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Research Article

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Determination of Acetamipride and Profenofos Residues in Cabbage Using QuECHERS Method in Sohag, Upper Egypt.

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ABSTRACT
Our investigation included cabbage leaves for insecticides residue analysis. We had been treated by the formulation acetamipride (Mosiplan 20% SP) and profenofos (Ictacron 72% EC) and residues were estimated by HPLC. The findings demonstrated that for each insecticide, a different amount of residues were recorded over the trial period. Acetamipride and profenofos had early deposits of 0.88 and 1.85 ppm on and in unwashed cabbage leaves, respectively. Acetamiprid and profenofos residues on unwashed cabbage leaves were 0.50 ppm and 1.07 ppm, respectively, after the first day of spraying. These reduced to 0.11 and 0.39 ppm on washed cabbage with tap water. To reduce the concentration of pesticide residues in cabbage leaves, it is vital to wash the leaves with tap water as residue loss increases with the amount of time that has passed after spraying began until the end of the trial period. It indicates that acetamiprid was degraded faster than profenofos. These variations in the rate of disappearance of various insecticides could be related to variations in chemical formulation and structure as well as application rates. The safety time after which cabbage plants sprayed with acetamipride and profenofos may be picked up was the first and seventh days, respectively, according to maximum residues limits (MRLs).
Keywords: Cabbage, Residues, Acetamipride, Profenofos, QuEChERS

INTRODUCTION

Vegetables are essential to human nutrition and health, because they include minerals, micronutrients, vitamins, and antioxidants. Poisonous substances known as pesticides are used to prevent, eliminate, or control pests throughout the production, processing, transportation, and marketing of food. Food safety has a big impact on human health, which is why people are more worried about eating safe food. Pesticide residues in food are one of the most recent problems to draw public attention on a global scale (Inobeme et al., 2020), especially in vegetables crops. Certain agricultural crops cannot be cleaned of pesticide residues by washing or cooking, indicating that the pesticide may have permeated the crops. The worry of the impact of increasing population on food security has led to a further increase in the use of pesticides in agriculture as a result of the world's population's rapid growth, which is expected to reach 8.5 billion people by 2030 (Liu et al., 2015). Europe, China, and subsequently the US are the three regions that use pesticides the most. Around 25% of the utilization is in African nations, with vegetable farming accounting for the majority of cases (De Bon, 2014). Due to their slow rate of degradation, some pesticides can bio accumulate up to 70,000 times their active components in the environment. Therefore, it is necessary for national authorities and regulators to control and enforce maximum residue limits (MRLs) through routine pesticide inspection (Kaushik et al., 2017). The maximum allowed level of pesticide residues (expressed in mg/kg) in food products and animal feeds that is both legally permissible and toxicologically acceptable is set by the Codex Alimentarius Commission (FAO, 2013). It represents the greatest residual ratio that might be anticipated if the crop was pesticide-treated in accordance with label recommendations and other allowed Good Agricultural Practices (GAP).

In order to reduce health risks, this research sought to examine the persistence of the aforementioned insecticide residues in cabbage plants and provide guidance on the pre-harvest interval (PHI) that should elapse after treatment and prior to commercialization. This study also aimed to shed light on how washing with tap water affected the elimination of pesticide traces from cabbage leaves.

MATERIALS AND METHODS

Insecticides used

Cabbage was cultivated under open field and sprayed by two insecticides, acetamipride (Mosiplan 20% SP) at 25 g / 100 L water and profenofos (Ictacron 72% EC) at

350mL/ 100 L water, respectively, according to Technical Recommendations for Agricultural Pest Control (2017) and determine the residues of these chemicals in cabbage. The experimental area was divided into plots of 42 m2 (1 / 100 Fed.). The layout of the experimental region was a fully randomised block. To act as a control, three plots were not given any treatment. The commercial production of cabbage involved manipulating all agricultural methods.

Insecticides bioassay

The rates listed above were used to apply the pesticides (recommended dose). A skilled operator applied the spray. A Knapsack-sprayer (Cp-3) with one nozzle that delivers (200 L/fed.) was used for the spraying, and it has proven to be sufficient to provide good coverage on the treated plants. The dissipation rate of the chemical on cabbage and the terminal residues in the finished products were investigated using residue trials carried out in accordance with crucial Good Agricultural Practices (GAPs).

Determination of acetamipride and profenofos residues in cabbage leaves

Extraction and clean-up processes

Ten heads of cabbage were collected randomly from each plot in open field and then taken two equal parts (500 g). To evaluate the impact of washing on the loss of the tested pesticides, the first half was washed three minutes with running tap water and then allowed to dry on clean paper for 30 minutes at room temperature. The second part was left untreated. The extraction and clean -up processes were carried out at the Water and Environment Laboratory in the Regional Center for the Development of Southern Upper Egypt - Quraman Island – Sohag. The samples were prepared with the QuEChERS method (Anastassiades, *et al.*, 2003).

A 50 mL PTFE centrifuge tube containing 10 grammes of homogenised cabbage sample was weighed, 10 mL of acetonitrile was added, the tube was vortexed for 1 minute, 4 grammes of anhydrous MgSO4 and 1 grammes of sodium chloride were added, the tube was vortexed for 30 seconds, and the mixture was centrifuged at 4000 rpm for 5 minutes. For cleanup, 1.0 mL of acetonitrile was placed into a 2.0 mL centrifuge tube. The dSPE tubes containing 25 mg PSA and 150 mg MgSO4 received an aliquot of 1 mL. The tubes were securely closed, vortexed for 30 s, and then centrifuged at a speed of about 4000 rpm for 5 min. In order to inject the mixed eluate into the HPLC, a 0.22-m nylon syringe filter was used.

Chromatographic conditions

The final determination of acetamiprid samples was carried out in HPLC. It system is an Agilent 1260 series with a photodiode array detector attached to an analytical column with dimensions of 150 mm 4.6 mm id, 5 m ODS. The mobile phase (acetonitrile 70% + water 30%) flow rate for acetamiprid was 1 ml/min, and the injection volume was 20 µl. A 205 nm detection wavelength was used. Under these conditions the retention time was 3.66 for, acetamiprid (Fig.1). While the HP6890 gas chromatograph outfitted with a flame photometric detector (FPD) was used for the final determination of profenofos Smples, a 30 m x 0.32 mm capillary column coated with a 0.25 m thick film of 14% cyanopropilsiloxane (PAS-1701) and a phosphorus filter were also used. The following was the oven temperature programme: 160 oC at start for 2 minutes. 6 oC every minute up to 260 oC, held for 30 minutes. The flow rate of the carrier gas (N2) was 4 ml/min. At 240 oC, a splitless injection of a 2-l volume was performed. The flow rates for the hydrogen and air used were 75 and 100 ml/min, respectively. 250 oC was the detector's temperature. In these circumstances, the retention time for profenofos was 5.276 minutes (Fig.2).

Recovery studies

Untreated cabbage leaves were strengthened by adding a standard solution of profenofos and acetamipride at concentrations ranging from 0.1 to 1.0 ppm. To validate the assay technique, the fortified samples were processed through each stage of the analytical method. The recovery percentages from fortified untreated samples were used to rectify the results (Table 1).

RESULTS AND DISCUSSION

Recovery percentages of acetamipride and profenofos

Table (1) displays the acetamipride and profenofos recoveries from fortified samples (50 g) at various processing steps. Acetamipride recovery rates ranged from 105.38 to 92.58%, whereas profenofos recovery rates ranged from 90.28 to 80.32%. The average of recoveries for acetamipride was 87.71 %, while for profenofos was 86.64 %. This result is compatible with Sallam and El-Nabarawy (2001), the same method in determination of chlorpyrifos-methyl, chlorpyrifos, and profenofos on moloukhia leaves. They found that recovery percentage of these insecticide were 86.52, 90.43, and 83.4%, respectively.

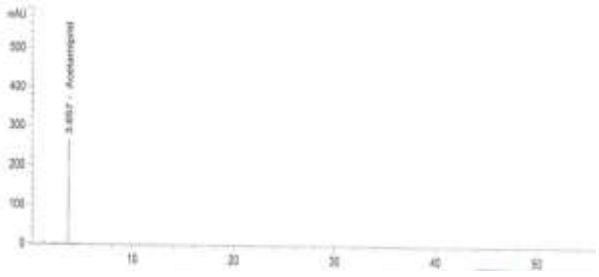

Figure 1. HPLC chromatogram of acetamiprid

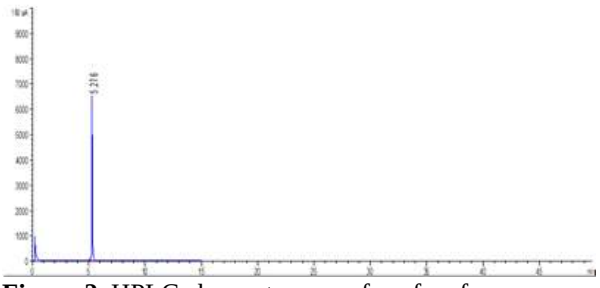

Figure 2. HPLC chromatogram of profenofos

Table 1. Recovery percentages of acetamiprid and profenofos in cabbage leaves.

Spiking level (mg/kg) (n*=3)	Acetamiprid		Profenofos	
	Mean recovery ±SD	% RSD	Mean recovery ±SD	%RSD
0.1	92.58±1.22	1.42	80.32±0.92	4.85
0.5	65.18±1.57	2.55	89.33±2.01	1.05
1	105.38±1.20	2.01	90.28±1.55	0.44
Average	87.71± 1.33	1.99	86.64±1.49	2.11

*: Number of replicates

Residues of acetamiprid and profenofos on and in cabbage leaves cultivated in open field.

The concentration of the initial deposits of profenofos and acetamiprid on unwashed cabbage leaves was 0.88 and 1.85 ppm, respectively, according to the findings in table (2) and figures (3 & 4). Within 24 hours of spraying, the residual levels fell to 0.50 and 1.07 ppm, respectively. The residues of acetamiprid dropped to 0.34, 0.12, 0.01, and undetectable after 3,5,7,12 and 15 days, respectively. The corresponding values for profenofos were 0.55, 0.08, 0.01, and undetectable ppm . With each pesticide, a different amount of residues was recorded over the testing period. These levels varied depending on the initial depositions, the speed at which the cabbage heads were exposed to external variables, and how the treated surface responded to the chemical used. Stevens, et al., (1988) demonstrated that uptake of pesticides on plant surface is affected by, the chemical composition, formulation, rate of insecticide employed, type of recipient surface, spraying equipment used, and climatic conditions particularly the ambient temperature, especi ally during pesticide application.

Data presented in Table (2) demonstrates that as time passed from the start of spraying to the completion of the trial period, residue loss grew. For acetamiprid and profenofos, the loss percentages after one day from the start of spraying were 43.18 and 42.16, respectively. More than 85% of the acetamiprid residues had gone by the fifth day. While with profenofos, the early deposits vanished to a greater than 95% extent. The same phenomenon took place with Shiboob, (1995), he discovered that after 12 days of spraying, profenofos residue loss percentages in tomato and cucumber fruits varied from 99.1 to 99.3%.

Acetamiprid and profenofos residues in unwashed cabbage leaves had half-lives calculated by Moye, et al., (1987), that were, respectively, 1.8 and 1.49 days. These results are in agreement with Abdalla, et al., (1993), who found that the half-life values (RL50) of profenofos residues was 3.2 days on tomato fruits, while [6], reported that profenofos on moloukhia leaves had a half-life of 52.08 hours.

It is obvious that acetamiprid was degraded faster than profenofos (Fig.3 & 4). These variations in the rate of disappearance of various insecticides could be related to variations in chemical formulation and structure as well as application rates. The same conclusion was mentioned by Sallam and El-Nabarawy (2001), that degradation rate, was correlated to the chemical structure of the tested compounds.

According to the maximum residues limits (MRLs) of acetamiprid (0.7 ppm) Codex (2012), and profenofos (0.01 ppm) EU (2017) in cabbage leaves. Acetamiprid and profenofos –sprayed cabbage leaves can be picked up after 1 and 7 days, respectively from spraying. The same conclusion was pointed out by El-Sayed, et al., (1977), they reported that according to their findings, there should be a one- to twelve-day waiting period between the spraying of insecticides and the harvesting of produce for commercialization in order to protect consumer safety and prevent health hazards.

Effect of washing process in removing on acetamiprid and profenofos residues from cabbage leaves.

Data in Table (2) indicated the great influence of washing with tap water in removing or elimination of acetamiprid and profenofos residues from sprayed cabbage leaves. Acetamiprid and profenofos residues on unwashed cabbage leaves were 0.50 and 1.07 ppm, respectively, after one day of spraying. On cabbage that had been washed with tap water, these were reduced to 0.11 and 0.39 ppm. The results showed that the amounts of acetamiprid and profenofos residues in washed cabbage were much lower than those in the unwashed cabbage. Washing process removed residues from 55.68 to 91.18 and 37.50 to 63.55 % respectively, for acetamiprid and profenofos on cabbage leaves. Such findings are in agreement with that obtained by Zhi-Yong Zhang, et al., (2007), they measure the quantities of pesticide residue (chlorpyrifos, p,p-DDT, cypermethrin, and chlorothalonil) in cabbage during the home preparation process by washing with various acetic acid and salt chloride concentrations as well as tap water. The findings demonstrated that the aforesaid pesticides lost to washing with acetic acid solutions (at 10% concentration for 20 min), NaCl solutions (at 10% concentration for 20 min), and tap water (for 20 min). To reduce or remove pesticide residues in agricultural products, many techniques and tools have been developed. These techniques include washing, chilling, peeling, boiling, and ozonation Li, P.-K, et al., (2004).

CONCLUSION

These results concluded that acetamiprid residue was reduced more quickly than profenofos on cabbage leaves that had been rinsed with tap water and left traces of both pesticides. Variances in chemical composition and structure as well as treatment rates may be responsible for these variances in the rate at which different insecticides vanish. The first and seventh days, respectively, were the safe periods after which cabbage plants treated with acetamipride and profenofos may be harvested.

Table 2. Residues of acetamiprid and profenofos on and in cabbage leaves cultivated in open field.

Days after spraying	Acetamiride				Profenofos			
	Unwashed		Washed		Unwashed		Washed	
	Residues (mg/kg)	% Loss	Residues (mg/kg)	% Loss	Residues (mg/kg)	% Loss	Residues (mg/kg)	% Loss
0	0.88	0.00	0.39	55.68	1.85	0.00	0.84	54.59
1	0.50	43.18	0.11	78.00	1.07	42.16	0.39	63.55
3	0.34	61.36	0.03	91.18	0.55	70.27	0.21	61.82
5	0.12	86.36	ND	---	0.08	95.67	0.05	37.50
7	0.01	98.86	ND	---	0.01	99.45	ND	---
12	ND	---	ND	---	ND	---	ND	---
15	ND	---	ND	---	ND	---	ND	---
18	ND	---	ND	---	ND	---	ND	---
MRL	0.7 Codex (2012)				0.01 EU (2017)			
PHI	1 day				7 days			
RL50	1.8		0.98		---			

ND = (Not detectable); MRL = (Maximum Residue Limits); PHI =(pre harvest intervals); RL50 =(Residue half-life)

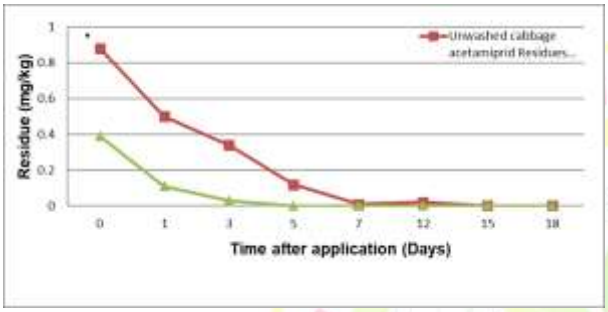

Figure 3. Persistence of acetamiprid residues on and in cabbage leaves

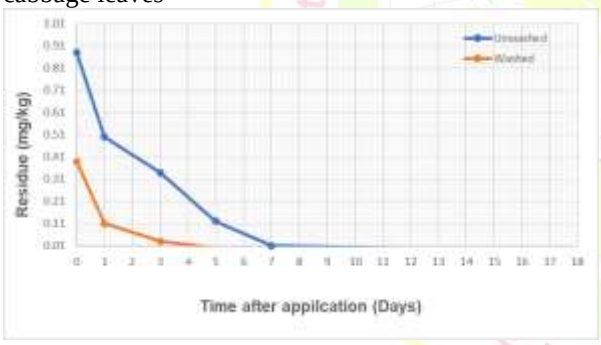

Figure 4. Persistence of profenofos residues on and in cabbage leaves

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