

A NEW ANALYTICAL METHOD COMBINING MICROWAVE ASSISTED MICELLAR EXTRACTION FOLLOWED BY SOLID PHASE MICROEXTRACTION FOR THE DETERMINATION OF ORGANOCHLORINE PESTICIDES IN MUD SAMPLES

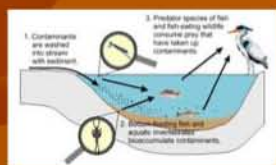
D. Vega Moreno, Z. Sosa Ferrera, J.J. Santana Rodríguez.

Department of Chemistry, Faculty of Marine Sciences, University of Las Palmas de Gran Canaria, 35017, Las Palmas de Gran Canaria, Spain. Tel: +34 92845 44 25, Fax: +34 928 45 29 22, e-mail: jsantana@dqui.ulpgc.es



INTRODUCTION

Organochlorine pesticides have bioaccumulative behaviour due to their high lipophilicity and persistence, with the subsequent risk to human health. In aquatic environment, they are removed from the water and adsorbed on the particulate matter due to their high affinity with organic matter, and finally accumulated on mud, which may play a role as a secondary contamination source.



- 1.- 4,4'-DDD
- 2.- dieldrin
- 3.- 4,4'-DDT
- 4.- 2,4'-DDT
- 5.- 4,4'-DDE
- 6.- aldrin

Organochlorine Pesticides Mix

Traditional methods for extracting organic pollutants from solid samples require typically time consuming, employ multistep procedures having high risk to lose analytes and use extensive amount of organic solvents. Microwave assisted micellar extraction (MAME) can be an alternative of these traditional methods.

However, due to the low concentration of analytes in mud samples, a preconcentration step, prior to their determination, is required. Solid phase microextraction (SPME) could be used for this purpose.

In this work, microwave assisted micellar extraction (MAME) followed by solid phase microextraction and HPLC-UV has been implemented for quantitative analysis of organochlorine pesticides in mud samples.

EXPERIMENTAL

2 g of mud sample was spiked with the pesticides mix with a final concentration of $0.8 \mu\text{g}\cdot\text{g}^{-1}$ (DDTs) and $1.6 \mu\text{g}\cdot\text{g}^{-1}$ (dieldrin and aldrin). For the extraction 8 ml of POLE (Polyoxyethylene 10 lauryl ether) micellar solution at 5% (v/v) was added to each mud sample. Mud samples were collected from 10 different regions of Gran Canaria Island (Spain).

RESULTS AND DISCUSSION

Microwave Assisted Micellar Extraction (MAME)

Spiked samples were introduced in Teflon vessels with the optimum volume of POLE solution and irradiated at the optimized conditions in a microwave oven. Surfactant extracts were then removed and filtrated.

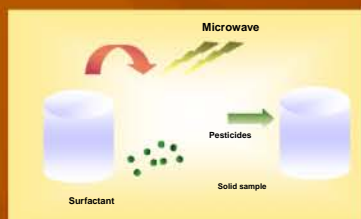


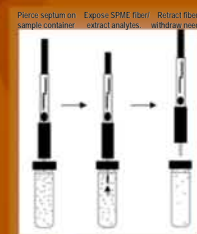
Diagram of MAME process

Optimum Conditions

Surfactant Volume	8 ml
Surfactant Concentration	5 % (v/v)
Power	750 W
Time	8 min

MAME-SPME procedure

As a second step, a $60 \mu\text{m}$ PDMS/DVB fiber was introduced in a solution with 5 ml of POLE with pesticides extract plus 7.5 ml of water in direct immersion.

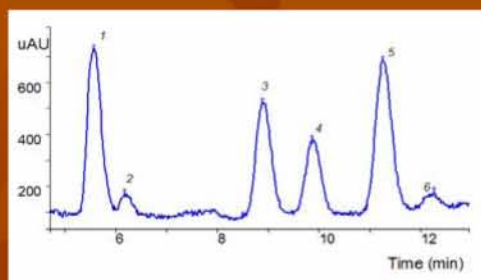


Optimum Conditions

Absorption Time	60 min
Desorption Time	8 min
Temperature	25 °C
NaCl Addition	0 %

Desorption was done in a methanol volume of 55 μl with fiber's average stirring. The extract was analyzed in a HPLC-UV system.

Analytical Parameters



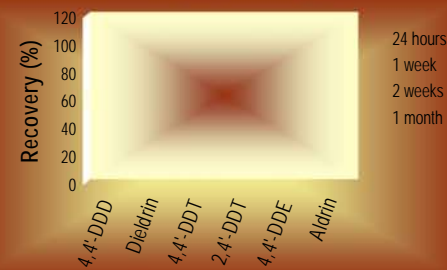
Chromatogram of an MAME-SPME extract of the mixture of six organochlorine pesticides

Pesticides	Recovery (%)	R.S.D. (n=6)	Detection Limit ($\text{ng}\cdot\text{g}^{-1}$)
4,4'-DDD	104.5	2.4	44
Dieldrin	91.1	8.4	104
4,4'-DDT	108.3	3.8	40
2,4'-DDT	98.1	2.7	84
4,4'-DDE	105.7	4.3	28
Aldrin	98.9	10.1	136

Applications to different mud samples

Recoveries (%) obtained after MAME-SPME-HPLC-UV procedure for the pesticides in ten different mud samples.

Mud samples	4,4'-DDD	Dieldrin	4,4'-DDT	2,4'-DDT	4,4'-DDE	Aldrin
Maspalomas	101.1	79.7	96.1	83.91	100.1	105.6
Las Palmas	105.1	99.5	109.9	103.9	117.7	93.3
Hoya Pozuelo	104.6	91.1	108.3	98.1	105.8	98.9
La Aldea	100.0	89.8	109.4	94.8	108.9	84.4
Sardina	87.4	81.9	89.9	82.2	87.9	83.3
Taurito	105.6	89.9	100.1	95.0	110.5	95.2
Agate	93.8	102.4	95.2	83.9	96.1	82.3
Jinamar	106.2	93.8	103.6	93.7	102.0	91.5
Salinetas	101.6	94.6	102.8	96.3	104.8	93.2
San Felipe	95.2	89.4	94.3	85.8	100.5	81.8



Recoveries of the organochlorine pesticides in a mud samples from Hoya Pozuelo for 24 hours, 1 week, 2 weeks and 1 month

CONCLUSIONS

Coupling MAME with SPME (an extraction-preconcentration technique) represents a viable clean-up procedure of the extract and it lets decrease the detection limit of the method.

The proposed method has been applied to different types and origin of mud samples, at different spiked times and to a certified soil with satisfactory results.

References

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