STUDYING OF SPECIFIC DISINTERGRATIVE CHARACTERISTIC FOR PESTICIDE RESIDUES THAT USED IN GREEN VEGETABLES IN THE ENVIRONMENT OF DALAT CITY

Le Tat Mua, Nguyen Tien Dat, Nguyen Van Minh, Ta Thi Tuyet Nhung, Truong Van Tai, Nguyen Ngoc Tuan and Nguyen Mong Sinh

Nuclear Research Institute

ABSTRACT: After pesticides are used, the disintegration occurs due to light, temperature, alkaline materials and bio-microorganism in soil and water. The disintegration rate depends on chemical properties of each pesticide and environmental conditions.

In this work, use of the method enable plant material to be extracted and cleaned up for gas chromatographic determination of residues of 4 compounds of the organophosphorus and pyrethroid groups as dimethoat, clopyrifos, methidation and cypermethrin compounds by one or the same procedure. Limit of detection and limit of quantitation are determinated in range of 5-10ng and 0.01- 0.05ppm. Recovery is in range 80-98%.

The transformation and disintegration rate in vegetables such as: Nozawana, Perilla and Spinach depends on the stability of pesticides used , the activities of enzyme in soil and water and weather conditions. Their process occurs fast when vegetables are in the interval of growth and in the condition of high temperature, moisture and light intensity.

The disintegration rate of 4 compounds of the organophosphorus and pyrethroid groups in vegetables occurs faster in dry season than rainy season.

INTRODUCTION

Use of the method enable plant material to be extracted and cleaned up for gas chromatographic determination of residues of 4 compounds of the organophosphorus and pyrethroid groups by one or the same procedure. The compounds are extracted with acetone. After dilution with water, the compounds are extracted with dichloromethane. The compounds are separated on activated carbon-silicagel column. The compounds are eluted with a mixture of dichloromethane, toluene and acetone. The compounds are identified and quantitated by gas chromatography using an electron capture detector.

EXPERIMENTS

Apparatus:

- Gas chromatography GC-17A V3.
- Analytical balance, micropipette, glassware, glass beakers. etc.

Reagents:

- All chemicals used were of analytical grade. Deionized water was obtained by processing distilled water in an ion exchange unit.
- Acetone, chemical pure, distilled in rotary evaporator at 40^0C
- Diclhoromethane, p.a. (Merck No. 6050)
- n-Hexan, Toluene for residue analysis

- Eluting mixture: dichloromethane+ toluene+ acetone 10:2:2 v/v/v
- Pesticide standard solution.
- Sodium chloride solution, saturated; sodium sulphate, active carbon, silicagel 0.063-0.200mm.

Nitrogen

Sample collection and sample preparation

More than 30 samples of 3 kinds of vegetables such as: Nozawana, Perilla and Spinach were collected from the vegestable farms of Dalat City and Ductrong District.

After being cleaned, the samples were cut into small piece and ground.

GC/ECD for Pesticide Residue Analysis

Procedure

Extraction

Weigh a 100g portion of sample into the beaker, add 200ml acetone and homogennize for 30 s. Rinse with acetone. Filter the homogenate in the Buchner funnel.

Shake the filtrate and measure its volume. Take one-fifth of filtrate and shake for 2 min with 250ml water, 25ml NaCl and 50ml CH_2Cl_2 in separator funnel. Repeat the step again. Combined the organic phases, dry on 30g Na_2SO_4 for 30 min. filter the dried extract through a filter paper. Rotery- evaporate the filtrate to about 2 ml. Dissolve the residue into 10ml CH_2Cl_2

Cleanup

Fill the chromatographic tube with CH_2Cl_2 to a level of 1cm. Slurry 5g silica gel in 15ml eluting mixture and pour the slurry into the column. Drain off the supernatant. Mix 15g silica gel and 1g active carbon into a beaker, and add 35ml eluting mixture.

Add the activated carbon / silica gel mixture onto the silica gel in the chromatographic column, by pouring it through a funnel. Use any eluate that has passed through the column for rinsing the flask. Drain the eluting mixture to a level 2 cm above the packing, and top the column with a total of 5 g sodium sulphate. Next, prewash the column with 50 ml eluting mixture. Transfer the dichloromethane solution quantitatively to the prepared column, completing the transfer with a total of 5ml dichloromethane. Collect liquid flowing through the column and subsequent eluate a 250ml round-bottomed flask. Elute the column with 140ml of eluting mixture. Evaporate the eluate to about 30ml. Trasfer it to a 50ml round-bottomed flask, and evaporate to about 2ml

Gas- chromatographic determination

Transfer the solution to a graduated cylinder and make up with n-hexan to 5ml. Inject 1µl of this solution into gas chromatograph.

Results

Table 1: Mean concentration of organophosphorus compound - chlorpyriphos in Nozawana, Perilla and Spinach samples (mg/kg.wet).

Date of harvest	Concentration (ppm)					
	Rainy season			*Dry season*		
	Spinach	Nozawana	Perilla	Spinach	Nozawana	Perilla
1	7.2	6.70	7.30	5.60	6.60	7.10
2	4.8	4.30	5.20	3.80	4.10	5.10
3	2.6	3.00	3.50	2.10	3.20	3.20
4	1.5	2.10	2.60	1.50	1.00	1.20
5	1.2	1.70	1.30	1.05	0.90	1.00
6	0.78	1.10	1.01	0.86	0.73	0.75
7	0.42	0.60	0.75	0.32	0.50	0.30
8	0.12	0.32	0.30	0.10	0.10	0.08
9	0.09	0.07	0.06	0.08	0.05	0.05
10	0.05	0.02	0.02	0.06	0.03	0.04
11	0.02	0.05	0.01	0.04	0.03	0.02
12	0.01	0.02	<0.01	0.02	0.02	0.01
13	<0.01	<0.01	<0.01	0.01	<0.01	<0.01
14	<0.01	<0.01	<0.01	0.01	<0.01	<0.01

Table 2: Mean concentration of organophosphorus compound - dimethoat in Nozawana, Perilla and Spinach samples (mg/kg.wet).

Date of harvest	Concentration (ppm)					
	Rainy season			*Dry season*		
	Spinach	Nozawana	Perilla	Spinach	Nozawana	Perilla
1	2.59	0.73	0.86	1.92	0.78	0.85
2	1.75	0.54	0.68	1.36	0.60	0.70
3	1.03	0.45	0.54	0.90	0.46	0.50
4	0.57	0.28	0.35	0.43	0.40	0.41
5	0.30	0.20	0.24	0.25	0.28	0.30
6	0.08	0.10	0.15	0.09	0.20	0.18
7	0.07	0.09	0.09	0.05	0.10	0.10

8	0.05	0.07	0.08	0.05	0.09	0.08
9	0.01	0.06	0.07	0.04	0.04	0.06
10	<0.01	0.02	0.05	0.03	0.02	0.05
11	<0.01	0.01	0.02	0.01	<0.01	0.03
12	<0.01	0.01	0.01	0.01	<0.01	0.01
13	<0.01	<0.01	<0.01	0.01	<0.01	<0.01
14	<0.01	<0.01	<0.01			

Table 3: Mean concentration of organophosphorus compound - methidathion in Nozawana, Perilla and Spinach samples (mg/kg.wet).

Date of harvest	**Concentration** (ppm)					
	Rainy season			***Dry season***		
	Spinach	Nozawana	Perilla	Spinach	Nozawana	Perilla
1	3.5	3.20	2.50	2.93	3.20	2.50
2	2.1	1.50	1.00	1.70	1.50	1.00
3	1.2	0.80	0.50	1.10	0.80	0.50
4	0.74	0.10	0.15	0.72	0.10	0.15
5	0.35	0.06	0.07	0.46	0.06	0.07
6	0.05	0.02	0.05	0.11	0.02	0.02
7	0.02	0.01	0.02	0.05	0.01	0.01
8	0.02	<0.01	0.01	0.03	<0.01	<0.01
9	0.01	<0.01	<0.01	0.02	<0.01	<0.01
10	0.01	<0.01	<0.01	0.01	<0.01	<0.01
11	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
12	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
13	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
14	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Table 4: Mean concentration of pyrethroid compound - cypermethrin in Nozawana, Perilla and Spinach samples (mg/kg.wet).

Date of harvest	**Concentration** (ppm)					
	Rainy season			***Dry season***		
	Spinach	Nozawana	Perilla	Spinach	Nozawana	Perilla
1	4.50	3.70	4.10	5.80	3.80	4.10
2	2.50	3.20	3.00	3.50	2.50	3.10

3	1.80	2.00	2.00	2.20	1.00	1.00
4	1.10	1.70	1.90	1.80	0.60	0.80
5	0.80	1.40	1.30	1.36	0.30	0.50
6	0.30	0.84	1.00	1.05	0.20	0.30
7	0.20	0.53	0.80	0.50	0.10	0.10
8	0.15	0.21	0.30	0.10	0.08	0.10
9	0.10	0.10	0.10	0.05	0.03	0.09
10	<0.05	<0.05	0.05	0.03	0.02	0.03
11	<0.05	<0.05	0.01	0.01	0.01	0.01
12	<0.05	<0.05	<0.05	<0.01	<0.01	<0.01
13	<0.05	<0.05	<0.05	<0.01	<0.01	<0.01
14	<0.05	<0.05	<0.05	<0.01	<0.01	<0.01
16				<0.01	<0.01	<0.01

CONCLUSIONS

Gas chromatography using an electron capture detector has become a valuable method for determination of 4 compounds of the organophosphorus and pyrethroid groups as dimethoat, chlopyrifos, methidation and cypermethrin compounds in vegetable samples. Limit of detection and limit of quantitation are determinated in range of 5-10ng and 0.01- 0.05ppm. Recovery is in range 80-98%. Precision and accuracy can be kept within 10-15%.

The transformation and disintegration rate in vegetables such as: Nozawana, Perilla and Spinach depends on the stability of pesticides used , the activities of enzyme in soil and water and weather conditions. Their process occurs fast when vegetables are in the interval of growth and in the condition of high temperature, moisture and light intensity.

The disintegration rate of 4 compounds of the organophosphorus and pyrethroid groups in vegetables collected from Dalat city and Ductrong district farms, such as: Nozawana, Perilla and Spinach depends on the stability of pesticides used , the activities of enzyme in soil and water and weather conditions, and occurs faster in dry season than rainy season.

REFERENCES

1. Manual of Pesticide Residue Analysis. Volume I, II. DFG Deutsche Fforchungs Gemeinschaft, 1997
2. AOAC Official Method 985.22. Organochlorine and Organophosphorous Pesticide Residues. Gas chromatographic Method. 1996
3. H.A. MOYE, Opportunities for pesticide residue analytical method development: the potential for aqueous extraction of pesticide residues from fruits and vegestables. Conference proceeding series, Eight International Congress of pesticide chemistry, American Chemistry Sosiety, Washington DC, 193-203,1995