, the degree of precision and accuracy was determined to be suitable for method validation by **Filho et al., (2006)**.

Table (1) provided a summary of the recovery outcomes. The residues from the spiked control samples were measured. According to [SANCO/12495/2013], the method is reliable with acetamiprid, imidacloprid, thiacloprid, and thiamethoxam analysis and demonstrate accurate and precise work. All computed mean's recoveries results ranged from 70% to 120% and RSD 20%.

Spiked M levels	Mean recovery (±SD for Acetamiprid	) RSD %	Mean recovery (±SD) for Imidacloprid	RSD %	Mean recovery (±SD) for Thiacloprid	RSD %	Mean recovery (±SD) for Thiamethoxam	RSD %
0.1	87.20±2.33	1.22	89.59±0.91	1.09	93.45±1.25	2.01	82.13±2.08	1.98
0.5	90.41±1.95	1.87	95.74±1.02	1.49	96.21±1.59	2.61	90.51±2.12	2.08
1	98.28±2.01	2.46	99.67±0.98	1.18	100.05±1.55	1.95	99.84±2.31	1.85

Table (1): Average recovery and repeatability accuracy of pesticides evaluated in tomatoes for HPLC-DAD analysis

According to **Rabea** *et al.* (2018), the recovery thiamethoxam rates were reported to be within 78-112% using the QuEChERS method and determination using the HPLC/DAD. Thiamethoxam's outstanding correlation coefficient of  $R^2$ >0.996 demonstrated high linearity, and the calibrated matrix-matched likewise displayed good linearity with determination coefficients of  $R^2$ > 0.98, at the spike levels (0.01 - 1 mg/kg) in pepper samples with relative standard deviations (RSDs) less than 3%,

# **3.6.** Determination of tested pesticides in tomato fruits using HPLC-DAD analysis.

**3.6.1 Determination of acetamiprid in tomato fruits.** The investigation of acetamiprid dissipation in tomato fruits in field circumstances followed the recognised methodology. After being applied for an hour, the initial acetamiprid deposit in tomatoes was 2.780.32 mg/kg. After one day of application, the value steadily dropped to 2.030.11 mg/kg; three, seven, and ten days later, it fell to 1.090.38, 0.400.22, and 0.080.16 mg/kg, respectively. After 15 days of application, there were no longer any acetamiprid residues. Acetamiprid's half-life was 2.46 days.

In tomato fruits, residue levels, RL50, MRL, and calculated PHI were displayed in (Table 2). According to the **(EU, 2019)** MRL, the estimated PHI values for the recommended dosage were 7 days. Due to environmental variables in an open field, like an increase in temperature, acetamiprid in tomatoes may disperse more quickly.

# **3.6.2.** Determination of imidacloprid in tomato fruits.

The developed method was applied to research imidacloprid dissipation in tomato fruits under real-world field conditions. After being applied for one hour, the imidacloprid initial deposit in tomato was 2.970.55 mg/kg. Following a day of administration, the rate gradually reduced to 2.140.39 mg/kg. Three, seven, and ten days later, the rate was 1.080.89, 0.220.14, and 0.070.96 mg/kg, respectively. After 15 days of application, imidacloprid residues were below detection thresholds.

Residue amounts, RL<sub>50</sub>, MRL and estimated PHI in tomato fruits were showed in (Table 3). Estimated PHI values according to MRL (Codex 2004) was 7 days for recommended dose. The half-life of imidacloprid was 2.35 days.

Table (2): Acetamipr	id residue levels and	dissipation	patterns in tom	atoes grown in o	open fields.

Intervals after application (d	ays) Residues (ppm)±S	D % Loss%	Persistence
initial*	$2.78\pm0.32$	0.00	100.0
1	$2.03\pm0.11$	26.97	70.03
3	$1.09\pm0.38$	60.79	39.21
7	$0.40\pm0.22$	85.61	14.39
10	$0.08\pm0.16$	97.12	2.88
15	ND	100.00	0.00
<b>RL</b> 50	2.46 days		
MRL	0.5 mg/kg (EU, 201	9)	

PHI (days)	7 days
RL 50: Half-life period. MRL: M	laximum residue level. PHI: Pre-harvest interval.

# **3.6.2.** Determination of imidacloprid in tomato fruits.

The developed method was applied to research imidacloprid dissipation in tomato fruits under real-world field conditions. After being applied for one hour, the imidacloprid initial deposit in tomato was 2.970.55 mg/kg. Following a day of administration, the rate gradually reduced to 2.140.39 mg/kg. Three, seven, and ten days later, the rate was 1.080.89,

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Residue amounts,  $RL_{50}$ , MRL and estimated PHI in tomato fruits were showed in (Table 3). Estimated PHI values according to MRL (Codex 2004) was 7 days for recommended dose. The half-life of imidacloprid was 2.35 days.

Intervals after application (days)	Residues (ppm)±SD	% Loss	% Persistence
initial*	$2.97\pm0.55$	0.00	100.0
1	$2.14\pm0.39$	27.94	72.06
3	$1.08\pm0.89$	63.63	36.37
7	$0.22\pm0.14$	92.59	7.41
10	$0.07 \pm 0.96$	97.64	2.36
15	ND	100.00	0.00
<b>RL</b> 50	2.35 days (Codex, 2004)		
MRL	0.5 mg/kg		
PHI (days)	7 days		

Table (3): Imidacloprid residue levels and dissipation patterns in tomatoes grown in an open field

RL 50: Half-life period. MRL: Maximum residue level. PHI: Pre-harvest interval.

#### 3.6.3. Determination of thiacloprid in tomato fruits.

The well-known technique was applied to look at how much thiacloprid decreased in tomato fruits in outdoor circumstances. After one hour of treatment, the initial thiacloprid deposit in tomato was 1.280.42 mg/kg. After one day of application, the rate at that time gradually decreased to 1.110.69 mg/kg. After three, seven, and ten days of treatment, the rate fell to 0.590.33, 0.170.61, and 0.050.28 mg/kg, respectively. After 15 days of use, the thiacloprid residues were below detection thresholds.

Residue amounts,  $RL_{50}$ , MRL and estimated PHI in tomato fruits were showed in (Table 4). Estimated PHI values according to **Codex (2007)** MRL was 7 days for recommended dose. The half-life of thiacloprid was 2.78 days,

Table (4): Thiacloprid residue levels and dissipation patterns in tomatoes grown in an open field.

Intervals after application (days)	Residues (ppm)±SD	% Loss	% Persistence
initial*	$1.28 \pm 0.42$	0.00	100.0
1	$1.11 \pm 0.69$	13.28	86.72
3	$0.59\pm0.33$	53.90	46.10
7	$0.17\pm0.61$	86.71	13.29
10	$0.05\pm0.28$	96.09	3.91
15	ND	100.00	0.00
$RL_{50}$	2.78 days		
MRL	0.5 mg/kg (Codex, 2007)		
PHI (days)	7 days		

RL 50: Half-life period. MRL: Maximum residue level. PHI: Pre-harvest interval

# **3.6.4.** Determination of thiamethoxam in tomato fruits

Results in Table (5) showed that thiamethoxam degenerated in tomato fruits. For 14 days, the thiamethoxam loss in tomato fruits was studied. After two hours of treatment, the first thiamethoxam deposit in tomato fruits was 0.97-1.13 mg/kg. Even yet, tomato residues of 0.88 to 0.88

mg/kg were present on the first day after spraying. On the third day after spraying, there was a rapid dissipation with a thiamethoxam residual of 0.57–0.29 mg/kg. The degradation persisted for another 7 days after treatment, reaching 0.14 0.48 mg/kg. Thiamethoxam residue on tomatoes was reduced to 0.04–0.74 mg/kg. On the fifteenth day following treatment, the residues had become undetectable. According to (EU,

<b>2017</b> ), the maximum residue limit (MRL) for thiamethoxam
in tomato fruits is 0.2 mg/Kg. In light of the results.

Intervals after application (days)	Residues (ppm)±SD	% Loss	% Persistence
initial*	$0.97 \pm 1.13$	0.00	100.0
1	$0.88\pm0.88$	9.27	90.73
3	$0.57\pm0.29$	41.23	58.77
7	$0.14\pm0.48$	85.56	14.44
10	$0.04\pm0.74$	95.87	4.13
15	ND	100.00	0.00
$\mathbf{RL}_{50}$	3.63 days		
MRL	0.2 mg/kg (EU, 2017)		
PHI (days)	7 days		

RL 50: Half-life period. MRL: Maximum residue level. PHI: Pre-harvest interval.

Our results were agreed with Badawy et al. (2019) investigation of the imidacloprid and acetamiprid residues in tomato fruits grown in greenhouses in Egypt. QuEChERS was used to extract and clean up the fruits, and then HPLC analysis was performed. With a determination coefficient  $(R^2)$  of greater than 0.99 over the concentration range of 0.0125-0.15 g/mL, the technique demonstrated satisfactory linearity. The approach was validated using a control tomato spiked at 5, 25, and 50 mg/kg. For acetamiprid and imidacloprid, respectively, the recovery ratios were 83.71, 94.52, and 97.49% and 88.59, 89.63, and 90.18%. Preharvest intervals (PHIs) were computed after studying the rates of dissipation of both insecticides. The half-lives of imidacloprid and acetamiprid were 1.30 and 2.07 days, respectively, and imidacloprid disappeared more quickly. Three and five days after application, respectively, acetamiprid and imidacloprid residue levels were below the preestablished European maximum residue limits (EU MRLs: 0.5 mg/kg).

Under field conditions, Li et al. (2022) observed how much of the pesticides thiacloprid and spirotetramat were remained in cowpeas. The presence of thiacloprid and spirotetramat residues in cowpeas was discovered using the QuEChERS technique and (HPLC-MS/MS). Relative standard deviations (RSDs) ranged from 2.1 to 9.5%, and recoveries ranged from 81.3 to 95.1%. Thiacloprid and spirotetramat in cowpeas were destroyed with half-lives of 1.14-1.54 and 1.25-2.79 days, respectively, according to the dissipation kinetics data. After two applications with a three-day PHI, the terminal residues of thiacloprid and spirotetramat were 0.0255-0.4570 mg kg1 and 0.0314-0.3070 mg/kg, respectively.

Dinotefuran and thiamethoxam had early deposits in pepper fruits of 6.59 and 1.38 mg/kg, respectively. Dinotefuran and thiamethoxam, the two pesticides that were tested on pepper fruits, had halflives of 2.11 and 3.11 days, respectively. The preharvest interval (PHI) was 11 days for thiamethoxam and 0.7 mg/kg for dinotefuran, respectively, based on maximum residue levels (MRL) of 0.01 mg/kg and 0.7 mg/kg, respectively, Rabea et al. (2018) while, Gong et al. (2012) discovered that the thiamethoxam half-life in tomatoes was 4.6 days, and that after one day, tomato residue levels were below the EU's maximum residue limit of 0.1 mg kg<sup>-1</sup>. Reddy et al. (2021) examined the rate at which thiamethoxam 25% WG dissipated in vegetable cowpea. The residues were measured using a liquid chromatography tandem mass spectrometer (LC-MS/ MS), and it was discovered that the average initial residue was 0.53 g g<sup>-1</sup>. Residues were reported to persist for up to three days until falling below detection level on fifth day. On other hand, Singh and Allam (2016) studied the behavior of thiamethoxam in tomato fruits and soil following application of the pesticide at the recommended dose and twice the recommended dose using GC-ECD and validated by GC-MS. In tomato, the first deposits were 0.11 and 0.18 g/g, respectively. The tomato fruits were exposed to recommended and double recommended doses of thiamethoxam for 7 and 10 days, respectively, before residue levels fell below the detection threshold (0.01). In tomato fruit samples, recoveries ranged from 85.61 to 98.63%, and the half-life values were 2.21 and 2.41

days. Pesticide residues in crops that are affected by meteorological conditions, dosage, and the intervals between application and harvest. However, high temperatures are the main element in reducing the amount of pesticides on the plant surface. Light also has a significant impact on how pesticides behave in the environment. Climatic conditions like sunlight and temperature influence how quickly sprayed pesticides dissipate. Additionally, the degradation of pesticides may be brought on by biological, chemical, or physical processes, or, if they are still present in the field, by dilution by crop growth (Waghulde *et al.*, 2011; Christensen, 2004).

### REFERENCES

- Abdel Ghani, S.B. and Hanafi, A.H. (2016). QuEChERS method combined with GC-MS for pesticide residues determination in water. J. Anal. Chem. 71:508-512. doi:10.1134/S1061934816050117.
- Anastassiades, M.; Lehotay, S.J.; Tajnbaher, D. and Schenck, J.F. (2003). Fast and easy multiresidues method employing acetonitrile extraction/partitioning and "dispersive solidphase extraction" for the determination of pesticide residues in produce. J. AOAC Int., 86:412-430.
- Badawy, M.E.I.; Ismail, A.M.E and Ibrahim, A.I.H. (2019). Quantitative analysis of acetamiprid and imidacloprid residues in tomato fruits under greenhouse conditions. Journal of Environmental Science and Health, Part B, 898-905.
- Christensen, H.B. (2004). Fungicides in food, analytical and food safety aspects. Ph.D. thesis. Danish Institute for Food and Veterinary Research, Denmark.
- Codex (2004). <u>CODEXALIMENTARIUS FAO-</u> <u>WHO</u>. http://www.fao.org/fao-whocodexalimentarius/codextexts/dbs/pestres/pesticide -detail/en/? p\_id=206.
- Codex (2007). <u>CODEXALIMENTARIUS FAO-</u> <u>WHO</u>. http://www.fao.org/fao-whocodexalimentarius/codextexts/dbs/pestres/pesticide -detail/en/? p\_id=223.
- EU (European Commission) (2017). <u>EU Pesticides</u> Database (v.2.2) Pesticide residue(s) and maximum residue levels (mg/kg) (europa.eu)

- EU (European Commission) (2019). <u>EU Pesticides</u> <u>Database</u> (v.2.2) Pesticide residue(s) and <u>maximum residue levels (mg/kg) (europa.eu)</u>
- Fenoll, J.; Ruiz, E.; Hellin, P.; Lacasa, A. and Flores, P. (2009). Dissipation rates of insecticides and fungicides in peppers grown in greenhouse and under cold storage conditions. Food Chem., 113: 727-732.
- Ferrer, C.; Lozano, A.; Aguera, A.; Jimenez, A. and Fernandez, A.R. (2011). Overcoming matrix effects using the dilution approach in multiresidue methods for fruits and vegetables. Journal of Chromatography A, 1218 (42): 7634-7639. DOI: https://doi.org/ 10.1016/j.chroma.2011.07.03316.
- Filho, M.A.; Navickiene, S. and Dorea, H.S. (2006). Development of MSPD Method for the Determination of Pesticide Residues in Tomato by GC-MS. J. Braz. Chem. Soc., 17 (5): 874-879. Doi: 10.1590/S0103-50532006000500009.
- Gong, Y.; Huang, W.; Zhang, Y.F. and Li, J.Z. (2012). Determination of thiamethoxam residues in tomato by high performance liquid chromatography with UV detection. Asian J. Chem., 24(11):5289-5291.
- Gupta, M.; Sharma, A. and Shanker, A. (2008). Dissipation of imidacloprid in orthodox tea and its transfer from made tea to infusion. Food Chem., 106 (1): 158-164.
- Li, K.; Chen, W.; Xiang, W.; Chen, T.; Zhang, M.; Ning, Y.; Liu, Y. and Chen, A. (2022). Determination, residue analysis and risk assessment of thiacloprid and spirotetramat in cowpeas under field conditions. Sci. Rep., 12, 3470. <u>https://doi.org/10.1038/s41598-022-</u> 07119-1.
- Palumbo, J.C. and Natwick, E.T. (2010). The Bagrada Bug (Hemiptera: Pentatomidae): A New Invasive Pest of Cole Crops in Arizona and California. Online. Plant Health Progress, 11(1): 50. doi: 10.1094/PHP-2010-0621-01-BR.
- Rabea, M.M.; Ibrahim, E.S.; Elhafny, D. and Bayoumi, M. A. (2018). Determination of Dinotefuran and Thiamethoxam Residues in Pepper Fruits under Greenhouse Conditions using the QuEChERS Method and HPLC/DAD. Egypt. J. Chem., 61(2): 249 – 257.
- Reddy, B.K.K.; Paul, A. and George, T. (2021). Dissipation kinetics and risk assessment of thiamethoxam 25 % WG residues in vegetable

cowpea. Indian J. Entomology., 83. DoI No.: 10.5958/0974-8172.2021.00132.2.

- Saber, A.N.; Malhat, F.M.; Badawy, H.M.A. and Barakat, D.A. (2016). Dissipation dynamic, residue distribution and processing factor of hexythiazox in strawberry fruits under open field condition. Food chemistry, 196: 1108-1116. DOI: https://doi.org/10.1016/j.foodchem.2015.10.05 2.
- SANCO/12495/2013, Method Validation and Quality Control Procedures for Pesticide Residues

Analysis in Food and Feed. Sanco, 4 (2).<u>https://ec.europa.</u> <u>eu/food/sites/food/files/plant/docs/pesticides</u> <u>mrl\_guidelines\_wrkdoc\_11945.pdf</u>.

- Singh, B. and Allam, R. (2016). Persistence of thiamethoxam in/on tomato fruits and soil. J. Insect Sci., 29 (1): 25-31.
- Waghulde, P.N.; Khatik, M.K.; Patil, V.T. and Patil, P.R. (2011). Persistence and dissipation of pesticides in chilly and okra at north Maharashtra region. Pest. Res. J., 23 (1): 23-26.

## سلوك وبقايا بعض مركبات النيونيكوتينويد المستخدمة ضد الحشرات الماصة على ثمار الطماطم تحت ظروف الحقل المفتوح احمد احمد الغنام' - داليا السيد الحفني' - عاطف طه حسين المصري'

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### الملخص العربى:

تهدف الدر اسة الحالية هو إلقاء نظرة على كيفية استخدام العديد من مركبات النيونيكوتينويد (أسيتاميبريد وإيميداكلوبريد وثياكلوبريد وثياميثوكسام) ضد الحشر ات الثاقبة الماصة في ثمار الطماطم والتي تتبدد وتبقى في البيئة بعد تطبيقها في الحقل المفتوح. تم استخدام تقنية QuEChERS لاستخلاص بقايا المبيدات في الطماطم المنزر عة في محافظة البحيرة - مصر خلال موسم ٢٠٢٠ وكذلك تحليلها باستخدام تقنية QuEChERS تم التحقق من صحة طريقة الاستخلاص والتحليل الكمي لمخلفات المبيدات المختبرة في ثمار الطماطم باستخدام DAC-DAD للثلاث مستويات ١.٩ و مجم/ كجم. أظهرت النتائج المتحصل عليها أن متوسطات الاسترجاع لكلا من الأسيتامبيريد ، الإيميداكلوبريد ، الثياكلوبريد ، والثياميثوكسام تتر اوح ممم/ كجم. أظهرت النتائج المتحصل عليها أن متوسطات الاسترجاع لكلا من الأسيتامبيريد ، الإيميداكلوبريد ، الثياكلوبريد ، والثياميثوكسام تتر اوح من ٢٠٢٠ (لي ٢٩,٨٤٪ ، ٩٩,٥٩ إلى ١٩,٦٩٪ ، ١٩،٥٥ إلى ٥٠٠ (١٠٢ إلى ٢٩,٨٤٪ على التوالي ، وكان الانحراف المعياري إلى ٢٩,٩٠ إلى ٢٢,٦١٪ وبالنسبة لمدى التركيزات من ٢٠٠ - ٥ مجم / لتر ، تم تحقيق خطية جيدة مع معامل تحديد (R2) من ١٩،٧ وثياميثوكسام في الحماطم الكمي الدور الدي التركيزات من ٢٠٠٠ - ٥ مجم / لتر ، تم تحقيق خطية جيدة مع معامل تحديد (D2) من ١٩،٧ وثياميثوكسام في الماطم المربيد و (LOQ) مجم / كجم. وفقًا للنتائج ، كانت فترات نصف عمر أسيتامبريد و إيميداكلوبريد وثيكلوبريد وثيكلوبريد وثيكلوبريد وثيكلوبريد وثيكلوبريد وينيا ويليد من الامر معال معياري النسبي (رجمال المالي من ٢,٦١ إلى ١٩,٨٤٪ ) من ١٩،٩ النسبي (رجمال مع معامل تحديد (LOQ) محم / حمل وقيًا للنتائج ، كانت فترات نصف عمر أسيتامبريد وإيميداكلوبريد وثيكلوبريد وثيكلوبريد وثيكلوبريد وثيكلوبريد وثيكلوبريد وثياميثوكسام في المالم من الدر الأصل الدرجة الأولى. كانت فترات نصف من النولي من الدولي من الدرجة من الرمال معار النائي من ١٢،٣

الكلمات المفتاحية: تبدد – متبقيات – المبيدات الحشرية – الطماطم – الحشرات الماصة – الكروماتوجر افي السائل