



# Journal of Applied Sciences

ISSN 1812-5654

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## One-step Extraction of Multiresidue Pesticides in Soil by Microwave-assisted Extraction Technique

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**Abstract:** A screening multi-residues method based on the Microwave-Assisted Extraction (MAE) technique has been optimized using soil samples collected from 15 regions in Saudi Arabia. This method was used to extract 12 pesticide residues with a broad range of physico-chemical properties in agricultural soils containing to Organophosphorous, Organochlorines, Pyrethroids and Carbamates mainly used in agriculture. All MAE factors affecting the extraction techniques (heating, pressure, power, time and solvent volume) of the targeted compounds were studied through experimental design to obtain a simple MAE method and evaluate the optimum extraction condition compared with traditional Soxhlet method for soil samples. The tested pesticide residues in the extracts of both techniques were analyzed by Gas Chromatography-Mass Spectrometer (GC-MS). The results were compared for the percentage of recovery, time consumption and volume of organic solvent used in each extraction procedure. The results indicated that the MAE method had the advantages resulting from the use of a low volume of organic solvent (acetone: hexane, 3:2), an unnecessary cleanup step and good efficiency to extract different groups of pesticides in soils at residual levels in 20 min, this compared with Soxhlet method. All the compounds extracted by MAE method were recovered in good yields and Minimum Detection Limits (MDL) ranging from 0.0001 to 0.004 mg kg<sup>-1</sup>. The MAE approach was efficient and faster than the Soxhlet method in determining 12 multi-residue pesticides with a broad range of physico-chemical properties in soils without cleanup of the extracts.

**Key words:** MAE, pesticides, residues, extraction, soil, GC-MS, soxhlet

### INTRODUCTION

The presence of pesticides as environmental contaminants has created concern about their fate and transport in natural waters, sediments and soils (Kizaa and Brown, 1998; Harner *et al.*, 1999; Jong-Hun and Smith, 2001; Hogendoorn *et al.*, 2001; Sun and Lee, 2003; Gong *et al.*, 2004). Pesticides wide use could lead to extensive pollution of the environment and constitutes a potential and/or deliberate risk to human health because some of these pesticides are classified as a probable human carcinogens (El-Saeid, 1999; Brock *et al.*, 2000; Saunders and Harper, 1994). Monitoring of pesticide residues in water and soils were reported by many investigators: Goncalves *et al.*, 2006; Manirakiza *et al.*, 2003; Oldal *et al.*, 2006; Shegunova *et al.*, 2006; Westbom *et al.*, 2008; Zhang *et al.*, 2006; Shen and Lee, 2003; Chen, 2010. In intensive agriculture, monitoring pesticides is necessary, but it is usually tedious and time-consuming, especially at the extraction stage (Williams, 1990; Saunders and Harper, 1994).

Pesticide determination in soil samples is carried out by different analytical methods such as Liquid Chromatography-Mass Spectrometry (LC-MS) or Gas Chromatography-Mass Spectrometry (GC-MS) even if the detection limits depends on matrices, GC-MS often provides better results than LC-MS in terms of LOD (limits of detection), except for water and liquid samples (Schreck *et al.*, 2008; Rashid, 2010).

Microwave-Assisted Extraction (MAE) is a recent extraction method that was successfully used during last years in the determination of a wide range of chemicals in many matrices. Therefore, a comparison between recent extraction methods with traditional extraction methods could be very useful to put forward new alternatives, which minimize work, time and expense (Pastor, 1997; Pateiro-Moure *et al.*, 2008).

Microwave-Assisted Extraction (MAE) technique was used by Ganzler *et al.* (1986) to extract anti-nutritive compounds from various plant materials. Since, then microwave methodologies have been adapted for other scientific applications, including the extraction of

pesticides (Fuentes *et al.*, 2006; Yuan *et al.*, 2006; Sporning *et al.*, 2005; Prados-Rosales *et al.*, 2002; Tavares *et al.*, 2005). Although, the advantages of these procedures include reduced solvent usage and shorter analysis time with less organic solvents, there is the possibility of a new application of Microwave-Assisted Extraction. Several publications have described its extraction methodologies for reduced solvent usage and shorter analysis time with less organic solvents purposes (Le Calves *et al.*, 2002; Patsias *et al.*, 2002; Barriada-Pereira *et al.*, 2003; Sun and Lee, 2003; Coscollà, 2009). Microwave energy was supplied to irradiate solvent/sample suspension for 30 sec several times each and it was found that MAE method was more efficient than Soxhlet (Singh *et al.*, 2007; Carvalho *et al.*, 2008; Schreck *et al.*, 2008).

In this study, we studied and described the application of a MAE techniques based on modified extraction approach. The performance of the MAE system was demonstrated by its application in the extraction of pesticide residues from soil samples. The advantage of this approach is to be able to extract solutes from water and soils without using much solvents, cleanup and short time at a low-cost and with simple operation.

## MATERIALS AND METHODS

The present study was carried out during the year of 2007 in the Laboratory of the Soil Sciences Department, King Saud University, Saudi Arabia.

**Chemicals and reagents:** Pesticide standards (Dimethoate, Chloroneb, Methomyl, Oxamyl, Toxaphen, DDT, DDE, Monocrotophos, Chlorpyrifos, Diazinon, Cypermethrin and Lindane) were provided by Chemservice, USA. All pesticide standards were of 98-99% purity. All the solvents (hexane and acetone) used were residue analysis grade. The stock working solutions were prepared in acetone.

**Preparation of spiked soil samples:** Spiked samples were prepared by adding an appropriate volume of spiking solution to 5 g of soil. The soil was spiked with a stock solution of the pesticides (5 mg L<sup>-1</sup>) prepared in Acetone. The spiked samples were prepared just before analysis, waiting approximately 30 min until solvent evaporation. Soil samples were extracted by two techniques, MAE and Soxhlet extraction (Barriada-Pereira *et al.*, 2003; Sporning *et al.*, 2005). The main purpose of this step was to calculate the average of the recovery percent of investigated pesticides by both extraction techniques.

**Table 1:** Optimum conditions of MAE technique for soil samples extraction

Parameters	Experimental conditions	Optimum conditions
Heating temperature (°C)	90-170	120
Pressure (psi)	60-120	80
MAE power (%)	40- 80	60
Solvent mixture		
Acetone: Hexane (v/v)	1:1; 2:1; 3:1; 3:2	3:2
Solvent volume (mL)	20-40	35
Extraction time (min)	15-30	20

**Minimum detection limit (MDL):** To determine the MDL and perform the GC-MS quantification using a four-point calibration curve plotting peak area versus ppm concentration of 12 pesticides using the dilution levels ranged from 0.0001-5.00 ppm (this sentence needs to be re-written).

**Extraction of pesticide residues by MAE:** The proposed EPA Method 3540C (USEPA, 1996) and CEM application note No. E003 (CEM Corporation, 1994) was modified and used while conducting the present study. A Microwave Assisted Extraction (MAE) system model MES-1000 (CEM Corporation, Matthews, NC, USA) with Lined Extraction Vessels (LEV) was used. This system consists of a 950 watt microwave instrument which has been specifically designed for use with organic solvents. Extraction vessels are double-walled vessels specifically adapted for use with organic solvents. Preliminary studies were performed to evaluate MAE efficiency for the effects of temperature, microwave power, extraction time and solvent volume (Table 1) in extracting different groups of pesticide residues from soil and water samples. For this purpose, 5.0 g of soil was weighed in a tetrafluoromethaxil (TFM) microwave extraction vessel with addition of 1 mL of an aqueous spiking solution containing mixture of the pesticide standard solution. Samples were equilibrated by shaking for 1 h before microwave extraction. Finally, all samples were extracted under different conditions as showed in the results to obtain the optimal MAE conditions with this procedure. The optimum Extraction conditions with high recovery was conducted with 5.0 g soil samples. The extraction solvent was 35 mL of Acetone : Hexane (3:2), pressure was 80 psi, microwave power was 60%, temperature was 120°C and time of extraction 20 min. After extraction, soil extracts were filtered and evaporated to dryness. The residues were re-dissolved and directly analyzed by Gas Chromatography- Mass Spectrometer (GC-MS) and clean chromatograms were obtained without any additional cleanup step.

**Extraction of pesticide residues by soxhlet:** The extraction was done by Soxhlet method (Barriada-Pereira *et al.*, 2003; Sporning *et al.*, 2005). A 5.0 g soil sample was extracted

with Acetone: Hexane (3:2) for 8 h. After extraction, soil samples were filtered and evaporated to dryness. The residues were re-dissolved in 1 mL of acetone and directly analyzed by Gas Chromatography-Mass Spectrometer (GC-MS). Clean chromatograms were obtained without any additional cleanup step.

**Gas Chromatography-Mass Selective (GC-MS) analysis:**

Both extracts of Pesticide residues in soil samples extract were performed and analyzed by a HP 5890 series II plus GC coupled to an HP 5972 Mass Selective Detector. The GC columns were a DB-5 fused silica capillary column (30 m×0.32 mm i.d., 1 µm film thickness; J and W Scientific, Folsom, CA). One micro liter of the soil sample extract was injected split less injector temperature of 250°C, on the GC-MS for analysis. The temperature program for the GC was as follows: isothermal for 1 min at 100°C, increased at a rate of 10°C min<sup>-1</sup> to 240°C and isothermal for 15 min. Helium was used as a carrier gas (1.2 mL min<sup>-1</sup>). Quantification was performed using a four-point calibration curve plotting peak area versus ppm concentration. The result was expressed in percentage recovery of pesticide.

**RESULTS AND DISCUSSION**

**Screening and optimum extraction factors of MAE:**

The application of MAE techniques based on modified extraction approaches were demonstrated by its application in the extraction of 12 pesticide residues with a broad range of physico-chemical properties from agricultural soil samples. Comparative study was carried out by Soxhlet extractor. The evaluation of the extraction efficiency of MAE was affected by 6 factors. These factors were defined to evaluate their contribution to pesticides extraction efficiency of spiked soil samples. The results of this part show the optimum extraction conditions (Table 1) with the average of high recovery ranging from 98.00±2.25 to 102.44±2.73 for all tested pesticides and low MDL.

**Minimum Detection Limits of pesticide residues (MDL):**

The MDL of all tested pesticide residues extracted by modified MAE technique compared with Soxhlet extractor and analyzed by GCMS were determined to evaluate the efficiency and availability of both extraction methods. The averages of MDL ranged from 0.0001 to 0.004 and from 0.002 to 0.012 mg kg<sup>-1</sup> for MAE and Soxhlet, respectively as shown in Table 2.

**Recovery (%) of pesticide residues in spiked soil samples:**

Recovery studies to validate MAE extraction

Table 2: MDL of tested pesticides in spiked soil samples extracted by MAE and Soxhlet and determined by GC-MS

Pesticides	MDLs (mg kg <sup>-1</sup> soil)	
	MAE	Soxhlet
Dimethoate	0.002	0.004
Chloroneb	0.003	0.012
Methomyl	0.002	0.007
Oxaryl	0.001	0.005
Toxaphen	0.001	0.003
pp-DDT	0.0001	0.002
pp-DDE	0.0001	0.005
Monocrotophos	0.002	0.010
Chlorpyrifos	0.002	0.004
Diazinon	0.004	0.011
Cypermethrin	0.002	0.004
Lindane	0.001	0.003

Table 3: Pesticide recovery (%) and relative standard deviation (RSD, %) of spiked soil samples extracted by MAE and Soxhlet and determined with GC-MS

Pesticides	Recovery±RSD (%)	
	MAE	Soxhlet
Dimethoate	99.64±1.67	96.66±2.09
Chloroneb	99.28±1.22	94.75±1.98
Methomyl	98.56±2.07	94.26±2.39
Oxaryl	98.70±2.48	93.18±2.27
Toxaphen	99.04±1.47	95.33±2.19
pp-DDT	102.44±1.55	97.09±2.36
pp-DDE	101.67±2.26	94.88±2.39
Monocrotophos	98.14±2.00	93.69±2.06
Chlorpyrifos	98.00±2.29	94.00±2.25
Diazinon	98.00±2.25	93.11±2.08
Cypermethrin	98.45±1.68	95.09±2.11
Lindane	101.55±1.22	96.80±2.59

RSD relative standard deviation

techniques were carried out with spiked soils obtained by adding a low volume of spiking pesticide solution in acetone to compare with Soxhlet extractor. From the comparison of both methods, it was found that higher recoveries were obtained by modified MAE than Soxhlet for all tested pesticides. On the other hand, the recoveries by Soxhlet were still good for Lindane, Toxaphen, DDT and Dimethoate under their respective optimum conditions. Therefore, MAE are suitable techniques for extracting all tested pesticides from spiked soil with recovery, % (±RSD) ranging from 98.00±2.25 to 102.44±2.73 and 93.11±2.08 to 97.09±2.36 % for Soxhlet extractor as shown in Table 3.

**Monitoring of pesticide residues in soil samples:**

By applying MAE in multiresidues, which we thus developed to obtain the optimum conditions, it was possible to extract 12 pesticides in soil samples (fresh, control or spiked) in 20 min. GC-MS was applied as analysis technique with high sensitivity of all 12 detected pesticides in this study (Al-Turki *et al.*, 2009).

Table 4: Averages of pesticide residues in soil samples (mg kg<sup>-1</sup> soil) extracted by MAE and determined with GC-MS

Pesticides	Residues concentration (mg kg <sup>-1</sup> soil)
Dimethoate	0.611
Chloroneb	0.258
Methomyl	0.259
Oxamyl	0.108
Toxaphen	0.151
pp-DDT	0.482
pp-DDE	0.432
Monocrotophos	0.052
Chlorpyrifos	0.180
Diazinon	0.025
Cypermethrin	0.153
Lindane	0.394

The results of studied pesticides was shown in Table 4 and showed that: Dimethoate was detected in 51.85% of total soil samples followed by Methomyl as 39.63%, Chloroneb as 34.44%. The most important notice in the results of soil analysis was the detection of the residue of DDT and one of their derivatives DDE. Both pesticide residues were detected in 4 regions namely, Al-Qatif, Al-Ahsa, Wadi Al-Dawaser and Gizan. Dimethoate was detected as the highest contaminant in Abha (0.002-1.220 mg kg<sup>-1</sup>) followed by DDT (0.960 mg kg<sup>-1</sup>) in Wadi Al-Dawaser. Meanwhile, Diazinon was detected as a lower contaminant. All detected pesticide residues were over the Maximum Residue Limits (MRLs) i.e., 0.1 mg kg<sup>-1</sup> except Monocrotophos and Diazinon. These results are in agreement with those reported in previous studies for the soil samples analyzed by MAE (Coscollà *et al.*, 2009; Carvalho *et al.*, 2008; Schreck *et al.*, 2008; Oldal *et al.*, 2006; Shegunova *et al.*, 2006; Westbom *et al.*, 2008; Chen, 2010).

### CONCLUSIONS

The described method is efficient and fast to determine multi-residue pesticides in soils. Most of the compounds studied were recovered in good yields with Relative Standard Deviations (RSDs) below 3.5%. The averages of MDL ranged from 0.0001 to 0.004 and 0.002 to 0.012 mg kg<sup>-1</sup> for MAE and Soxhlet techniques, respectively. MAE technique had more extraction advantages than Soxhlet such as short time, did not require refluxing large volumes of solvent, cleanup and permitting the simultaneous extraction of several samples. The modified MAE method was successfully applied to the extraction of 12 pesticide residues namely, Dimethoate, Chloroneb, Methomyl, Oxamyl, Toxaphen, DDT, DDE, Monocrotophos, Chlorpyrifos, Diazinon, Cypermethrin and Lindane from soils which were considered an appropriate index to establishing its applications.

### ACKNOWLEDGMENT

The authors would like to thank the Deanship of Scientific Research at King Saud University for financial support through Project # DSR- AR- 2- (14).

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