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Residues of phenthoate and chlorfenapyr in peach orchard

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ABSTRACT

The residual behavior of phenthoate and chlorfenapyr on peach was studied. In addition, the effect of some processing on the degradation of phenthoate and chlorfenapyr was also investigated. The results revealed that the rate of disappearance (loss) was faster in case of chlorfenapyr than phenthoate treatments. The half life values of phenthoate in unwashed peach fruits, leaves and soil were 2.63, 4.68 and 1.8 days, respectively, while in case of chlorfenapyr, these values were 1.0, 2.68 and 1.5 days, respectively. Rinsing treated fruits with tap water removed considerable amounts of phenthoate and chlorfenapyr. The dislodgeable amounts of the pesticide residues by washing decreased as the residues on the fruits decreased by time elapsing. Unwashed and washed fruits could be safely consumed by consumer after 21 and 14 days of phenthoate and chlorfenapyr, respectively. Processed jam from peach fruits collected one hour after spraying either with phenthoate or chlorfenapyr could be used safely directly after processing.

INTRODUCTION

Peach fruits are considered one of the important fruits for human consumption and exportation in Egypt. Peach trees are liable to be infested with different insect pests and mites that cause significant injury and reduce final yield. Therefore, phenthoate (Cidial 50% EC) (non-systemic organophosphorus insecticide) is recommended to be applied against certain pests on several crops such as apricot, apple, fig, peach, pear, plum and olive (Li *et al.*, 2002 and Attala, 2006). Chlorfenapyr (Challenger 36% SC) is a novel broad-spectrum insecticide miticide currently registered in different countries for the control of various insect and mite pests on different field crops; ornamentals, vegetables and fruit crops. This compound is effective against different insect pests and mites which are

typically resistant to carbamate, organophosphate, and pyrethroid insecticides as well as chitin synthesis inhibitors (Cao *et al*., 2005).

The present work was conducted to determine the residues of phenthoate and chlorfenapyr in peach leaves, fruits and soil. The effects of certain household processes on the removal of phenthoate and chlorfenapyr residues from treated fruits were also studied.

MATERIALS AND METHODS

I- Residues of phenthoate and chlorfenapyr on and in peach fruits, leaves and soil:

1- Field experiments: To study the residual behavior of phenthoate and chlorfenapyr on peach, a filed experiment was carried out in a private orchard (garden) of peach located in EL-Sadat city, Minnufiya Governorate during 2007 season. The tested insecticides and their rates of application were:

A) Phenthoate 50% EC (Cidial) 200 cm^3 / 100 liter of water.
B) Chlorfenapyr 36% SC (Challenger) 40 cm^3 / 100 liter of water.

Peach trees *Prunus persica* varity Florida Prince were treated with phenthoate and chlorfenapyr on April 2, 2007. Each treatment was carried out on 9 trees. Representative samples from soil surfaces, leaves and fruits were taken at random after one hour, 1, 3, 7, 10, 14 and 21 days of treatment for residue analysis.

To assess the effect of household processing, fruit samples were divided into two sub samples: the first sub sample was washed with tap water and let for air drying, while the second one was left without washing. Fruit samples taken after one hour were divided into three sub samples: the third sub sample was washed with tap water, air dried and subjected to jam preparing processes.

- The jam was prepared by cooking weighed amount of peach fruits with sugar (1:1 w/w), after which the jam was cooled to room temperature and acidulated with citric acid (3 g/kg sugar), filled on glass jars and kept for residue analysis (EL-Behissy *et al*., 2001 and Kurz *et al*., 2008).

2- Residues analysis:

2.1. Extraction and clean up of soil samples: Soil was crushed into a hummer mill and then sieved to obtain the same size granules (40 – 120 mesh) as mentioned by Cao *et al.* (2005) and 50 g soil was subjected to extraction and clean up procedures.

Residues of phenthoate and chlorfenapyr were extracted from soil and the extract was cleaned up according to the procedures of Abo-El-Soud *et al.* (1995), where the pesticides were extracted from soil samples by socking overnight with a mixture of acetone and water 70:30 (v/v) and then shaken for one hour on an electric shaker.

2.2. Extraction and clean up of plant samples:

2.2.1 Phenthoate residues: Fruit samples (50 g) and leaf samples (20 g) treated with phenthoate were extracted as mentioned by Mollhof (1975), this method modified to use methanol instead of acetone as a solvent for the extraction of phenthoate. Methanol extract was then partitioned with methylene chloride. The dry extract was then subjected to the clean up procedure suggested by Mills *et al.* (1972) using florisil chromatographic column where phenthoate residues were eluted using a mixture of methylene chloride (50%): n-hexane (48.5%): acetonitrile (1.5%).

2.2.2. Chlorfenapyr residues: Peach samples treated with chlorfenapyr were extracted and cleaned up according to the procedures of Abo-El-Soud *et al.* (1995). Fruit or leaf samples were extracted with distilled acetone, then the extract was partitioned with methylene chloride. The concentrated extract was mixed with activated charcoal and sodium sulphate after dissolving in n-hexane, and the slurry was allowed to settle, then the clear layer was chromatographed through 5g silica gel column chromatography where chlorfenapyr was eluted with n-hexane.

2.3. Final determination

2.3.1. Gas chromatography conditions for quantitative analysis of phenthoate: Agilent Technologies 7890A gas chromatograph equipped with flame photometric detector (FPD) operated in the phosphorus mode (526 nm filter) was used for determination of phenthoate. The column was HP-5 (30 m × 0.32 mm × 0. 25 µm film thickness) coated with 5% phenyl

and 95% dimethylpolysiloxane. Injector temperature was 250°C, detector temperature was 250 °C, column temperature was 230 °C. Gases flow rates were 60, 30 and 30 ml/min. for nitrogen, hydrogen and air, respectively (Hegazy *et al.*, 2006). Under the previous conditions, phenthoate retention time was 3.14 min.

2.3.2. HPLC conditions for quantitative analysis of chlorfenapyr: Agilent 1100 HPLC with UV photo diode array detector (DAD) has proven to be a good alternative for chlorfenapyr determination. Chromatographic separation in Zorbax SB - C_{18} column (4.6×150 mm). The detection at 260 nm wavelength offers suitable chromatograms for the quantification of chlorfenapyr. The mobile phase was methanol: water (80: 20 v/v) with 1 ml min^{-1} flow rate. The column temperature was kept at 25 °C. The volume of the injection loop was 20 µl (Cao *et al.*, 2005). Under the previous conditions, chlorfenapyr retention time was 4.4 min.

Jam processed from peach fruit contaminated with phenthoate or chlorfenapyr was extracted, cleaned up and the residues were determined by GC and HPLC, respectively, as previously mentioned with the exception that 50 ml distilled water was added to the jam sample during the extraction steps.

II. Recovery studies: Untreated control samples of soil, jam processed, peach fruits and leaves were spiked with known amounts of phenthoate and chlorfenapyr prior to extraction and clean up for recovery tests of each pesticide. The spiking levels were 0.5, 1 and 5 ppm. These samples passed through the entire process of extraction then cleaned up and analyzed as previously described (2.1 and 2.2). Following such techniques the average rate of recovery for phenthoate and chlorfenpyr from spiked control samples of fruits, plant leaves, soil and jam was calculated. The obtained results were corrected according to the recovery % (Table 1).

III. Statistical analysis: All data were subjected to statistical analysis according to Timme and Fisher (1980). The half life ($t_{1/2}$) was calculated mathematically according to equation's of Moye *et al.* (1987) from the following equation:

$t_{1/2}$= Ln 2/K

K_0= (1/t_x). Ln a/bx., where:

K_0: is the degradation rate constant at the intervals in hour
K: is the mean of K_0.
a: the residue level at the initial time (zero time).
bx: is the residue level at the successive intervals in hour

Table (1): The average recovery percentages of phenthoate and chlorfenapyr from spiked samples of peach fruits, leaves, soil and jam.

Pesticides	Sample types	Added concentration (mg/kg)	Mean detected concentration (mg/kg)	Mean recovery %
chlorfenapyr	Soil	5	4.509 ± 0.0085	90.18
		1	0.940 ± 0.0057	94.00
		0.5	0.490 ± 0.0057	98.00
	Peach fruit	5	5.044 ± 0.07	100.88
		1	1.054 ± 0.0083	105.38
		0.5	0.535±0.00049	106.91
	Leaves	5	4.709 ± 0.071	94.19
		1	0.966 ± 0.0064	96.60
		0.5	0.479 ± 0.0056	95.79
	Jam	5	4.479 ± 0.023	89.59
		1	0.920 ± 0.015	92.68
		0.5	0.460 ± 0.0028	93.87
phenthoate	Soil	5	4.545 ± 0.025	90.54
		1	0.929 ± 0.007	92.90
		0.5	0.469 ± 0.0035	93.87
	Peach fruit	5	4.518 ± 0.016	90.36
		1	0.948 ± 0.0064	94.81
		0.5	0.483 ± 0.0028	96.53
	Leaves	5	4.569 ± 0.04	91.38
		1	0.939 ± 0.00071	93.88
		0.5	0.497± 0.0035	99.33
	Jam	5	4.695 ± 0.027	93.89
		1	0.970 ± 0.0014	97.05
		0.5	o.480 ± 0.0007	96.89

RESULTS AND DISCUSSION

Residues of phenthoate and chlorfenapyr on and in peach fruits, leaves and soil:

1. Residues of phenthoate on and in peach fruits:

Data presented in Table (2) show the residues dissipation of phenthoate in unwashed and washed fruits as well as the effect of the processing of peach fruits. In case of unwashed fruits, the results show that initial amounts of phenthoate in unwashed fruits as determined after one hours of application were 2.045 ± 0.023 mg/kg. Such amount decreased to be 1.75 ± 0.152, 0.982 ± 0.087, 0.472 ± 0.093, 0.39 ± 0.049, 0.23 ± 0.088 and 0.047 ± 0.002 mg/kg after 1, 3, 7, 10, 14 and 21 days from spraying, respectively recording 14.43, 51.98, 76.92, 80.93, 88.75 and 97.70% loss after 1, 3, 7, 10, 14, and 21 days of application, respectively.

The effect of washing with tap water on the values of phenthoate residues detected in peach fruits was estimated and presented in Table (2). It is obvious that the initial amount decreased to be 1.185 ± 0.216 mg/kg indicating 42.05 % loss by washing. Data also show that phenthoate dissipated from washed fruits and reached to 0.047 ± 0.002 mg/kg after 21 days from the onset of insecticidal treatment. The loss percentages of phenthoate residues ranged from 42.05 to 2.61 due to washing the treated peach fruits with tap water. Data also revealed that processing of peach fruits to jam decreased the residues of phenthoate to be undetectable amounts (UND).

2. Residues of phenthoate on and in peach leaves and soil:

Table (3) shows that the leaves had 8.805 ± 0.022 mg/kg initial deposit. The residues were dropped to 6.115 ± 0.045 mg/kg recording 30.55 % loss after 1 day of spraying. Thereafter the residues were gradually decreased by time. The residues reached 0.305 ± 0.091 mg/kg indicating 96.54 % loss of initial deposit after 21 days of spraying.

Table: (2) Effect of washing and processing to jam on phenthoate residues on and in peach fruits.

Days after treatment	Unwashed fruits		Washed fruits		Processed fruits (jam)	
	Residues (mg/kg)	% loss	Residues (mg/kg)	% reduction	Residues (mg/kg)	% reduction
*Initial	2.045 ± 0.023	0.00	1.185 ± 0.216	42.05	**UND	~ 100
1	1.75 ± 0.152	14.43	1.21 ± 0.044	30.86		
3	0.982 ± 0.087	51.98	0.76 ± 0.052	22.61		
7	0.472 ± 0.093	76.92	0.42 ± 0.032	11.02		
10	0.39 ± 0.049	80.93	0.369 ± 0.011	5.38		
14	0.23 ± 0.088	88.75	0.224 ± 0.007	2.61		
21	0.047 ± 0.002	97.70	0.047 ± 0.002	0.00		
t½ in days	2.63					
MRL	0.1 ppm					
PHI in days	21					

*Initial = one hour after treatment
**UND = undetectable amounts
LOD (limit of detection) = 0.001ppm

Table: (3) Residues of phenthoate on and in peach leaves and in soil under peach treated trees.

Days after treatment	leaves		Soil	
	Residues (mg/kg)	% loss	Residues (mg/kg)	% loss
*Initial	8.805 ± 0.022	0.00	0.461±0.011	0.00
1	6.115 ± 0.045	30.55	0.458±0.073	0.65
3	5.39 ± 0.064	38.28	0.076±0.002	83.51
7	3.695 ± 0.098	58.04	0.049±0.009	89.37
10	2.935 ± 0.066	66.67	**UND	~ 100
14	0.978 ± 0.031	88.89	UND	~ 100
21	0.305±0.091	96.54	UND	~ 100
t½ in days	4.68		1.8	

*Initial = one hour after treatment
**UND = undetectable amounts
LOD (limit of detection) = 0.001ppm

With regard to phenthoate in soil (surface layer, 10-15cm) under treated peach trees, data in Table (3) revealed that the initial amount of phenthoate

in soil after one hour of spraying was 0.461 ± 0.011 mg/kg. The residues were rapidly decreased during the first week after spraying and reached 0.049 ± 0.009 mg/kg indicating 89.37% loss. Half-life values of phenthoate residues in unwashed peach fruits, leaves and in soil were 2.63, 4.68 and 1.8 days, respectively (Tables 2 and 3).

The aforementioned results show that unwashed and washed peach fruits could be safely used for human consumption after 21 days of spraying (PHI=21days) where the residues of phenthoate in such fruits were below the maximum residue level (0.1 mg/kg) which established by, FAO/WHO www.mhlw.go.jp/english/topics/mrls/second/dl/mrls33.pdf. Processed jam from peach fruits collected 1 hour after spraying could be used safely directly after processing.

It is worthy to note that in loss of phenthoate in peach fruits and leaves. more than 50% took place 3 and 7 days following application, respectively. This result revealed that phenthoate was dissipated faster from peach fruits than leaves.

3. Residues of chlorfenapyr on and in peach fruits:

Data presented in Table (4) show that the initial deposit of chlorfenapyr in unwashed peach fruits as determined after 1 hour of spraying was 7.035 ± 0.20 mg/kg. The residues of chlorfenapyr were decreased by time and reached 0.056 ± 0.004 mg/kg after 21 days of spraying, indicating 99.20 % loss of initial deposit.

Data in the same table revealed that washing process of peach fruits previously sprayed with chlorfenapyr decreased the residues by 35.75 to 2.47 %. However, the loss by washing was 35.75, 25.07, 19.24, 10.56,4.55 and 2.47 % from samples collected after 0, 1, 3, 7, 10, 14 and 21 days of spraying, respectively.

Regarding the effect of processing the treated peach fruits on the residues of chlorfenapyr (Table, 4), undetectable amount of chlorfenapyr was recorded in processed jam of treated peach fruits collected after one hour of spraying.

4. Residues of chlorfenapyer on and in peach leaves and soil :

Results in Table (5) represent the amount of chlorfenapyr residues and its persistence through the period of study on and in peach leaves and in soil (surface layer, 10-15cm) under treated peach trees. The initial deposits on and in leaves and in soil were 13.455 ± 0.53 and 4.36 ± 0.98 mg/kg, and decreased to 8.045 ± 0.24 and 3.12 ± 0.039 mg/kg one day after treatment, respectively. These amounts gradually decreased with time lapse to 0.26 ± 0.02 mg/kg and undetectable amount after 21 days of spraying. The loss percentages of chlorfenapyer residues ranged from 40.21 to 98.07 % and 28.44 to 99.95 % in leaves and soil, respectively.

The half life values of chlorfenapyr residues in unwashed peach fruits, peach leaves and soil samples were 1, 2.68 and 1.50 days, respectively (Tables 4 and 5). The maximum residue level (MRL) of chlorfenapyr in peach fruits as established by Australia New Zealand Food Standards Code was 1 ppm (http://www.foodstandards.gov.au/_srcfiles/Standard_1_4_2_MRLs_part_2_Sched_1_A-L_v116.pdf). On the other hand, the residues of chlorfenapyr in unwashed peach fruits and washed fruits were 0.81±0.09 and 0.79±0.07 after 14 days of spraying. Accordingly, peach fruits could be marketed safely after 14 days of spraying (PHI=14 days). Jam processed from peach fruits which collected one hour after spraying could be safely used for human consumption directly after processing. Degradation of chlorfenapyr in peach fruits was faster than in the leaves.

The initial deposits of the two tested pesticides, their dissipation during the whole experimental period, the rate of decomposition, the effect of washing the fruits with tap water, the half-life, as well as the safety periods greatly differed depending on the chemical structure of pesticides as well as the crop. Such fluctuations in these criteria have been confirmed with other investigators working on the residues of the two tested pesticides as well as other pesticides on the same crop or other crops. Howell and George (1984) stated that at harvest, chlorfenapyr residues on peaches were 7-11 times as high on twigs as on fruit. Two applications left a residue level in fruit of 0.053 ppm, just over the 0.05 ppm tolerance limit. Yonce and Brady (1984) reported that when chlorpyrifos was applied on peach trees at 0.9 or 1.8 g

Table: (4) Effect of washing and processing to jam on chlorfenapyr residues on and in peach fruits.

Days after treatment	Unwashed fruits		Washed fruits		Processed fruits (jam)	
	Residues (mg/kg)	% loss	Residues (mg/kg)	% reduction	Residues (mg/kg)	% reduction
*Initial	7.035 ± 0.2	0.00	4.52 ± 0.5	35.75	**UND	~ 100
1	3.51 ± 0.03	50.10	2.63 ± 0.08	25.07		
3	2.91 ± 0.08	58.64	2.35 ± 0.09	19.24		
7	1.61 ± 0.01	77.11	1.44 ± 0.02	10.56		
10	1.32 ± 0.06	81.24	1.26 ± 0.05	4.55		
14	0.81 ± 0.09	88.49	0.79 ± 0.07	2.47		
21	0.056 ± 0.004	99.20	0.056 ± 0.004	0.00		
t½ in days	1.0					
MRL	1					
PHI in days	14					

*Initial = one hour after treatment
**UND = undetectable amounts
LOD (limit of detection) = 0.01ppm

Table: (5) Residues of chlorfenapyr on and in peach leaves and in soil under treated trees.

Days after treatment	leaves		Soil	
	Residues (mg/kg)	% loss	Residues (mg/kg)	% loss
*Initial	13.455 ± 0.53	0.00	4.36±0.98	0.00
1	8.045 ± 0.24	40.21	3.12 ± 0.039	28.44
3	5.83 ± 0.84	56.67	1.26 ± 0.047	71.10
7	4.11 ± 0.33	69.45	0.56 ± 0.049	87.16
10	2.98 ± 0.09	77.85	0.14 ± 0.082	96.79
14	1.92 ± 0.01	85.73	0.002 ± 0.001	99.95
21	0.26 ± 0.02	98.07	**UND	~ 100
t½ in days	2.68		1.50	

*Initial = one hour after treatment
**UND = undetectable amounts
LOD (limit of detection) = 0.01ppm

a.i./litre, its concentration on peaches was reduced to 0.05 ppm (the current tolerance level) or less at 5 and 8 weeks, respectively after treatment. Adzhemyan *et al.* (1985) found that residues of phosalone in peach fruits had fallen to the MRL after one week of spraying, and had disappeared after 20 days. Hameed *et al.* (1985) reported that the waiting period before the peach fruit could be safely consumed was 14-16 days after fenitrothion treatment. Kashyap and Hameed (1986) noticed that washing methyl-parathion, fentrothion, trichlophon and fenthion treated peach fruits could not be consumed earlier than 7 days after spraying. Washed malathion treated fruits were safe to eat after one day. Karageorgiev (1987) stated that chlorpyrifos residues were declined rapidly in treated cherries, pears, peaches and apple. The waiting period for these fruits was 21 days after treatment. Zidan *et al.* (1991) studied the effect of jam processing on the elimination of insecticide residues (fenitrothion and fenpropathrin) from fig fruits. They stated that jam product was free of insecticide residues. Lentza-Rizos and Chitzanidis (1996) determined dicloran residues in peach fruits and found that washing of fruits removed about 40 % of the residues present before washing. Marudov *et al.* (1999) reported that washing with water, sodium hydrogen carbonate or sodium hydroxide solutions for peach fruits removed Dursban residues from 49.9 to 54.8 %. Navarro *et al.* (2001) found that heat process destroyed the remaining acephate residues in peach. Tsatsakis *et al.* (2002) reported that the residues levels of methyl parathion on fruits after 4 days were lower than the maximum residue limit indicating a safe harvest. Cao *et al.* (2005) found that the half-lives of chlorfenapyr nanoformulation and suspension concentration in soil were about 4.3 days and 3.9 days, respectively. The degradation rate of chlorfenapyr nanoformulation was faster than that suspension concentration and chlorfenapyr nanoformulation was safer than suspension concentration. Balinova *et al.* (2006) investigated the effect of fruit processing on pesticide residues in peaches, and the results revealed that thermal treatment substantially reduced organophosphate (chlorpyrifos-methyl and fenitrothion) residues. Beena *et al.* (2007) estimated the pesticide residues in some popular brands of jam. The contamination observed was 100% due to organochlorines and 25-75% to synthetic pyrethroids and organophosphates, the residues of any pesticide did not exceed the MRL values prescribed for fruits.

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متبقيات الفينثويت والكلورفينابير فى بستان الخوخ

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تم معاملة أشجار الخوخ المثمرة بمبيدى الفينثويت والكلورفينابير فى بستان خاص بمدينة السادات بمحافظة المنوفية خلال موسم النمو 2007 وقد أوضحت النتائج ما يلى:

1- كان معدل اختفاء مبيد الكلورفينابير أسرع من الفينثيويت.

2- كانت فترة نصف العمر 2.36 ، 1 ، 4.68 يوم لمركب الفينثويت وكانت 2.68 ، 1.8 ، 1.5 يوم بالنسبة لمركب الكلورفينابير وذلك فى الثمار الغير مغسولة والأوراق والتربة أسفل الأشجار المعاملة على التوالى.

3- أدت عملية الغسيل إلى إزالة كميات من المبيدين تراوحت نسبتها من 2.61-37.16% مع الفينثيويت 2.47-35.75 % مع الكلورفينابير .

4- يمكن تناول الثمار الغير مغسولة والمغسولة بعد فترة 21 يوم فى حالة مبيد الفينثيويت ،14 يوم فى حالة مبيد الكلورفينابير.

5- وجد أن تصنيع ثمار الخوخ المعاملة بالمبيدين إلى مربى أدى إلى عدم وجود تركيزات من المتبقيات يمكن تقديرها.