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Microwave-Assisted Extraction for the Simultaneous Determination of Thiamethoxam, Imidacloprid, and Carbendazim Residues in Fresh and Cooked Vegetable Samples

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Microwave-assisted extraction (MAE) was carried out for the simultaneous determination of the insecticides thiamethoxam [(*EZ*)-3-(2-chloro-1,3-thiazol-5-ylmethyl)-5-methyl-1,3,5-oxadiazinan-4-ylidene(nitro)amine], imidacloprid [1-(6-chloro-3-pyridylmethyl)-*N*-nitroimidazolidin-2-ylideneamine], and the fungicide carbendazim (methyl benzimidazol-2-ylcarbamate) in vegetable samples. Five crop samples consisting of cabbage, tomatoes, chilies, potatoes, and peppers were fortified with the three pesticides and subjected to MAE followed by cleanup to remove coextractives prior to analysis by high-performance liquid chromatography. Using the selected microwave exposure time and power setting, the recoveries of the three pesticides from the fortified vegetable samples ranged from 68.1 to 106%. The corresponding recoveries for samples processed simultaneously but without microwave exposure ranged from 37.2 to 61.4%. The recoveries by MAE were comparable to those obtained by the conventional blender extraction technique. The precision of the MAE method was demonstrated by relative standard deviations of <7% for the three pesticides. The cooked cabbage and tomato samples showed no breakdown of the parent compounds, and the recoveries of three pesticides were comparable to those obtained with the uncooked samples.

KEYWORDS: Microwave-assisted extraction; thiamethoxam; imidacloprid; carbendazim; vegetable crops; multiresidue analysis; cooking; HPLC analysis

INTRODUCTION

The traditional Soxhlet or shake flask liquid−solid extraction procedures applied to pesticide residues in crop matrixes are time consuming, labor intensive, and use large amounts (i.e., >200 mL per sample) of organic solvents. Microwave-assisted extraction (MAE) has been used by analysts in the past few years as a refined technique to facilitate the extraction of pesticide residues from various food products. The use of a conventional commercial household microwave oven to extract parathion and bromophos residues from solid matrixes such as maize, soya bean, fava bean, walnut, and cotton seed was first reported by Ganzler and Salgo (*1*), where microwave energy was supplied to irradiate a solvent/sample suspension. It was found that the MAE method was more efficient in performance and solvent use than Soxhlet extraction (SE). Pylypiw et al. (*2*) used a scientific microwave extraction apparatus and reported on the MEA extraction of several pesticides from crops such as beets, cucumbers, lettuce, peppers, and tomatoes. The recoveries and reproducibility of the microwave method compared favorably with the conventional blender extraction. Falqui-Cao et al. (*3*) used a focused MAE technique for analyzing residues of five pesticides in strawberries.

As part of an ongoing program addressing the extraction of pesticide residues from different matrixes using MAE, this study deals with multiresidue analysis of pesticide residues in vegetable samples. Because the cost of the scientific equipment associated with MAE is considerable, we used in our study a commercial household microwave unit. The main advantage of using the latter, particularly in the developing countries, is the low cost of the equipment, short extraction time, and low use of solvents. Our recent work has demonstrated that MAE extraction using a commercial household microwave unit is a viable alternative to SE of chlorothalonil in coffee beans from Ethiopia (*4*) and fenitrothion in white and black beans from Senegal (*5*). It was further shown that the use of MAE and accelerated solvent extraction (ASE) in the extraction of chlorothalonil from coffee beans was more convenient, faster, and less solvent intensive relative to SE (*4*). However, the cost of the ASE instrument was considered prohibitive for some laboratories budgets, particularly in developing countries.

The insecticides thiamethoxam [(*EZ*)-3-(2-chloro-1,3-thiazol-5-ylmethyl)-5-methyl-1,3,5-oxadiazinan-4-ylidene(nitro)-amine], imidacloprid [1-(6-chloro-3-pyridylmethyl)-*N*-nitro-

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imidazolidin-2-ylideneamine], and the fungicide carbendazim (methyl benzimidazol-2-ylcarbamate) are simultaneously applied during the growing season in different vegetable crops in India to control insects and fungal diseases. Although high-performance liquid chromatographic (HPLC) methods have been reported for the residue determination for the individual compounds (*6*), there is as yet no published method available for the simultaneous determination of these pesticides in vegetable crops. In a previous paper, we reported simultaneous determination of carbaryl, malathion, fenitrothion, and diazinon residues in sesame seeds obtained from an Ethiopian field crop (*7*). This study is an extension of our ongoing research on MAE of pesticide residues from crop and vegetable samples using a commercial household microwave unit. Herein, we report the results obtained for simultaneous determination of thiamethoxam, imidacloprid, and carbendazim residues in cabbage, tomatoes, chilies, potatoes, and peppers. MAE was used for the extraction of pesticide residues followed by cleanup and subsequent analysis by HPLC. The residues levels of the pesticides under study after the cooking process were also investigated for cabbage and tomato samples.

MATERIALS AND METHODS

Chemicals. All solvents used were of HPLC grade (Burdick and Jackson, McGraw Park, IL) and used as received. Analytical grade imidacloprid was obtained from Bayer (Mumbai, India), thiamethoxam was obtained from Syngenta (Mumbai, India), and carbendazim was obtained from Rallis India Ltd. (Bangalore, India). Stock solutions of each pesticide (10 mg/100 mL) were prepared in acetonitrile except for carbendazim, which was prepared in acidic acetonitrile (1% 0.1 N HCl). One milliliter of each stock solution was pipetted and taken in a 10 mL volumetric flask, and the volume was made with acetonitrile up to the mark to obtain a stock standard mixture of the three pesticides containing 10 μg/mL of each. This served as a stock standard mixture and was used to obtain working solutions (1 and 0.05 μg/mL) by serial dilution.

Vegetable Samples. Cabbage, tomatoes, chilies, potatoes, and peppers were obtained from local grocery stores. Each vegetable sample was chopped into small pieces and mixed. A 20 g vegetable sample was weighed into a beaker and fortified with an aliquot of the pesticide working standard mixture to obtain the desired concentration. Cabbage and tomato samples were fortified to obtain pesticide concentrations of 0.1, 0.5, and 1.0 μg/g, while chilies, potatoes, and peppers were fortified at the 1.0 μg/g level only. The solvent was allowed to evaporate for 24 h, and the sample was then ground and thoroughly mixed using a mortar and pestle. The control vegetable samples, with no pesticide fortification, were processed following a similar stepwise procedure. All of the recovery experiments were conducted in triplicate except chilies, potatoes, and peppers samples without MAE, which were run in duplicate as described below.

MAE. The use of microwave energy for extraction of pesticide residues with flammable organic solvents can pose serious hazards. Microwave energy will heat liquids and increase pressures in vessels. If the sample seal or "safety bottle" leaks, potential explosive or flammable concentrations of solvent may be present within the apparatus. Under these conditions, sparks within the microwave oven could result in explosions or fires. To minimize such hazards, it is critical that safety methods be followed exactly. It is also recommended that equipment with appropriate safety features recently acquired in the authors' laboratory should be used for future work.

A commercial household microwave oven (Sharp model R-530CW) was used for all of the MAE extractions. The microwave oven was operated with a 60 Hz single-phase output of 1100 W. A 1.0 g portion of the fortified or control vegetable sample was placed in a 25 mL glass vial followed by the addition of 10 mL of acetone. The vial was tightly sealed using a Teflon-lined screw cap. The contents in the vial were vigorously shaken by hand, and the vial was subsequently placed in a plastic safety container (wide mouth 500 mL capacity with cap, high-density polyethylene). The safety bottle was closed and then placed at the center of the microwave oven. Extraction was performed for 30 s at a power level of 50%. After extraction, the glass bottle was kept closed, cooled in a refrigerator for 5 min, and vigorously shaken by hand, and finally, the contents of the bottle were allowed to cool to room temperature before opening. A 5 mL aliquot of the supernatant extract was carefully removed with a 9 in. glass pipet into a 5 mL conical glass centrifuge tube (13 mm $\times$ 135 mm) and evaporated to 0.5 mL with a gentle stream of dry nitrogen gas.

Fortified and control vegetable samples prepared in glass bottles as described above were simultaneously processed side by side without MAE. The contents of the bottle were frequently and vigorously shaken by hand, allowed to stand on the bench, and then processed as described above. In preliminary experiments, it was observed that occasional vigorous shaking of the samples in the bottle for even a longer period ($\sim$30 min) did not result in any noticeable increase in recoveries of the three pesticides used in this study.

Blender Extraction. A fortified or control vegetable sample was weighed (10.0 g) and blended in a Waring blender (model 33BL79, Waring Product Division, New Hartford, CT) with 100 mL of acetone for 15 min. The content was filtered on a Buchner funnel with suction. The solid residues on the funnel were carefully transferred to the blender and blended again with 50 mL of acetone. The blended material was filtered, and the solid residue was washed with 25 mL of acetone. The combined extract was evaporated on a rotary vacuum evaporator and then concentrated to $\sim$2 mL. The latter was then quantitatively transferred to a 5.0 mL graduated centrifuge glass tube, and the volume was made up to the mark with acetone. A 0.5 mL aliquot extract was used for cleanup.

Cooked Samples. The fortified or control vegetable sample was weighed (5 g) into a 200 mL beaker, and 20 mL of double-distilled water was added. The beaker was loosely covered with a plastic lid, placed in the center of the microwave oven, and cooked for 2 min at a power level of 100%. More water (10 mL) was added, and cooking was continued for another 5 min. After it was cooked, the sample was removed from the oven and cooled to room temperature. The cooked material was subsequently ground using a mortar and pestle. The sample was then processed as described in the previous section.

Cleanup. The concentrated acetone extract (0.5 mL) obtained from MAE, or without MAE, or blender extraction as described above was diluted to 15 mL with 10% aqueous sodium chloride. The diluted extract was partitioned with hexane three times sequentially (10 mL + 10 mL + 5 mL). The hexane portions were discarded, and the aqueous portion was extracted three times sequentially with dichloromethane (10 mL + 10 mL + 5 mL). The dichloromethane extracts were combined and dried over anhydrous sodium sulfate (1 g) and concentrated to a final volume of $\sim$2 mL under reduced pressure on a rotary flash evaporator (30 °C). The solution was then quantitatively transferred to a 5 mL conical glass centrifuge glass tube with the help of a glass pipet. The dichloromethane was evaporated under a gentle stream of dry nitrogen gas and solvent exchanged with acetonitrile for the complete removal of dichloromethane. Finally, the concentrated extract was further diluted with acetonitrile:water (1:1) to bring the final volume to 1.0 mL. The contents were vortex mixed and filtered through a 13 mm $\times$ 0.45 μm filter (Supelco, Iso-Disk Filters, PTFE-13-4) prior to analysis. Before it was filtered, the final solution was diluted or concentrated when necessary so that the injected volume contained an amount of the pesticides within the linear range of the ultraviolet (UV) detector.

HPLC. The HPLC (Hewlett-Packard model 1100 binary pump and model 1100 UV detector) was equipped with a variable UV detector set to 270 nm and a Rheodyne (Coprati, CA) model 1070 injector with a fixed 20 μL sample loop. A Whatman (Clifton, NJ) stainless steel analytical column (25 cm $\times$ 9.4 mm) packed with Partisil 10 ODS-1 (10 μm particle size) stationary phase was used at ambient temperature. A Whatman stainless steel guard column containing pelicular C18 preceded the analytical column. The mobile phase consisted of (1:1) acetonitrile−water (containing 0.01% orthophosphoric acid). All separations were performed at a flow rate of 1.0 mL/min. Injection of 20 μL was performed in the HPLC for quantitative analysis. A four-point calibration curve was constructed from the peak area of the calibration run. The HPLC detector response was linear in the range of 0.1−2.0

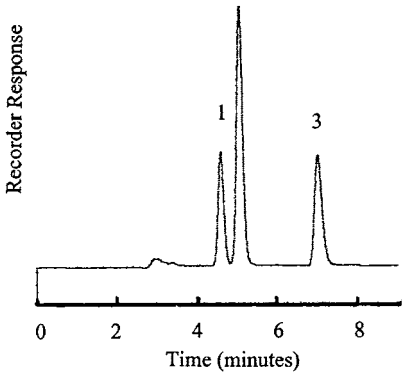

Figure 1. HPLC chromatograms of the reference standards: 1, thiamethoxam; 2, imidacloprid; and 3, carbendazim.

μg/mL, which covers the respective concentration range expected for the three pesticides under study during routine residue analysis. The concentration of the pesticide residues in the extract was determined by comparing the peak area with that of reference standard. Under the experimental conditions described, the HPLC retention times for thiamethoxam, imidacloprid, and carbendazim were 4.5, 5.0, and 7.1 min, respectively (**Figure 1**). Although the observed maximum absorption wavelengths for thiamethoxam, imidacloprid, and carbendazim were 256, 270, and 286 nm, respectively, the results were evaluated with detection at 270 nm in all cases. This simplified the data handling, and very few interfering peaks at the retention times of the pesticides were observed for the quantification of the three pesticides at this wavelength.

Identification and Confirmation. Identity of the desired peak was confirmed by comparing its retention time with that of the reference standard and subsequently confirmed in selected extracts by HPLC/mass spectrometry (MS). A Waters Corporation (Milford, MA) 2690 Separations Module HPLC system coupled to a Waters Micromass ZQ 2000 single-quadrupole mass spectrometer using electrospray ionization in both positive and negative ion modes was employed. The mass spectrometer voltage was optimized for the detection of the three pesticides.

Instrument (IDL) and Estimated Method Detection Limit (EMDL). The IDL is treated as the minimum concentration of pure pesticide that can be reliably detected by the HPLC system used in this study under the stated conditions of analysis. The IDLs for thiomethoxam, imidacloprid, and carbendazim were estimated through 10 repetitive injections of a standard solution containing 0.4 μg/mL of each pesticide as follows:

$$\text{IDL } (\mu\text{g/mL}) = \text{SD} \times t_{95}$$

where SD is the standard deviation of the peak areas for the replicate injections and t_{95} is the Student's t at the 95% level of confidence.

The EMDLs for each pesticide were estimated from the IDLs as follows:

$$\text{EMDL } (\mu\text{g/g}) = \frac{\text{IDL} \times 0.020 \times 100}{M \times \%\ \text{rec}}$$

where M is the mass of the sample (g) and % rec is the average percent recovery of the pesticide in the method. The 0.020 term in the equation refers to the 0.020 mL fixed volume sample loop of the HPLC instrument.

RESULTS AND DISCUSSION

The IDL and EMDL values for the three pesticides under study are shown in **Table 1**. By comparison with the pure standard solutions, the vegetable-containing samples showed matrix diminishment effects for the three pesticides. Samples with pesticide concentrations lower than the EMDLs were considered not quantifiable.

Table 1. IDL and EMDL of Thiamethoxam, Imidacloprid, and Carbendazim by HPLC

	thiamethoxam	imidacloprid	carbendazim
	matrix		
	IDL (μg/mL)		
pure standard	0.020	0.015	0.018
	vegetable matrix		
	EMDL (μg/g)		
cabbage	0.044	0.038	0.048
tomatoes	0.040	0.033	0.045
chilies	0.045	0.030	0.032
peppers	0.038	0.037	0.041
potatoes	0.039	0.039	0.043

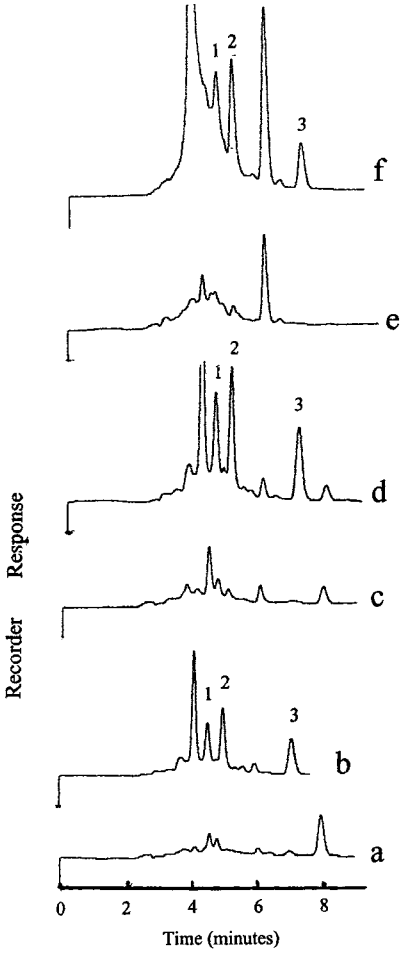

Figure 2. HPLC chromatograms of extracts from cabbage without and after MAE. (**a**) Control cabbage without MAE; (**b**) fortified cabbage without MAE; (**c**) control cabbage after MAE; (**d**) fortified cabbage after MAE; (**e**) control cooked cabbage after MAE; and (**f**) fortified cooked cabbage after MAE. Refer to **Figure 1** for peak labels.

In the preliminary experiments, solvents and/or solvent mixtures that were commonly used in conventional MAE were tried in the microwave system using closed glass bottles described earlier. Hexane/acetone (1:1) and acetone reached the maximum temperature within 30−60 s. Acetone was chosen in subsequent experiments since this solvent yielded higher recoveries of the pesticides. MAE was performed for 30, 60, 90, and 120 s at 50 and 70% power. Heating at 50% power was preferred because the higher power setting often caused a rapid boiling that resulted in a break in the seal of the lid resulting in the loss of sample. The time required to achieve quantitative recoveries was chosen at 30 s, and the results were

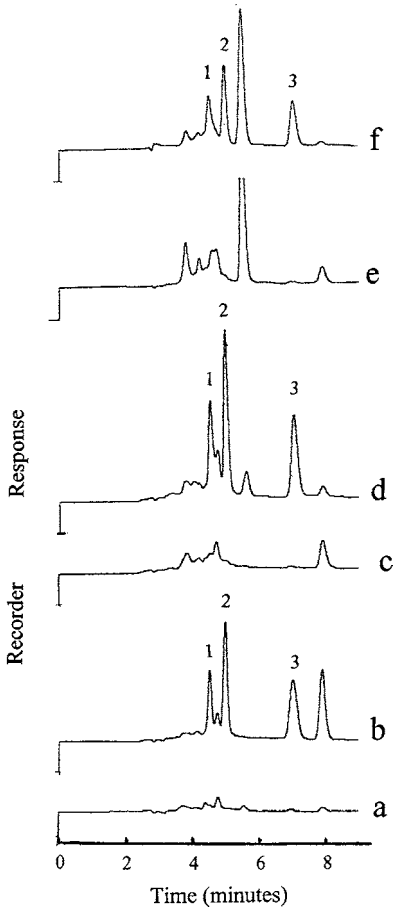

Figure 3. HPLC chromatograms of extracts from tomatoes without and after MAE. (**a**) Control tomatoes without MAE; (**b**) fortified tomatoes without MAE; (**c**) control tomatoes after MAE; (**d**) fortified tomatoes after MAE; (**e**) control cooked tomatoes after MAE; and (**f**) fortified cooked tomatoes after MAE. Refer to **Figure 1** for peak labels.

comparable with those obtained at longer times. Therefore, the choice of 30 s at 50% power setting for microwave extraction was a compromise parameter with the intent of developing a multiresidue method for the three pesticides from the vegetable samples under study. Under the experimental conditions tested, no breakdown of thiamethoxam, imidacloprid, and carbendazim was observed.

An initial attempt to clean up the extracts obtained from MEA of the fortified vegetable samples was performed using both C_{18} and ENV^+ (Jones Chromatography, Lakewood, CO) cartridges. The concentrated acetone MAE extract (0.5 mL) described above was diluted with 10% aqueous sodium chloride solution and passed through the cartridge. To remove the chlorophyll contents from extract effectively, 0.45 μm filters coupled with solid phase extraction (SPE) cartridges were also tried. A number of elution solvent systems including CH_3CN, CH_3CN-H_2O (1:1), and/or CH_3OH were tried. However, in each case, low recoveries were obtained and considerable interfering coextractive HPLC peaks were present in the chromatograms. Therefore, it was necessary to devise an alternative cleanup procedure that would remove most of the interfering coextractives from the extracts prior to HPLC analysis and provide better recoveries.

The hexane partition cleanup procedure developed in this study effectively removed interfering coextractives for HPLC analysis and was more effective than the SPE cartridges. **Figures 2−4** show the HPLC chromatograms of the three pesticides from extraction of the fortified and control cabbage, tomatoes, chilies, potatoes, and peppers. No interfering peaks were observed for the quantification of the three pesticides. In the case of MEA extracts of cooked cabbage, a peak was always found at an early retention time, but as such, it did not affect the pesticide peaks used in quantitation.

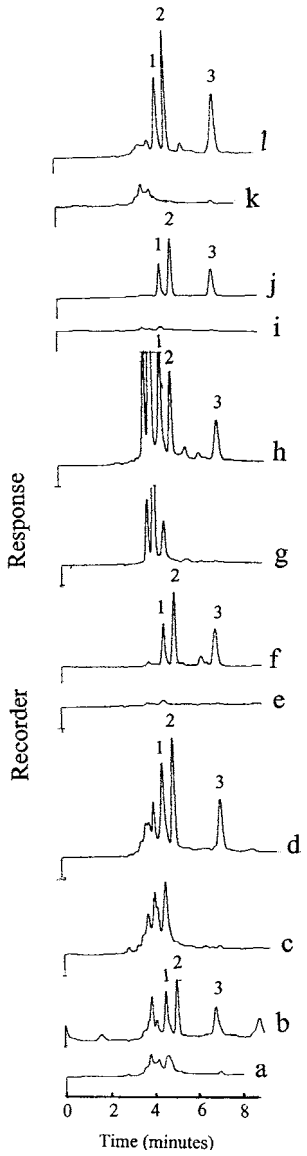

Figure 4. HPLC chromatograms of extracts from chilies, potatoes, and peppers without and after MAE. (**a**) Control chilies without MAE; (**b**) fortified chilies without MAE; (**c**) control chilies after MAE; (**d**) fortified chilies after MAE; (**e**) control potatoes without MAE; (**f**) fortified potatoes without MAE; (**g**) control potatoes after MAE; (**h**) fortified potatoes after MAE; (**i**) control peppers without MAE; (**j**) fortified peppers without MAE; (**k**) control peppers after MAE; and (**l**) fortified peppers after MAE. Refer to **Figure 1** for peak labels.

Table 2 summarizes the recoveries of the three pesticides from fortified cabbage and tomatoes at the 0.1, 0.5, and 1.0 μg/g levels. The mean recoveries of the three pesticides by MAE for cabbage and tomato ranged from 68.3 to 99.7 and 68.1 to 100%, respectively. The corresponding values from samples simultaneously processed without microwave extraction were 44.1−60.9 and 44.1−60.7%, respectively. The recoveries by MAE were comparable to those obtained by the conventional blender extraction technique. The recoveries obtained from cabbage and tomato fortified at 1.0 μg/g level by the later technique ranged from 70.1 to 90.2 and 79.7 to 94.0%,

Table 2. Percent Mean Recoveries and RSD of Thiamethoxam, Imidacloprid, and Carbendazim from Fortified Cabbage and Tomato without and after Microwave Extraction

fortification level (μg/g)	thiamethoxam		imidacloprid		carbendazim	
	MAE	no MAE	MAE	no MAE	MAE	no MAE
			cabbage			
0.1	81.7 (6.6)	44.1 (9.9)	77.0 (7.1)	58.1 (2.1)	68.3 (4.6)	47.0 (1.2)
0.5	93.6 (7.1)	47.1 (2.1)	76.8 (3.6)	53.8 (4.7)	72.8 (3.7)	52.1 (1.9)
1.0	99.7 (6.6)	51.7 (1.4)	82.7 (3.0)	60.9 (2.6)	74.7 (3.0)	49.2 (2.9)
overall mean	91.7 (6.8)	47.6 (4.4)	78.8 (4.6)	57.6 (3.1)	71.9 (3.7)	49.4 (2.0)
			tomato			
0.1	100 (9.2)	60.7 (5.2)	92.8 (5.2)	52.5 (2.8)	68.1 (7.6)	44.1 (4.2)
0.5	98.8 (2.2)	58.6 (4.3)	87.7 (5.1)	51.1 (3.7)	80.7 (3.8)	47.0 (3.2)
1.0	97.2 (1.4)	56.5 (1.8)	89.3 (3.7)	48.6 (1.3)	81.0 (2.0)	50.9 (1.3)
overall mean	98.7 (4.2)	58.6 (3.8)	89.9 (4.7)	50.9 (2.6)	76.6 (4.5)	47.3 (2.9)

Table 3. Percent Mean Recoveries of Thiamethoxam, Imidacloprid, and Carbendazim from Chilies, Peppers, and Potatoes Fortified at 1.0 μg/g Level without and after Microwave Extraction

	thiamethoxam		imidacloprid		carbendazim	
vegetable	MAE	no MAE	MAE	no MAE	MAE	no MAE
chilies	89.9 (6.3)	60.4	99.5 (6.5)	37.2	106 (3.2)	45.0
peppers	106 (6.7)	61.4	82.0 (4.8)	48.2	82.1 (1.8)	45.2
potatoes	104 (2.1)	57.1	76.3 (2.3)	42.3	78.9 (6.8)	42.7

Table 4. Percent Mean Recoveries and RSD of Thiamethoxam, Imidacloprid, and Carbendazim from Cabbage, Tomatoes, Chilies, Peppers, and Potatoes Fortified at 1.0 μg/g Level with Blender Extraction

vegetable	thiamethoxam	imidacloprid	carbendazim
cabbage	90.2 (2.3)	81.3 (6.7)	70.1 (1.9)
tomatoes	94.0 (6.8)	90.2 (1.8)	79.7 (4.3)
chilies	85.3 (6.3)	92.4 (6.5)	92.2 (2.4)
peppers	92.1 (2.3)	81.3 (6.8)	81.5 (1.8)
potatoes	97.5 (1.8)	76.1 (4.8)	72.9 (2.9)

respectively (**Table 4**). The blender extraction method is considered similar to those normally used for the routine residue analysis of the three pesticides from vegetable crops. However, when compared to MAE, the blender extraction method is time consuming and uses a considerably large volume of solvent. The percent recoveries of thiamethoxam from cabbage and tomato of residues were much better by MAE and blender extraction than those for the other two pesticides.

As seen in **Table 3**, the recoveries of the three pesticides after MEA from chilies, peppers, and potatoes fortified at 1.0 μg/g level ranged from 89.9 to 106, 82.0 to 106, and 76.3 to 104%, respectively. The corresponding values for samples processed simultaneously but not subjected to MAE were 37.2−60.4, 45.2−61.4, and 42.3−57.1%, respectively. The recoveries obtained from chilies, peppers, and potatoes fortified at the 1.0 μg/g level by the three pesticides and subjected to blender extraction ranged from 85.3 to 92.4, 81.3 to 92.1, and 72.9 to 97.5%, respectively (**Table 4**). The results in **Tables 2** and **3** illustrate the effectiveness of MAE in obtaining satisfactory recoveries of the three pesticides from the vegetable samples used in this study. Overall, the precision can be described by a relative standard deviation (RSD) of <7% for the three pesticides used in this study.

The cabbage and tomato samples fortified at the 1.0 μg/g level were cooked as described earlier. The procedure used was considered to simulate the condition normally used in household cooking. The percent recoveries after MEA of the cooked samples for thiamethoxam, imidacloprid, and carbendazim were 106, 91.0, and 51.0, respectively. The corresponding values for tomatoes were 102, 91.6, and 64.1, respectively. Cooking of cabbage and tomatoes appears to show a considerable loss of carbendazim residues. The presence of the parent pesticide residues in the cooked samples was confirmed by HPLC/MS, and no breakdown products were observed for any of the pesticides.

The method presented here demonstrates that MAE is an efficient tool for simultaneous extraction of pesticides residues from vegetable samples without showing strong matrix effects normally observed in SE. The sample preparation and MAE processes described here appear to be simple and time-saving procedures. The MAE involves the use of relatively small amounts of solvents and therefore is environmentally friendly. This method provides an attractive approach with detection limits at sub parts per million concentrations and could be extended to additional crops and pesticides that could be present at very low concentrations.

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LITERATURE CITED

(1) Ganzler, K.; Salgó, A.; Valkó, K. Microwave extraction: A novel sample preparation method for chromatography. *J. Chromatogr. A* **1986**, *371*, 299−306.

(2) Pylypiw, H. M., Jr.; Arsenault, T. L.; Thetford, C. M.; Mattina, M. J. I. Suitability of Microwave-assisted extraction for multiresidue pesticide analysis of produce. *J. Agric. Food Chem.* **1997**, *45* (9), 3522−3528.

(3) Falqui-Cao, C.; Wang, Z.; Urruty, L.; Pommier, J.-J.; Montury, M. Focused microwave assistance for extracting some pesticide residues from strawberries into water before their determination by SPME/HPLC/DAD. *J. Agric. Food Chem.* **2001**, *49* (11), 5092−5097.

(4) Negeri, B. O.; Foster, G. D.; Khan, S. U. Determination of Chlorothalonil Residues in Coffee. *Toxicol. Environ. Chem.* **2000**, *77*, 41−47.

(5) Diagne, R. G.; Foster, G. D.; Khan, S. U. Comparison of Soxhlet and microwave-assisted extraction for the determination of fenitrothion residues in beans. *J. Agric. Food Chem*. **2002**, *50*, 3204−3207.

(6) *The Pesticide Manual*, 10th ed.; Crop Protection Publication; The Royal Society of Chemistry: Cambridge, U.K., 1994.

(7) Gebreegzi, Y. T.; Foster, G. D.; Khan, S. U. Simultaneous determination of carbaryl, malathion, fenitrothion, and diazinon residues in sesame seeds (*Sesamum indicum* L.). *J. Agric. Food Chem.* **2000**, *48*, 5165−5168.

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