

## Dissipation of Pymetrozine and Penconazole On and In Pea Fruits

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### Abstract

Estimate the residues of pymetrozine and penconazole in pea fruits were determined by using GC-ECD and HPLC-DAD with QuEChERS method. The application was carried out using knapsack sprayer equipped with one nozzle. The samples were taken after one hour, 1, 3, 5, 8, 11, 15 and 21 days of treatment. Obtained data revealed that the residual level of pymetrozine was less than the maximum residue limit (MRL= 0.02 mg/kg) which recommended by Codex Alimentation Commission, as well as penconazole (MRL= 0.05mg/kg).Data showed that penconazole residues were high persistence ( $t_{1/2}=3.6$  days) than pymetrozine residues ( $t_{1/2}=2.5$  days) on pea fruits respectively. The pre-harvest interval (PHI) of pymetrozine and penconazole were 8 and 12days after treatments for pea fruits respectively. Results of household processing (washing with water, peeling and boiling) of pea fruits indicated that there are some safety methods to decrease the residues level of pymetrozine and penconazole.

**Key words:** residues, pymetrozine ,penconazole , pea fruits, household processing.

### 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 (*El-Lakwah et al .1995* ) Persistence is the ability of a pesticide to remain present and active in its original form for an extended period before degrading. A chemical's persistence is described in terms of its half-life, a comparative measure of the time needed for the chemical to degrade. The longer a pesticide's half-life, the more persistent the pesticide residues are sometimes desirable because they provide long term pest control and reduce the need for repeated applications (*Pradhan et al .2013*). Fruits and vegetables play an important role in human nutrition and health; they constitute an important part of our daily diet. They are important sources of carbohydrates, vitamins, trace minerals, and antioxidants. Therefore, they can be contaminated by pesticides used for the protection of their culture. The use of pesticides to control pests in fruits and vegetables can lead to the presence of pesticide residues. The level of these residues can be below the maximum residue limit (MRL) if good agricultural practices (GAP) were used. The presence of residues with level exceeding MRLs should be interpreted as violation of GAP. In many reports, pesticide residues are present in the majority of fruits and vegetables; they are more detected in fruits than in vegetables. The percentage of exceeding MRLs is less than 20% in most monitoring programs. The risk assessment for long-term and short-term exposure must be done for all pesticides detected to ensure consumer's health protection (*Mebdoua 2018*)

Garden pea (*Pisum Sativum*, L) is extensively grown vegetables all over the country. It is rich in protein, carbohydrate, vitamin A and C, calcium and phosphorus. It also contains small quantity of iron. The crop is attacked by multitude of insect pests and diseases. Pesticide residues are reduced by processing or household preparation stages such as washing, peeling and cooking. The effect of processing on residues has been seen to vary with both crop and pesticides (*Burchat et al.1998*). Penconazole was effective in controlling a broad spectrum of fungi diseases; it is a systemic triazole fungicide with protective and curative actions. It is recommended under commercial formulation topas 10% EC for controlling powdery mildew in pea vegetables, especially aphids and white fly. (*Shen et al. 2012*) Chemical Family: Pyridine Azomethines It is a novel insecticide with selective activity against homopteran insects and was recommended for control aphid of vegetables in Egypt The mode of action of pymetrozine in insects has not been precisely determined biochemically, but it may involve effects on neuroregulation or nerve-muscle interaction. Physiologically, it appears to act by preventing these insects from inserting their stylus into the plant tissue (*Linders et al.2000*). The QuEChERS method covers a very wide analyte scope such as highly polar pesticides, and highly acidic and basic ones. This method involves extraction with acetonitrile and partitioning after the addition of a salt mixture. The extract in acetonitrile could be directly injected in GC and HPLC. The maximum residue limits (MRL) regulations require a pre-harvest interval (PHI) to ensure the dissipation of pesticide below the proposed MRL at harvest time (*Karmakar & Kulhestha, 2009*).

Therefore, to ensure food safety and protecting the environment field dissipation studies on pesticide persistence in food stuffs and pesticide residue behaviour in agricultural fields are needed. this study was conducted to estimate the residues of these pesticides in the open field and estimate the residues of these pesticides after some household equipment washing, peeling, cooking, as well as residues in the soil under treated plants and compared to the level of residues limits allowed globally.

#### **Materials and Methods**

##### **1: Pesticides**

**Table 1. Pesticides Tested**

| Pesticides            | Fungicide   | insecticide  |
|-----------------------|---|--|
| Common name           | Penconazole   | Pymetrozine  |
| Trade name            | Topas 100 10% EC  | Chess 50% WG   |
| Manufacturer          | Syngenta Agro   | Syngenta Agro  |
| Rate of use           | 25cc/100 L  | 20gm/100 L   |
| Pests                 | Powdery mildew  | Aphid  |
| Chemical name (IUPAC) | 1-[2-(2,4-dichloro-henyl)-phenyl]-1H-[1,2,4] triazole<br>CIPAC No : 446 | 6-methyl-4-[(E)(pyridin-3ylmethylene)amino]-4,5-dihydro-2H-[1,2,4]-triazin-3(2H)-one<br>pyridinylmethylene)amino]-1,2,4-triazin-3(2H)-one<br>Pyridine azomethine |
| Chemical group        | Triazole  |  |
| Structural formula    |   |  |

##### **2: Chemicals and Reagents:**

All reagents and solvents used were analytical reagent grade supplied by Algommuria Company for Trading Chemicals and Medical Appliances - Egypt.

##### **3: Spraying:**

The application was carried out using knapsack sprayer (20 L) equipped with one nozzle

##### **4: Design of the experiments:**

The field tests were carried out at the farm of: Al - Ajmaeen – Ibshway - Fayoum Governorate, Egypt. Name of varieties: pea *Pisum sp.* (Aintesar -1) were planted on 1<sup>st</sup> December 2017. The experiment were designed in the following ways (42 rows -4 m×25 cm/each ) 18 rows for each pesticide in addition to 6 rows for control. the experimental design was a complete randomized block, each treatment includes three replicates. Irrigation and fertilization were made according to the crop schedule.

##### **5: Sampling:**

###### **5.1: plant samples**

After the application of the pesticides on the plants, the samples (one kg) of the peas plant, fruits were

Therefore, to ensure food safety and protecting the environment field dissipation studies on pesticide persistence in food stuffs and pesticide residue behaviour in agricultural fields are needed. this study was conducted to estimate the residues of these pesticides in the open field and estimate the residues of these pesticides after some household equipment washing, peeling, cooking, as well as residues in the soil under treated plants and compared to the level of residues limits allowed globally.

collected from each treatment randomly and from non-treated. The samples of plants were collected at zero time (one hour after the treatment) on 7th April 2017, then 1, 3, 5, 8, 12, 15 and 21 days after the treatment. All samples were placed in bags and transported in ice boxes to laboratory chopped using a food cutter, all undesired parts involved in samples were avoided, and then the seeds were removed out of pods of peas. Sub sampling 50gm/each were performed at the laboratory, and kept in deep freezing until analysis.

##### **5.2: Soil samples**

Samples of soil were collected randomly from the depth at 5 cm form the area (one kg) under the treated and untreated plants at intervals of zero time (one hour after the treatment) on 17 April 2017, then 1, 3, 5, 8, 12, 15, and 21 days after the treatment.

##### **6: Extraction and clean up:**

###### **6.1: Plant samples:**

Plant samples were extracted and cleaned up by using QuEChERS Quick, Easy, Cheap, Effective, Rugged, and Safe, the QuEChERS method is based on work done and published by Anastasias et al

(2016). QuEChERS was developed using an extraction method for pesticides in fruits and vegetables, coupled with a cleanup method that removes sugars, lipids, organic acids, sterols, proteins, pigments, and excess water. This technique offers a user-friendly alternative to traditional liquid and solid phase extractions. The process involves two simple steps. First, the homogenized samples are extracted and partitioned using an organic solvent and salt solution. Then, the supernatant is further extracted and cleaned using a dispersive solid phase extraction. This is done as follows ten (10) g previously homogenized samples, were placed in a 50 ml centrifuge tube and shaken vigorously for 30 s and then vortex for 1 min. After 10 acetonitrile was added, the centrifuge tube was capped and agitated for 3 min. After adding 4 g anhydrous magnesium sulfate and 1 g sodium chloride, the sample was mixed vigorously by overtaxing for 1 min and centrifuge extracted for 5 min at 3600 rpm. 1 ml of acetonitrile layer was transferred into a 2 ml micro-centrifuge tube containing 50 mg PSA sorbent and 150 mg anhydrous magnesium sulfate. The sample was mixed vigorously by overtaxing for 1 min and centrifuge extracted for 2 min at 4000 rpm (Zhang et al. 2012). The residues of pymetrozine and Penconazole were re-dissolved with 2ml of ethyl acetate and determined by GC for Penconazole and HPLC for pymetrozine.

#### **6.2: Soil samples:**

Residue of pymetrozine and Penconazole were extracted from soil samples as follows: 50g representative soil sample was transferred into a 500 ml stopper conical flask and extracted by shaking mechanically with 150 mL of ethyl acetate for 30 min. The extract was carefully decanted and filtered through a clean pad of cotton into 100 mL graduated cylinder. The filtrate was concentrated by using a rotary vacuum evaporator at 35°C. The residues were re-dissolved with 2ml of ethyl acetate and determined by GC for Penconazole and HPLC for pymetrozine.

#### **7: Determination of household preparation effects**

##### **7.1: Washing:**

The pea samples were washed after 0, 1, and 3 days of spraying by tap water for 3 minutes with gentle hand wash (*Walter et al., 2000*) and then analysed as described above.

##### **7.2: Peeling**

pea were peeled after 0, 1, and 3 days of spraying.

##### **7.3:Cooking procedure:**

Pea samples after 0, 1, and 3 days from spraying were cooked for 15 min at the boiling temperature and analysed as described above.

#### **8: GC and HPLC Determination**

##### **8.1: GC Analysis conditions**

Penconazole was determined by Agilent 6890 series gas chromatography (GC) system equipped with an Agilent 7673 auto-sampler, an electron- capture detector. A 30 m × 0.32 mm capillary column coated with a 0.25 µm thick film of 5 % phenyl methyl polysiloxane (HP-5) from Hewlett and Packard was used in combination with the following oven temperature program:Initial temperature 230°C for 2 min, 6°C/min up to 280°C and held for 1 min. Nitrogen Carrier gas 4 ml/min., split less injection of a 1µl volume was carried out, the detector and injector temperatures was 300°C under these conditions the retention times was 10. min for penconazole .

#### **8.2: HPLC Analysis conditions**

Pymetrozine residues were estimated by Agilent 1100 HPLC equipped with diode array detector. A hyper cell ODS analytical column (150 mm × 4.6mm i.d, 5µl) was used. The mobile phase was (methanol/acetonitrile/water) (45/40/15, v/v/v) with a flow rate of 0.8 ml/min., and the injection volume was 20µl. Detection wavelength set at 240 nm. At this condition the retention time of pymetrozine was 7.9 min.

#### **9: Recovery studies:**

Recovery studies were carried out to define the efficacy and the limit of quantization of the method used. The untreated samples of pods, peels, seeds and soil were fortified with pesticides used penconazole and pymetrozine solutions level 1, 0.5 and 0.1 mg kg<sup>-1</sup>, then the procedures of extraction, cleaning up and determination were performed as previously mentioned. Averages of recovery from samples were illustrated in Table 2, the recovery values were calculated according to the following formula:

$$\text{Recovery \%} = [(\mu\text{g pesticide residue/g. sample found}) / (\mu\text{g pesticide residue/g. sample added})] \times 100$$

#### **10: Residues calculation:**

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

$$\text{Ps.B.V/Pst.G.C} \times F$$

*Where:*

$$F=100/R \text{ recovery factor}$$

Pst = standard peak area. R =average of recovery.

V = final volume of sample solution (ml).

Ps = sample peak area.

B = amount of standard injected (ng).

G = sample weight

C = volume of sample solution injected (µl).

#### **11: Half-life times (t<sub>1/2</sub>) calculation;**

The dissipation kinetics of pesticide residues were determined by plotting residue against elapsed time of application, and equation of best curve fit with maximum coefficients of determination (R<sup>2</sup>) was determined. For dissipation of targeted pesticides in the samples, exponential relationship was found to be

applicable corresponding to the general first-order kinetics equation:

$$Ct = C0e^{-kt}$$

**Where** Ct represents the concentration of the pesticide residue at the time of t, C0 represents the initial deposits after application 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 pesticides.

**Half-life times (t<sub>1/2</sub>)** and the degradation behavior of penconazole and pymetrozine were calculated

mathematically according to *Moye et al. (1987)*. Using the following equation:

$$RL50 (t_{1/2}) = \ln 2 / K = 0.6932 / K$$

$$K = 1 / tx * \ln a / bx$$

**Where:-**

K = rate of decomposition.

tx = time in days.

a = initial residue .

bx = residue at x time\|

**Table 2.** Recovery percentage of pymetrozine and penconazole in / on (Peels – Seed) of peas fruits'

| Fortified level (mg/kg)(n=5) | Recovery (%) |             |             |             |
|------------------------------|--------------|-------------|-------------|-------------|
|                              | Peels        |             | seeds       |             |
|                              | pymetrozine  | penconazole | pymetrozine | penconazole |
| 1.0                          | 100          | 91.24       | 100         | 99.2        |
| 0.5                          | 99.31        | 96.01       | 96.94       | 100         |
| 0.1                          | 93.21        | 88.31       | 91.03       | 100         |

## Results and discussion

### 1: Recovery results

The accuracy of the proposed methods was evaluated via the recovery experiment with fortified samples at three fortification levels, 1, 0.5 and 0.1mgkg<sup>-1</sup>, each replicated three times. Results in **Table (2)** showed that the recovery values of pymetrozine were 100, 99.31 and 93.21% in pea peels, respectively and 100, 96.94 and 91.03% in pea seeds, respectively. The average recovery percentages of penconazole were 91.24, 96.01 and 88.31% in pea peels, respectively. While, in pea seeds were 99.2, 100 and 100%, respectively. The recovery values of pymetrozine in squash were 87.01, 90.34 and 85.21%, respectively. However, the recovery values of penconazole were found to be 91.34, 94.91 and 90.54%, respectively. Where, The recovery values of pymetrozine in pepper 99.1, 90.01 and 83.71% and penconazole 100, 100 and 95.01% respectively according to document No.SANCO / 12495/2011 (**European Commission 2011**), the recovery rate should be between 70 and 120%, and the results in this study are within the acceptable range for residue determination (*Cabras et al.1990 and Khay et al. 2008*).

### 2: Persistence of Pesticide Residues in / on Peas Fruits:

#### 2.1- Pymetrozine:

Results in **Table (3)** and **Fig (1)** showed the concentration of initial deposits of pymetrozine in and on pea's pods. Data indicated that the initial deposits after one hour after treatment was 2.08 mgkg<sup>-1</sup> this value begins to decrease to 1.99, 0.78,

0.63, 0.02 and 0.01 mgkg<sup>-1</sup> after 1,3,5,8 and 12 days after treatments , respectively. The loss percentages of pymetrozine were 4.33, 62.5, 69.71, 99.04 and 99.52 % respectively. While, between 1 and 15 days after spraying a gradual decrease was observed in residues for pymetrozine it was not detected in pea's pods, 15 days after treatment.

In Table (3) the half-life (T<sub>1/2</sub>), which is the time required for a quantity pesticide to reduce to half of its initial value, was differed from one to the others. On pea's pods, half-life for pymetrozine was 2.5 days after pymetrozine application. The maximum residue limits (MRLs) were 0.02 mg/kg, so the peas could be safely after 8 days of the treatment **Fig (2)**. The results are in agreement with *Gao et. al (2019)* residues of quinalphos reached below detection limit of 0.05 mgkg<sup>-1</sup> after 10 days at recommended dosage. For risk assessment studies, the 10th day wills be safe for consumers for consumption of green pea.

#### 2.2: Penconazole :

The results presented in **Table (3)** and **Fig. (1)** indicate that the initial deposits (one hour after treatment) of penconazole in peas was 3.121 mgkg<sup>-1</sup> than this value begins to decrease to 2.34, 1.85, 1.01, 0.78, 0.03 and 0.009 mgkg<sup>-1</sup> after 1,3,5,8 , 12 and 15 days from treatments, respectively, The loss percentages of penconazole were 25.02, 40.72, 67.64, 75, 99.04 and 99.71 % while, from the samples that were taken 21 days after treatment be no detectable of penconazole .and the half-life value was 3.6 days on pods. The maximum residue limits (MRLs) is 0.05 mgkg<sup>-1</sup>, so the peas could be safely EATEN after 12 days form treatment **Fig (3)**.

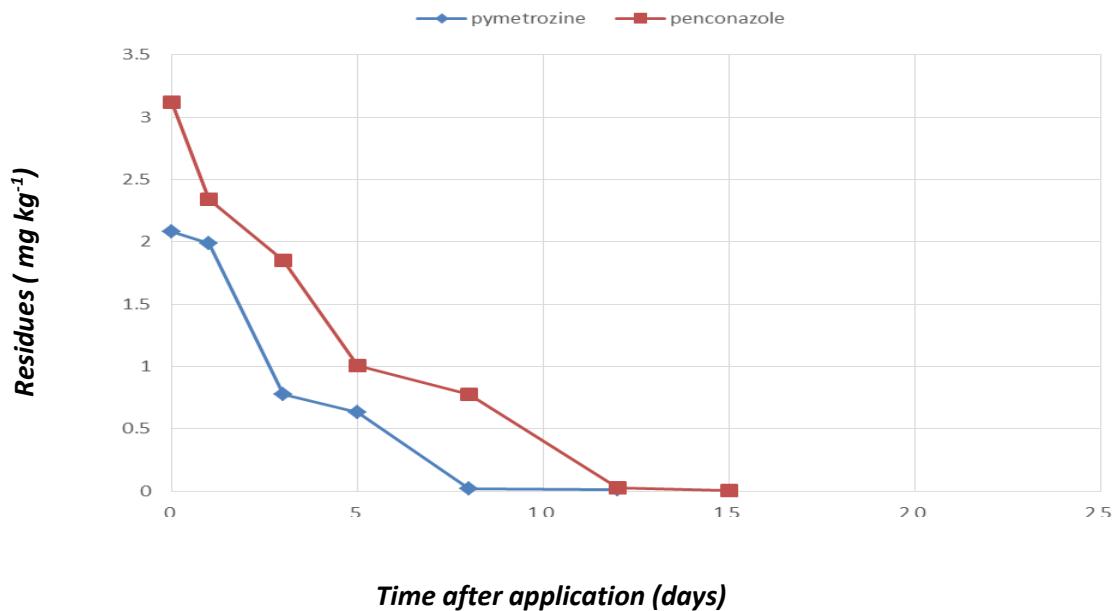
**Table 3.** Pymetrozine and penconazole residues in/on peas fruits.

| Time after treatment (days) | Pymetrozine        |        |               | penconazole       |       |               |
|-----------------------------|--------------------|--------|---------------|-------------------|-------|---------------|
|                             | Residues mg/kg± SE | Loss % | Persistence % | Residues mg/kg±SE | Loss% | persistence % |
| 0*                          | 2.08 ± 0.43        | 0      | 100           | 3.121± 0.72       | 0     | 100           |
| 1                           | 1.99 ± 0.31        | 4.33   | 95.67         | 2.34 ± 0.50       | 25.02 | 74.98         |
| 3                           | 0.78 ± 0.07        | 62.5   | 37.5          | 1.85 ± 0.09       | 40.72 | 59.28         |
| 5                           | 0.63 ± 0.31        | 69.71  | 30.29         | 1.01 ± 0.11       | 67.64 | 32.36         |
| 8                           | 0.02 ± 0.01        | 99.04  | 0.96          | 0.78 ± 0.26       | 75    | 25            |
| 12                          | 0.01 ± 0.01        | 99.52  | 0.48          | 0.03 ± 0.01       | 99.04 | 0.96          |
| 15                          | ND                 | 100    | 0             | 0.009 ± 0.00      | 99.71 | 0.29          |
| 21                          | ND                 | 100    | 0             | ND                | 100   | 0             |
| T <sub>1/2</sub> (days)     | 2.5                |        |               | 3.6               |       |               |
| MRLs (mg/kg)                | 0.02               |        |               | 0.05              |       |               |
| PHI                         | 8                  |        |               | 12                |       |               |

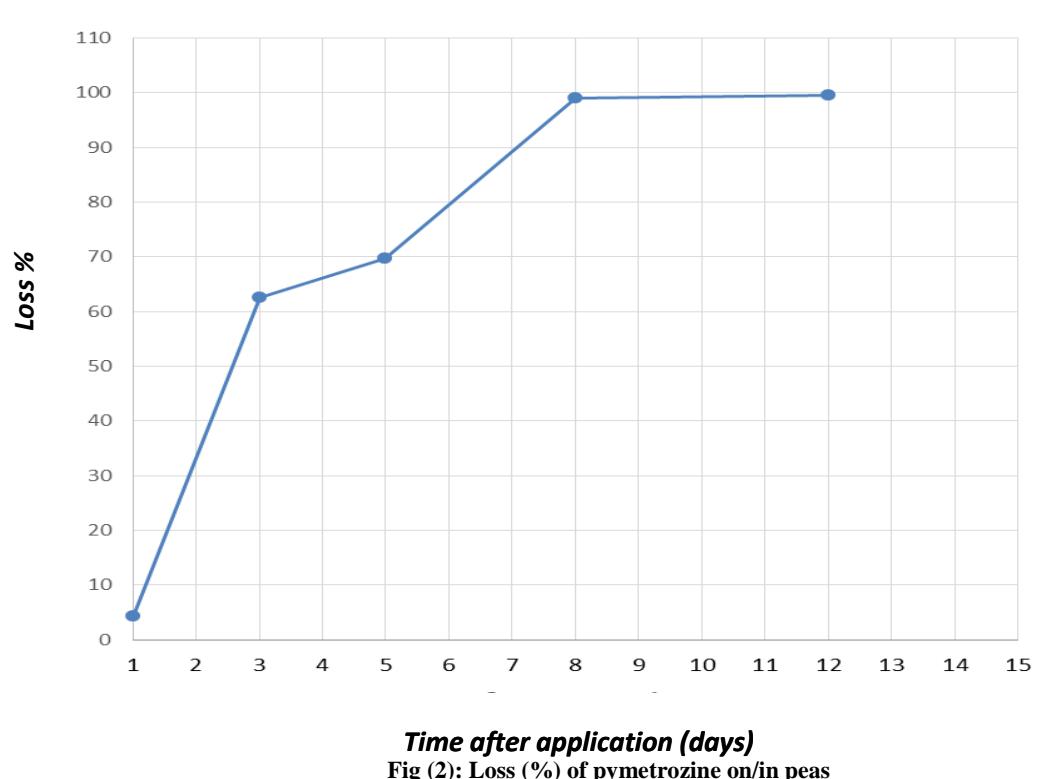
\* Samples were taken after one hour of application

Half-life (t<sub>1/2</sub>) Half life value MRLs : Maximum residual limits

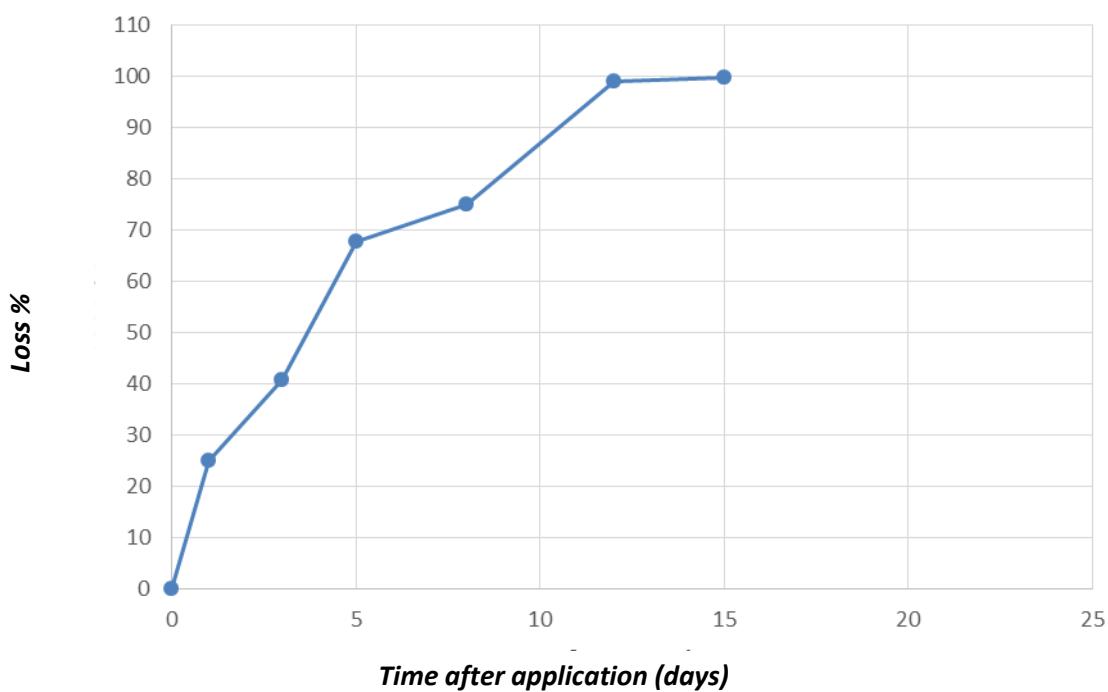
PHI: Pre-harvest intervals ND: Not Detected SE: Standard Error



**Fig (1):** Pymetrozine and penconazole residues in/on peas fruits.



**Fig (2): Loss (%) of pymetrozine on/in peas**



**Fig (3): Loss (%) for penconazole on/in peas**

**Table 4.** Effect of washing, peeling and boiling on residues of pymetrozine in and on peas

| Time | Before preparations | Washed pods              |              | Seed              |              | Peels             |              | Boiling           |              |
|------|---------------------|--------------------------|--------------|-------------------|--------------|-------------------|--------------|-------------------|--------------|
|      |                     | mg/kg± SE                | Loss %       | mg/kg±SE          | Loss%        | mg/kg±SE          | Loss %       | mg/kg±SE          | Loss %       |
| 0*   | <b>2.08 ± 0.43</b>  | <b>0.85±0.<br/>233</b>   | <b>0</b>     | <b>0.052±0.00</b> | <b>0</b>     | <b>1.94±0.470</b> | <b>0</b>     | <b>0.03±0.027</b> | <b>0</b>     |
| 1    | <b>1.99 ± 0.31</b>  | <b>0.131±0.<br/>.059</b> | <b>84.59</b> | <b>0.061±0.00</b> | <b>83.8%</b> | <b>1.58±0.480</b> | <b>18.57</b> | <b>0.02±0.007</b> | <b>33.33</b> |
| 3    | <b>0.78 ± 0.07</b>  | <b>0.08±0.<br/>049</b>   | <b>90.59</b> | <b>0.03±0.013</b> | <b>0</b>     | <b>0.73±0.153</b> | <b>62.37</b> | <b>ND</b>         | <b>0</b>     |

**Table 5.** Effect of washing, peeling and boiling on residues of penconazole in / on peas

| Time day | Before preparations | Washed pods             |              | Seeds                   |             | Peel                    |              | Boiling                 |          |
|----------|---------------------|-------------------------|--------------|-------------------------|-------------|-------------------------|--------------|-------------------------|----------|
|          |                     | mgkg <sup>-1</sup> ± SE | Loss %       | mgkg <sup>-1</sup> ± SE | Loss %      | mgkg <sup>-1</sup> ± SE | Loss %       | mgkg <sup>-1</sup> ± SE | Loss %   |
| 0        | <b>3.121± 0.72</b>  | <b>1.94±0.<br/>.307</b> | <b>0</b>     | <b>0.039±0.00</b>       | <b>0</b>    | <b>3.012±0.72</b>       | <b>0</b>     | <b>0.056±0.01</b>       | <b>0</b> |
| 1        | <b>2.34 ± 0.50</b>  | <b>0.981±<br/>0.513</b> | <b>49.43</b> | <b>0.035±0.00</b>       | <b>10.2</b> | <b>1.981±0.21</b>       | <b>34.22</b> | <b>0</b>                | <b>0</b> |
| 3        | <b>1.85 ± 0.09</b>  | <b>0.324±<br/>0.037</b> | <b>83.3</b>  | <b>0.017±0.00</b>       | <b>56.4</b> | <b>1.07±0.033</b>       | <b>64.47</b> | <b>0</b>                | <b>0</b> |

\* Samples were taken after one hour of application

ND: Not Detected SE: Standard Error

### 3: The effect of washing, peeling and boiling on residues of pymetrozine and penconazole

#### 3.1: Pymetrozine

Data in Table (4) clearly show the effect of home processing like washing with tap water, peeling and boiling on the reduction of pymetrozine residues in peas fruits (pod, seed and peels). Result indicated that during the samples 1 h, one and three days after spraying, the removal percentages of pymetrozine residue by washing was 84.59% after one day from the application, then the reduction was reached 90.59 % after 3 days . On the other hand, the removal percentages of pymetrozine in seeds ranged where 14.75 and 50.81% after one and three days after application respectively. While the removal percentages due to peeling were 0, 18.57 and 62.37% after one hour, 1 day and 3 days of the treatment, respectively. Boiling removed 33.33% after one day from the application, and the pymetrozine pesticide residues were completely demolished in the samples collected after 3days. The initial amounts of pymetrozine in unwashed pods were higher than that in washed pods, seeds and peels, respectively. The results also showed that there were significant different between the peeling , the washing and boiling process, the boiling recess decreased the amount of pymetrozine residue much more than peeling and washing.

#### 3.2: Penconazole

Data in Table (5) showed showed that the effect of home processing on the reduction of penconazole residues in peas pods. The removal percentage of penconazole residue by washing

ranged between 49.43 and 83.3 % respectively. While the removal percentages by peeling were 0, 34.22 and 64.47 % after one hour, 1 day and 3 days of treatment, respectively. The highest removal percentage was 100% for the boiled samples collected after one day from the penconazole application. The results also showed that there were significant differences between the peeling, washing and boiling process, the boiling process was the most effective for reducing the residues penconazole on much more than peeling and washing. These results are quite comparable with those reported by many investigators regardless of experiments were carried out under different climate. *Joshi et.al (2015)* concluded that processing substantially lowers the residues of pesticides in garden pea. It was found that washing; boiling and cooking process minimized the pesticide residues of twelve pesticides in the range of 3.77-39.46, 6.46-87.32 and 42.97-98.20 per cent, respectively. The percentage reductions in the present study are supported by both early and most recent publications. These reductions are extremely important in evaluating the risk associated with ingestion of pesticide residues, especially in vegetables, which are eaten by almost all income groups' people. The best methods for removal of these contaminants' pesticide residues in these plant products from food of plant origin are food processing technologies, which affect the levels of pesticide residues to various degrees. *Lozowicka and Jnkowska (2014)and (Ali et al 2020)* On the other hand, the different levels of initial deposits of both tested pesticides in peas due to many factors; evaporation of the surface residue which is

dependent on temperature condition, chemical or biochemical decomposition, metabolism and photolysis. (*Shokr and Nasr, 2006*).

The above results seem to show that the half-life value of pymetrozine and penconazole were 2.5 and 3.6 days in peas. These results are quite comparable with those reported by many investigators regardless of experiments were carried out under different climate conditions, geographical locations, time of application, application technique, concentrations, type of formulation and plant species. who studied the residues of tetriconazole and penconazole on and in some vegetable crops grown in greenhouse and found that the half-life values of these fungicides were 1.78 and 1.5 day in cucumber fruits. (*Barakat et al., 2006*) showed that diniconazole initial deposit and half-life value in the whole green pods of peas were 1.165 ppm and 72 hours, respectively. (*Romeh et al. 2001*) evaluate the effects of household processing such as: washing with water, washing with 10% of vinegar solution and washing with 10% of sodium bicarbonate solution on pesticide residue levels of acetamiprid, azoxystrobin, diflubenzuron, dimethoate, fipronil, imidacloprid, procymidone and thiamethoxam, in tomato samples (*Graziela C. R. M. Andrade, 2015*). Dissipation of penconazole was estimated in tomatoes fruits cultivated in field using QuEChERS method for sample preparation and high-performance liquid chromatography with diode array detector. Following one application of normal dose 25 mL/100 L water, the average initial deposits of penconazole were observed to be 0.74 and 1.21 mg/kg for tomatoes fruits and soil, respectively. (*Abd-Alrahman and Ahmed 2012*) investigated the persistence of diniconazole and etoxazole in broad bean pods, peels, seeds and soil. Samples were taken after zero, 1, 3, 5, 7, 9, 14 and 21 days after pesticides application. (*Shams EL Din et al. 2015*) Study effect the washing with water or soaking in solutions of salt and some chemicals e.g. Chlorine, chlorine dioxide, hydrogen peroxide, ozone, acetic acid, hydroxy peracetic acid, iprodione and detergents are reported to be highly effective in reducing the level of pesticides. Preparatory steps like peeling, trimming etc. Remove the residues from outer portions.

#### **4. The studied pesticides in Soil under pea's plants:**

##### **4.1: Pymetrozine**

**Table (6)** summarized that pymetrozine one hour after application in soil had high initial residues under pea plants. The maximum residue limits (MRLs) of pymetrozine in soil under pea plants were 0.31 mg/kg. The pymetrozine residues reduced to 0.151, 0.021 mg/kg after one and three days of application, respectively and not detected in soil under pea plants after 15 days from applications.

##### **4.2: Penconazole**

Results in **Table (7)** showed the concentration of initial deposits of penconazole on soil under pea plants one hour after application was 0.316 mg/kg. Samples of soil under pea plants were taken one day after application contained 0.056 mg/kg with loss 82.28% of the initial amounts of penconazole. The residues reduced to 0.031, 0.024 and 0.005 mg/kg after 3, 15 and 21 days after treatment, respectively. The loss percentage of penconazole on soil under pea plants were 90.19 and 98.42%, between days 3 and 21 days after treatment respectively.

**Table 6.** Residue of pymetrozine on soil under pea plants

| Time<br>day | mg/kg <sup>±</sup> SE | Loss%        | persistence % |
|-------------|-----------------------|--------------|---------------|
| 0*          | <b>0.31±0.013</b>     | <b>0</b>     | <b>100</b>    |
| 1           | <b>0.151±0.008</b>    | <b>51.2</b>  | <b>48.8</b>   |
| 3           | <b>0.021±0.009</b>    | <b>93.22</b> | <b>6.78</b>   |
| 15          | <b>ND</b>             | <b>100</b>   | <b>0</b>      |

**Table 7.** Residue of penconazole on soil under pea plants

| Time<br>day | mg/kg <sup>±</sup> SE | Loss %       | persistence % |
|-------------|-----------------------|--------------|---------------|
| 0*          | <b>0.316±0.009</b>    | <b>0</b>     | <b>100</b>    |
| 1           | <b>0.056±0.002</b>    | <b>82.28</b> | <b>17.72</b>  |
| 3           | <b>0.031±0.027</b>    | <b>90.19</b> | <b>9.81</b>   |
| 15          | <b>0.024±0.013</b>    | <b>92.41</b> | <b>7.59</b>   |
| 21          | <b>0.005±0.001</b>    | <b>98.42</b> | <b>1.58</b>   |

\* Samples were taken after one hour of application

ND: Not Detected SE: Standard Error

#### **Conclusion**

In these study residues on peas collected after one hour, 1, 3, 5, 8, 12, 15 and 21 days from last spray. This study obtained that the residue level of pymetrozine and penconazole were less than the Maximum Residue Level which recommended by Codex Alimentation Commission. Also, this study showed that penconazole high persistence than pymetrozine. the pre-harvest interval (PHI) of pymetrozine and penconazole were 15 and 12 days after application for peas fruits respectively. Household operations on peas had a clear effect on the destruction of pesticide residues. Pesticide residues were gradually destroyed in the soil samples

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## متبييات البيميتروزين والبينكونازول على وفي ثمار البازلاء

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تم تقدير بقايا البيميتروزين والبينكونازول في ثمار البازلاء باستخدام QuEChERS HPLC-DAD مع طريقة GC-ECD. تم تنفيذ التطبيق باستخدام رشاش محمول على الظهر مزود بفوهة واحدة. تم أخذ العينات بعد ساعة و 1 و 3 و 5 و 8 و 11 و 15 و 21 يوماً من العلاج. أظهرت البيانات التي تم الحصول عليها أن المستوى المتبقى من البيميتروزين كان أقل من الحد الأقصى للمخلفات ( $MRL = 0.02$  مجم / كجم) الذي أوصت به هيئة الرقابة على الغذاء ، وكذلك البينكونازول ( $MRL = 0.05$  مجم / كجم) ، وأظهرت البيانات أن بقايا البينكونازول كانت ذات مستوى عالي من الثبات وكانت فترة نصف عمر المبيد ( $t_{1/2} = 3.6$  أيام) بينما سجل البيميتروزين ( $t_{1/2} = 2.5$  يوم) على ثمار البازلاء على التوالي. كانت فترة ما قبل الحصاد (PHI) لكل من البيميتروزين والبينكونازول 8 و 12 يوماً بعد معاملة ثمار البازلاء على التوالي. وأشارت نتائج المعالجة المنزلية (الغسيل بالماء ، النقشير والغليان) لثمار البازلاء إلى وجود بعض طرق الأمان لتقليل مستوى مخلفات البيميتروزين والبينكونازول.