Egypt. J. Chem. **Vol. 65,** No. SI:13B pp. 25 - 31 (2022)

Egyptian Journal of Chemistry

http://ejchem.journals.ekb.eg/

Dissipation of Cyflumetofen and Sulfoxaflor Residues in Cucumber fruits (*Cucumis sativus*) under Greenhouse Conditions using the QuEChERS Method and HPLC/DAD

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Abstract

Determination of cyflumetofen and sulfoxaflor residues in treated cucumber samples (*Cucumis sativus*), which were collected randomly 0, 1, 2, 3, 5, 7, 10, and 15 days after treatment using quick, easy, cheap, effective, rugged, safe (QuEChERS) and cleanup step utilizing dispersive solid-phase extraction (DSPE) and followed by determination high-performance liquid chromatography with diode-array detection (HPLC/DAD). Cyflumetofen was recovered within 87.60-99.43%, while sulfoxaflor was recovered within 80.85-100.14%, with relative standard deviations (RSDs), ranging from 3.97-6.21% and 3.46-5.94% respectively, in cucumber samples at the spike levels (0.1 - 1 mg/kg). Good linearity was achieved for cyflumetofen with an excellent correlation coefficient of R^2> 0.99and sulfoxaflor also showed R^2> 0.98. The initial deposits of cyflumetofen and sulfoxaflor averaged 1.44 and 2.29 mg/kg, respectively. The half-life period (RL_{50}) of cyflumetofen and sulfoxaflor were 0.60 and 1.31 days, respectively. According to the maximum residue level (MRL), which reached 0.4 mg/kg for cyflumetofen and 0.5 mg/kg for sulfoxaflor, the pre-harvest interval (PHI) was 3 days and 7 days, respectively.

Keywords: Cyflumetofen; Sulfoxaflor; Dissipation; QuEChERS; HPLC/DAD and Cucumber.

1. Introduction

Cucumber (*Cucumis sativus*) is one of the most popular greenhouse vegetable products growing worldwide. Cucumber, which is related to melons, such as watermelon, cantaloupe, and honeydew, is characterized by a relatively low-calorie food at just about 15 calories per cup and about 95% water. They contain high levels of lignans, vitamin K, cucurbitacins, and their derivatives (triterpenoids), flavonoids (apigenin, luteolin, quercetin, and kaempferol), antioxidants such as beta carotene and vitamins C, and B, among other trace elements and minerals [1, 2]. With such a high level of water content and the bonus of naturally occurring nutrients and trace minerals, cucumber could be a great supplement to drinking water or even serve as an alternative to consuming sports drinks.

Cucumber plants are infested with several insect and mite pests. Aphids, such as *Aphis gossypii* (Glover) (Homoptera: Aphididae) and the two-spotted spider mite *Tetranychus urticae* (Koch). (Acari: Tetranychidae), are considered among the most important pests that infest cucumber plants under greenhouse conditions [3, 4, 5, 6]. Recently, growing vegetables under protected cultivation in Egypt has been expanding rapidly. The number of single-arch greenhouses reached about twenty thousand, where about 12000 (60%) are used only for cucumber production [7]. The climate in greenhouses is essentially warm, humid, and wind-free which encourages different kinds of pest development causing serious damage and high yield losses [8].

Cyflumetofen, 2-methoxyethyl(RS)-2-(4-tert-butylphenyl)-2- cyano-3-oxo-3-(-trifluoro-o-toll) propionate, was developed by OtsukaAgriTechno (Osaka, Japan) in 2007, and its chemical structure was shown in Fig. 1. Cyflumetofen was a relatively novel benzoyl acetonitrile acaricide, new inhibitors of complex I of the mitochondrial electron transport chain with activity as pesticides and active against

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DOI: 10.21608/EJCHEM.2022.100756.4884

phytophagous mites especially spider mites at all development stages and can play a suitable role in integrated pest management systems (IPM) without cross-resistance to existing acaricides [9]. Meanwhile, this product is safe for predatory phytoseiidae mites as well as other non-target organisms [10,11]. Cyflumetofen shows excellent efficacy for the control of a wide range of pests in fruits, ornamentals, vegetables, and other crops. The use of cyflumetofen has increased dramatically in recent years to control spider mites under greenhouse conditions [12].

Sulfoxaflor was developed by Dow AgroSciences in 2010. It was the novel sulfoximine type [13,14]. Unlike, the neonicotinoids and other nAChR-acting insecticides, sulfoxaflor has a unique action on nicotinic acetylcholine receptor nAChR in the insect nervous system [15,16]. Sulfoxaflor was classified to Group 4C by the Insecticide Resistance Action Committee [17]. Due to its special action mechanism, it has been demonstrated an excellent control on sucking pests, lying within the families Aleyrodidae, Aphididae, Delphacidae, Margarodidae, and Miridae, even resistant populations [15,18]. In China, it has been registered as a 50% WDG against *Aphis Lucorum* (Meyer-Dur) infesting cotton crop (http://www.chinapesticide.gov.cn). Under field conditions, the relatively high longevity of *A. lucorum* adults (up to 30 days) [19] and insecticide degradation possibly induce sub-lethal effects [20].

Many farmers, who cultivate the cucumber for export purposes, have used chemical pesticides with low MRL (Maximum Residue Limits) of main export target countries or simultaneous multi-residue analysis in Egypt has not been established. Accordingly, cyflumetofen and sulfoxaflor are selected to determine the PHI (pre-harvest interval) in cucumber growing in a greenhouse. In addition, checkup weather is suitable or not for main export target countries.

Evaluate pesticide residues in food matrices. This is a tremendous challenge mainly because the amounts of analytes are small compared to the huge quantities of interfering substances that strongly interact with analytes [21]. QuEChERS has been developed as a new sample preparation methodology between 2000 and 2002 for pesticide multiresidue analysis [22]. Generic extraction procedures, like the QuEChERS method and ultra-high-performance liquid chromatography systems combined with polar embedded C18 phases, providing excellent peak shape and good resolution, enabled us to detect a wide spectrum of compounds that belong to different pesticides classes and chemical properties in each sample [22, 23].

The objective of the present work was to determine the dissipation behavior and the residues of cyflumetofen and sulfoxaflor in cucumber fruits under greenhouse conditions.

2. Materials and Methods

2.1. Chemicals and reagents

Certified reference standards of cyflumetofen Fig.1 and sulfoxaflor Fig.2 were obtained from Central Agricultural Pesticides Laboratory (CAPL), Egypt. Acetonitrile of HPLC grade was purchased from Merck, and bulk primary secondary amine (PSA) sorbent (Bondesil-PSA, 40 µm) was bought from Subiaco. Anhydrous magnesium sulfate and sodium chloride were purchased from Merck. Anhydrous magnesium sulfate and sodium chloride were activated by heating at 250ºC for 4 h in the oven before use and kept in desiccators. Pesticide technical formulations Cyflumetofen (Danisaraba 20% SC), was supplied by Arista Life Science Company, Egypt with an application rate of 30 ml/100 L. Sulfoxaflor (Closer 24% SC) was supplied by Dow Chemical Company, Egypt with an application rate of 10 ml/100 L.

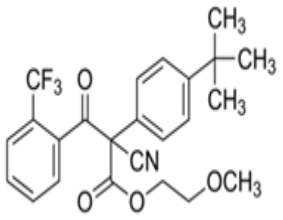

Fig.1 Structure of cyflumetofen

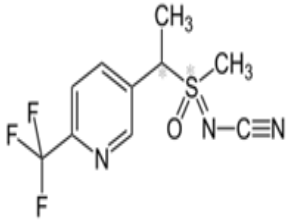

Fig.2 Structure of sulfoxaflor

2.2. Instrument conditions

The HPLC system, an Agilent 1260 series equipped with an analytical column (150 mm × 4.6 mm id × 5 µm ODS) attached to a photodiode array detector. The flow rate of the mobile phase (acetonitrile 30% + water 70%) was 1 ml/min and the injection volume was 20 µl. The detection wavelength was set at 210 nm for sulfoxaflor. The retention time for sulfoxaflor was 2.3 min., while the mobile phase flow rate (acetonitrile 70% + water 30 %) was 1 ml/min for Cyflumetofen and the injection volume was 20 µl. The detection wavelength was set at 225 nm. The retention time for Cyflumetofen was 9.5 min.

2.3. Standard preparation

Stock solution: Each pesticide's 1000 ug/ml reference standard solution was prepared in acetonitrile. Intermediate solutions: 100 ug/ml mixture standards were prepared by diluting stock

solution in acetonitrile. Calibration solutions: Calibration mixtures of concentration levels 0.05, 0.1, 0.5, 1, 5 and 10 ug/ml were prepared in acetonitrile.

2.4. Field experiments

Field experiments were carried out in the experimental Farm of Faculty of Agric., Cairo Univ. in 2019. The soil of the greenhouse was well prepared before the plantation of cucumber var., (Alnafis). The experimental area was divided into four plots (treatments), each of which contained 50 plants. The four plots were sprayed with the recommended rate of danisaraba 20% SC at 30 ml/100 L and closer to 24% SC at 10 ml/100 L. A manual compressor sprayer (20 liters capacity) was used for pesticides applications. Each treatment was replicated four times according to a complete randomized block design.

2.5. Sampling and storage

Fruit samples were collected after 40 days from planting seeds of cucumber. Samples were representative of all plants in the area, where two Kg of cucumber fruits were randomly collected from each untreated and treated area with cyflumetofen and sulfoxaflor. First clean samples of cucumber were collected from the control area, and then treatment of plants started on the previously mentioned dates, and sampling took place 2 h after application of the initial deposits and repeated 1, 2, 3, 5, 7, 10, and 15 days afterward to study the dissipation of the tested pesticides. Field samples were transported in iceboxes to the laboratory and stored at -5 °C.

2.6. Extraction and clean up

The samples were prepared with the QuEChERS method [21,22]. 10g of homogenized cucumber sample was weighed into a 50 ml PTFE centrifuge tube. After that, 10 ml of acetonitrile were added. The tube was vigorously hand-shaken for 1 min, where 4g of anhydrous $MgSO_4$ plus 1 g of sodium chloride were added. The tube was hand-shaken for 30 s., and the mixture was centrifuged at ≤4000 rpm for 5 min. Acetonitrile 1.0 ml was transferred into a 2.0 ml centrifuge tube for cleanup. An aliquot of 1.0 ml was transferred into the DSPE tubes containing 25 mg PSA and 150 mg $MgSO_4$). The tubes were well capped and vortexed for 30 s., then centrifuged for 5min at ≤4000 rpm. The combined eluate was filtered through a 0.22 µm nylon syringe filter into an auto-sampler vial for HPLC injection.

3. Method validation

The method linearity was calculated from the results directly proportional to the concentration of tested pesticide in the solvent. Linearity was assessed by the correlation coefficient (R^2) resulting from the five-point calibration curve at levels of 0.05, 0.1, 0.5, 1, 5, and 10 ug/ml prepared in acetonitrile). Matrix-matched calibration was used to compensate for the matrix effects. The matrix effects were defined as the influence of one or more co-extracted components from the sample on the measurement of tested pesticide concentration. The presence of these effects is demonstrated by comparing the response produced from the tested pesticide in a pure solvent solution with the samples were first extracted and then spiked with a tested pesticide in the same solvent at the same concentration levels (0.05, 0.1, 0.5, 1, 5 and 10 mg/kg).

Matrix effects (%ME) were calculated using the following equation:

$$\text{ME \%} = \frac{\text{M matrix} - \text{M solvent}}{\text{M solvent}} X100\%$$

Where

ME: The matrix effect

M matrix: Slope of the calibration curve in a matrix.

M solvent: Slope of the calibration curve in the pure solvent.

The trueness, means of recovery study was carried out on untreated cucumber samples by fortifying five replicates of the samples with tested pesticides standards at three levels ranging from 0.1 to 1 mg/kg by spiking 10 g of blank samples with the standard solution. The obtained mean recoveries range from 87.60% to 99.43% with relative standard deviation (RSDs) ranging from 3.97 to 6.21 for Cyflumetofen and range from 80.85% to 100.14% with relative standard deviation (RSDs) ranged from 3.46 to 5.94 for sulfoxaflor [23]. The obtained mean recoveries were within the acceptable range (70-120%). So, the value indicates that the method was sensitive and able to detect and quantify the analyte at low levels, and it is suitable for the determination of tested pesticide residue in cucumber.

Trueness was calculated using the following equation:

$\% R = (X/\mu) \times 100$

%R: recovery percentage

X: experimental concentration of cyflumetofen and sulfoxaflor mg/kg

µ: calculated concentration of cyflumetofen and sulfoxaflor mg/kg

The repeatability precision (RSDr) involved repeat recovery levels (0.1, 0.5, and 1 mg/kg), five replicates for each level per day on three different days. The (RSDr) value ranged from 4.58 - 6.21% for Cyflumetofen and 4.81-5.94% for sulfoxaflor [23] the obtained (RSDr) value was within the acceptable range ≤20%.

RSDr = (SD / M) × 100

Where

SD: standard deviation of the replicates.

M: the mean value of the recovery.

4. Statistical analysis

The dissipation kinetics of cyflumetofen and sulfoxaflor residues in cucumber was determined by plotting residue concentration against elapsed time after application and equations of best curve fit with maximum coefficients of determination (R^2) were determined. For dissipation of cyflumetofen and sulfoxaflor in cucumber, exponential relationships were found to be applied corresponding to the general first-order kinetics equation: $Ct=C0e^{-kt}$

Where Ct represents the concentration of the pesticide residue at the time of t, C0 represents the initial deposits after application, k is the constant rate of pesticide dissipation per day. This equation determined the dissipation half-life periods (t1/2=ln 2/k), [24, 25, 26] of the studied insecticides.

5. Results and Discussion

5.1. Validation Method

The evaluations of calibration curve linearity of cyflumetofen and sulfoxaflor were done based on the injection of standard solutions prepared in pure organic solvent methanol in series at 0.05, 0.1, 0.5, 1, 5, and 10 mg/kg for HPLC injection. Standard calibration curves of cyflumetofen and sulfoxaflor were constructed by plotting analyte concentrations against peak areas. The correlation coefficient (R^2= 0.99 and R^2= 0.98).

In the pesticide residues analysis study, the injected sample contained large amounts of the unavoidably present co-extractives responsible for the matrix effects occurring on the injector. The matrix effect was investigated by comparing the slopes of calibration curves at 0.05, 0.1, 0.5, 1, 5, and 10 mg/kg of cyflumetofen and sulfoxaflor in cucumber in pure solvent. The ME% could be either negative or positive and would be classified into three categories: no matrix effect (between -20% and 20%), medium matrix effect (between -50% and -20%), and strong matrix effect (below -50% or above 50%) [27]. The obtained results showed that the matrix effect for cyflumetofen and sulfoxaflor was 40.97% and -14.84% in cucumber Fruits, for which there was no interfering endogenous peak appeared and did not significantly suppress or enhance the response of the instrument.

The lowest validated level of cyflumetofen and sulfoxaflor with acceptable precision and trueness LOQ was 0.1 ug/g for HPLC injection analysis in cucumber, respectively. According to [21], the LOQ values are acceptable where LOQ ≤. MRL 0.1ug/g for cyflumetofen and sulfoxaflor.

The trueness, means of recovery was carried out in 5 replicates at 3 fortification levels (1, 0.5, and 0.1 mg/kg) by spiking 10 g of blank samples with a standard solution. The obtained mean recoveries ranged from 87.60% to 99.43% with relative standard deviation (RSDs) ranging from 3.97 to 6.21 for cyflumetofen and range from 80.85% to 100.14%with relative standard deviation (RSDs) ranged from 3.46 to 5.94 for Sulfoxaflor. According to [23], the obtained mean recoveries were within the acceptable range (70-120%). So, the value indicates that the method was sensitive and able to detect and quantify the analyte at low levels, and it is suitable for the determination of tested pesticide residue in cucumber.

The repeatability precision (RSDr) involved repeat recovery levels (0.1, 0.5, and 1 mg/kg), five replicates for each level per day on three different days. The (RSDr) value ranged from 4.58to 6.21 for Cyflumetofen and 4.81to 5.94 for sulfoxaflor According to (SANTE, 2017) the obtained (RSDr) value was within the acceptable range ≤20%.

5.2. Dissipation of Cyflumetofen and Sulfoxaflor in cucumber fruits

Dissipation of cyflumetofen and sulfoxaflor in cucumber was studied for 15 days. The dissipation pattern of cyflumetofen and sulfoxaflor in cucumber at different sampling intervals (0, 1, 2, 3, 5, 7, 10, and 15 days) is presented in table (1, 2) and fig. (3) The initial deposit of cyflumetofen and sulfoxaflor in cucumber was 1.44 mg/kg and 2.29 mg/kg two hours after application. The cucumber residue of cyflumetofen and sulfoxaflor decreased to 0.85 mg/kg and 1.95 mg/kg with 40.97% and 14.84% loss. The rapid dissipation was found on the third day after spraying, which cyflumetofen and sulfoxaflor residue reached 0.26 mg/kg and 1.08 mg/kg with 52.83% and 81.94% loss. The degradation continued to reach 0.05 mg/kg and 0.82 mg/kg with 96.52% and 64.19% loss after 5 days after application. The residues of cyflumetofen and sulfoxaflor on cucumber. The residues became undetectable on the 15th day after application. Based on the previous results, the calculated half-life period (RL_{50}) of cyflumetofen and sulfoxaflor on cucumber was 0.26 and 1.08 days. The maximum residue limit (MRL) of cyflumetofen and

sulfoxaflor on cucumber are 0.4 mg/Kg and 0.5 mg/Kg according to [28]. The results presented showed that cucumber can be consumed safely by humans after 3 days and 7 days from spraying with cyflumetofen and sulfoxaflor.

Table 1. Dissipation behavior of sulfoxaflor residue after application in cucumber fruits.

Time intervals (days)	Sulfoxaflor Residues (µg/g)±SD	Loss %
0	2.29±0.16	0.00
1	1.95±0.35	14.84
2	1.31±0.44	42.79
3	1.08±0.36	52.83
5	0.82±0.16	64.19
7	ND	---
10	ND	---
15	ND	---
MRL	0.5 (EU 2018)	
PHI	7 days	
LOD	0.05	
LOQ	0.1	

Table 2. Dissipation behavior of Cyflumetofen residue after application in cucumber fruits.

Time intervals (days)	Cyflumetofen Residues (µg/g) ± SD	% Loss
0	1.44±1.24	0.00
1	0.85±1.01	40.97
2	0.60±2.97	58.33
3	0.26±2.42	81.94
5	0.05±0.92	96.52
7	ND	---
10	ND	---
15	ND	---
MRL	0.4 (EU 2020)	
PHI	3 days	
LOD	0.05	
LOQ	0.1	

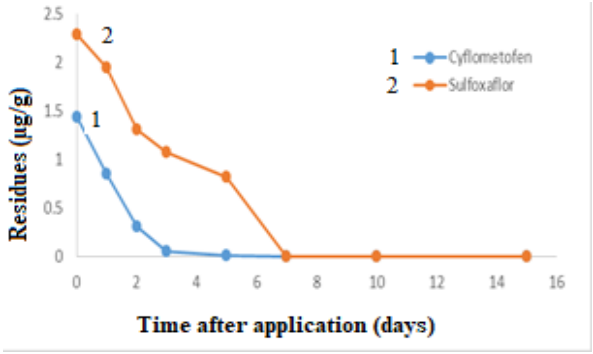

Fig. 3. Dissipation behavior curve of sulfoxaflor and Cyflumetofen after application in cucumber fruits.

5.3. Discussion

The evaluations of calibration curve linearity of cyflumetofen and sulfoxaflor were done with HPLC injection. The correlation coefficient (R^2= 0.99 and R^2= 0.98). The results showed that the matrix effect for cyflumetofen and sulfoxaflor was -40.97% and -14.84% in cucumber, respectively. Which indicated that no interfering endogenous peak appeared and did not significantly suppress or enhance the response of the instrument.

The residual amount of cyflumetofen and sulfoxaflor were rapidly decreased during the third day of spraying followed by gradually decreasing and full dissipation until the end of the experimental period, the lowest dissipation value was on the 5th day of application, while the highest value occurred in the first 24 hours of spraying. The cucumber could be used safely for human consumption after 3 days and 7 days from spraying with cyflumetofen and sulfoxaflor, because of the residues below the MRL of cyflumetofen and sulfoxaflor on cucumber fruits.

The highest dissipation rate of cyflumetofen and sulfoxaflor per day were found on the first day, a quick decline of cyflumetofen and sulfoxaflor concentration mainly during the first three days was observed followed by a gradual decrease until the 7th day after treatment. In the same concept, [29] reported that 3 days and 7 days after application by cyflumetofen and sulfoxaflor are necessary to meet the European MRL requirements and zero days for the USA requirements. On the other hand, [30] found a maximum of 0.26 mg/kg and 0.82 mg/kg of cyflumetofen and sulfoxaflor was detected in leek sample sprayed three times at 3 days and 7 days before harvest intervals.

The effect of environmental conditions, dosage, and the interval training between application and harvest just on the dissipation of pesticide residues in crops, as well as the rapid dissipation of initially applied pesticide, is dependent on a range of environmental exposures such as sunlight and temperature; however, high temperature is reported to be the most important factor in reducing pesticides from the plant surface, and light plays an essential part in pesticide behavior in the environment. Pesticides may also be gradually declining to biological, chemical, or physical processes, or, if remaining in the field, due to dispersion by crop growth. [31, 32, 33,34].

6. Conclusion

In this work, an HPLC injection analytical method based on QuEChERS sample pretreatment procedures was used for the determination of cyflumetofen and sulfoxaflor in cucumber fruits. The developed method is easy and compatible with cyflumetofen and sulfoxaflor residue analyses. The mean recoveries ranged from 87.60% to 99.43% and 80.85% to 100.14% respectively, and repeatability of the method, expressed as the relative standard deviation, was lower than 3%. The calculated half-life period (RL_{50}) of tested pesticides on cucumber fruits were 0.60 and 1.31 days respectively, for cyflumetofen and sulfoxaflor. According to the maximum residue limit (MRL) [35,36], the pre-harvest interval (PHI) of cyflumetofen and sulfoxaflor cucumber was 3 days and 7 days after the treatment, respectively.

7. Conflicts of interest

There are no conflicts to declare.

8. Acknowledgments

The Faculty of Agriculture, Cairo University, Egypt, supported this work.

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