Journal of Pollution Effects & Control
ISSN: 2375-4397

Nasr, et al., J Pollut Eff Cont 2014, 2:1
DOI: 10.4172/2375-4397.1000107

Research Article Open Access

Determination of Imidacloprid and Tetraconazol Residues in Cucumber Fruits

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Rec Date: Feb 03, 2014, **Acc Date:** Apr 17, 2014, **Pub Date:** May 06, 2014

Abstract

The experimental design was a complete randomized blocks design with two treatments for imidacloprid and tetraconazole in addition to control. Each treatment includes three replicates. The application was carried out using knapsack sprayer equipped with one nozzle. Its residues on and in cucumber fruits collected after one hour, 1, 3, 5, 8, 11, 15 and 21 days from last spray were lower than the maximum residue limit (MRL) of imidacloprid and tetraconazole on cucumber fruits. The results obtained revealed that the residual level of imidacloprid was less than the maximum residual level (MRL=1 mg/kg) which recommended by Codex Alimentration Commission, and the residual level of tetraconazole was less than the maximum residual level (MRL=0.2 mg/kg) which recommended by Codex Alimentration Commission. Also, the results showed that tetraconazole high persistence ($t_{1/2}$=1.4 days) than imidacloprid ($t_{1/2}$=2.2 days) on cucumber fruits.

Keywords: Imidacloprid; Tetraconazol; Residues; Cucumber plants

Introduction

Vegetables are susceptible to insect and disease attacks, so pesticides are widely used. Therefore, residues of pesticides in raw foods could affect the ultimate consumers especially when freshly consumed. Many studies were carried out on pesticide residues in vegetables [1]. Cucumber plant Cucumis sativus is one of the most important vegetable crops in Egypt, this vegetable infested by several pests causing serious quantitative and qualitative damages. "Imidacloprid and tetraconazole" are pesticides recommended for use on cucumber to control pests.

Imidacloprid is an extensively used insecticide for crop protection in the world wide from the last decade due to its low soil persistence and insecticidal activity at low application rate [2]. It is fastest growing in sales as insecticide globally because of its low selectivity for insects and apparent safety for humans [3,4]. Tetraconazole belongs to azole group of chemicals and has low acute toxicity. It is broad-spectrum systemic fungicide. It has been registered in Egypt and various countries [5]. This fungicide is steroid demethylation inhibitors acting mainly on the vegetative stages of fungi by blocking the mycelial growth either inside or on the surface of the host plant. Tetraconazole is effective in controlling a broad spectrum of diseases such as powdery mildew and scab on fruit [6].

Hence, exposure to these insecticides may involve a large segment of the population, which includes agriculture workers and their families, those living in proximity to farms/orchards, and the general population who may be exposed through home application of pesticides or via residues on food [7-9]. Since the literature concerning the analysis of the residues of these pesticides in different matrices and their residues is limited. Therefore the present study was undertaken to determine the residues of "imidacloprid, tetraconazole" and their persistence in and on cucumber fruits in the open field.

Materials and Methods

Field experiments and sampling method

The experimental design was a complete randomized blocks design with two treatments for imidacloprid and tetraconazole in addition to control (42 m^2). Each treatment includes three replicates. Cucumber variety Prince f 1 was cultivated at El-Mahmoudia area, El-Behera Governorate, Egypt. The cucumber plants were transplanted on June 2009 on 42 rows (4 m×25 cm), 18 rows for each pesticide in addition to 6 rows for control.

Treatments	Added amount (µg)	Found amount (µg)	% Recovery	% Recovery average
Imidacloprid	25	28.98	115.92	117.5 ± 1.59
	50	59.55	119.1	
Tetraconazole	25	21.547	86.19	91.62 ± 5.43
	35	33.97	97.05	

Table 1: Recovery percentage of imidacloprid and tetraconazole from cucumber fruits

Cucumber plant at fruiting stage were treated with imidacloprid (20% S.C) on July 2009, at rate of 125 gai/ha (recommended dose) and tetraconazole (10% E.C) at rate of 50 g ai/ha (recommended dose), respectively to protect cucumber plant from insect infestation (whitefly) and powdery mildew, according to pest control program, Ministry of Agriculture, Arabic Republic of Egypt (2001). The application was carried out using knapsack sprayer equipped with one nozzle. The two insecticides were sprayed at dates presented in Table 1.

Cucumber fruits samples were collected from each replicate (one Kg) at time intervals of one hour after application (zero time), 1, 3, 5,

8, 11, 15 and 21 days after treatment with imidacloprid, as well as at 1 h after spraying and 1, 3, 5, 8, 11 and 15 days after treatment with tetraconazole. As soon as the fruits were picked up, they were transferred to the laboratory. The collected representative samples were placed in aluminum foil and kept deep frozen until analyses.

Analytical procedures

Imidacloprid insecticide extraction: Method of Blass [10] was used for extraction of imidacloprid from cucumber fruits with slight modification of substituting methanol instead of acetonitrile in extraction. Fifty grams of cucumber fruits were blended at high speed for about 3 minutes with 200 ml methanol and filtered. After evaporation of methanol extract by means of a vacuum rotary evaporator, the aqueous reminder was treated with 50 ml saturated sodium chloride solution and transferred into a separatory funnel and shaken vigorously with 100 ml n-hexane. Then the organic phase was discarded and the aqueous was shaken with 100, 50, and 50 ml methylene chloride in separatory funnel. The lower methylene chloride phases were collected and subsequently dried over anhydrous sodium sulfate. Then it was evaporated just to dryness using a rotary evaporator at 40°C.

Clean-up of extracts: Method of Blass [10] was followed for cleaning-up of the extracted samples of imidacloprid. The chromatographic column was prepared by adding 20 ml of HPLC-grade ethyl acetate to topped glass column (with a plug of glass wool) followed by 4.5 g of 5% deactivated florisil (60-100 mesh) as slurry in HPLC-grade ethyl acetate. The dry extract was dissolved in 5 ml of ethyl acetate and quantitatively transferred to the top of column. The column was eluted with 20 ml HPLC-grade ethyl acetate which was discarded. The imidacloprid was eluted from the column with 25 ml of HPLC-grade acetonitrile which was evaporated just to dryness as previously described and redissolved in an appropriate volume of HPLC-grade acetronitrile. Finally, the extract was filtered through a 13 mm, 0.45 µm nylon filter into a glass stopper test tube, then the residues were ready for analysis by HPLC.

HPLC determination: Estimation of imidacloprid residues were performed by Perkin Elmer, series 200 High pressure liquid chromatography equipped with diode array UV detector (wavelength of 270 nm) in the central laboratory of faculty of pharmacy. The column used was sphere-5 (C18) 5 µm, 220 mm×4.6 mm. And the mobile phase was (acetonitrile/water) (40/60 v/v). The flow rate used was 0.8 ml/min., and the injection volume was 20 µl. At this condition the retention time of imidacloprid was 3.2 min.

Tetraconazole fungicide extraction: Method of Mollhoff [11] was adopted for extraction of tetraconazole from cucumber fruits; methanol was used instead of acetone. Fifty grams samples were placed in the blender cup and a constant amount of methanol (2 ml/gram fruits) were added, then blended for three minutes and filtered. Extracts were shaken successively with 100, 50 and 50 ml of methylene chloride in separatory funnel after adding 40 ml of sodium chloride solution (20%); then the water phase discarded. The combined methylene chloride phases were dried by filtration through anhydrous sodium sulfate. Then, it was evaporated just to dryness using a rotary evaporated at 40°C.

Clean-up of extracts: The extract of tetraconazole was then cleaned up using ammonium chloride solution and filtered through hyflo-supercell as discribed by Johnson [12]. The coagulation solution was prepared by dissolving 0.5 g of ammonium chloride and 1.0 ml ortho-phosphoric acid 85% in 400 ml distilled water. A fresh solution was prepared daily. The residue was dissolved in 5 ml methanol and thoroughly mixed with 10 ml freshly prepared coagulating solution, then quantitatively transferred and filtered through a chromatographic column (25 cm i.d. × 40 cm length) packed with a 2.5 cm layer of hyflo-supercell. The quantity transfer step was repeated four times. The extract was collected in 250 ml separatory funnel and partitioned three times with 100, 50 and 50 ml chloroform. The combined chloroform was dried through anhydrous sodium sulfate and then evaporated to dryness using a rotary evaporator at 35°C. To concentrate, 10 ml of acetone was added and re concentrated. Repeated the same procedure with additional 10 ml portion of acetone. The acetone re-concentration was done in order to remove the amount of chloroform in the extract which affects the performance of Electron Capture Detector (ECD). The residues were then dissolved in a known volume of ethyl acetate for GLC determination with Electron Capture Detector (ECD).

GLC determination: Quantitative analysis of tetraconazole residues were performed by Perkin Elmer, series 4500 plus gas chromatograph (GLC), equipped with electron capture detector (ECD) in central laboratory of faculty of science. The column was RT X R–CL Pesticides (30 mx0.25 mmx0.25 µm film thicknesses) and the injection port temperature was 250°C, the column temperature was 280°C and the detector temperature 250°C. The carrier gas was nitrogen at a flow rate of 1 ml/min., and the injection volume was 1 µl. Under these conditions, the retention time (Rt) for tetraconazole was 20.8 min.

Calculated Half Life Values: Half life time ($t_{1/2}$) in days was calculated for the two tested pesticides according to the equation of Moye [13].

$t_{1/2} = In_2 / K = 0.693 / K$

$K = 1 / t \times In\ a / m$

Where: K=Apparent rate constant. t=Time in days. m=Residue at x time. a=Initial residue

Recovery studies: Untreated samples of cucumber fruits were spiked with known amounts of tetraconazole and imidacloprid prior to extraction and cleanup for recovery tests of each pesticide. These samples were passed through the entire process of extraction then clean up and analyzed as previously described. Following such techniques the rate of recovery for tetraconazole and imidacloprid residues. The recovery values were calculated, the obtained results of residues determination were corrected according to the recovery percentages.

Statistical Analysis

All obtained data were statistically analyzed using Statistical analysis (SAS) software program [14]. Data were analyzed as factorial arrangement of kind of emulsifying and storage period in complete randomized design with three replicates. Comparisons among the means of different treatments were achieved using the least significant difference procedure (LSD) at P= 0.05 and 0.01 level as illustrated by Al-Rawi and Khalaf-Allah [15].

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Results

Recovery of imidacloprid and tetraconazol

Recovery experiments were performed in order to study the efficiency of the analytical method. The procedures which were used for the determination of tetraconazole and imidacloprid residues in and on cucumber fruits were applied for cucumber samples fortified with known amounts from each pesticide.

Time after application(day)	Residues* (mg/kg) ± SE	% Residues loss
0 (after 1 hour)	0.943 ± 0.0153	0
1	0.365 ± 0.0104	61.29
3	0.271 ± 0.0116	71.61
5	0.226 ± 0.0137	76.03
8	0.086 ± 0.0008	90.88
11	0.049 ± 0.0046	94.8
15	0.028 ± 0.0025	97.03
21	0.013 ± 0.0001	98.62

Table 2: Residual decay of imidacloprid on and in cucumber fruits at different time intervals (*Mean of three replicates)

Results in Table 1 showed that the recovery percentage of tetraconazole and imidacloprid were 91.62% and 117.5% respectively. These results in agreement with those obtained by Amadeo et al. [16] who reported that the rates of recovery for imidacloprid were 123, 114, and 102% in pepper, tomato and cucumber fruits, respectively. Salim [17] mentioned that the recovery percentage for imidacloprid was 113% in tomato fruits. Alfonso et al. [18] indicated that the average of recovery rates of acetamiprid, imidacloprid, thiacloprid and thiamethoxam were ranged between (80 to 105%) and (73 to 102) at the two levels of 0.1 and 1.0 mg/kg for each standard pesticide, respectively, in peach, pear, courgette, celery and apricot. Eiki et al. [19] found that the average rates of recovery for imidacloprid were 113.3, 88.0, 82.7 and 87.5% in cucumber, eggplant, lettuce and green pepper, respectively. Amer et al. [20] determined the residues of tetraconazole and diniconazole fungicides in tomatoes and green beans. The recovery percentage of both fungicides was greater than 90% for both plant samples. The determination limit of the method was 0.001 mg/kg for both fungicides. Spiroudi et al. [21] indicated that the recovery percentage of tetraconazole from sugar beet roots and leaves was found in the range of 86-111 and 78-103%, respectively. In addition, [22] developed a sensitive gas chromatographic method using Electron Capture Detector (ECD) for the determination of tetraconazole fungicide residue in sugar beet foliage and roots. The recovery percentage of tetraconazole in foliage were 86.01- 90.98% and 90.10-93.52% in roots of sugar beet. All results included in residue studies were corrected according the obtained percentage of recovery, for both pesticides.

Persistence of Pesticides Residues on and in Cucumber Fruits

Imidacloprid: Data in Table 2 indicate the amount of imidacloprid residues on and in cucumber fruits taken after different time intervals from the last foliage applications. The initial deposit (one hour after application) of imidacloprid was 0.943 mg/kg. Then the detected residue amounts were 0.365, 0.271, 0.226, 0.086, 0.049, 0.028 and 0.013 mg/kg after 1, 3, 5, 8, 11, 15 and 21 days of treatment, respectively. However the results indicate that the percent rates of loss were 61.29, 71.61, 76.03, 90.88, 94.8, 97.03 and 98.62% after 1, 3, 5, 8, 11, 15 and 21 days of treatment, respectively. The calculated half-life ($t_{1/2}$) value of this insecticide was 2.2 days. The behavior of imidacloprid residue was carried out on cucumber to evaluate the diluting effect on the residue caused by plant growth during the experiment. The residue amount at one day was 0.98 mg/kg, corresponding to 59% from the initial dose. The residues declined rapidly, after 7 days later. The decrease of residues was not significantly ascribed to the growth diluting effect but caused by degradation of the pesticide itself. The theoretical half- life in cucumber (0.97 day) was shorter than in the rice plant. This would be attributed to the fact that rice plants could absorb imidacloprid from the treated zone continuously, whereas the cucumber was injected directly to the stem at once. On the other hand, no imidacloprid residues were detected one hour after application, on roots of sugar beet plants, and then appeared after one day of application (0.004) mg/kg. Also, this residues value was found as it is after 3 and 6 days from application. Fossen [23] reported that imidacloprid is rapidly moved through plant tissues after application, and can be present in detectable concentrations in tissues such as leaves, vascular fluids, and imidacloprid is a systemic pesticide with physical/chemical properties that allow residues to move into treated plants and then throughout the plant via xylem transport and translamilar between leaf surfaces movement.

Some reference which contained residues of imidacloprid on different vegetables

Imidacloprid: Imidacloprid 200 SL was applied at the recommended rate (30.0 g ai ha^{-1}) and its double (60.0 gai ha^{-1}). The average initial deposits of imidacloprid on the cucumber fruits were found to be 1.93 and 3.65 mg kg^{-1} at the single and double dosages, respectively. Results showed that Imidacloprid was rapidly dissipated in cucumbers following a first order reaction kinetics at both application rates (Table 3).

Time after application(day)	Residues* (mg/kg) ± SE	% Residues loss
zero time (after 1 hour)	0.174 ± 0.0004	0
1	0.104 ± 0.0003	40.41
3	0.037 ± 0.0001	78.81
5	0.009 ± 0.0002	95.06
8	0.005 ± 0.0003	97.18
11	0.002 ± 0.0001	99.02
15	ND	

Table 3: Residual decay of tetraconazole on and in cucumber fruits at different time intervals (*Mean of three replicates)

The amount of dissipation in 21 days was 94.48% and 99.18% for, respectively, the single and double dosages. Residues of imidacloprid dissipated below the maximum residue limit (MRL) of 1 mg kg^{-1} in 3 days. Half-life ($T_{1/2}$) for degradation of imidacloprid in cucumber was observed to be 3.40 and 2.70 days at the single and double dosages, respectively. A waiting period of 3 days is suggested for safe consumption of cucumber. Also, results showed that the dissipation was dependent on the initial application dose and followed a first order rate kinetics [24].

Imidacloprid (Konfedor®) was sprayed on tomato, cucumber and pepper in three greenhouses using the same concentration that farmers used to spray their crops, concentration of imidacloprid was 14.5 mg/L. Samples from different crops were collected of spraying, five days, ten days and twenty days later. Samples were taken from fruits, leaves and roots. Results obtained from this study indicates that the residues of imidacloprid was higher than the quantities of residues that determined by previous researchers. Also results showed that the quantities of residues were higher than the maximum residue levels (MRLs) in the samples that are collected on the first, the fifth and the tenth day of spraying [25].

Fruits within the first 24 hours after application decreased to 0.1038 mg/kg with percentage of loss 40.41%. The rapid degradation continued for tetraconazole to reach 0.0369 mg/kg with 78.81% loss after 3 days of application. The decrease in the amounts of tetraconazole recovered 5 days after application continued to be 0.0086 mg/kg with 95.06% loss. After 8 days, there was a small decline to 0.0049 mg/kg, which continued after 11 days to 0.0017 mg/kg with percentage of loss 99.02%, At the last day of this experiment (15^{th} day), residue of tetraconazole was not detected (below detection limit 0.002 mg/kg). The obtained residual half-life value ($t_{1/2}$) of tetraconazole on and in cucumber fruits was 1.4 day. These results agree with those obtained by Khalafallah et al. [6] who determined the fate of tetraconazole residues on greenhouses cucumber by a gas chromatographic method using NPD detector. Tetraconazole residues in cucumber after 7 days were 0.025 and 0.061 mg/kg for the two application doses 4.0 and 8.0 g a.i./ha, respectively, with a half-life of 7 days.

Some reference which contained residues of tetraconazole on different vegetables

Jun [26] determined tetraconazole residues in fruits and vegetables. A 10 g homogenized sample was mixed with 10 mL ethyl acetate, shaken vigorously for 3 min, stored at −20°C for 15 min, and then vortexed vigorously for 1 min; 1 g NaCl and 4 g anhydrous $MgSO_4$ were added. The clean-up was carried out by applying dispersive solid-phase with 150 mg $MgSO_4$ and 50 mg primary secondary amine. Three precursor product ion transitions for tetraconazole were measured and evaluated to provide the maximum degree of confidence. Average recoveries in fruits and vegetables at three levels (0.005, 0.05 and 0.5 mg/kg) ranged from 85.53% to 110.66% with relative standard deviations (RSDr) from 1.3% to 17.5%. The LODs ranged from 0.002 to 0.004 µg/kg, and LOQs ranged from 0.006 to 0.012 µg/kg. This method was also applied to determine tetraconazole residue in cucumber dissipation experiment under field conditions. The half-lives of tetraconazole in cucumber were in the range of 2.1-3.1 days.

Discussion

The different levels of initial deposits of both tested pesticides on fruits of cucumber mainly due to many factors; the ratio of surface to mass area and character of treated surface, smooth or rough and waxy or non-waxy [27]. Systemic and non-systemic character of both compounds, high wax content of fruit surface and hydrophilic-lipophilic balance of investigated pesticides controlled the penetrability of applied agrochemicals into fruit tissues [28,29]. Degradation and dissipation residues of imidacloprid and tetraconazole from cucumber fruits happened because the initial deposits and residues at different intervals of these pesticides are influenced by different factors: evaporation of the surface residue which is dependent on temperature condition, biological dilution which is dependent on the increase mass of fruits, chemical or biochemical decomposition, metabolism and photolysis. Great interest to note that the same factors were studied by several investigators. Christensen [30] reported that the decline of pesticides may due to biological, chemical or physical processes, or if still in the field, due to dilution by growth of the crop. Plant growth particularly for fruits is also responsible to a great extent for decreasing the pesticide residue concentrations due to growth dilution effects [31]. In addition, the rapid dissipation of originally applied pesticide are dependent on a variety of environmental factors such as sunlight and temperature [32]. However, high temperature is reported to the major factor in reducing the pesticides from plant surface [33]. Light plays an important role in the behavior of pesticide in the environment [34]. In this respect, several investigations have been carried out to examine the residual behavior of these pesticides on treated plants. Fossen [22] reported that imidacloprid is rapidly moved through plant tissues after application, and can be present in detectable concentrations in tissues such as leaves, vascular fluids, and pollen. Imidacloprid is a systemic pesticide with physical/chemical properties that allow residues to move into treated plants and then throughout the plant via xylem transport and translamilar (between leaf surfaces) movement Khalfallah et al. [6] studied dissipation of tetraconazole on greenhouse-grown cucumber, and pointed out that its dissipation could mainly be attributed to degradation by chemical and physical properties and less by growth dilution effects when the cucumber plants were almost mature. This study showed that tetraconazole high persistence than imidacloprid on cucumber fruits, the data also imidacloprid residues in cucumber after 1 day from application were 61.29%, but tetraconazole residues that reached to 40.41% from the initial deposit.

Conclusion

In this study residues on and in cucumber fruits collected after one hour, 1, 3, 5, 8, 11, 15 and 21 days from last spray. This study obtained that the residue level of imidacloprid and tetraconazole were less than the Maximum Residue Level which recommended by Codex Alimentration Commission. Also, this study showed that tetraconazole high persistence than imidacloprid on cucumber fruits, but imidacloprid residues in cucumber after 1 day from application were 61.29%, and tetraconazole residues that reached to 40.41% from the initial deposit.

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