

Indoxacarb Residue Analysis, Dissipation and Field Efficacy on Sugar Beet Applied for *Spodoptera Littoralis* Infestation

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Abstract: The severity of *Spodoptera littoralis* from attacking edible plants especially vegetables that required heavy insecticide application, insecticide residue analysis must take place to serve health communities. In this experiment pest infestations monitoring on the sugar beet plants per leaf were completed before and during the three doses of indoxacarb application. Percentages of plant damage reduction data showed fluctuation between samples ranged about more than 90% representing good efficacy on the three levels of concentration tested. The reduction percentages of the *S.littoralis* larvae per leaf were ranged between 98.2 % to 100 at 1 day after application to 50 and 65.6 % at 14 days after 3 doses of indoxacarb applications. Residues of indoxacarb in the root, leaf, and soil were analyzed using QuEChERS method, average recoveries of the all samples were ranged between 96.14 % for soils, 93.86 for leaves and 92.1 % for root. The calibration curves were with a good linearity, exclusive concentration ranges, and had a coefficient of determination (r^2) of 0.990 or better. The limit of detection (LOD) and limit of quantification (LOQ) of this method were in 0.05 and 0.3 $\mu\text{g/g}$ lower than the maximum residue limits (MRL) 0.1. The residue levels of indoxacarb on sugar beet after three doses of application were 0.21 ± 0.07 , undetectable and 0.27 ± 0.07 ppm at 0 days for leaf, root, and soil at the standard dose of application, respectively. Results obtained were 0.39 ± 0.09 , undetectable and 0.53 ± 0.12 for the double dose and were 0.58 ± 0.12 , undetectable and 0.77 ± 0.21 for the triple dose. The average initial deposits were estimated as 0.2, UD and 0.3 ppm for leaf, root and soil and 0.4, UD and 0.53 ppm and 0.58, UD and 0.79 ppm, respectively, for the standard dose, double and triple. The biological half-lives of indoxacarb in sugar beet were 2.3, 2.48 and 2.49 days for leaf, root, and soil respectively, for the standard dose, and the PHI for root was about 3 days for the standard and double applications and 5 days for the triple dose.

Keywords: Indoxacarb, residue analysis, dissipation, sugar beet, *Spodoptera littoralis*, infestation.

1.Introduction

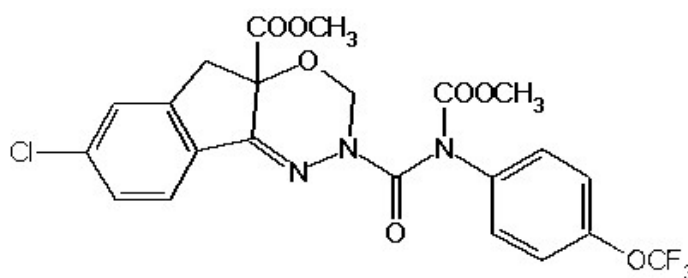
Indoxacarb ($\text{C}_{22}\text{H}_{17}\text{ClF}_3\text{N}_3\text{O}_7$) is an oxadiazine insecticide that is broad spectrum, highly effective, low mammalian toxicity, nonsystemic, organophosphate replacement insecticide and considered as reduce risk insecticide, and recently registered for the use on vegetables to control lepidopteran, sucking mouthparts pests and others (Wing *et al.*, 2000 and Urvashi *et al.*, 2012). Indoxacarb is acted by blocking the sodium channel in insect neurons (Lahm *et al.*, 2000).

Sugar beet, plant *Beta vulgaris L.* is the most vital crop as well as sugarcane in Egypt representing 50% of sugar production. In Egypt, the lower yield per feddan is one of the major production problems. The cotton leaf worm *Spodoptera littoralis* (Boisd.) that habitually extensive attacks many Egyptian fields and other country crops exclusively vegetables lead to lowering yield. Constantly and heavily is infesting sugar beet during the growing season. The high population density requires intensive use of existed insecticides affecting the grounds of the development of resistance to many registered pesticide (Said *et al.* 2012). The pest monitoring is to gather information on insect pest population status within a limited time and space, subsequently, provide information for making management decision about detecting pest emergence spread, and generations by counting some insects per plant into the area of investigation, (Shelton and Trumble, 1990). Recently, all research work about the insecticides that have a unique mode of action, and environmentally friendly and less harmful is beneficial to represent as new alternative compounds to reduce the extensive pesticide uses and less polluting the environment (Lahm *et al.*, 2000). This work aimed to investigate the field efficacy of indoxacarb insecticide about its biological

half-life and residue patterns during sugar beet cultivation and the final residue levels at harvest at high infestation levels of the Egyptian cotton leafworm and its dissipation pattern in the soil in this growing season.

2.Materials and methods

2.1.Insecticide and preparations:



Indoxacarb (Avaunt 15% EC) formulations were originated from Egyptian central agricultural pesticide laboratory. The insecticide solutions were prepared by dilution with distilled water at three doses that freshly required.

2.2.Sugar beet material:

Sugar beet seeds used in this study introduced from Sugar Croup Research Institute, Agriculture Research Center, Egypt, Demapoly cultivar. Seeds were sown at nursery plantation in 10-10-2016 and transplant in the open field at 30-10-2016 at Dokki in Giza Governorate. Seedlings sown in hills, 30 cm apart and every thirty days from sowing fertilization were applied. The plots were irrigated every 10 days intervals. Soil surface was covered by herbicide application to control weed after 50 days from sowing.

2.3. Experimental conditions:

The experimental field area was 120 m² were divided into four plots (each plot were 30 m²), 3 rows and 45 plants. The plots were laid out as three treatments, including three replicates to each treatment and the control plot. Insecticide treatments were applied with the commercial products according to the standard for sugar beet in Egypt indoxacarb (Avuant 15% EC) at standard dose application of 150.75 mL/feddan, 310.5 mL/feddan and 470.25 mL/feddan representing treatment one, two and three. Initially; pest status monitoring began from transplanting in the open field and during the experiment, and the action threshold of all pests monitored was considered. The control plot was maintained at a safe distance. The treatment insecticide was applied when infestation by *Spodoptera littoralis* reached 10 egg-masses/ 100 sugar beet plant as a threshold level. Applications were implemented using a knapsack sprayer 20 L Size with a spray volume of 0.4 L/ m² of a field planted. During all the experimental period, the average relative humidity ranged from 60% to 85%, with maximum and minimum average daily temperatures of 17°C to 27°C from October 2016 to May 2017, respectively.

2.4. Pest Monitoring and Field Sampling of *S. littoralis* infestation:

For detecting plant damage, pest emergence pattern or generation peaks, the relative population densities per area monitoring began from cultivation date till damage reaches the threshold level. All other larvae in the lepidoptera complex were monitored. Percent of infestation level and plant damage were recorded during the growing period. Samples (5 plants/plots), were taken randomly and periodically twice a week before threshold reach infestation and after threshold. Subsequently, after application, samples were collected at five times, 1, 3, 5, 7 and 10 days after treatment and then weekly till significant survival was observed in larvae exposed to the insecticide-treated leaves. Plants were carefully examined for counting the larvae at the pre and post-treatment, and the survived larvae were collected from each treatment plots and taken to the lab for adult emergence percentage exploration.

2.5. Data analysis:

The experiment planned as complete block design about three replicate for each treatment. Data were analyzed by analysis of variance (ANOVA) using IBM-SPSS-20 statistical programme (IBM-SPSS 20, 2011). Means of treatments were separated by Tukey test and the least significant difference test at 5% level, and the LSD was used. Indoxacarb efficacy and percentage of plant damage reduction by treatments were calculated comparing with the check using Henderson and Telton (1955) formula:

$$\% \text{ reductions} = 1 - (\text{treatment after} \times \text{control before} / \text{treatment before} \times \text{control after} \times 100).$$

2.6. Analytical procedures for indoxacarb insecticide residues:

2.6.1. Sampling of leaves:

After the spray of the tested insecticide, samples of sugar beet leaves were taken randomly from each replicate at intervals of zero time (2 h after application) 1, 3, 5 and 7 days, and stored at -20°C until used for analysis.

2.6.2. Sampling of soil and roots:

Representative samples of soil and roots were collected randomly from the depth at 5 cm from the area under the treat-

ed or untreated plants at intervals of zero-day (2h after application), 1, 3, 5 and 7 days, and stored at -20 °C until used for analysis. Also at harvest (200 days from sowing), root sample from each plot was used to estimate the indoxacarb residue on root yields.

2.7. Extraction and clean up (Indoxacarb)

2.7.1. Reagent and chemicals

Indoxacarb reference standard ($\geq 99.9\%$ purity) was purchased from Dr. Ehrestorfer Augsburg, Germany. All organic solvents used in this work were of HPLC analytical grade and purchased from Scharlau (Barcelona, Spain). Solvents suitability were ensured by running the reagent blank along with actual analysis. Analytical grade sodium chloride was obtained from El-Naser Pharmaceutical Chemicals Co. (Cairo, Egypt). Analytical grade anhydrous magnesium sulfate was purchased from Merck (Germany), and activated by heating at 400°C for 4 h in a muffle furnace. Then it was cooled and kept in a desiccators' before use. Graphitized carbon black (GCB) and primary, secondary amine (PSA, 40 μ m Bondesil) were obtained from Supelco (Bellefonte, PA).

2.7.2. QuEChERS methods for residue analysis:

The QuEChERS method has distinguishable advantages as Takkar and many scientists said that a rapid, simple and an efficient method for the determination of indoxacarb in cauliflower and soil samples was developed and validated using QuEChERS technique (Quick, Easy, Cheap, Effective, Rugged and Safe (Takkar *et al.*, 2011).

2.7.2.1. Extraction:

The samples were comminuted using the laboratory blender, and representative homogenized (15g) of each was then placed into 50 mL polyethylene tube. Samples were extracted and cleaned up immediately after sampling. Fifteen mL of acetonitrile & 1% acetic acid were added to each tube. The samples were well shaken using a vortex mixer at maximum speed. Afterwards, 6 g of anhydrous magnesium sulfate and 1.5 g of sodium chloride were added, then extract by shaking vigorously on vortex for 5 min and centrifuged for 10 min at 4,000 rpm (Hassan *et al.*, 2013).

2.7.2.2. Cleaning-up:

An aliquot of 4 mL was transferred from the supernatant to a new clean 15-mL centrifuge tube containing 100 mg PSA and 600 mg anhydrous magnesium sulfate. The samples were again vortexed for 3 min and then centrifuged for 10 min at 4,000 rpm. An aliquot of 2 mL was filtered through a 0.2 μ m PTFE filter (Millipore, USA). The sample was then ready for the final analysis in LC system (Anastassiades and Lehotay, 2003).

2.7.2.3. HPLC analysis:

The measurements were performed with an Agilent 1100 HPLC system (USA), with quaternary pump, manual injector (Rheodyne), thermostat compartment for the column and photodiode array detector. The chromatographic column was C18 Zorbax XDE (250 mm x 4.6 mm, 5 μ m). The column was kept at room temperature. The flow rate of mobile phase (acetonitrile/water = 50/50 v/v) was 0.8 mL/min., and the injection volume was 20 μ L. Detection wavelength of indoxacarb was set at 225 nm. The retention time of indoxacarb was 8.26 min. Residues were estimated by comparison of the peak area of standards with that of the unknown or spiked samples run under identical conditions:

$$\text{Recovery value} = \mu\text{g insecticide} / \text{g sample found} / \mu\text{g insecticide} / \text{g sample added}.$$

2.7.2.4. Insecticide residue calculation:

The residues were calculated by applying the following equation of Mollhoff (1975).

$$\text{ppm} = \frac{\text{ps.B.V}}{\text{pst.G.C}} \times \text{F} \quad \text{Where:}$$

F=100/R (recovery factor) pst=standard peak area

R=average of recovery. V=final of the sample solution. (ml).

ps = sample peak area. B= amount injected of standard solution (ng)

G= sample weight (g) C=amount of sample solution injected.

2.7.2.5. Half-life Calculation:

Half-life time ($t_{1/2}$) of indoxacarb residues were calculated according to Moye *et al.* (1987). The dissipation kinetics of indoxacarb residues were determined by plotting residue concentration against elapsed time after application and equation of the best curve fit with maximum coefficients of determination (R^2) was determined. For dissipation of insecticide in sugar beet and soil, was consistently applicable to the kinetics equation: $C_t = C_0 e^{-kt}$.

Where C_t represents the concentration of the pesticide residue at the time of t , C_0 represents the initial deposits after application and k is the constant rate of pesticide disappearance per day. From this equation, the dissipation half-life periods ($t_{1/2} = \ln(2)/k$) of the studied insecticide.

3. Results and Discussions

3.1. The field persistence of the tested compound residues:

Percentages of plant damage reduction data were in Table (1), and showed up and down fluctuation between samples of days after application, but clearly, most of values were more than 90% representing good efficacy on the three levels of indoxacarb concentrations tested and the incidence of the plant damage. Table (2) show the reduction percentage of the *S. littoralis* larvae per leaf calculated with Hinderson and Tilton equation (1955), and results were ranged between 98.2 % to 100 at 1 day after application to 50 and 65.6 % at 14 days after application. Results proved that the efficacy of indoxacarb against *S. littoralis* on sugar beet plant was decreased to 50% after two weeks from the first application that needs another application to get IBM-SPSS 20 computer program ANOVA multivariate based Leven's test, Duncan and fit of mean differences showed significant differences were found between the three concentrations, each other's, and between sample days itself and between treatment against control as

follows: F=0.102, DF=2 and Sig=0.903), (F=0.189, DF=4 and Sig= 0.943) and (F=0.009, DF= 2 and Sig=0.091), respectively.

The efficacy of any application must be affected by the duration or persistence of the residual effect of the proper insecticide application that always affected by treatment number required for 10 days to affect emerged moths and the insecticide dose-mortality response for the specific pest.

In general, the efficacies of most of the insecticide to control main pests were more than 90%. Nagal *et al.* (2016) point that the efficacy on *Spodoptera litura* on bell pepper showed the highest mean reduction was recorded in flubendiamide (96.41%), followed by emamectin benzoate (94.82%), indoxacarb (93.54%), bifenthrin (93.22%) and chlorantraniliprole (85.92%). Saghfi and Valizadegan (2014) study effects of pyridalyl on *Spodoptera exigua* instars larvae loss on sugar beet plants grown in pots, were 95%, 92.5%, and 80%, respectively at LC₅₀ was 485, 791 and 1280 ppm, respectively, estimated after 72 h of treatment. All the result present were leads to the main point that the application to control *S. species* must be in vital ages.

Insecticide efficacy may differ between each group of chemicals other as well as Said *et al.* (2012), in a sugar beet field study the efficiency of the biocides against *S. littoralis* were compared and arranged as follows viruset, agerin, brofect, protecto 63.64 %, 51.61, 46.38%, 27.14% reduction in the initial kill after five days, respectively, but lannate, cord, and flaxe within eleven days after treatment was permanently equal 100 % reduction, respectively, but IGR'S, toponon, runner, and demelin were gave around 88 % reduction, respectively, after one day of treatment. Ibrahim (2014) conducted efficacy experiment at sugar beet fields in two season at Shennu village (Kafr El- Sheikh Governorate) proved that IGR's caused high reductions in *S. littoralis* larvae populations about 93.39 and 94.98% as well as the conventional one (94.88%) but provided low reduction in nature enemy.

3.2. Indoxacarb Residues in sugar beet leaves, roots, and soil:

The excellent sensitivity and selectivity of any method depended on the precise quantitation and identification at low levels with a minimum run time, insecticide recovery level and calibration curve linearity and accurate.

3.2.1. Indoxacarb Recovery level in sugar beet leaf, root, and soil:

Data percentage of indoxacarb recovery from plant leaf and soil were demonstrated in a table (3). Average recoveries of spiked samples were 96.14 % for soils, 93.86 for leaves and

Table (1): Reduction percentages of Plant damage conferring *S. littoralis* status.

Plant damage reduction percentage at indicated days after indoxacarb treatment (DAT) during 2017						
treatments \ DAT	1DAT(22 Feb)	5DAT(27 Feb)	10DAT(4 mar)	15DAT(9 mar)	20DAT(14mar)	25DAT(19mar)
150.75 ml/feddan	87	89.34	88.67	89.7	88.4	84
310.5 ml/feddan	97	94	93.34	92.4	91	90.4
470.25 ml/feddan	99.67	99.34	99	98	96.7	96.4

Table (2): Indoxacarb efficacy at three doses on *S. littoralis* infected sugar beet plant.

Reduction percentages (±SE) of <i>Spodoptera littoralis</i> Larvae at indicated days after treatment during 2017							
treatments \ Dates	22Feb 1D	27Feb 5D	4 Mar 10D	9Mar 15D	14Mar 20D	% Adult emergence	YieldWeight
150.75 ml/feddan	98.2±0.55	90.9±2.77a	81.5±0.52	58.1±2.3	50.0±1.9	12.3±1.94	61 ton/fed
31.5 ml/feddan	100±0a	95.3±1.38	83.1±0.47	64.3±1.4	50.0±3.1	6.3±0.73	84 ton/fed
47.25 ml/feddan	100±0a	99.0±0.27a	87.2±0.36	69.6±1.8	65.6±2.3	1.6±0.73	44 ton/fed
Control	--	--	--	--	--	71.6±2.67	110 ton/fed

Means followed by the same letter are not significantly different, Tukey's HSD ($\alpha=0.05$).

Means and standard error are expressed as percentage reduction of *S. littoralis*.

Table (3): Recovery and Spike level of the analytical method for indoxacarb in sugar beet.

Samples	Spike level (mg/kg)	% Recovery	Average
leaves	0.01	92.2±0.019	93.86%
	0.05	94.1±0.013	
	0.1	95.3±0.017	
Roots	0.01	88.7±0.025	92.1%
	0.05	93.1±0.023	
	0.1	94.7±0.021	
Soils	0.01	98.06±0.014	96.41%
	0.05	96.03±0.021	
	0.1	95.16±0.019	

92.1 % for root. These results were similar to most of the broadcasted results recorded by *Urvashi et al. (2012)* in indoxacarb residues in cabbage following three applications of Indoxacarb at 52.2 and 104.4 g.a.i./ha. The average recoveries of indoxacarb on cabbage for fortification levels of 0.01, 0.05 and 0.1 mg/kg were observed to be 83.93, 89.86 and 95.40%, respectively. This proposed method was successfully applied to different fruit samples, and satisfactory recoveries, ranging from 78 to 118 %, were obtained, besides the relative standard deviations (RSDs), were in the range of 0.2–11.7% (*Bedassa et al., 2015*).

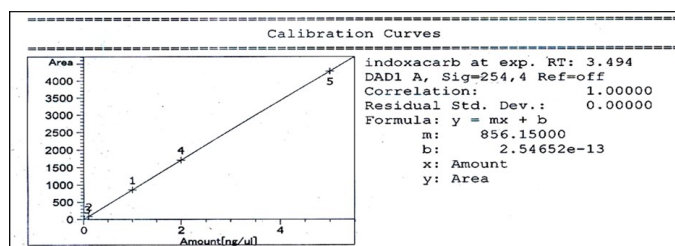
3.2.2. Calibration curve :

In this study, the calibration curve was building using the sugar beet sample with a good linearity (Fig. (1)), exclusive concentration ranges, and had a coefficient of determination (r^2) of 0.99. The limits of detection (LOD) and limit of quantification (LOQ) of this method were 0.05 and 0.3 mg/kg, respectively, which lower than the (MRL) 0.1 mg/kg in sugar beet root (*EFSA, 2009*).

In this respect, *Urvashi et al. (2012)* proved that the LOQ for indoxacarb on cabbage was 0.01 mg/kg. The initial deposits were 0.18 and 0.39 mg kg⁻¹, respectively, at single and double the application rate. These residues dissipated below its LOQ of 0.01 mg kg⁻¹ after 7 and 10 days, respectively, at single and double dosages and the Half-life of them was 2.88 and 1.92 days, respectively.

3.2.3. Dissipation of Indoxacarb in sugar beet:

The present study has screened the level of indoxacarb residues in sugar beet root samples at harvest and immediately after application and intervals to assess the danger of the present residues to the consumer. The residue levels of indoxacarb on sugar beet after three levels of application were demonstrated in tables (4, 5 and 6). These tables show that values of indoxacarb residues were 0.21 ± 0.07, undetectable, and 0.27 ± 0.07 ppm at 0 days for leaf, root, and soil at the standard dose of application. Otherwise, indoxacarb residues in roots were detected after 24 hours of application; it was 0.24±0.08 ppm. This could be regarded as the fact that indoxacarb did not spray directly on the root. The results were 0.39 ± 0.09, unde-

**Fig (1): Linearity of Indoxacarb calibration curve at 1-5 ng/mL of concentrations.****Table (4): Indoxcarb residues after single dose application (ppm).**

Day	Sample	Leaf	Root	Soil
0		0.21±0.07	UD	0.27±0.07
1		0.14±0.06	0.24±0.08	0.17±0.06
3		0.04±0.03	0.06±0.03	0.07±0.04
5		0±0.0	0.02±0.1	0.03±0.01
7		0±0.0	0±0.0	0.01±0.01
Half-life		2.306 day	2.133 day	2.492 day
PHI		3 day	3day	-----
χ^2			0.9356 #	

Insignificant difference between different half-lives using χ^2 probability test at P < 0.05, UD undetectable.

tectable, and 0.53 ± 0.12 for the double dose, otherwise it was 0.44 ± 0.4 ppm in sugar beet root after one day of application. Same observations were observed for indoxycarb residues after the triple dose of application where it was 0.58±0.12, undetectable, and 0.77 ±0.21 ppm and 0.68±0.14 in root after 24 hours of application. Likewise figures (2, 3, and 4) shows the dissipation behavior of indoxacarb on sugar beet leaf, root, and soil with the three tested doses for the standard dose 150.75 mL/feddan, double 310.5 ml/feddan and triple 470.25 mL/feddan.

The biological half-lives of indoxacarb in sugar beet were 2.306, 2.133 and 2.492 days for leaf, root, and soil, respectively for the standard dose. Although different applied concentration and pesticide appeared in the tested samples, it is noticed that there is insignificant difference between the calculated half-lives in the studied parameters. This result was too smaller than that recorded by *Adriana (2003)*, who reported that field dissipation half-lives of indoxacarb are variable, ranging from 16 to 114 days. This could be as a result of microbial degradation of indoxacarb which is an important degradation pathway in the soil. Indoxacarb undergoes rapid decomposition in terrestrial environments through microbial degradation (*Brugger, 1997*). Otherwise, in simulated sunlight, the Half-life was 4.5 days (*FAO, 2009*). After double dose application, the half-lives were 2.525, 2.571 and 2.61 days for leaves, and roots, respectively (Table (5) and Fig. (3)). Otherwise, in triple dosed samples, it was 2.586, 2.406 and 2.412 days (table (6) and Fig. (4)). Table (4) showed that in case of recommended dose as well as in double dose, the PHI was 3 days in leaves, soil and roots where the residue was less than MRL(0.1 mg/kg). Otherwise, if dose applied more than the recommended dose or double, this forced farms to increase the safe time for crop collection (5days).

These results were similar to some researches around indoxacarb residues, as *Yoon et al. (2013)*, who stated that the

Table (5): Indoxcarb residues after double dose application (ppm).

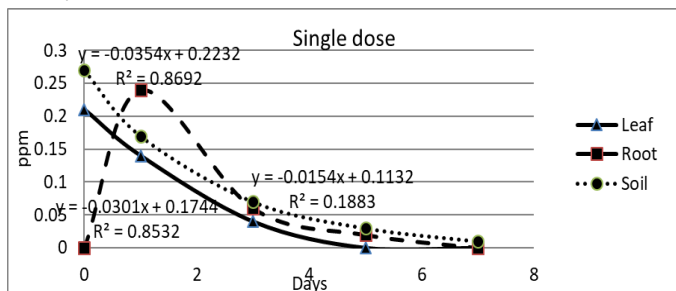
Day	Sample	Leaf	Root	Soil
0		0.39±0.09	UD	0.53±0.12
1		0.27±0.07	0.44±0.11	0.38±0.09
3		0.075±0.03	0.09±0.03	0.12±0.07
5		0.042±0.02	0.05±0.02	0.054±0.03
7		0.023±0.01	0.026±0.01	0.03±0.01
Half-life		2.525 day	2.571 day	2.61 day
PHI		3day	3day	-----
χ^2			0.8314 #	

Insignificant difference between different half-lives using χ^2 probability test at P < 0.05, UD undetectable.

Table (6): Indoxacarb residues after Triple dose application (ppm).

Sample	Leaf	Root	Soil
Day			
0	0.58±0.12	UD	0.77±0.21
1	0.39±0.09	0.68±0.14	0.47±0.08
3	0.16±0.05	0.17±0.08	0.19±0.07
5	0.061±0.03	0.061±0.06	0.062±0.04
7	0.02±0.01	0.03±0.01	0.04±0.01
Half-life	2.586 day	2.406 day	2.412 day
PHI	5day	5day	-----
χ^2		0.8746 #	

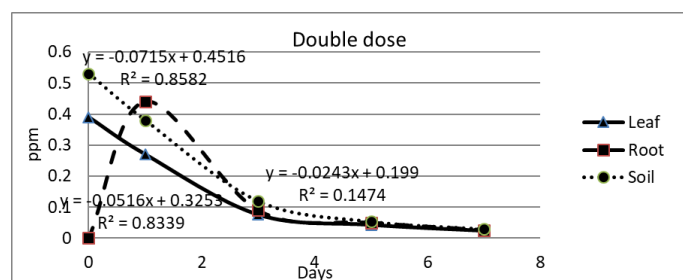
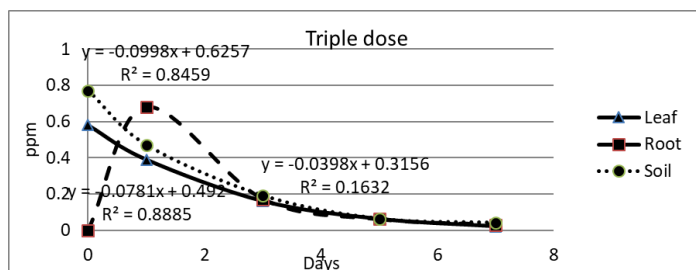
Insignificant difference between different half-lives using χ^2 probability test at $P < 0.05$, UD undetectable.

**Fig. (2): Dissipation behavior of indoxacarb (ppm) after single dose application.**

biological half-lives of indoxacarb in cauliflower were 6.33 days for the standard dose, and 6.26 days for the double dose, respectively. The initial and persisting concentrations of indoxacarb were all below the MRL for broccoli (1.0 mg/kg). **Takkar (2011)** found that recoveries at four concentrations of 0.01, 0.05, 0.1 and 0.2 mg/kg ranged from 87% to 96% were achieved, the initial deposits of 0.23 and 0.45 mg/kg were observed after last application of indoxacarb at 52.2 and 104.4 g.a.i./ ha at recommended and double the recommended dosages, respectively, (**Shim et al., 2007**). Chlorflazuron was sprayed onto pear trees at the recommended dose, residue determination was evidenced by its good linearity (>0.995) in the concentration range between 0.2 and 10 $\mu\text{g/mL}$. The recoveries at two different fortification levels were 0.05 and 0.25 ppm, ranged from 84.9 ± 3.2 to 94.3 ± 10.6 .

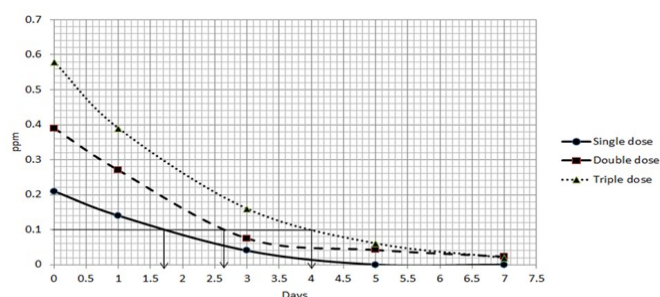
Considering the residues of the tested compound in sugar beet roots at harvest time, it was found that no insecticide residues could be detected in these roots. So, sugar beet roots at harvest time could be used directly after harvesting.

In conclusion, it is advised to increase the PHI after indoxacarb application by 5 days to be sure from crop to be free from the studied insecticide as resulting from pesticide misuse.

**Fig. (3): Dissipation behavior of indoxacarb (ppm) after double dose application****Fig. (4): Dissipation behavior of indoxacarb (ppm) after Triple dose application.**

References

- Adriana M. (2003).** Environmental Fate Of Indoxacarb. Environmental Monitoring Branch Department of Pesticide Regulation 1001 I Street Sacramento, CA 95812-4015. <http://www.cdpr.ca.gov/docs/emon/pubs/fatememo/indoxacarb.pdf>
- Anastassiades, M. and Lehotay, S.J. (2003).** Fast and easy multiresidues method employing acetonitrile traction/partitioning and "dispersive solid-phase extraction" for the determination of pesticide residues in produce. J. of AOAC Int., 86: 412 – 431.
- Bedassa, T.; Gure, A. and Megersa, N. (2015).** Modified QuEChERS Method for the Determination of Multiclass Pesticide Residues in Fruit Samples Utilizing High-Performance Liquid Chromatography. Food Anal. Methods 8:2020–2027.
- Brugger, K.E., and Kannuck, R.M. (1997).** Tier 1 Environmental Risk Assessment of DPX-MP062 and Competitive Insecticides in the USA. Dupont Agriculture Products Document No. AMR 4635-97.
- EFSA (2009).** Modification of the existing MRLs for indoxacarb in cherries and sugar beets, Prepared by the Pesticides Unit (PRAPeR). EFSA Scientific Report (2009) 324, 1-30, Issued on 7 July 2009. <http://onlinelibrary.wiley.com/doi/10.2903/j.efsa.2009.324r/pdf>
- FAO (2009).** FAO Specifications And Evaluations For Agricultural Pesticides Indoxacarb, FAO/WHO Evaluation Report On Indoxacarb, http://www.fao.org/fileadmin/templates/agphome/documents/Pests_Pesticides/Specs/Indoxacarb09.pdf.
- Gao, M.; Mu, W.; Wang, W.; Zhou, C. and Xiuhuan, Li (2014).** Resistance mechanisms and risk assessment regarding indoxacarb in the beet armyworm, *Spodoptera exigua*. Phytoparasitica, 42:585–594.
- Hassan, E.; Neven Ahmed and Arief, M. (2013).** Dissipation and Residues of Penconazole in Grape Fruits. World Journal of Analytical Chemistry, 1 (3), 28-30.

**Fig. (5): The sloping curve of leaf residues after different indoxacarb application.**

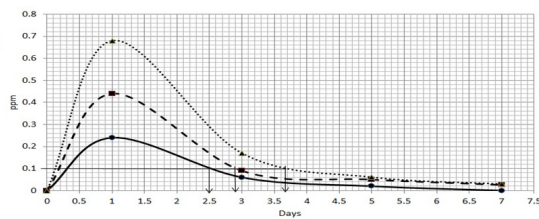


Fig. (6): The sloping curve of root residues after different indoxacarb application.

- IBM-SPSS 20 (2011).** IBM SPSS, *Statistical Package for Social Science*. Standard Version, Copyright SPSS Inc., 1989-2011, All Rights Reserved, IBM license, Copyright © IBM & SPSS Inc.
- Ibrahim, A.SH.M. (2014).** Role of Insect growth regulators (IGRs) For Cotton Leafworm; *Spodoptera Littoralis* Boisd. and conserving to some predators in Sugar beet Fields. J. Plant Prot. and Path., Mansoura Univ., Vol. 5 (12):1089 – 1096.
- Lahm, G.P.; McCann S.F.; Harrison C.R.; Stevenson, T.M. and Shapiro, R. (2000).** Evolution of the sodium channel blocking insecticides: the discovery of indoxacarb. Agrochemical discovery, Chap 3, pp 20–34.
- Loomis, R.S. (1966).** Armyworms and planting date. J. Am. Soc. Sugar beet Tech., 14: 177. (c.f. Akil, 1974).
- Mollhoff, E. (1975).** Method for gas-chromatographic determination of residues of tokuthion and its oxon in plants and soil samples. Pflanzenschutz-Nachrichten Bayer, 28(3): 382-387.
- Moye, H.A.; Malagodi, M.H.L Yoh,J.; Leabee, G.I Ku,C.C. and Wislocki, P.G. (1987).** Residues of avermectin bla rotational crops and soils following soil treatment with (C14) avermectin bla. J. Agric.Food Chem., 35:859-864.
- Nagal,G.; Verma,K.S. and Rathore, L. (2016).** Management of *Spodoptera litura* (Fabricius) through some novel insecticides and biopesticides on Bell pepper

under polyhouse environment. Advances in Life Sciences 5(3):1081-1084.

- Saghfi, M. and Valizadegan, O. (2014).** Study the Effects of Pyridalyl on Larvae of *Spodoptera exigua* (Hubner) at First, Second and Third Ages During 72 Hours in Laboratory Conditions. Int.J.Curr.Microbiol.App.Sci, 3(4): 310-315.
- Said, A.A.A.; Shaheen, F.A.H.; Sherief, E.A.H. and Fouad, H.A.M. (2012).** Estimation of certain compound against cotton leafworm, *Spodoptera littoralis* (boisd.) On sugar beet plants. J. Plant Prot. and Path., Mansoura Univ., Vol. 3 (12): 1321 – 1330.
- Shelton, A.M. and Trumble, J.T. (1990).** Monitoring insect populations. CRC Handbook of pest management in agriculture, Vol 11, p: 45-62.
- Shim, J.-H. Abd El-Aty, A.-M.; Choi, K.-H. and Choi, Y.-S. (2007).** Post-harvest HPLC determination of chlorfluazuron residues in pears treated with different programs. Biomed. Chromatogr. 21: 695–700.
- Takkar, R., Sahoo, S. K.; Singh, G.; Mandal,K.; Battu, R. S.; Singh, B. (2011).** Persistence of Indoxacarb on Cauliflower (*Brassica oleracea* var. botrytis. L.) and Its Risk Assessment. American Journal of Analytical Chemistry, 69-76.
- Urvashi, Gagan Jyot, S. K. Sahoo, Sarabjit Kaur, R. S. Battu, Balwinder Singh (2012).** Estimation of Indoxacarb Residues by QuEChERS Technique and Its Degradation Pattern in Cabbage. Bull. Environ. Contam. Toxicol. 88:372–376.
- Wing, K.D., Sacher, M., Kagaya, Y., Tsurubuchi, Y., Mulderig, L., Connair, M. and Schnee, M. (2000).** Bioactivation and mode of action of the oxadiazine indoxacarb in insects. Crop Protection, 19: 537-545.
- Yoon, J.-Y.; Park, J.-H.; Moon, H.-R.; Han, G.-T. and Lee, K.-S. (2013).** Residue patterns of indoxacarb and pyridalyl in treated cauliflower. Agric. Sciences Vol.4, No.3, 111-116.

تحليل متبقيات مبيد الاندوكسكارب وتقدير ثباته في التربة وتقدير فعاليتها في مكافحه دوده ورق القطن علي نبات بنجر السكر

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مركز البحوث الزراعية - المعمل المركزى للمبيدات - الدقى - الجيزة - مصر

الملخص العربي

تهاجم دوده ورق القطن معظم المحاصيل والخضروات المصرية بشراسة لذا يجب مكافحتها بالمبيدات بطريقة دوريه فعاله وامنه علي صحه المواطن . لذا يستلزم عمل تحليلات دقيقه لمتبقيات المبيدات لهذه الخضروات قبل اتاحتها للمستهلك. لذلك تم عمل تجربه علي نباتات بنجر السكر بفحص الافات التي تصيب النبات منذ زراعته تلقيه رشه من مبيد الاندوكسكارب بثلاث جرعات هي الموصي بها كمعدل حقلي ومضاعفاتها مرتين في بدايه اصابه النبات بدوده ورق القطن . اظهرت النتائج نسبه النقص في تشوهات النباتات المصابه يتراوح بين 90% بينما نسبه النقص في اصابه النباتات بالافه 98 % الي 100 بعد يوم واحد من الرش علي مستوي الثلاث تركيزات المستخدمه من المبيد المذكور. بينما تصل الي 50 % بعد 14 يوم من الرش.

و قد تم تحليل المتبقيات لهذا المبيد في الاوراق والتربه والجذور فور الرش وعلي فترات اخذ العينات وايضا بعد الحصاد اسفرت عن قيمه استرجاع بين 96.14 لعينات التربه و 92.1 لعينات الجذور و 93.86 لعينات الاوراق و اوضح منحني المعايير لهذا المبيد حدود الكشف وحدود دقه التقدير كانت في حدود 0.05 و 0.3 ميكروجرام/جم. و بينما كانت قيم المتبقي 0.27 ± 0.07 و قيمه غير مقدره و 0.21 ± 0.07 جذء في المليون علي التوالي للاوراق والتربه والجذور عند صفر يوم اي مباشره بعد الرش بساعتين وذلك باستخدام الجرعه الموصي بها . اما باستخدام ضعف الجرعه مره فكانت القيم كذلك 0.09 ± 0.39 و قيمه غير مقدره و 0.12 ± 0.53 و باستخدام ضعف الجرعه مرتين كانت 0.12 ± 0.58 و قيمه غير مقدره و 0.21 ± 0.77 علي التوالي ايضا. اوضحت النتائج ايضا ان فتره نصف العمر البيولوجيه للمبيد 2.3 و 2.48 و 2.49 للجرعه الموصي بها وفتره ما قبل الحصاد (فتره الامان) كانت 3 يوم لنفس الجرعه بينما 5 يوم في الجرعه الثالثه . انه تم تقدير متبقي المبيد المستخدم في الجذور عند الحصاد ولم يوجد به متبقي لذلك يمكن استخدامه بامان للاستهلاك.