Original Article

The Use of Solid Phase Extraction for Pesticide Residue Analysis of Crops: An Alternative to Liquid-Liquid Partitioning

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Solid-phase extraction with bonded silica sorbents has been developed as an alternative to liquid-liquid partitioning cleanup in the pesticide residue analysis of crops. Elution patterns and recovery from an octadecyl (C18) sorbent were determined for 17 organophosphorus, nine carbamates and three other pesticides with the sample matrix from rice grain. The recovery was good for all compounds whose properties were water-soluble at below 25,000 mg/l. The sample loading test proved that a cartridge containing only 0.5 g of sorbent mass was capable of trace enrichment of analytes with a sample matrix from 20 g of grain. Among various types of bonded silica sorbents, octyl (C8), ethyl (C2) and cyclohexyl (CH) sorbents were superior to others in retention and selective elution. The new technique was compared with the conventional method which requires liquid-liquid partitioning. The availability of the technique was also evaluated in several fruits and vegetables.

INTRODUCTION

The analysis of pesticides in crops usually requires several purification and trace enrichment steps in order to quantitatively separate analytes from coextracted materials from samples. Liquid-liquid partitioning is generally used to cleanup sample extracts. However, a main disadvantage of this technique is emulsion formation, which makes the recovery of analytes difficult.

In recent years a new technique, solid-phase extraction with bonded silica sorbents, has been developed for extraction and concentration of organic compounds from liquid samples. This technique has become popular in the analysis of pharmaceutical fluids and environmental waters.¹⁻⁵⁾ However, there are only a few cases in which this technique was applied to the pesticide residue analysis of crop samples.^{6,7)}

In the present study the feasibility of the solid-phase extraction method was studied with seven different bonded silica sorbents as an alternative to liquid-liquid partitioning cleanup in the pesticide residue analysis of crops.

MATERIALS AND METHODS

1. Reagents

All solvents used in this study were of pesticide residue analysis grade. Water was obtained with a Milli-Q purifier system (Millipore). All other chemicals were of reagent grade.

Disposable solid-phase extraction cartridges (Bond Elut®) were purchased from Analytichem International. Octadecyl (C18), octyl (C8), ethyl (C2), methyl (C1), phenyl (PH), cyclohexyl (CH) and cyanopropyl (CN) cartridges each containing 0.5 g sorbent mass were used to examine their applicability to the

pesticide residue analysis of crops (For the practical application to routine residue analysis, cartridges with 1.0 g sorbent mass are used). Sep-Pak® silica and Florisil cartridges (Waters) were used for additional cleanup.

Analytical standards of 17 organophosphorus, nine carbamates, and three other pesticides were purchased from Wako Pure Chemical Ind. Co., Ltd. or obtained from the Agriculture Chemicals Inspection Station in Japan. Each compound was weighed accurately and dissolved in acetone to prepare a stock solution containing 0.4 mg/ml. A mixed standard solution of organophosphorus pesticides (+metalaxyl) and one of carbamates (+oxadiazon and fenvalerate) were prepared from the stock solution by adjusting each test compound to 0.1 to 2 ppm and used to spike crop samples.

2. Apparatus

Analyses were done by GC (Hewlett-Packard Model 5890A) with an N-P detector. Test compounds were analyzed on a 15-m $\times$ 0.53-mm i.d. DB-17 (J & W Scientific)-fused silica capillary column with a film thickness of 1 μ m and/or a 30-m $\times$ 0.53-mm i.d. SPB-5 (Supelco, Inc.)-fused silica capillary column a film thickness of 0.5 μ m. The column temperature for organophosphorus pesticides (+metalaxyl), which were analyzed on both columns, was 140°C in the initial stage and programmed at 4°C/min to 260°C. That for carbamate pesticides (+oxadiazon and fenvalerate), which were analyzed on a DB-17 column, was 140°C and programmed at 4°C/min to 170°C and then at 10°C/min to 260°C with an 8-min hold. The injection and detector temperatures were 250°C and 280°C, respectively. The flow rate of helium carrier gas was 10 ml/min and that of NPD gas was air 115 ml/min and hydrogen 3.5 ml/min.

3. Procedure

3.1 Sample preparation and extraction

Crops containing little water such as rice grain were ground to powder. Crops containing much water such as fruits and vegetables were chopped and then homogenized.

A powder sample (20 g) was weighed into an Erlenmeyer flask (300-ml). To this 1/15 M

phosphate buffer (pH=8.05: 20 ml), 0.1 N AgNO₃ (0.2 ml) only for the analysis of carbamate pesticides, and acetonitrile (80 ml) were added, and the contents were macerated at high speed for 1 min and shaken on a reciprocating shaker for 20 min. The extract was filtered through a funnel with suction into a flask (300-ml), and the residue was washed with acetonitrile (50 ml). The filtrates were combined and filled up to 200 ml with acetonitrile.

A homogenized sample (50 g) was weighed into an Erlenmeyer flask (300-ml). To this 1/15 M phosphate buffer (pH=8.05: 25 ml), 0.1 N AgNO₃ (0.3 ml) and acetonitrile (150 ml) were added, and shaken on a reciprocating shaker for 20 min. The extract was filtered through a funnel (if needed, a layer of Celite was placed on the filter paper) with suction into a flask (300-ml), and the residue was washed with acetonitrile (50 ml). The filtrates were combined and filled up to 250 ml with acetonitrile.

An aliquot of the extract (equivalent to 5–20 g of a sample) was concentrated in vacuum to remove the bulk of the organic solvent, and the concentrated crude extract was applied to a disposable cartridge for solid-phase extraction or a separatory funnel for liquid-liquid partitioning.

3.2 Solid-phase extraction

Solid-phase extraction was done as follows: cartridges were inserted into specially designed holes of a rubber stopper affixed to a glass tank, to which a vacuum was applied. The cartridges were first conditioned by adding 3–5 ml of methanol, followed by 3–5 ml of water. Since it was important that the cartridges did not dry up, no vacuum was left after each liquid had passed through the head of the cartridges.

A crude sample extract (equivalent to 5–20 g sample) was passed through the cartridges slowly so that the effluent could form distinct drops and the analyte could be retained on the sorbent with the nonpolar mechanism.⁹⁾ After the sample solution passed through the cartridges, the glassware that had held the sample was rinsed with 4–5 ml washing solution at the highest concentration of the organic solvent (acetonitrile) that do not elute the analyte in the cartridges. The solvent was also passed

through the cartridges.

Finally, the analytes in the cartridges were eluted by the following two elution procedures: one, the cartridges were eluted with 4–5 ml eluting solution at a concentration of the organic solvent (acetonitrile) high enough to elute the analytes. The other, air was drawn through the cartridges for 20–30 sec to dry the cartridges, followed by passing 4–5 ml of the organic solvent (acetonitrile) to elute the analytes.

The eluted solution from the cartridges was evaporated to dryness with a rotary vacuum evaporator. For additional cleanup with Sep-Pak® cartridges, the residue was dissolved in 2 ml of 30% acetone in hexane (v/v) for the analysis of organophosphorus pesticides (+metaxyl) or 30% ethyl acetate in hexane (v/v) for carbamate pesticides (+oxadiazon and fenvalerate).

3.3 Liquid-liquid partitioning

A crude sample extract (equivalent to 10 g grain sample) was transferred to a 300-ml separatory funnel. A 100 ml portion of 5% NaCl was added to the solution, and the mixture was extracted twice with 60 ml of dichloromethane. The dichloromethane extract was dried by passing through a bed of anhydrous sodium sulfate, combined in a 300-ml round-bottom flask, and evaporated to dryness. The residue in the flask was dissolved in 2 ml of 30% ethyl acetate in hexane for additional cleanup with Sep-Pak® silica cartridges.

3.4 Cleanup with Sep-Pak® cartridges

For the analysis of 17 organophosphorus pesticides and metaxyl, a sample dissolved in 30% acetone in hexane was applied to a Sep-Pak® Florisil cartridge, while for nine carbamates plus oxadiazon and fenvalerate, a sample dissolved in 30% ethyl acetate in hexane was applied to Sep-Pak® silica cartridges. The cartridges were washed before use with 5 ml each of the above corresponding solutions. The chromatograph was developed by adding 6 ml each of the above corresponding solutions, and each eluted solution was collected in a 20-ml test tube. The solution was evaporated to dryness, and the residue was dissolved in 1–10 ml of acetone for GC analysis.

3.5 Quantitation by GC

Test samples were spiked with an appro-

priate amount of the internal standard iprodion for the quantitation of carbamates (+metaxyl) and EPBP for organophosphorus pesticides (+oxadiazon and fenvalerate) immediately prior to GC analysis. The standards, solutions of the standard mixture at 0.5–2.0 ng/ μ l, were injected into GC, and the response factors for each pesticide relative to the internal standard were determined with an integrator. The concentration of a pesticide was then calculated from the calibration curve obtained with the standards.

RESULTS AND DISCUSSION

1. Elution Patterns and Recovery of 29 Pesticides from C18 Sorbents

The extraction efficiency of bonded silica sorbents was examined for 29 pesticides (each 5 μ g; 17 organophosphorus, nine carbamate and three other pesticides) spiked into extracts (5 g eq) from rice grain and applied to a C18 cartridge which was expected to interact most strongly with the analytes in the hydrophobic bonded phase. Twenty-nine pesticides were chosen purposely in a wide range of water solubility and molecular weight to analyze multi-residues of organophosphorus and carbamate pesticides in rice grain. The elution pattern and percent recovery of each pesticide from the cartridge are shown in Table 1.

With C18 sorbents the recovery was acceptable for almost all compounds but low for some compounds. Except for monocrotophos and trichlorfon (DEP), the recovery of organophosphorus pesticides was 80–101%, and that of carbamate compounds 85–100%. The compounds recovered in large amounts were water-soluble at below 25,000 mg/l.

In order to improve the extraction efficiency of the compounds whose recovery was low, NaCl (0.06–0.6 g), a salting-out agent aimed to decrease the water solubility of the compounds, was added to the crude sample extract (ca. 4 ml, eq 5 g sample). The addition of NaCl did not improve the recovery, however.

From the result in Table 1, it appears that there is a correlation between the elution pattern and the water solubility or molecular weight of a compound: the lower the water solubility of the compound or the greater the molecular weight of the compound, the higher

Table 1 Elution patterns and recovery of 29 pesticides from C18 sorbent.

Pesticides	M. W.	Solubility ^{a)} (mg/l)	Elution with % CH ₃ CN in water					
			20	40	60	80	100	Total
(% recovery)								
P 1: Monocrotophos	223	1000000						nd ^{e)}
P 2: Trichlorfon (DEP)	257	154000	17					17
P 3: Dimethoate	229	25000	27	53				80
O 1: Metalaxyl	279	7100		30	51			81
C 1: Metolcarb (MTMC)	165	2600		85				85
C 2: Propoxur (PHC)	209	2000		92				92
C 3: Fenobucarb (BPMC)	207	610			89			89
C 4: Xylcarb (MPMC)	179	580		90				90
C 5: XMC ^{d)}	179	470		88				88
P 4: Phenthoate (PAP)	320	200			81	12		93
P 5: Malathion	330	145			94	1		95
P 6: Dimethylvinphos (E)	331.5	130			83			83
P 7: Dimethylvinphos (Z)	331.5	130			95			95
C 6: Carbaryl (NAC)	201	120		97				97
C 7: Isoprocarb (MIPC)	193	ss ^{b)}		73	14			87
P 8: Fenthion (MPP)·SO ₂	310	ss		24	77			101
P 9: Pyridaphenthion	340	ss			93	2		95
P10: EPN	323	is ^{c)}			40	53		93
P11: Diazinon	304	40			43	48		91
P12: Phosmet (PMP)	317	22			96	2		98
P13: Fenitrothion (MEP)	277	14			88			88
P14: Tetrachlorvinphos (E)	366	11			83	10		93
P15: Tetrachlorvinphos (Z)	366	11			95			95
P16: Chlorpyrifos-methyl	322	4			27	59	2	88
C 8: Benfuracarb	410	8			3	97		100
P17: Isoxathion	313	2			48	44	2	94
O 2: Fenvalerate	420	1				87	2	89
O 3: Oxadiazon	345	0.7				97		97
C 9: Carbosulfan	380	0.3				68	23	91

P1-17: organophosphorus pesticides, C1-9: carbamate pesticides, O1-3: others.

^{a)} water solubility. ^{b)} slightly soluble. ^{c)} insoluble. ^{d)} 3, 5-xylcarb methylcarbamate. ^{e)} nd: not detected.

Method: A grain extract (eq. 5g) spiked with each compound at 1.0 ppm was applied to a C18 cartridge with 0.5g sorbent mass. The cartridge was washed with 5 ml of water and eluted with 5 ml of each solution above (20-100% acetonitrile in water).

the concentration of acetonitrile required for elution. This may be reasonable in view of the mechanism of solid-phase extraction (with bonded silica sorbents) because it is based on nonpolar interactions between the carbon-hydrogen bonds of sorbent functional groups and the carbon-hydrogen bonds of an analyte.⁸⁾

2. Recovery from Seven Bonded Silica Sorbents

Seven bonded silica sorbents were examined for the extraction efficiency to nine carbamates (5 µg) spiked into extracts (5 g eq) from

rice grain. The results are shown in Table 2.

In sorbent extraction, the goal is to keep an analyte on a sorbent strongly enough not to move through the sorbent until eluting solution is introduced, and to elute the analyte from the sorbent in the smallest volume of eluting solution.⁹⁾ Judging from this point of view, C18, C8, C2, CH and PH sorbents are useful for the analysis of carbamates, retaining the analytes firmly on the sorbents. C8, C2 and CH sorbents are superior to the others, selectively eluting the analytes together by a

Table 2 Elution patterns and recovery of carbamates from seven sorbents.

<Sorbent> Pesticide	Elution with % CH ₃ CN in water					
	20	40	60	80	100	Total
(<C18>)						
C1: Metolcarb (MTMC)		85				85
C2: Propoxur (PHC)		92				92
C3: Fenobucarb (BPMC)			89			89
C4: Xylylcarb (MPMC)		90				90
C5: XMC		88				88
C6: Carbaryl (NAC)		97				97
C7: Isoprocarb (MIPC)		73	14			87
C8: Benfuracarb			3	97		100
C9: Carbosulfan				68	23	91
(<C8>)						
C1: Metolcarb (MTMC)		82				82
C2: Propoxur (PHC)		94				94
C3: Fenobucarb (BPMC)			88			88
C4: Xylylcarb (MPMC)		89				89
C5: XMC		87				87
C6: Carbaryl (NAC)		107				107
C7: Isoprocarb (MIPC)		58	31			89
C8: Benfuracarb			25	78		103
C9: Carbosulfan				93		93
(<C2>)						
C1: Metolcarb (MTMC)		84				84
C2: Propoxur (PHC)		92				92
C3: Fenobucarb (BPMC)			87			87
C4: Xylylcarb (MPMC)		90				90
C5: XMC		89				89
C6: Carbaryl (NAC)		97				97
C7: Isoprocarb (MIPC)		80	7			87
C8: Benfuracarb			102	5		107
C9: Carbosulfan			7	85		92
(<C1>)						
C1: Metolcarb (MTMC)	20	62				82
C2: Propoxur (PHC)	7	87				94
C3: Fenobucarb (BPMC)		62	29			91
C4: Xylylcarb (MPMC)		93				93
C5: XMC		90				90
C6: Carbaryl (NAC)		100				100
C7: Isoprocarb (MIPC)		86				86
C8: Benfuracarb			107			107
C9: Carbosulfan			13	77		90
(<CH>)						
C1: Metolcarb (MTMC)		81				81
C2: Propoxur (PHC)		88	3			91
C3: Fenobucarb (BPMC)			86			86
C4: Xylylcarb (MPMC)		82	6			88
C5: XMC		78	8			86
C6: Carbaryl (NAC)		80	16			96
C7: Isoprocarb (MIPC)		35	48			83
C8: Benfuracarb			27	77		104
C9: Carbosulfan			4	85		89

Table 2 (continued)

<Sorbent> Pesticide	Elution with % CH ₃ CN in water					
	20	40	60	80	100	Total
				(% recovery)		
<PH>						
C1: Metolcarb (MTMC)	8	74				82
C2: Propoxur (PHC)		92				92
C3: Fenobucarb (BPMC)		35	54			89
C4: Xylylcarb (MPMC)		87	3			90
C5: XMC		84	4			88
C6: Carbaryl (NAC)		96	7			103
C7: Isoprocarb (MIPC)		76	9			85
C8: Benfuracarb			92	10		102
C9: Carbosulfan			15	78		93
<CN>						
C1: Metolcarb (MTMC)	71	6				77
C2: Propoxur (PHC)	79	10				89
C3: Fenobucarb (BPMC)		79	4			83
C4: Xylylcarb (MPMC)	24	61				85
C5: XMC	20	64				84
C6: Carbaryl (NAC)	4	93				97
C7: Isoprocarb (MIPC)	5	77				82
C8: Benfuracarb			102			102
C9: Carbosulfan		4	82			86

Method: A grain extract (eq. 5 g) spiked with nine carbamates at 1.0 ppm was applied to seven different phase cartridges with 0.5 g of each sorbent mass.

The cartridges were washed with 5 ml of water and eluted with 5 ml of each solution above (20–100% acetonitrile in water).

limited eluting fraction, thus elution may be favorable to multi-residue analysis, in particular.

3. Difference in the Amount of Samples Loaded

The effect of the amount of a sample loaded on pesticide retention on a sorbent was evaluated by using nine carbamates (10 μg) spiked into crude extracts from 2, 5, 10 and 20 g of rice grain and a C18 sorbent that was expected to be capable of the strongest retention. The results in elution pattern and percent recovery are shown in Table 3.

There was no appreciable difference in the elution pattern and the recovery of carbamates among the four different sample loadings, although benfuracarb (BC) and carbosulfan (CS) were liable to be eluted at a lower concentration of acetonitrile in eluting solution, with an increase in the loading amount. It was demonstrated that the C18 cartridge with

only 0.5 g of the sorbent mass was capable of trace enrichment of the carbamates with the sample matrix from 20 g rice grain.

4. Recovery from Fruits and Vegetables

Recovery of nine carbamates (10 μ g) spiked into crude extracts (eq 10 g sample) from melon, apple, cabbage, eggplant and green pepper at 1.0 ppm was tested. Solid-phase extraction was done as follows: a spiked crude extract was applied to the C8 cartridge suitable for multi-residue analysis of carbamates as mentioned in Table 2, washed with 5 ml each of water and 20% acetonitrile in water (v/v), and eluted with 5 ml of 80% acetonitrile in water (v/v).

Table 4 summarizes the recovery data. For all compounds, the recovery was acceptable from every crop. The recovery of carbosulfan was relatively low from apples, cabbage and eggplants, which may be due to its lability under acidic and basic conditions.

Table 3 Recovery of carbamates from grain samples different in the amount.

<g Sample> Pesticide	Elution with % CH ₃ CN in water					Total
	20	40	60	80	100	
(% recovery)						
<hr/>						
<2g>						
C1: Metolcarb (MTMC)		82				82
C2: Propoxur (PHC)		104				104
C3: Fenobucarb (BPMC)		8	79			87
C4: Xylylcarb (MPMC)		99				99
C5: XMC		95				95
C6: Carbaryl (NAC)		97				97
C7: Isoprocarb (MIPC)		78	11			89
C8: Benfuracarb			7	99		106
C9: Carbosulfan				59	41	100
<5g>						
C1: Metolcarb (MTMC)		76				76
C2: Propoxur (PHC)		99				99
C3: Fenobucarb (BPMC)		7	93			100
C4: Xylylcarb (MPMC)		108				108
C5: XMC		88	3			91
C6: Carbaryl (NAC)		90	3			93
C7: Isoprocarb (MIPC)		66	16			82
C8: Benfuracarb			18	89		107
C9: Carbosulfan				83	16	99
<10g>						
C1: Metolcarb (MTMC)		78				78
C2: Propoxur (PHC)		102				102
C3: Fenobucarb (BPMC)			91			91
C4: Xylylcarb (MPMC)		93				93
C5: XMC		88				88
C6: Carbaryl (NAC)		88				88
C7: Isoprocarb (MIPC)		75	7			82
C8: Benfuracarb			23	79		102
C9: Carbosulfan				88		88
<20g>						
C1: Metolcarb (MTMC)		74				74
C2: Propoxur (PHC)		93				93
C3: Fenobucarb (BPMC)		25	68			93
C4: Xylylcarb (MPMC)		93	3			96
C5: XMC		83	5			88
C6: Carbaryl (NAC)		82	6			88
C7: Isoprocarb (MIPC)		72	15			87
C8: Benfuracarb			80	27		107
C9: Carbosulfan			6	84		90

Method: Grain extracts (eq. 5–20 g) spiked with 10 µg each of carbamate were applied to C18 cartridges with 0.5 g sorbent mass.

The cartridges were washed with 5 ml of water and eluted with 5 ml of each of solution above (20–100% acetonitrile in water).

5. Comparison with the Conventional Method

The new technique was compared with the conventional method which requires liquid-liquid partitioning. Sample extracts (eq 10 g

sample) from rice grain were spiked with nine carbamates at 1.0 ppm and then analyzed by the new method using a C8 cartridge or by the approved conventional method.⁹⁾ The results

Table 4 Recovery of carbamates from fruits and vegetables.

Pesticide	Fruits and vegetables (% recovery)				
	Melon	Apple	Cabbage	Eggplant	Green pepper
Metolcarb (MTMC)	69	71	76	76	73
Propoxur (PHC)	86	92	93	96	87
Fenobucarb (BPMC)	79	79	83	86	82
Xylylcarb (MPMC)	89	88	91	93	91
XMC	83	82	85	88	86
Carbaryl (NAC)	96	95	99	101	97
Isoprocarb (MIPC)	75	75	80	82	82
Benfuracarb	95	90	97	92	89
Carbosulfan	83	70	76	70	83

Method: Each sample extract (eq. 10 g) spiked with nine carbamates at 1.0 ppm was applied to a C8 cartridge with 0.5 g sorbent mass.

The cartridge was washed with 5 ml each of water and 20% acetonitrile in water and eluted with 5 ml of 80% acetonitrile in water.

Table 5 Comparison with liquid-liquid partitioning.

Pesticide	% recovery (CV %; $n=4$)	
	Solid-phase extraction	Liquid-liquid partitioning
Metolcarb (MTMC)	75.2 (6.2)	81.1 (9.9)
Propoxur (PHC)	88.0 (3.5)	80.9 (7.7)
Fenobucarb (BPMC)	87.5 (2.2)	80.1 (8.3)
Xylylcarb (MPMC)	88.9 (3.5)	81.4 (8.5)
XMC	86.3 (2.1)	79.4 (8.4)
Carbaryl (NAC)	90.6 (4.0)	79.9 (6.1)
Isoprocarb (MIPC)	85.7 (3.1)	82.2 (12)
Benfuracarb	79.7 (4.2)	82.9 (2.9)
Carbosulfan	73.6 (3.4)	76.4 (6.7)

Solid-phase extraction method: A grain extract (eq 10 g) spiked with nine carbamates at 1 ppm was applied to a C8 cartridge with 0.5 g sorbent mass. The cartridge was washed with 5 ml each of water and 20% acetonitrile in water and eluted with 5 ml of 80% acetonitrile in water.

Liquid-liquid partitioning method: A grain extract (eq 10 g) spiked with nine carbamates at 1 ppm was applied to liquid-liquid partitioning with 2 × 80 ml dichloromethane.

are shown in Table 5.

The recovery by the solid-phase extraction method almost equaled that by the liquid-liquid partitioning method. However, the recovery values by the new method had a lower coefficient of variation than those by the conventional method. A possible explana-

tion of this is that the extraction was more selective by the new method than by liquid-liquid partitioning.

CONCLUSION

Solid-phase extraction with bonded silica sorbents is an applicable method as an alternative to liquid-liquid partitioning cleanup in the pesticide residue analysis of crops. Proper applying, post-washing and elution techniques may be determined depending on the chemical/physical properties of a pesticide, the selection of sorbents bonded with several functional groups and sample matrix.

Solid-phase extraction has the following advantages in comparison with liquid-liquid partitioning: because the amount of a solvent required for cleanup is greatly reduced, it saves time by shortening the evaporative concentration step and minimizes the analyst's exposure to the injurious solvent. By selective extraction a final elute has a fewer co-extractive interferences than by liquid-liquid partitioning. This makes subsequent cleanup steps easier and as a result, it can improve the accuracy and precision of analysis.

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要 約

固相抽出法の農薬作物残留分析における利用— 液液分配の代替として

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化学結合型シリカゲルを用いた固相抽出法の農薬作物

残留分析における(液液分配の代替としての)有用性について検討した。C18 固相抽出カラムにおける農薬成分の溶出パターンと回収率を, 水溶解度の異なる 29 種類の農薬(有機リン剤 17 種, カーバメート剤 9 種, その他 3 種)を用いて調べたところ, 水溶解度 25,000 mg/l 以下の化合物(27 種類)はいずれもカラムに保持され, カラムからの回収も良好(80~101%)であった。保持に及ぼす狭雑物の量的な影響を玄米抽出液(2~20 g 相当)を用いて検討したが, 同カラム(固相充填量 0.5 g)に保持されたカーバメート系化合物の溶出パターンに大きな変化は認められなかった。C8, C2, CH の各固相は, 保持能力と溶出パターンの点で他の固相(C18, C1, PH, CN)よりも優れていた。液液分配との比較や各種作物残留分析への応用についても検討した。本法は簡便, 迅速な上に液液分配よりも高い精製効果と再現性が期待できることから, 農薬の作物残留分析における液液分配の代替として有効な手法と考えられる。