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Determination of organochlorine and pyrethroid pesticides in fruit and vegetables using SAX/PSA clean-up column

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Abstract

A study was conducted to investigate the organochlorine and pyrethroid pesticide residues in fruit and vegetables from market in Malaysia. Gas chromatography with electron capture detector (GC-ECD) was used to determine the concentrations of pesticide residues and SAX/PSA was used as a clean-up. Cypermethrin was detected in 38 of 302 vegetable samples with a mean value of 0.47 mg/kg. The mean value of cypermethrin for tomato, chinese parsley, chinese celery, chilli, brinjal, french beans, green mustard and capsicum ranged from 0.16 to 1.48 mg/kg. The mean values for all samples were below the maximum residue limits (MRLs) allowed by the Malaysian Food Regulations except for brinjal. None of the 206 fruit samples tested showed any pesticide residue.

Keywords: Organochlorine and pyrethroid pesticides; Fruit and vegetables; Clean-up

1. Introduction

Most of organochlorine pesticides have been banned because they are highly persistent insecticides, but their residues still appear as pollutants in food as well as in the environment (Abou-Arab, 1999). Pyrethroid insecticides have greater photostability, enhanced insecticidal activity, and relatively low toxicity as compared with organochlorine and organophosphorus insecticides (Pang, Fan, & Chao, 1994a, 1994b). According to Tomlin as quoted by Colume, Cardenas, Gallego, and Valcarcel (2001a, 2001b), pyrethroids and organochlorine pesticides are non-systemic as they cannot penetrate into the plant. The extraction of these pesticides is rather simple compared to the systemic organophosphate and carbamate pesticides because they form a deposit on the surface of the leaf after the treatment. According to Baker and Bottomley as quoted by Pang et al. (1994a, 1994b), pyrethroid insecticides actually is a mixture of more than one isomer, which is different from organophosphates and carbamates. The pyrethroid potential absorption depends on structural formula, lipophilicity and concentration of the active ingredients.

Pesticides are widely used in fruit and vegetables because of their susceptibility to insect and disease attacks. Therefore, residues of pesticide could affect the ultimate consumers especially when these commodities are freshly consumed. The total dietary intake of pesticide residues that remain on agricultural commodities are known as carcinogens and/or toxins, and therefore, it is desirable to reduce these residues. The levels of pesticide residues are controlled by the maximum residue limits (MRLs), which are established by each country and sometimes cause conflicts because residue levels

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acceptable in one country could be unacceptable in another (Torres, Pico, & Manes, 1996). The required rates of application may vary, under different agricultural and climatic conditions, from country to country, and between regions of the same country (Torres, Pico, & Manes, 1997).

Many countries have given high priority to monitor and control pesticide residues on treated crops. Methods were developed and implemented as part of regulatory programs to detect products that exceed MRLs (Bennett, Chung, & Lee, 1997). In Malaysia, the MRLs of pesticide residues for specific commodities are established in Schedule 16 of the Malaysia Food Regulations, 1985. In fruit and vegetables, analytical methods are needed to screen, quantify, and confirm pesticide residues for both research and regulatory purposes (Torres et al., 1996).

Most determinations of organochlorine and pyrethroid compounds have been developed using chromatographic techniques because of their high resolution capacity and the availability of selective detectors (Fernandez, Pico, & Manes, 2001), mainly gas chromatography (GC) with electron capture detector (ECD) because of the halogen atoms in their chemical structure (Colume et al., 2001a, 2001b). GC-ECD has been widely used in the determination of organochlorine and pyrethroid pesticides in fruit and vegetables (Adeyeye & Osibanjo, 1999; Colume et al., 2001a, 2001b; Fernandez-Alba, Valverde, Aguera, & Contreras, 1994; Gelsomino, Petrovicova, Tiburtini, Magnani, & Felici, 1997; Muccio et al., 1997), human milk and infant formulas (Pico, Viana, Font, & Manes, 1995), soil (Concha-Grana et al., 2004), milk and dairy products (Pandit, Sharma, Srivastava, & Sahu, 2002), beeswax (Jimenez, Bernal, del Nozal, & Alonso, 2004), egg (Schenck and Donoghue, 2000) and ethoxylated lanolin (Polese, Sannomiya, de Oliveira Sader, & Ribeiro, 2000).

Gas chromatography/mass spectrometry (GC/MS) with selected ion monitoring (SIM) was chosen because of its capability for sensitive and specific detection (Sannino, Mambriani, Bandini, & Bolzoni, 1996). GC–MS has been used in confirmation studies of pesticide residue in fruit and vegetable (Chen & Wang, 1996; Colume et al., 2001a, 2001b; Fernandez-Alba et al., 1994; Gelsomino et al., 1997). Confirmatory analysis was needed due to the large probability of false positive results obtained by GC-ECD (Gelsomino et al., 1997).

The purpose of this study was to determine the presence of organochlorine and pyrethroid pesticide residues in fruit and vegetable samples purchased from two local markets in Sungai Buloh and Selayang, Malaysia. The SAX/PSA was used as a clean-up column to remove interferences in the sample extract before determination using GC-ECD. Any detected residues were confirmed using GC–MS to prevent any misinterpretation of results.

2. Materials and methods

2.1. Samples

The fruit samples used in this study included pear, orange, apple, star fruits, mango, guava, watermelon, grape, banana and papaya, while the vegetable samples included green mustard, lettuce, celery, cauliflower, broccoli, cabbage, tomato, chili, capsicum, brinjal, french beans and carrot. Samples were purchased from a local market at Sungai Buloh and Selayang. The sample size was at least one kg for small and medium sized of fresh product and always included ten units of pears, oranges or apples. The minimum weight for large sample sizes was 2 kg (for examples broccoli and cabbage), where the unit was generally more than 250 g (Codex Alimentarius, 2000).

The portion of raw agricultural commodity prepared as the analytical sample for determination of pesticide residues was carried out according to the Codex Alimentarius, Volume 2A, Part 1-2000. A representative portion of the analytical sample was blended using a food processor and mixed thoroughly. The homogenized samples were stored at –21 °C. Prior to use, the samples were thawed at 4 °C overnight.

2.2. Reagents

Pesticide-grade ethyl acetate, acetone, *n*-hexane and anhydrous sodium sulfate were obtained from Merck (Darmstadt, Germany). Pesticide standards of gamma-HCH, heptachlor, aldrin, dieldrin, endrin, captafol, permethrin, cypermethrin and fenvalerate were purchased from Dr. Ehrenstorfer (Ausberg, Germany). Individual stock standard solution of pesticide was prepared by dissolving 50–60 mg of each compound in 50 mL of ethyl acetate and stored in amber bottles at 4 °C. A mixed standard solution was prepared from the stock solutions with a concentration of 10 mg/L. A series of calibration standards were prepared by diluting 10 mg/L of the mixed standard solution to produce a final concentration of 0.05, 0.1, 0.2 and 0.5 mg/L in ethyl acetate.

2.3. Solid phase extraction

A solid phase extraction vacuum manifold (International Sorbent Technology, UK) was used for simultaneous extraction of the samples. A mixed-mode disposable column of SAX/PSA (6 mL) containing 1 g of sorbent was purchased from International Sorbent Technology (IST), UK. The columns were conditioned by passing 5 mL of a mixture solution of acetone:*n*-hexane (3:7, v/v) through the column. The sorbent was not allowed to dry during the conditioning and sample loading steps.

2.4. Analytical procedure

Twenty grams of the homogenized sample was mixed with 80 mL of ethyl acetate and 50–100 g of sodium sulfate prior to extraction. The mixture was blended for 3 min and the extract was filtered on Advantec 5A filter paper into an evaporation flask. The sample was re-extracted with another 50 mL of ethyl acetate and filtered into the same flask. The filtrate was evaporated under vacuum using a rotary evaporator (Laborota 4002, Heidolph) at 40 °C to about 1 mL and dried-up using a stream of nitrogen gas. The residue was reconstituted into 5.0 mL using a solvent mixture of acetone:*n*-hexane (3:7, v/v) and centrifuged for 1 min at $156 \times g$ to separate any particles. Three mL of the extract was transferred to the SAX/PSA clean-up column and pesticides were eluted with 5 mL of acetone:*n*-hexane (3:7) drop by drop into a 10 mL graduated test tube and concentrated to about 3 mL using a sample concentrator (Techne). The extract was made-up to 3.0 mL with the same solvent mixture and the pesticides determined by gas chromatography coupled to an electron capture detector (GC-ECD). To avoid false positive, a GC–MS analysis was used to confirm the presence of any pesticide residue.

2.5. Recovery rate and limits of detection

Grape samples were fortified at 0.01, 0.02 and 0.1 mg/kg, by adding 5.0 mL of a mixed standard solution. The recovery rate was replicated three times and the data are presented in Table 1. The table shows that the recovery rate for nine pesticides were within acceptable range except captafol. The method is applicable for the determination of eight pesticides in fruit and vegetable samples. The low recovery for captafol may be due to the analyte retained in the clean-up column.

The limits of detection (LODs), calculated by using a signal-to-noise (S/N) ratio of 3, were in the range between 0.003 and 0.015 mg/kg as shown in Table 2. The quantity of each pesticide was calculated by applying the external standard method.

Table 1
Analytical recoveries (%) ± SD of six organochlorine and three pyrethroid pesticides in grape samples at 0.01, 0.02 and 0.1 mg/kg fortification levels ($n = 3$)

Pesticides	Analytical recoveries (%) ± SD at different fortification levels (mg/kg)		
	0.01	0.02	0.1
Gamma-HCH	96.0 ± 9.5	96.1 ± 6.3	97.1 ± 5.6
Heptachlor	102.4 ± 9.2	95.4 ± 7.3	98.7 ± 7.6
Aldrin	88.9 ± 10.6	99.6 ± 9.8	104.1 ± 5.5
Dieldrin	102.4 ± 10.4	91.6 ± 10.4	97.4 ± 4.9
Endrin	99.9 ± 9.6	100.8 ± 7.9	103.2 ± 6.4
Captafol	54.0 ± 27.5	61.2 ± 19.6	66.5 ± 16.1
Permethrin	92.7 ± 10.9	97.4 ± 5.4	96.4 ± 5.5
Cypermethrin	101.7 ± 9.3	101.8 ± 8.3	81.7 ± 7.5
Fenvalerate	103.1 ± 9.7	101.2 ± 6.1	101.8 ± 3.4

Table 2
The retention time, LOD and LOQ in mg/kg of grape sample

Pesticides	Retention time (min)	LOD (mg/kg)	LOQ (mg/kg)
Gamma HCH	13.51	0.015	0.05
Heptachlor	15.13	0.015	0.05
Aldrin	15.89	0.003	0.01
Dieldrin	18.15	0.003	0.01
Endrin	18.71	0.003	0.01
Captafol	20.92	0.015	0.05
Permethrin	27.06 27.57	0.015	0.05
Cypermethrin	31.26 31.83 32.24 32.47	0.015	0.05
Fenvalerate	37.03 38.66	0.015	0.05

2.6. Gas chromatographic determination

The Agilent HP 6890 N gas chromatograph equipped with an ECD and with a 30 m × 0.25 mm ID × 0.25 μm film thickness capillary column coated with cross-linked 5% phenyl methyl polysiloxane was used. The injection port temperature was 250 °C and the detector temperature was 300 °C. The column temperature was programmed as follows: the initial temperature of 100 °C was increased at a rate of 10 °C/min up to 250 °C and held for 25 min, from 250 to 300 °C at a rate of 50 °C/min was used and held for 5 min at the final temperature. Helium carrier gas at a flow rate of 1.2 ml/min was used. Two microliters of the extract was injected and the peak area was compared (sum of the peak areas of all isomers of pyrethroid pesticides) to that of the calibration standards to determine the residue quantitatively.

2.7. Gas chromatographic–mass spectrometry (GC–MS) confirmation

A Shimadzu GC 17 A system equipped with a GCMS-QP 5050A quadrupole mass spectrometer; 30 m × 0.25 mm × 0.25 μm DB-5-MS column was used for the experiment. The temperature program was: initial temperature 60 °C held for 2 min, 8 °C/min ramp to 220 °C and finally 15 °C/min to 250 °C, held for 20 min; a transfer line temperature of 280 °C; injection temperature of 250 °C; ionization temperature of 150 °C and a column flow of 1.8 mL/min. The injection mode was splitless and the injection volume was 2.0 μL. The mass spectrometer with electron impact (EI) was used in selected ion monitoring (SIM) mode and three ions were selected and monitored for each pesticide. Retention times, molecular weight, selected ion for SIM of nine studied pesticides are listed in Table 3. Detected pesticides were confirmed by matching their

Table 3
The retention time, molecular weight and fragment ions selected for confirmation of pesticides using GC–MS

Pesticide	Retention time (min)	M/W	M/Z1	M/Z2	M/Z3
Gamma-HCH	19.92	288	219	217	181
Heptachlor	21.25	370	337	272	237
Aldrin	22.15	362	293	263	221
Dieldrin	24.32	381	277	263	237
Endrin	24.78	382	345	279	263
Captafol	26.63	347	183	150	107
Permethrin	31.64	390	390	183	163
Permethrin	32.05				
Cypermethrin	34.49	416	415	197	181
Cypermethrin	34.92				
Cypermethrin	35.24				
Cypermethrin	35.42				
Fenvalerate	39.60	420	419	225	181
Fenvalerate	40.92				

retention time and fragment ions with those of the standards using the SIM mode.

3. Results and discussion

GC-ECD provided good responses even at very low concentrations because of its selective and sensitive detectors (Gelsomino et al., 1997). GC–MS operating in SIM mode, allowed higher instrumental sensitivity and lower detection limits and was applied for confirmation of peak identity. Three fragment ions were selected for each pesticide used in this study. The sensitivity of the assay decreased when the numbers of ions monitored increased (Stajnbaher & Zupaneie-Kralj, 2003). The use of target ions and their retention time allowed to positively confirm of the pesticide identity.

Table 4 shows a total of 508 samples consisting of different commodities that were analysed for six organochlorine (gamma-HCH, heptachlor, aldrin, dieldrin, endrin, captafol) and three pyrethroid (permethrin, cypermethrin, fenvalerate) pesticides. The residue was detected in 7.5% of the total samples (38 from 508 samples). The residue was only found in vegetable samples. None of the 206 fruit samples examined contained any pesticide residue. The pesticide residue was detected in tomato (30%), chinese parsley (30%), chinese celery (25%), chilli (23.3%), brinjal (20%), french beans (15%), green mustard (6.7%) and capsicum (4.5%).

The residue detected in all samples was cypermethrin only. Each detected cypermethrin was confirmed by matching its retention time and fragment ions with the cypermethrin standard. The mean levels of nine pesticides in fruit and vegetable samples are presented in Table 5. The table shows that cypermethrin residue was detected in 38 vegetable samples with a mean level of 0.47 mg/kg. Other pesticides detected in this study were not found in any fruit or vegetable samples.

The mean levels of cypermethrin detected in 38 vegetable samples are summarized in Table 6. The table shows that chinese celery contained the highest mean value of 1.48 mg/kg followed by chinese parsley (0.68 mg/kg), brinjal (0.37 mg/kg), capsicum (0.36 mg/kg), tomato (0.29 mg/

Table 5
The mean levels of six organochlorine and three pyrethroid pesticides in vegetable and fruit samples in Sungai Buloh and Selayang area (Malaysia)

Pesticides	Vegetable ($n = 302$)		Fruit ($n = 206$)	
	Mean value (mg/kg)	No. of positive samples	Mean value (mg/kg)	No. of positive samples
Gamma-HCH	ND	–	ND	–
Heptachlor	ND	–	ND	–
Aldrin	ND	–	ND	–
Dieldrin	ND	–	ND	–
Endrin	ND	–	ND	–
Captafol	ND	–	ND	–
Permethrin	ND	–	ND	–
Cypermethrin	0.47	38	ND	–
Fenvalerate	ND	–	ND	–

Table 4
Number of fruit and vegetable samples analysed and number of samples with pesticide residue detected

Commodity	Vegetables		Commodity	Fruits	
	No. of samples analysed	No. of samples with residue		No. of samples analysed	No. of samples with residue
Tomato	30	9	Orange	30	0
Chinese parsley	20	6	Grape	30	0
Chinese celery	16	4	Apple	25	0
Chili	30	7	Pear	20	0
Brinjal	30	6	Guava	20	0
French beans	20	3	Banana	18	0
Green mustard	30	2	Watermelon	20	0
Capsicum	22	1	Mango	25	0
Lettuce	30	0	Papaya	18	0
Broccoli	25	0			
Cabbage	24	0			
Carrot	25	0			
Total	302	38	Total	206	0

Table 6
The levels of cypermethrin detected in vegetable samples

Samples	Mean value (mg/kg)	No. of positive samples	No. of samples above MRL[a]	MRL[a] (mg/kg)
Tomato	0.29	9	1	0.5
Chinese parsley	0.68	6	0	2
Chinese celery	1.48	4	1	2
Chilli	0.22	7	1	0.5
Brinjal	0.37	6	5	0.2
French beans	0.16	3	0	0.5
Green mustard	0.24	2	0	1
Capsicum	0.36	1	0	0.5

[a] Malaysia Food Regulations, 1985.

kg), green mustard (0.24 mg/kg), chilli (0.22 mg/kg) and french beans (0.16 mg/kg). According to the Malaysia Food Regulations (1985), the MRL of cypermethrin for leafy vegetables is 2 mg/kg. Therefore, the mean value for chinese celery was lower than the MRL. But, the mean value for brinjal was found exceeding the MRL. The mean value for other samples was below the MRL.

4. Conclusion

The results showed that pyrethroid pesticide was only present in vegetable samples. No pyrethroid and organochlorine pesticides were present in fruit samples analysed. The use of cypermethrin in vegetable samples may be due to its greater photostability and relatively low toxicity as compared with organochlorine and organophosphorus insecticides. Organochlorine, permethrin and fenvalerate were not detected suggesting that these pesticides were not in common use in fruit and vegetable samples found in this region. In order to minimize health risk as well as for enforcement activities, monitoring of pesticide residues is increasingly important and essential.

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