



Analysis of Fenpyroximate Residues in Eggplant, *Aubergine (Solanum melongena L.)* During Crop Production Cycle by HPLC and Determination of Its Biological Activity.

Shiry S. Takla¹, Farida M. S. E. El-Dars², A.S. Amien³, and Marguerite A. Rizk¹

1-Plant Protection Research Institute, Agriculture Research Center, Dokki, Giza, Egypt.

2-Chemistry Department, Faculty of Science, Helwan University, Ain Helwan, Helwan, 11795 Cairo, Egypt.

3- Chemistry Department, Faculty of Science, Benha University, Qalubiya Governorate

*Email: shirysobhey63@yahoo.com

ARTICLE INFO

Article History

Received:31/3/2020

Accepted:2/5/2020

Keywords:

Fenpyroximate
(*Ortus*) (5%);
insecticide
residue; HPLC;
eggplant

ABSTRACT

A method for determination of Fenpyroximate (*Ortus*) (5%) residues in eggplant (*Solanum melongena L.*) by HPLC is described. The analysis was done using HPLC equipped with a UV-Vis detector at 254 nm. The degradation rate of Fenpyroximate was studied and the results indicated that final residue in eggplant reached 20.1% after 21 days which was considered safe for human and animal consumption, despite its insecticide effectively on eggplant pests.

INTRODUCTION

Fenpyroximate (also known as *Ortus*): IUPAC (tert-butyl (E)- α -(1,3dimethyl-5-phenoxy-pyrazol 4ylmethyleneamino-oxy)-p-toluato) is an acaricide belonging to the phenoxy-pyrazole group, with selective activity on phytophagous species (Hamaguchi *et al.* 1990, Malhat *et al.* 2014). Fenpyroximate (acaricide) is widely used in prophylactic treatment of mite infestation of many fruits and vegetables.

The analysis of fenpyroximate was previously described concerning extraction and determination steps, and the role of instrumentation infra-structure and facilities available (Halvorsen *et al.* 2000; Sannino *et al.* 2004 and Xu *et al.* 2013). However, relatively few data are available regarding the fate of fenpyroximate under field conditions (Naik and Dethé 2009; Sherif *et al.* 2012).

The persistence of pesticides and the fact that residues remain in food may pose potential health hazards to consumers. Therefore, to ensure food safety and environmental protection, investigations need to focus on the proper use of pesticides in terms of authorization, registration, and compliance with maximum residue limits (MRL). Toward this end, field dissipation studies on pesticide persistence in foodstuff and on pesticide residue behavior in agricultural fields are needed (Malhat *et al.* 2014). Thus, this study aims to identify the chemical constituents' of Fenpyroximate using HPLC and to determine its residues in eggplant leaves and fruits. As well, to determine the efficiency

of the biological activity on this plant pests.

MATERIALS AND METHODS

Insecticide:

Ortus (5% SC) was obtained from Shura Company, Egypt. Fenpyroximate is used as an acaricide and insecticide at the recommended dose of (50 cm³/100-liter water) for Cotton, Eggplant, and Grapes. The physicochemical properties of Fenpyroximate are shown in (Table 1). Fenpyroximate Standard solution was prepared as 100 cm³/1-liter water and Fenpyroximate working solution was at the recommended dose of 50 cm³/100-liter water.

Field trial:

Experiments were carried out during winter and summer season of 2017- 2018 at fields near Benha, Qalubiya Governorate, Egypt. Eggplant (*Solanum melongena L.*) was planted in an area of (1st m²). The area was divided into two equal plots. The experiment plot was cleared; prepared and suitable seeds of eggplant were cultivated. Treatments were done in completely randomized blocks design and were replicated four times. Each block was separated from the other by 50 cm blank area. The growing plants were sprayed with the Fenpyroximate at the recommended dose of (50 cm³/100-liter) water using a (25-liter capacity) plastic drum sprayer and the control crop was sprayed with water. Plants from each treatment were combined and placed in individual plastic bags. Besides the determination of pesticide residues, a sample of the crop and leaves from each treatment were taken before and after to indicate the pest count.

Method Validation for Fenpyroximate Residues Determination:

Before the determination of Fenpyroximate residues in eggplant, the developed method was validated in terms of linearity of the instrument response and the concentration range limits, accuracy (spike and recovery), the limit of detection (LOD), the limit of quantification (LOQ), inter and intra-day precision and stability studies as per ICH guidelines (2005) using HPLC, GC and UV-Vis spectroscopy. HPLC analysis was performed on an Agilent 1100 HPLC equipped with a diode-array detector (Agilent, USA), reverse-phase C₁₈ HPLC hypersil column that was maintained at 25° C. The standard parameters for HPLC instrument during the determination of Fenpyroximate are summarized in Table 1.

Table 1: Standard conditions for HPLC determination of Fenpyroximate (*Ortus*) and its residues in the tested crop

Pesticide	Column Type	Injection Volume	LOD	Mobile Phase	Retention time	Wave length (λ)
Fenpyroximate	Hypersil C ₁₈ (150 μm × 4.5 i.D.)	20 μL	0.5 ng/ml	Methanol: water (80:20 v/v)	2.764 min	254 nm

To construct a calibration curve for this chemical (Fig. 1), five standard solutions of Fenpyroximate (10.0 - 100 μg /L) were prepared and analyzed using HPLC. The standards were injected at the beginning and end of each run, and each standard was injected at a minimum of five times. Regression equations were generated using the peak area responses versus the respective concentrations for the construction of a calibration line. The concentration of Fenpyroximate in the samples was determined by substituting the peak area responses of the sample into the applicable regression equation.

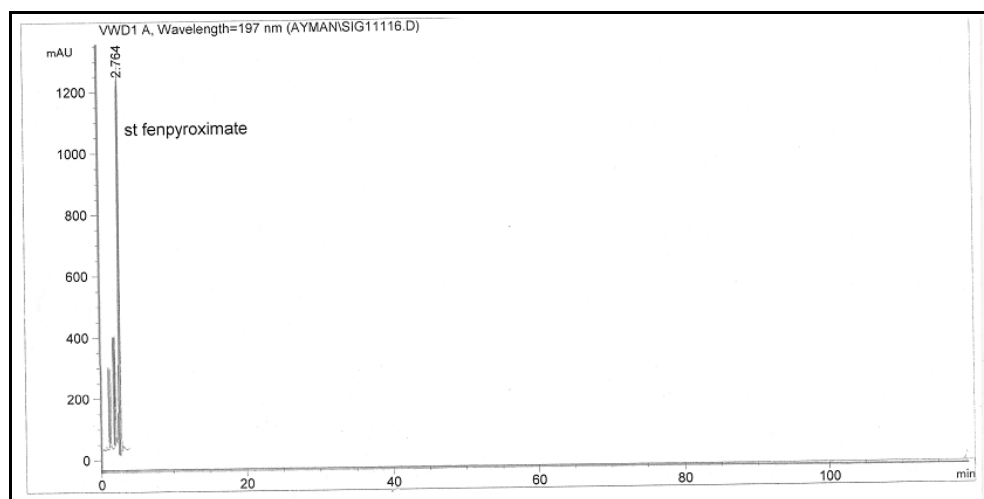


Fig. 1: HPLC chromatogram of standard Fenpyroximate.

To determine linearity, pesticide working standard solutions of concentrations ranging between 0.5 – 400.0 ng/ml were injected in HPLC. For the accuracy of the method, samples of untreated eggplant were fortified with Fenpyroximate standard solutions (5, 10, 20, 30, and 40 ppm). Before the extraction step, the fortified samples were allowed to settle for 30 min. Samples were then processed according to the following extraction procedure. Five replicates for each concentration were analyzed to validate and evaluate the accuracy of the method. The limit of detection (LOD) and limit of quantification (LOQ) of the overall method were calculated as concentration giving a signal-to-noise ratio of 3 ($S/N = 3$) and 10 ($S/N = 10$), respectively.

Determination of Fenpyroximate (Ortus) Residues In Treated Crops Using HPLC:

Representative samples were taken randomly after 0 and 1 hour and 1, 3, 7, 14, and 21 days of spraying Ortus from the leaves and fruits. Residue extraction and sample cleanup were carried out at room temperature (25 °C) and according to the following procedure (Cao *et al.* 2005 and Sobhey, 2014). 50 g of vegetable sample was cut, grounded and was extracted with 150 mL acetone (5 times) in a stopper separating conical funnel by shaking for 1 hour. The extracts were filtered with Whatman No.1 filter paper and were concentrated using a vacuum rotator evaporator at 55°C until the final volume reached 10 mL. The sample was transferred to a separator funnel containing 100 mL of 4% sodium chloride and the residue was extracted by liquid-liquid partitioning with dichloromethane three times with a volume 50, 30 and 30 mL, respectively. The organic phase was combined passed over anhydrous sodium sulfate to remove water and was further concentrated using a water bath to a final sample volume of 2mL for column chromatography (Cao *et al.*, 2005 and Sobhey, 2014).

Sample Clean-up:

The concentrated extract was transferred quantitatively to a glass beaker with 20 mL of n-hexane and mixed well with 2 g activated charcoal, 2 g anhydrous sodium sulfate, and the slurry was allowed to settle. The clear layer of the slurry was transferred to a suitable chromatographic column (300 mm × 30mm id) fitted with a stopcock and packed with silica gel and was allowed to pass slowly through the column (30 drops /min). The charcoal mixture was washed 6 times with 20 mL n-hexane and passed through the column. The combined extract was evaporated to dryness under vacuum and transferred quantitatively with methanol to a 10 mL volumetric flask for injection into the HPLC (Cao *et al.*, 2005 and Sobhey, 2014).

Sampling Program for Pest Count on Sampled Crops and Leaves Sampling Before and After Treatment Technique:

To study the effect of Fenpyroximate on controlling sucking pests attacking eggplant plants, samples (40 leaves) were taken randomly before treatment and after 1, 3, 7, 14, and 21 days after treatment and from control samples. Plants from each treatment were combined and placed individually in plastic bags. All sucking pests were counted per 1-inch² area for the incidence of spider mites (*Tetranychus urticae*), egg stage of spider mites (*Tetranychus urticae*), the nymph of whitefly (*Bamisia tabaci*), Thrips (*Thrips tabaci*), Leafhopper Jassid (*Empoasca. Sp.*). The initial effect of the different spray methods was estimated after 1 day from the application. The accumulated general reduction was also estimated for counting carried out after 21 days from each application. Percentage of population reduction for each pest species/ each treatment was calculated according to Henderson's formula (Henderson and Tilton, 1955) as follows:

$$\text{Percentage reduction} = \left[1 - \frac{T_a \times C_b}{T_b \times C_a} \right] \times 100$$

Where,

Ta: number after treatment in the treated plot.

Tb: number before treatment in the treated plot.

Ca: number after treatment in the check plot.

Cb: number before treatment in check plot

RESULTS AND DISCUSSION

Method Validation for Fenpyroximate Determination on Eggplant:

A.) Linearity, LOD and LOQ:

To determine linearity, pesticide working standard solutions of concentrations ranging between 0.5 – 400 ng/ml were injected in HPLC. A good linear relationships and coefficients of determination ($R^2 > 0.99998$) were obtained over the concentration ranges of 0.5 – 400 ng/ml. The limit of detection (LOD) and limit of quantification (LOQ) of the overall method were calculated as concentration giving a signal-to-noise ratio of 3 (S/N =3) and 10 (S/N =10), respectively. LOD and LOQ are obtained in this study 0.5 and 400 ng/ml respectively, (Table 2) for fenpyroximate on eggplant, (SANTE, 2015).

Table 2. Linearity parameters for the analytical determination of Fenpyroximate using HPLC

Parameters	Value
Linearity range	0.5 – 400 ng/ml
Correlation coefficient(R^2)	0.99998
Slope	13.28443
Intercept	4.02×10^{-1}
Regression equation ,Where Y :Area , X :concentration , m:Slope , b:intercept	$Y = (13.28443 \times X) + 4.02 \times 10^{-1}$
LOD	0.5 ng/ml
LOQ	400 ng/ml

B.) Spike and Recovery Accuracy Test of Fenpyroximate:

From the data in Table (3), the mean recovery values were between 89.52% and 100% which indicates that the method was accurate. Residues corrected according to the average recovery, Islam *et al.* (2009). These values were satisfactory for residue analysis and of the same order obtained when using more complicated methodologies.

Table 3: Accuracy (spike and Recovery %) for HPLC method for Fenpyroximate determination in Eggplant blank:

Injected standard Fenpyroximate concentration	Area [MAU S]	Recovery %	Retention time (min)
5 ppm	4916.78	100 %	2.764
10 ppm	591.55	96.26 %	2.786
20 ppm	1152.22	93.736 %	2.778
30 ppm	1650.46	89.52 %	2.735
40 ppm	2430.32	98.86 %	2.766

C.) Determination of Fenpyroximate (Ortus) Residues in Treated Eggplant Using the Proposed HPLC Method:

Ortus residues present in the eggplant samples were identified and quantified with reference to standard pesticides (Ortus). The residue levels of pesticide found at different time intervals on different samples are listed in Table (4).

Table 4: Determination of Fenpyroximate residues (ppm) in eggplant

Time	Height LU	Area [MAU S]	Recovery%	Recovered Ortus Conc (ppm)
0	1182.37012	4445.75488	100.0 %.	5
1 hour	157.36868	1185.9224	81.969 %.	4.09845
1 day	110.53994	959.77698	74.40 %.	3.72
3 day	92.36627	523.81281	49.52 %.	2.476
5day	76.18311	368.20447	38.82 %.	1.941
7 day	43.90411	206.544	28.62 %.	1.431
14 day	28.06931	103.73469	24.68 %.	1.234
21 day	8.56608	68.04003	20.09 %.	1.0045

Figures (2 and 3) show that fenpyroximate (ortus) residues were detected in all eggplant samples collected from the field in Benha, Qalubiya Governorate at time = 0. After 1 hour of treatment, the residue was 81.969% which was equivalent to 4.09845 ppm. After 1-day treatment, the pesticide residue was 74.40% equivalent to 3.72 ppm. After 3 days of treatment, the pesticide residue was 49.52 % which was equivalent to 2.476 ppm. After 5-day treatment, the pesticide residue was 38.82% which was equivalent to 1.941 ppm. After 7 days, the pesticide residue was 28.62% which was equivalent to 1.431 ppm. After 14 days of treatment, the pesticide residue was 24.68% which was equivalent to 1.234 ppm. The data indicated that the residues of Fenpyroximate in eggplant decreased with the longer time interval of sampling after spraying. After 21 days, the residue was only 20.09% which was equivalent to 1.0045 ppm.

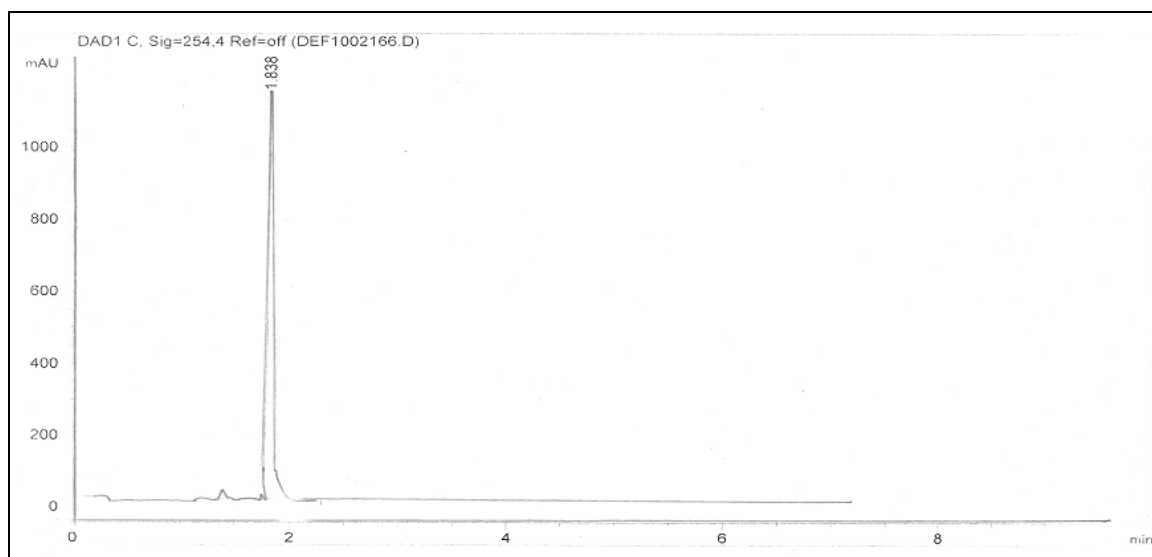


Fig. 2. Chromatogram of Fenpyroximate (ppm) Residues in eggplant sample at time=0

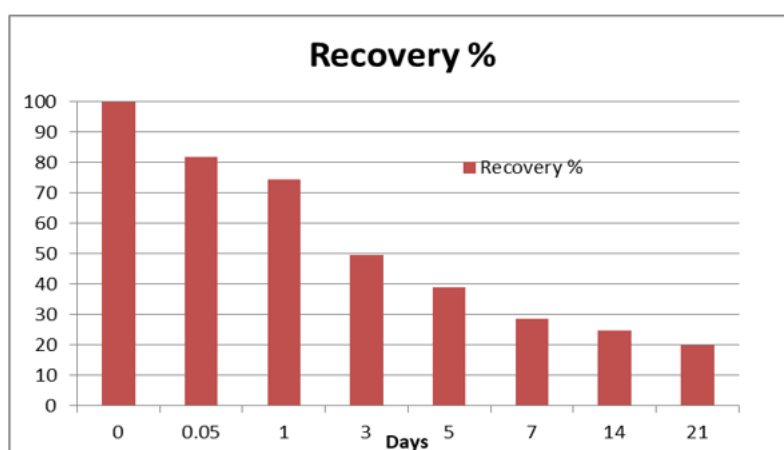


Fig.3. Residue Recovery from Eggplant after spraying days

The results agree with that of Mahmoud (2004) which indicated that no residues were detected in plants after 15 days of application. Hence, the plants could be marketed and consumed safely after that period. The residue half-life values in leaves were 1.6 days and 1.3 days in the green bean.

Malhat *et al.* (2014) showed that the half-life of Fenpyroximate on grapes was approximately 3.5 days at both recommended and twice the recommended dosage in an open field.

Also, Meijs (2008) stated that many or all properties of the pesticide may change when the formulation is changed. Since the physical-chemical properties are very important parameters for the different aspects of the assessment, determination of all physical-chemical properties has to be done thoroughly as relevant information for the calculation of the efficacy and the side-effects of the new pesticide.

Wellings (2006) has indicated that the use of C18 columns provides good results for the determination of fenpyroximate because no derivation step was needed (Cao *et al.* 2005; Wellings, 2006 and Kandil *et al.* 2011).

Degradation Kinetics of Fenpyroximate in Eggplant:

Table (5) and Figure (4) show the amount of Fenpyroximate residue determined in eggplant fruits over the testing period. It has to be noted that after 21 days of pesticide application, the amount of residue was undetectable. This is in accordance with the

findings of Mahmoud (2004); no residues were detected in plants after 15 days from application. Figure (5) shows that the pesticides followed a first-order degradation rate of 2.304×10^{-1} / day.

Table 5: fenpyroximate residue (mg/kg) on eggplant over the test period

Time day	Residue (mg/kg) C	Ln C
0.1	1.74	0.5539
1	1.15	0.1398
3	0.56	-0.5798
7	0.05	-2.9957
14	0.02	-3.9120
21	0.0	-

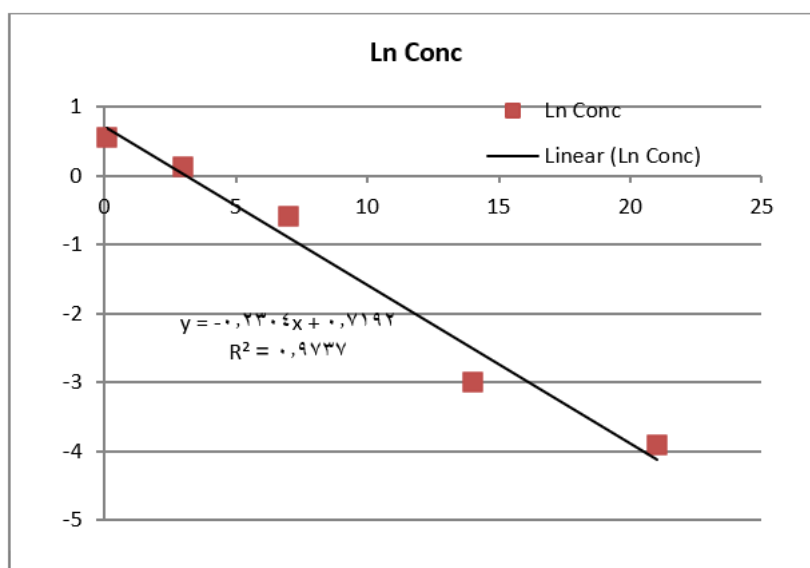


Fig. 4: The stability of Fenpyroximate solution (5%) with time

Analysis of Fenpyroximate (Ortus) Using FTIR:

The biochemical content of Fenpyroximate (ortus) was investigated using FTIR spectroscopy. Figure (5) shows the representative FTIR spectra obtained from ortus in the $(3441.02 \text{ to } 474.39 \text{ cm}^{-1})$ region. The frequency ranges from 566.13 cm^{-1} peaks are represents the sulfonyl chloride SO_2 stretching vibration, the presence of carboxylic acid and amines. FTIR spectra identify active ingredients and impurities which included (Solvents, Emulsifiers, Spreaders, Stickers, Buffers, Thickeners, Boirs, Synergists, and Abrasives). Table (6) shows the presence of phytochemical compounds in Fenpyroximate extract such as $-\text{NH}_2$ in aromatic amines, primary amines, and amides at 3441.02 cm^{-1} , $\text{C}=\text{O}$ in Ketones at 1713.08 cm^{-1} , OH in carboxylic acid at 1414.09 cm^{-1} , $\text{C}-\text{O}-\text{C}$ in esters, lactones at 1249.75 cm^{-1} , SO_3H in sulfonic acids at 1074.97 cm^{-1} , Pyridines at 635.43 cm^{-1} , $\text{C}-\text{I}$ in iodo-compounds at 587.00 cm^{-1} , $\text{C}-\text{N}-\text{C}$ in amines at 495.62 cm^{-1} , naphthaline at 474.39 cm^{-1} . Impurities in Ortus may include (Solvents, Emulsifiers, Spreaders, Stickers, Buffers, Thickeners, Boirs, Synergists, and Abrasives).

The presence of a phytochemical companied in Fenpyroximate (ortus) extract agrees with other investigators (Iglesias *et al.* 2011; Angelo and Zodrow, 2011).

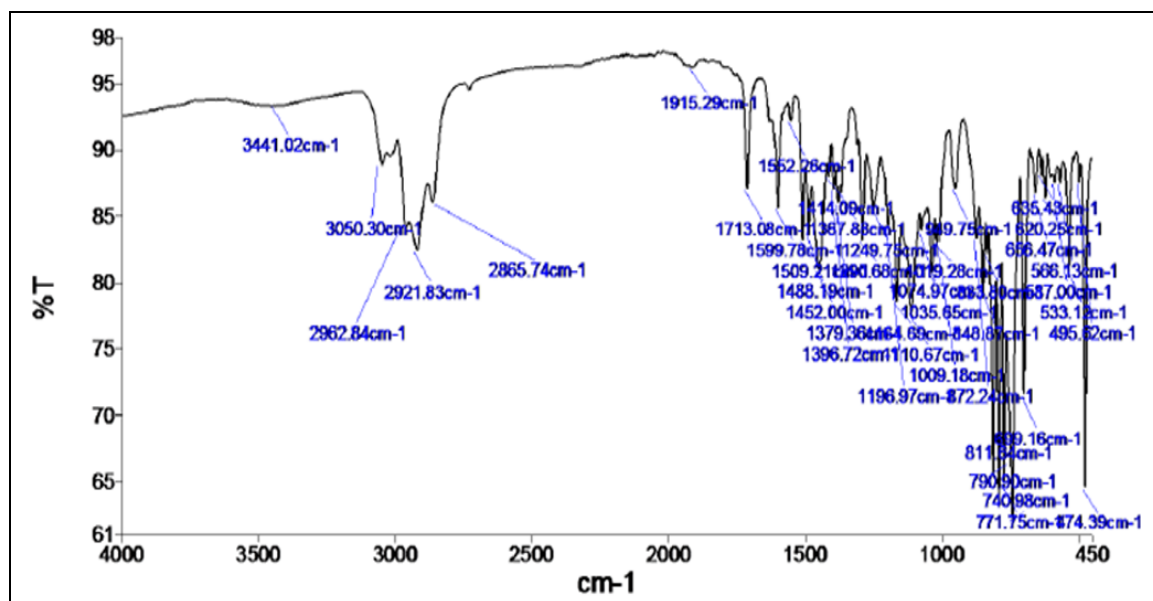


Fig.5. FTIR Spectrum of Ortus

Table 6. Determination of Fenpyroximate analyzed by FT-IR procedure developed.

Peak Number	X (cm ⁻¹)
1)-NH ₂ in aromatic amines, primary amines, and amides	3441.02
2) Substituted benzene rings	1915.29
3) C=O in Ketones	1713.08
4)COO- in carboxylic acid salts	1599.78
5) NO ₂ in aliphatic nitro compounds	1552.26
6) Benzene ring in aromatic compounds	1509.21
7) OH in carboxylic acid	1414.09
8) C-F in aliphatic fluoro compounds	1290.68
9) C-O-C in esters, lactones	1249.75
10)SO ₂ -in sulfones	1164.69
11) Si-O-Si in siloxanes	1110.67
12) SO ₃ H in sulfonic acids	1074.97
13) P-O-C in organophospho compounds	1035.65
14) R-NH ₂ primary amines	848.87
15) C-Cl in chloro compounds	833.80
16) O-C=O in carboxylic acid	699.16
17) Pyridines	635.43
18) C-Co-C in ketones	620.25
19)C-I in iodo compounds	587.00
20) SO ₂ in sulfonyl chlorides	566.13
21) C-N-C in amines	495.62
22) naphthaline	474.39

Biological Activity of Ortus 5%sc (Fenpyroximate):

Field observation showed that five insect pest species were found in eggplant plots throughout the growing season; i.e. Spider mites (*Tetranychus urticae*); Whitefly (*Bemisia tabaci*); Thrips (*Thrips tabaci*) and Leafhopper Jassid (*Empoasca. Sp*).

Table 7: Effect of Ortus 5%Sc (Fenpyroximate) on pests invested eggplant crops in winter 2017.

Pest	No. of pests before treatment/ leaf	Initial The effect after 1 day	R%	3 day	7 day	14 day	21 day	Total	mean	R%
Spider Mite Adult Stage	78	5	94.29	3	5	7	40	55	13.75	88.38
Control	89	100	-----	12	13	14	15	540	135	-----
Spider Mite Egg Stage	120	5	95.04	20	10	5	30	65	16.25	88.72
Control	125	105	-----	135	160	185	120	600	150	-----
White Fly	107	5	95.72	10	5	5	20	40	10	93.52
Control	110	120	-----	135	150	170	180	635	158.7	-----
Jassid	247	10	96.11	20	30	30	40	120	30	90.21
Control	250	260	-----	280	300	320	340	1240	310	-----
Thrips	87	10	89.66	50	20	20	20	110	27.5	79.68
Control	90	100	-----	120	130	150	160	560	140	-----

Table 8: Effect of Ortus 5%sc (Fenpyroximate) on pests invested eggplant crops in summer 2018.

pests	No. of pests Before treatment/ leaf	Initial The effect after 1 day	R%	3 day	7 day	14 day	21 day	Total	mean	R%
spider mite adult stage	57	10	86.84	1	3	4	4	12	3	91.58
Control	60	80	-----	30	40	40	40	150	37.5	-----
spider mite egg stage	65	20	64.11	10	0	0	0	10	2.5	94.87
Control	70	60	-	60	50	60	40	210	52.5	-----
White fly	60	10	77.77	0	0	10	20	30	7.5	60.00
Control	80	60	-----	40	40	20	0	100	25	-----
Jassid	90	20	62.96	20	10	10	20	60	15	64.91
Control	100	60	-----	60	60	50	20	190	47.5	-----
Thrips	217	40	80.69	50	20	10	6	86	21.5	51.56
Control	220	210	-----	100	20	10	50	180	45	-----

Data in Tables (7 and 8) show the effect of Ortus success in controlling pests invested eggplant in winter and summer. Ortus (Fenpyroximate) succeeded in controlling spider mites egg stage Table (7 and 8) which gave (95.04% and 64.10%) initial effect in winter and summer, respectively, and decreased to 88.7% residual effect in winter increased to 94.87% in summer. The compounds tested significantly reduced spider mites (*Tetranychus urticae*) population on eggplant compared with the check. Regarding the initial effect, Fenpyroximate was more effective in controlling mite mobile stages in winter but residual it's more in summer. Tayyib *et al.* (2005) in Pakistan evaluated new insecticides for controlling *T. urticae* on cotton. They indicated that fenpyroximate gave (63.75%), while the dicofol and azocyclotin gave less than 50% mortality.

Also, Ortus (Fenpyroximate) was successful against Whitefly (*Bemisia tabaci*). Table (7 and 8) gave 95.71% initial effect and 93.52% residual effect in winter which decreased to 77.77% initial effect and 60.00% residual effects in summer.

The reduction of leafhopper (*Jassid*) population on eggplant crop spray with Ortus (Fenpyroximate), the initial effect of 96.11% in winter decreased to 62.96% in summer. Also, the same result which reduction after 21 days 90.21% in winter decreased to 64.91% in summer.

Regarding the initial effect, (Fenpyroximate) ortus (on the day after spraying) against Thrips (*Thrips tabaci*) on eggplant was 89.66% in winter reduced to 80.69% in summer. While after 21 days, the reduction of thrips reduced to 79.68% and 51.56% in winter and summer, respectively.

This study indicated that the time of application had a considerable effect on the efficiency of the pesticide under field conditions. This agrees with Gennari *et al.* (1985), who found that further, several environmental factors, particularly temperature, precipitation (and humidity), and air movement affect pesticide persistence.

Desai *et al.* (2014) indicated that Fenpyroximate 5 EC at the lower dose i.e., 25 g a.i./ha can be taken advantage of the management of leafhopper as well as spider mite infestation on cotton. It remained effective up to 15 days of application in controlling both the pests. Similar findings were reported by the Muhammad *et al.*, (2012) on spider mite infesting cotton, Singh, and Singh (2005) on *T. Urticae* infesting okra and Naik *et al.* (2009) on *Tetranychus* infesting brinjal. Murugesan and Kavitha (2009) reported the effectiveness of imidacloprid against leafhopper infesting cotton.

Results obtained that Fenpyroximate provided rapid "stop feeding" action thus minimizing crop damage. Also, it provided a long-lasting control when applied at the recommended dose rate. As well, it inhibits oviposition of females, which further increases the length of control.

Conclusion:

The present work investigated the presence of Fenpyroximate residues on eggplant. The results indicated the stability of the applied chemical formulation under the recommended dose of 50 cm³/100-liter water. In studying pesticide residues on treated crops, it is desirable to determine the intervals required between applications and harvest (safety period). The final degradation rate recovery of area residue in eggplant the fruits reached 20.09% after 21 days which was considered safe for human beings and animal consumption.

REFERENCES

- Angelo, J.A.D. and Zodrow, E. (2011). Chemometric study of functional groups in different layers of *Trigono carpus grandis* ovules (Pennsylvanian seed fern, Canada). 2011, NEW OPEN ACCESS JO Results in organic Geochemistry, Volume 42(9): 1039-1054.
- Cao, Y.; Chen, J.; Wang, Y.; Liang, J.; Chen, L. and Lu, Y. (2005). HPLC/UV analysis of chlorfenapyr residues in cabbage and soil to study by the dynamics of different formulations. Science of the Total Environment. Volume 350, Issues 1–3, 1 November 2005, Pages 38-46.
- Desai, H.R.; Sojitra, R.S.; Patel, C.J.; Maisuria, I.M. and Kumar, V. (2014). Field evaluation for bio-efficacy of fenpyroximate 5 EC against leaf hopper and spider mite infesting cotton and their safety to natural enemies. Advance Research Journal of Crop Improvement 2014 Vol.5 No.2 pp.172-175 ref.11
- FAO and WHO. (2015). Pesticide residues in food. Report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Core Assessment Group. FAO Plant Production and Protection Paper, 223. Rome. Retrieved from <http://www.fao.org/3/a-i5186e.pdf>
- Gennari, M.E.; Zannini, A.; Cignetti, C.; Bicchi, A. D.; Amato, M.; Taccheo, C.; Spessotto, M.; De-Paoli and Flori, P. (1985). Vinclozolin Decay on Different Grape Vines in Four Different Italian Areas. Journal of Agricultural and Food Chemistry, 33: 1232–1237.

- Henderson, C.F. and Tilton, E.W. (1955). Tests with acaricides against the brown wheat mite. Entomology Research Branch, Agriculture Research Services, V.S.D.A., and ELVIN, 48: 157–161.
- Halvorsen, B.; Thomsen, C.; Greibrokk, T. and Lundanes, E. (2000). “Determination of Fenpyroximate in Apples by Supercritical Fluid Extraction and Packed Capillary Liquid Chromatography with UV Detection”. Journal of Chromatography A Volume 880, Issues 1–2, 2 June 2000, Pages 121-128.
- Hamaguchi, H.; Ohshima, T.H.; Takaishi, A. Y.; Konno, T. and Kajihara, O. (1990) “Synthesis and Acaricidal Activities of Fenpyroximate (NNI-850) and its Related Compounds.” In Abstract Papers of the 7th International Congress of Pesticide Chemistry, Vol. 1. Hamburg: 5–10.
- Iglesias, J.V.; Grozovski, V.V.; Herrero, E. and Feliu, J. M. (2011). Adsorption of Formic Acid and Its Role as Intermediate in Formic Acid Oxidation on Platinum Electrodes. ChemPhysChem Volume 12, Issue 9, pages 1641-1644. June 20, 2011.
- ICH guidelines (2005) International Conference on Harmonisation. ICH-harmonised-tripartite-guideline: validation of analytical procedures: text and methodology, Q2 (R1), Geneva; <http://www.ich.org/LOB/media/MEDIA417.pdf>, 2005 (Parent Guideline dated 27 October 1994, Complementary Guideline on Methodology dated 6 November 1996 incorporated in November 2005).
- Islam, S.; Afrin, N.; Hossain, M. S.; Nahar, N.; Mosihuzzaman, M. and Mamun, M.I.R. (2009). Analysis of some pesticide residues in cauliflower by High-Performance Liquid Chromatography. American Journal of Environmental Sciences 2009 Vol.5, No.3 pp.325-329 ref.14.
- Kandil, M.A.; Swelam, E.S.; Abu-Zahw, M.M. and Shalby, M.A. (2011). Affect of Light and Temperature on Chlorfenapyr and Identification of its main degradation products. Research Journal of Environmental Toxicology, 5(5): 316-322, 2011.
- Mahmoud, H.A. (2004). Biochemical studies on the behavior of some pesticides in Broad bean crop. D.Sc. Thesis, Faculty Of Agriculture, Cairo University.
- Malhat, F.; Badawy, H.; Barakat, D. and Saber, A. (2014). “Residues, Dissipation and Safety Evaluation of Chromafenozide in Strawberry under Open Field Condition.”. Food Chemistry Volume 152, 1 June 2014, Pages 18-22
- Meijs, W.H.M.A. (2008). New type of formulations and new application techniques; consequences for the authorization of pesticides. (New pesticide formulations and applications for reduced environmental impact.) Environmentalist 18(1): 5-8.
- Muhammad, A.; Muhammad, H.B.; Muhammad, D.G.; Muhammad, K.Z.; Khan, M.A. and Liaquat, A. (2012). Evaluation of some acaricides against two-spotted spider mites, *tetranychus urticae* Koch (acari: tetranychidae) on cotton crop under laboratory and field conditions. Pakistan Entomologist 2012 Vol.34 No.2 pp.125-129 ref.21
- Murugesan, N. and Kavitha, A. (2009). Seed treatment with *Pseudomonas fluorescens*, plant products, and synthetic insecticides against the leafhopper, *Amrasca devastans* (Distant) in cotton. Journal of Biopesticides, 2(1): 22-25 (2009).
- Naik, R.V. K. and Dethle, M. (2009). “Studies on Residues of Flufenzin and Fenpyroximate on Brinjal.” International Journal of Plant Protection 2009 Vol.2_No.1 pp.38-41 ref.5.
- Sannino, A.; Bolzoni, L. and Bandini, M. (2004). “Application of Liquid Chromatography with Electro sprays Tandem Mass Spectrometry to the Determination of a New Generation of Pesticides in Processed Fruits and Vegetables.” Journal of Chromatography A Volume 1036, Issue 2, 21 May 2004, Pages 161-169

- SANTE (2015). Guidance document on analytical quality control and method validation procedures for pesticides residues analysis in food and feed. Supersedes SANCO/12571/2013.
- Sherif, H.; Almaz, M. and Osama, I. (2012). "Determination of Degradation Rate of Acaricide Fenpyroximate in Apple, Citrus, and Grape by HPLC-DAD." Food Analytical Methods volume 5, pages 306–311, (2012).
- Singh, S.P. and Singh, R.N. (2005). Efficacy of some pesticides against spider mite, *Tetranychus urticae* Koch and its Predatory mite, *Amblyseius longis pinosus* (Evans). Resistant Pest Management, News Letter. 14(2): 7-10, 2005.
- Sobhey, S.T. (2014) Selected pesticides used in the control of mites during the crop production cycle. M.Sc. chemistry department, faculty of Science, Helwan University, 125 pp.
- Tayyib, M.; Sohail, A.S.; Murtaza, A. and Jamil, F.F. (2005). Efficacy of some new chemistry insecticides for controlling the sucking insect pests and mites on cotton. Pakistan Entomol, 2005, Vol. 27 (1): 63-66
- Wellings, D.A. (2006). A Practical Handbook of Preparative HPLC. Elsevier Publisher, Radarweg 29, PO Box 211, and 1000 AEA Amsterdam the Netherlands: 17 – 22.
- Xu, D.L.; Shengyu, C. L.; Dajie, L.; Jinchang, Z.; Zhigang, F.Y. and Yu, Z. (2013). "Determination of Ten Pesticides of Pyrazoles and Pyrroles in Tea by Accelerated Solvent Extraction Coupled with Gas Chromatography-Tandem Mass Spectrometry." Chinese Journal of Chromatography 01 Mar 2013, 31: 218–222.

ARABIC SUMMARY

تحليل الاثار المتبقية من مبيد الاورتنس (الفينبيرواكسيمات) فى الباذنجان اثناء فترة الانتاج بجهاز التحليل HPLC ومتابعة نشاطه الحيوى

شيرى صبحى تكللا¹, فريدة محمد سعد الدين الدرس², علاء السيد احمد امين³, مارجرىت عدلى رزق¹

١ - معهد بحوث وقاية النباتات- مركز البحوث الزراعية- بالدقى-مصر

٢- قسم الكيمياء- كلية العلوم جامعة حلوان

٣- قسم الكيمياء - كلية العلوم جامعة بنها

تهدف هذه الدراسة الى تحليل متبقيات المبيد الحشرى الاورتنس(الفينبيرواكسيمات٥%) ومتابعة نشاطه الحيوى على نبات الباذنجان عن طريق جهاز التحليل الكروماتوجرافى HPLC و UV وذلك عن طريق عامل الفصل الميثانول والماء بنسبة ٨٠:٢٠ ثم يتبع ذلك الاستخراج والتحليل الجزئى المائى و يليه مرحلة التنظيف وذلك عند طول موجى ٢٥٤ نانوميتر. وقد وجد ان معدل التفسير لمادة الفينبيرواكسيمات الموجودة فى مبيد الاورتنس ٥% على الباذنجان ٠,٠٢ ملى جرام / كيلوجرام بعد ١٤ يوم من الرش وتنتهى اثاره نهائيا بعد ٢١ يوم وهذا المعدل يعتبر الاكثر امانا للاستهلاك الادمى والحيوانى.

تضمنت خطة الدراسة النقاط الاتية

١. تحديد المبيد المستخدم والمحصول.

٢. دراسة الطرق العملية التحليلية المناسبة لاستخراج بقايا المبيد من المنتجات الزراعية.

٣. تقييم بقايا المبيد فى العينات المجمعة بطرق الفصل الكروماتوجرافى عن طريق جهاز HPLC

ويمكن تلخيص النتائج المتحصل عليها فيما يلى:

اولا: تحليل متبقيات مبيد الاورتنس بواسطة جهاز HPLC واعطت احسن النتائج لانها لا تحتاج الى اى خطوات جزئية. ثانيا: لقد اوضحت النتائج ان الاورتنس ٥% يخضع فى التحكم وقتل العنكبوت الاحمر بنسبة ٨٨,٣٨% شتاء و ٩١,٥٨% صيفا. وبالنسبة لبيض العنكبوت الاحمر ٨٨,٧٢% شتاء و ٩٤,٨٧% صيفا. واما الذبابة البيضاء ٩٣,٥٢% شتاء و ٦٠% صيفا بينما الجاست ٩٠,٢١% شتاء و ٦٤,٩١% صيفا وسجل الترس ٧٩,٦٨% شتاء و ٥١,٥٦% صيفا.

ونظرا لوجود نسبة قليلة من متبقيات الاورتنس لذا ينصح بغسل النبات بماء الصنبور قبل استخدامه ليصبح أكثر امانا للاستخدام الادمى.