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DETERMINATION OF THE RESIDUES OF ALPHA-CYPERMETHRIN, INDOXACARB AND LUFENURON ON ORANGE AND TANGERINE.

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ABSTRACT

Alpha-cypermethrin, indoxacarb, and lufenuron insecticide residues were studied in oranges and tangerine after their application in open fields in Egypt. This was done using QuEChERS for extraction and clean-up, the integration of technology with HPLC and GC equipments. When validating a method for the analysis of insecticides in commodities, there are certain criteria that must be met. These include linearity range, limit of quantitation (LOQ), and accuracy regarding precision and dependability.

In our testing, we found that the recovery rates of the method at different fortification levels (0.01, 0.1, and 1.0 mg/kg) ranged from 91% to 103% for the insecticides used in both crops. Results showed that the zero time (initial) of alpha-cypermethrin, indoxacarb, and lufenuron in orange were 2.55, 1.88, 3.1, and 2.67, 1.92, and 3.4 ppm in tangerine. The percentage of losses in residues was greater in tangerine than orange.

The half-lives of Alpha-Cypermethrin, indoxacarb, and lufenuron were 2.4, 4.2, and 4.71 days in orange and 2.5, 4.6, and 5.69 days in tangerine. Contaminated orange and tangerine could be consumed safely after two-week treatment.

Conclusively, *from these results it could be concluded that the percentage of losses in residues was greater in tangerine than orange, and contaminated orange and tangerine could be consumed safely after two-week treatment.*

Key Words: Indoxacarb, Lufenuron, Alpha-Cypermethrin, Orange, Tangerine Residues.

INTRODUCTION

Citrus fruits, including oranges and tangerines, constitute a significant portion of Egypt's agricultural exports, particularly to the European Union. Insecticides are crucial in contemporary agriculture for managing crop pests and ensuring food security. Key factors in the identification of novel insecticides include high potency and selectivity, as well as low residual effects and resistance **(Lamberth *et al*., 2013).** Citrus fruits serve as food and juice sources, while the peel is utilized for extracting essential oils employed in various perfumes and pharmaceuticals. Pesticides may be harmful to non-target creatures. **(World Health Organization [WHO], 2018).**

Lufenuron has been widely utilized in citrus fruits, particularly to control the red and citrus rust mites, as well as the Asian citrus mites. It is also a very good pesticide for reducing pest’s resistant to pyrethroids and organophosphates because of their importance in the mechanism of action **(Moya *et al*., 2010; Navarro-Llopis *et al*., 2004).** Lufenuron is an insecticide that was first approved for use on a wide range of crops to suppress the larvae of several insect pests. Lufenuron suppresses chitin synthesis, most likely by enzymatic interference, preventing the larvae from molting. Alpha-cypermethrin is a widely used pyrethroid pesticide. It has little water solubility, is relatively volatile, and, according to its chemical properties, does not leach into groundwater. However, it is classified as a significant water pollutant. This is due to its modest persistence in soil and its ability to remain in water. It is extremely poisonous to mammals and a known irritant. It is reasonably harmless to birds but extremely harmful to most aquatic species and bees. Alpha-cypermethrin is somewhat poisonous to earthworms. It is effective against a diverse spectrum of insect hosts in a variety of crops. Alpha-cypermethrin has both contact and intestinal activity and can be used to control insects in a variety°of crops, including cereals, cotton, fruits, vegetables, floriculture, oilseeds, sugar beet, tea, tobacco, and vines. Indoxacarb is an oxadiazine insecticide that inhibits voltage-gated sodium channels. It has been designed to be effective against scale insect larvae.

The beet armyworm is one of the scale pests that indoxacarb helps to control. Consequently, citrus fruits still contain a lot of pesticide residue, which could be harmful to people's health. European Union (EU) directives (Council directives 76/895/EEC, 86/362/EEC, 86/363/EEC, and 90/642/EEC) and Swiss legislation are among the rules put in place to protect people's health from pesticide residues and keep food safe for customers. Pesticide maximum residue limits (MRLs) in food have been set by Swiss regulation **(Swiss regulation RS 817.021.23)**. Many efforts have been made to increase the value of citrus by-products; for example, they can be used as animal feed, composted, or used in bio refinery processes to produce ethanol, biogas, and biofuels (**Zema *et al*., 2018**.). While the yearly use of pesticides—numbering in the millions of tons—helps cut down on food waste due to weeds, pests, and diseases, it also poses risks to ecosystems, wildlife, and human health (**Yang *et al*., 2023**). In addition, misapplication or other causes can cause pesticides and their residues to be carried to regions not intended for treatment via spray drift and runoff. According to the US Environmental Protection Agency [US EPA, 2019]) and **(Moeller, 2019)**, when growing citrus fruits, it is necessary to use pesticides in areas where these diseases and pests are common **(Besil *et al.*, 2018; EC, 2005; Li *et al*., 2012)**.

So, the present work was undertaken with a view to studying the behavior of three pesticides represented to various pesticide groups in fresh orange and tangerine fruits under field conditions.

MATERIALS AND METHODS

I- *Pesticide selected:*

The pesticides used and usage rate for commercial formulation were:

a. alpha-Cypermethrin (**Alpha –Syper 10% EC** at the rate of 250 cm3/100 Liter water.
b. indoxacarb (**Camvaal 15% EC** at the rate of 100 cm^3/100 Liter water.
c. lufenuron (**Match 5% EC** at the rate of 160 cm^3/100 Liter water.

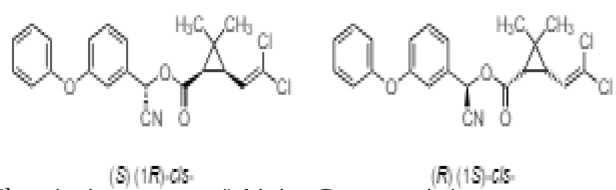

Fig. 1. Chemical structure of Alpha-Cypermethrin

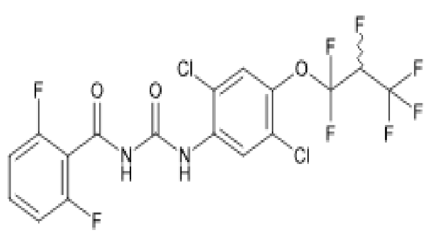

Fig. 2. Chemical structure of Indoxacarb

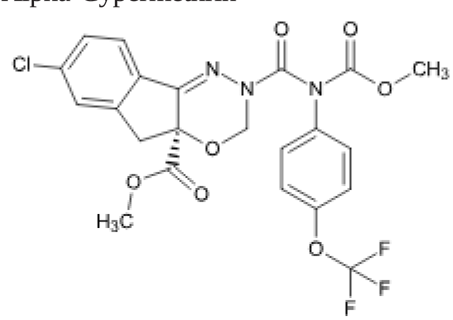

Fig. 3. Chemical structure of lufenuron

The pesticides were acquired from the Plant Protection Research Institute, Dokki, Giza, Egypt, and analyzed in the Central Agricultural Pesticide Laboratory, Agricultural Research Center, Dokki, Giza, Egypt.

2- ***Field experiment and sampling:***

Experiments were carried out at a private citrus field (Valencia Summer Orange and Tangerine Murcott variety) located at Wady El MulaK, Ismailia district, Ismailia governorate, Egypt. The experimental tree was 15 years' old, the height of the trees 1.5 meters, and it was organized in a randomized block design with three replicates, featuring three trees in each replicate for every treatment as well as the untreated control.

Pesticide application rates were used based on the Ministry of Agriculture and Land Reclamation's recommendations for Alpha-Cypermethrin, Indoxacarb and lufenuron in grapes. Orange and tangerine trees were sprayed once throughout the fruiting period with a backpack hand sprayer equipped with a single nozzle. To determine the residues of the investigated pesticides, representative samples of orange and tangerine 2 kilogram fruits were randomly collected from the experimental plots at intervals of 2 hours, 1, 2, and 3 weeks following application.

3- Extraction, cleanup techniques, and residue determination:

Orange and tangerine samples were extracted and purified utilizing the QuEChERS method **(Anastassiades *et al*., 2003)**. A 50 ml centrifuge tube was filled with homogenized oranges and tangerines (10 g of fruit), 15 milliliters of acetonitrile, acidified with 1.0% acetic acid were poured into a centrifuge tube and agitated vigorously for a minimum of one minute. Subsequently, added 4 grams of anhydrous magnesium sulfate and 1 gram of sodium acetate and the mixture was vigorously agitated for 5 minutes. The mixture was subsequently centrifuged at 3000 rpm for approximately 5 minutes. A 5 ml aliquot of the purified liquid was put into a 15-ml centrifuge tube and subsequently combined with 50 mg of PSA, 10 mg of graphitized carbon black, and 150 mg of magnesium sulfate. The tube was subsequently centrifuged at 6,000 rpm for a duration of 10 minutes. The liquid containing indoxacarb and lufenuron was collected for HPLC analysis using an Agilent 1260 HPLC. The mobile phase for indoxacarb was acetonitrile/water at a ratio of 75:25 with a wavelength of 254 nm, while for lufenuron, it was acetonitrile/water at a ratio of 65:35 with a wavelength of 230 nm. The mobile phase was administered at a flow rate of 1 ml per minute with an injection volume of 20 µl. Under these conditions, the optimal separation rate and sensitivity were achieved with retention durations of 3.531 and

5.084 minutes for Indoxacarb. For the Agilent Technologies alpha-Cypermethrin 7890A Gas Chromatograph with an Electron Capture Detector (ECD) in the specified operating conditions:

Column: DB-17 (15 m × 0.32 mm × 0.52 µm film thickness).

Column temperature: 240 °C for one minute, then increased to 300 °C at a rate of 10 °C/min. 1.5 ml/min nitrogen as the carrier gas, with N_2/Air makeup gas at 30 ml/min.

Injector temperature: 280 °C.

Detector temperature: 300 °C.

Carrier gas: (N2).

Purge flow rate: 9 mL/min.

4- *Recovery rates and statistical analysis*

In order to assess the efficiency of the extraction, purification, and final analysis methods, three untreated fruit samples were fortified with known concentrations (0.01, 0.1, and 1 mg/kg) of the active component from the solutions of the three pesticides being examined. As could be noticed in Table (1) recovery percentages were ranged between 96-101,97-103 (alpha-cypermethrin),94-99, 98-101 (indoxacarb) and 91-96, 92-98%(lufenuron) on orange and tangerine respectively. All acquired data underwent statistical analysis and were visually represented in accordance with **Timme and Fisher (1980)**. The half-life ($t_{1/2}$) was calculated mathematically according to **Moye *et al.* (1987)** from the following equations:

$$t_{1/2} = \text{Ln } 2/K$$

$$K_0 = (1/t_x).\ \text{Ln } a/bx.$$

Where:

K_0: is the degradation rate constant at the intervals in hours, K: is the mean of $K_{0,}$ a: the residue level at the initial time (zero time), bx: is the residue level at x time.

The amount of residues is calculated by the following equation according to **Brennecke (1985)**:

$$R = \frac{Fa.Wst.Vend}{Fst.G.Vi} F$$

Where:

F_a: Peak area of sample solution, F_{st}: Peak area of standard solution, W_{st}: Amount injected of standard solution (ng),G: Samples weight (g), V_{end}: Terminal volume of sample solution, V_i: Portion of terminal volume injected (µl), F: Recovery factor: 100 / % of recovery.

Table 1. Average of recovery rates of the three pesticides under study.

Pesticide	Sample types	Spiking level (mg./kg.)	Average detected concentration (mg/kg)	Mean recovery %
Alpha-Cypermethrin	**Orange**	1 0.1 0.01	0.96±0.213 0.1±0.0175 0.0101±0.0019	96 100 101
	Tangerine	1 0.1 0.01	0.97±0.0035 0.0101±0.0021 0.0103±0.0012	97 101 103
Indoxacarb	**Orange**	1 0.1 0.01	0.94±0.031 0.97±0.001 0.0099±0.0019	94 97 99
	Tangerine	1 0.1 0.01	0.98±0.027 0.099±0.0041 0.0101±0.0033	98 99 101
lufenuron	**Orange**	1 0.1 0.01	0.91±0.25 0.093±0.0248 0.0096±0.0031	91 93 96
	Tangerine	1 0.1 0.01	0.92±0.015 0.095±0.029 0.0098±0.0073	92 95 98

RESULTS AND DISCUSSION

Residues and the correlated criteria of alpha-cypermethrin-indoxacarb and Lufenuron pesticides are presented in Tables (2, 3 and 4) and Figures. (4, 5 and 6). It is obvious that the initial quantities measured 2 hrs.' spraying Orange and Tangerine were 2.55, 2.67 - 1.88, 1.92, and 3.1, 3.4 mg/kg for alpha-cypermethrin, indoxacarb, and lufenuron, respectively. It is obvious to note that there is a positive relationship between the absorption of the evaluated insecticides on the recipient surface of orange and tangerine and their use rate. Also, the initial amounts in tangerine fruits were, however, higher than orange fruits. This finding is a result of the fact that tangerine fruits have a larger surface area per unit weight than orange fruits.

Data also, revealed that during the experimental period, levels of the tested insecticide residues were much higher decreased with alpha-cypermethrin treatment than that indoxacarb and lufenuron ones. The Figures of the rate of pesticide degradation as well as the half-lives (t½) reinforce this occurrence; nevertheless, the figures of degradation rates in the case of orange are greater than the residue of tangerine, and the inverse of the case can be seen with the figures of (t½). After one week of spraying, the above-mentioned initial amounts were degraded in treatments of alpha-cypermethrin faster than that of indoxacarb and lufenuron ones and reached 0.51,0.55 (alpha-cypermethrin), 0.81,0.83 (indoxacarb), and 1.95,2.2 mg/kg (lufenuron) in orange and tangerine, respectively. Recording loss percentages ranged between 79.4-80%, 56.77-56.9%, and 35.29-37.09% with alpha-cypermethrin-indoxaarb and lufenuron, respectively.

After two weeks' undetectable amounts of alpha-cypermethrin were recorded in the tow tested fruits, recording 100% loss (table 2). While in indoxacarb treatments, it was found that 0.03 and 0.04 mg/kg indoxacarb were determined, indicating 98.4% and 97.92% loss in orange and tangerine fruits, respectively (table 3). At the same time, residues of lufenuron determined two weeks after treatment were 0.42 and 0.98 mg/kg, recording loss percentages of 86.45 and 85.98% in orange and tangerine fruits, respectively (table 4). At the third week after treatment, undetectable amounts of indoxacarb and lufenuron were recorded (Tables 3 and 4), indicating 100% loss in the two tested fruits (Tables 3 and 4).

Likewise, our results agree with those obtained regarding the characteristics of the pesticide, such as its total stability, volatility, solubility, and formulation, the sort of crops being treated, and the timing and location of pesticide application **(Malhat *et al*., 2016; Malhat *et al*., 2017** and **Bletsou *et al.*, 2013**).

Furthermore, the growth dilution factor can also impact pesticide residue concentrations while still in the field. Plant growth can decrease concentrations of pesticide residues as a result of growth dilution effects **(El-Hefny and Abdel-Hamid, 2017).** On the other hand, our results disagree with **(Li *et al*., 2022; Liu *et al.*, 2016; Wang *et al.*, 2023; Wang *et al.*, 2021b; Zhao *et al.*, 2020).** In the cultivation of citrus, pesticides that remain in the plant for a long period with a pre-harvest interval (PHI) like lufenuron and indoxacarb estimated to be higher than pesticides with a shorter interval, such as pyrethroids, should not be used to ensure no contamination and low pesticide residues.

Table 2. Residues of alpha-cypermethrin detected in orange and tangerine.

intervals (weeks)	Residues (mg/kg)	% Loss	% Persistence	Residues (mg/kg)	% Loss	% Persistence
	Orange			Tangerine		
initial	2.55±0.04	0	100	2.67±0.02	0	100
1	0.51±0.03	80	20	0.55±0.04	79.4	20.6
2	UND	100	0	UND	100	0
3	UND	100	0	UND	100	0
EU MRL(mg/kg)	2			2		
PHI (days)	2			2.1		
t½ (days)	2.4			2.5		

UND = Undetectable Amounts, t½= Half-life, MRL= Maximum Residue Limit, PHI= Pre harvest Interval

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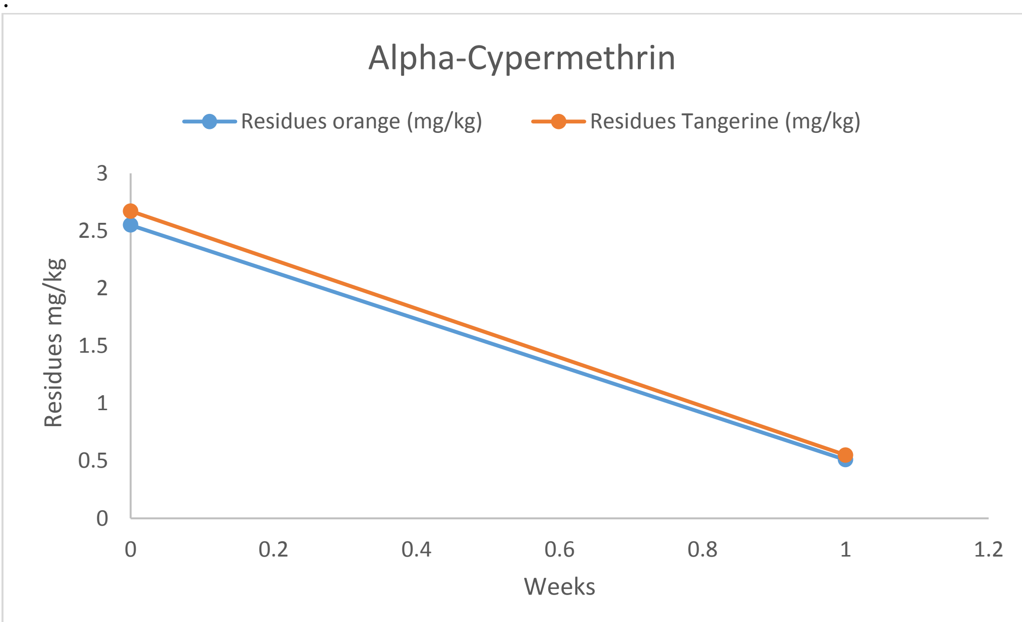

Figure 4: Dissipation behavior of alpha-cypermethrin in orange and tangerine under field conditions.

Table 3. Residues of indoxacarb detected in orange and tangerine

Intervals (weeks)	**Residues (mg/kg)**	**% Loss**	**% Persistence**	**Residues (mg/kg)**	**% Loss**	**% Persistence**
	Orange			**Tangerine**		
initial*	1.88±0.06	0	100	1.92±0.04	0	100
1	0.81±0.02	56.91	43.09	0.83±0.02	56.77	43.23
2	0.03±0.01	98.4	1.6	0.04±0.03	97.92	2.08
3	UND	100	0	UND	100	0
EU MRL(mg/kg)	0.01			0.01		
PHI (days)	15.3			15.5		
t½ (days)	4.2			4.6		

UND = Undetectable Amounts, t½= Half-life, MRL= Maximum Residue Limit, PHI= Pre harvest Interval.

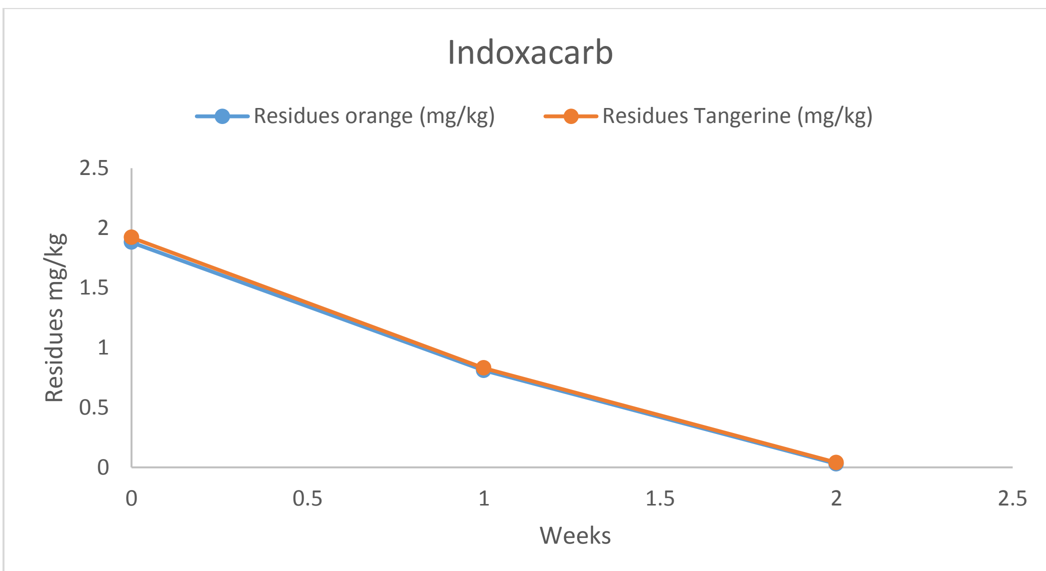

Figure 5: Dissipation behavior of indoxacarb in orange and tangerine under field conditions.

Table 4. Residues of lufenuron detected in orange and tangerine.

intervals (weeks)	Residues (mg/kg)	% Loss	% Persistence	Residues (mg/kg)	% Loss	% Persistence
	Orange			**Tangerine**		
initial*	3.1±0.4	0	100	3.4±0.2	0	100
1	1.95±0.05	37.09	62.91	2.2±0.1	35.29	64.71
2	0.42±0.02	86.45	13.55	0.48±0.01	85.88	14.12
3	UND	100	0	UND	100	0
EU MRL(mg/kg)	0.01			0.01		
PHI (days)	18.3			19.2		
t½ (days)	4.71			5.69		

UND = Undetectable Amounts, t½= Half-life, MRL= Maximum Residue Limit, PHI= Pre harvest Interval.

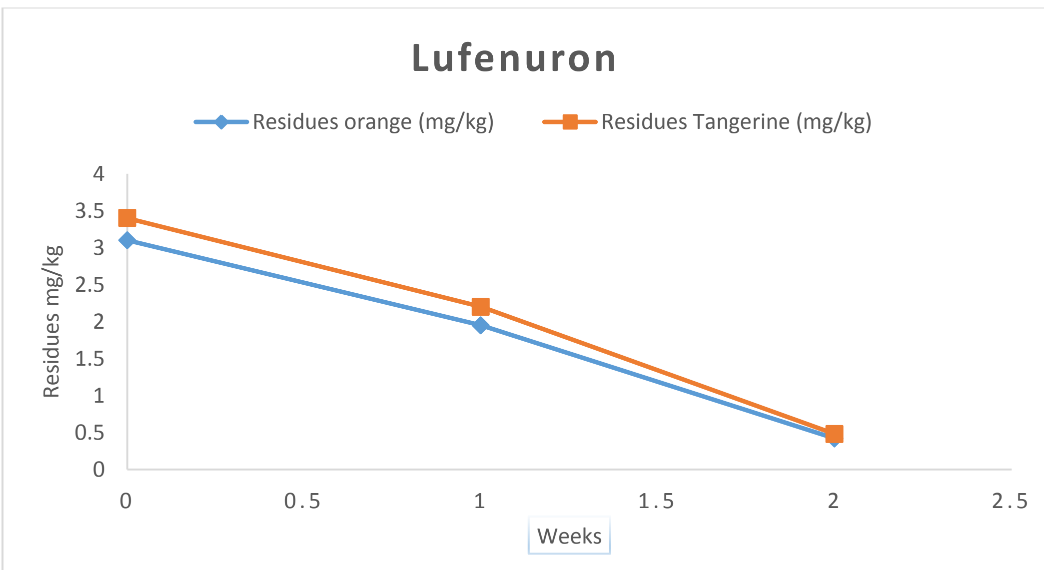

Figure 6: Dissipation behavior of lufenuron in orange and tangerine under field conditions

In conclusion:

The research successfully isolated residues of alpha-cypermethrin, indoxacarb, and Lufenuron in oranges and tangerines utilizing the QuEChERS technology in conjunction with HPLC and GC. The findings demonstrated satisfactory accuracy and precision. The proposed methodology was corroborated in compliance with the **SANTE/12682/2019** criteria. The established approach met the recovery criteria and was successfully employed to investigate the dissipation kinetics of Alpha-Cypermethrin, indoxacarb, and lufenuron in oranges and tangerines in field conditions. The degradation rates of three pesticides applied to oranges and tangerines were assessed under field circumstances in Egypt. The results indicated that the degradation rate of three insecticides was more rapid on oranges than on tangerines, with calculated half-lives (t0.5) of [2.4, 4.2, and 4.71 days for Alpha-Cypermethrin, indoxacarb, and lufenuron in oranges, respectively, while in tangerines, the half-lives for alpha-cypermethrin, indoxacarb, and lufenuron were 2.5, 4.6, and 5.69 days, respectively.

Complete dissipation of residues was observed below the detectable level at the recommended rate of application on both oranges than tangerine by the 2 days. The pre-harvest interval (PHI) for alpha-cypermethrin on oranges and tangerine and 15.3–15.5 days for indoxacarb and for lufenuron were 18.3–19.2 days in oranges and tangerine based on the European Union maximum residue levels (MRL) after treatment at the recommended dose. The residues were found to be under the detectable level of maximum residue levels (2 mg/kg) for alpha-cypermethrin and 0.01 mg/kg for indoxacarb and lufenuron in both commodities, in oranges and tangerine, respectively; therefore, we can say that Alpha-Cypermethrin is more safe to use than indoxacarb and lufenuron in oranges and Tangerine orchard. Especially in exports abroad.

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تقدير متبقيات ألفا سايبرمثرين – إندوكساكارب ولوفينورون على البرتقال واليوسفي.

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٢ – معهد بحوث وقاية النبات ، الدقي ، جيزة ، مصر

٣ – قسم تلوث البيئة ، ومتبقيات المبيدات ، معمل المبيدات الحشرية الزراعية ، مركز البحوث الزراعية ، الدقي ، الجيزة ، مصر

أجري هذا البحث لتقدير متبقيات مستحضرات ألفا سايبرمثرين (ألفا سايبر ١٠٪ EC بمعدل ٢٥٠ سم٣ / ١٠٠ لتر ماء) إندوكساكارب (كامفال ١٥٪ EC بمعدل ١٠٠ سم٣ / ١٠٠ لتر ماء) و لوفينورون (ماتش ٥٪ EC بمعدل ١٦٠ سم٣ / ١٠٠ لتر ماء) على محصولى البرتقال واليوسفى فى وادى الملاك بمحافظة الأسماعيلية، بعد تطبيقه بالمعدل الموصي به في الحقول المصرية المفتوحة. وقد تم ذلك باستخدام QuEChERS للاستخلاص والتنظيف، مع أجهزة HPLC - GC عند التحقق من صحة طريقة لتحليل المبيدات الحشرية في الثمار، هناك معايير معينة يجب الوفاء بها. وتشمل هذه الخطية، والحد الكمى (LOQ)، والدقة فيما يتعلق بمعدل الأسترجاع. في اختبارنا، وجدنا أن معدلات الأسترجاع للطريقة عند مستويات استرجاع المختلفة (٠.٠١، ٠.١، و ١.٠ مجم / كجم) تتراوح من ٩١٪ إلى ١٠٣٪ للمبيدات الحشرية المستخدمة في كلا المحصولين. أظهرت النتائج أن بعد الرش مباشرة لألفا سايبرمثرين وإندوكساكارب ولوفينورون في البرتقال كان ٢.٥٥ و١.٨٨ و٣.١ و٢.٦٧ و١.٩٢ و٣.٤ جزء في المليون في اليوسفي. وكانت نسبة الخفض في متبقيات المبيدات أكبر في اليوسفي منها في البرتقال. وكانت فترات نصف العمر لألفا سايبرمثرين وإندوكساكارب ولوفينورون ٢.٤ و٤.٢ و٤.٧١ يومًا في البرتقال و٢.٥ و٤.٦ و٥.٦٩ يومًا في اليوسفي. ويمكن استهلاك البرتقال واليوسفي الملوثين بأمان بعد الأنتظار لمدة أسبوعين قبل جمع المحصول

التوصية: وكانت نسبة الخفض في متبقيات المبيدات أكبر في اليوسفي منها في البرتقال ، ويمكن استهلاك البرتقال واليوسفي الملوثين بأمان بعد الأنتظار لمدة أسبوعين قبل جمع المحصول

. **الكلمات المفتاحية :**: متبقيات – QuEChERS – ألفا سايبرمثرين - إندوكساكارب -لوفينورون - البرتقال - اليوسقى