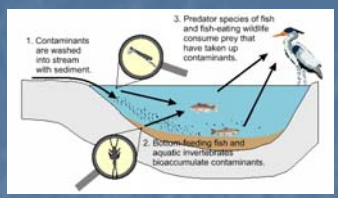
COUPLING MICROWAVE ASSISTED MICELLAR EXTRACTION AND SPME FOR THE DETERMINATION OF PESTICIDES IN SOILS

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

A non-ionic surfactant solution with microwave assisted extraction has been used for the extraction of several pesticides from agricultural soils and coupled after the extraction with SPME.

Organochlorine pesticides are presented in soils in spite of being prohibited since more than two decades due to their high persistence in the environment. As a consequence the pesticides are presented too in the whole food chain.



SOIL CHARACTERISTICS

	Culture Type	pH	Organic Matter (%)	% Clay
Valleseco Type I	Potatoes	*4,84*	*4,4*	*32,06*
Valleseco Type II	Potatoes	*3,91*	*6,2*	*47,29*
Tafira Type III	Garden	*8,3*	*4,8*	*23,8*
Sta Brígida Type IV	Pine Forest	*5,9*	*3,9*	*12,8*

EXPERIMENTAL

Pesticides Mix

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

A 2 g of soil sample was spiked with the pesticides mix with a final concentration of 300 ng·g^{-1} (DDTs) and 500 ng·g^{-1} (dieldrin). For the extraction was added to the soils a micellar solution of POLE (polyoxyethylene 10 lauryl ether).

Optimized Variables for MAME

For the variables optimization was used a multiparametric analysis.

Microwave's Power and Time

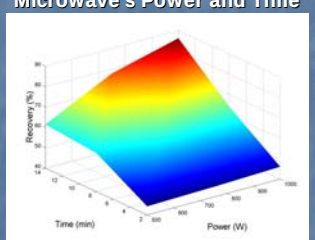
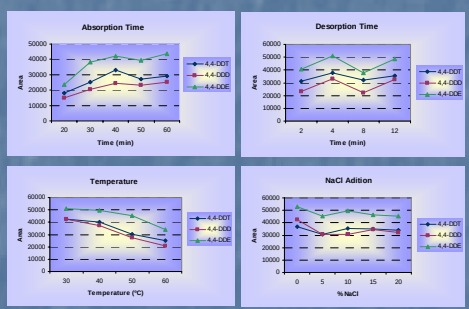


Figure 1. Response Surface Diagram where is represented the recoveryof the compound 4,4'-DDT versus Power and Time

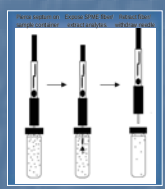
Optimum Conditions	
Surfactant Volume	8 ml
Surfactant Concentration	5 %
Power	1000 W
Time	14 min

EXTRACTION SPME WITH POLE

Optimized Variables for SPME



As a second step, a PDMS/DVB fiber was introduced in a solution with 2 ml of POLE with pesticides extract plus 2 ml of water.



Optimum Conditions	
Absorption Time	40 min
Desorption Time	4 min
Temperature	30°C
NaCl Adition	0 %

RESULTS AND DISCUSSION

Pesticides	R.S.D. (n=6)	Detection Limit (ng·g^{-1})
4,4'-DDD	7.43	17
Dieldrin	5.51	24
4,4'-DDT	7.28	18
2,4'-DDT	8.42	21
4,4'-DDE	7.94	14

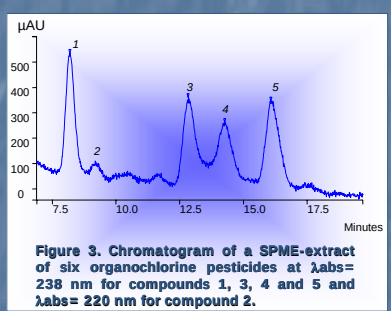


Figure 3. Chromatogram of a SPME-extract of six organochlorine pesticides at λabs= 238 nm for compounds 1, 3, 4 and 5 and λabs= 220 nm for compound 2.

ANALYTICAL APPLICATIONS

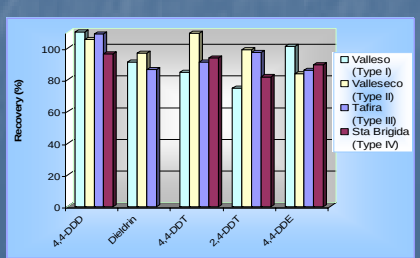


Figure 4. Recoveries of the mix of 5 organochlorine pesticides from 4 differents types soils samples

CONCLUSIONS

Microwave assisted extraction of pesticides using surfactant solutions is a procedure with several advantages: is an efficient method, less time consuming, green method, low cost and their compatibility with mobile phase used in HPLC.

Coupling this method with SPME extraction represents a viable clean-up procedure of the extract and it lets decrease the detection limit of the method.

The method has been applied to four different types of soils at different spiked times and to a certified soil with satisfactory results.

References

1. M. Maroni, C. Colosio, A. Ferioli, A. Fait. *Toxicology* **143** (2000) 5-8
2. H.M. Shivaramaiah, I.O.A. Odeh, I.V. Kennedy, J.H.Skerritt, *Journal of Agricultural and Food Chemistry,* **50** (2002), 5360-5367
3. Jacob de Boer, Robin J. Law. *J. of Chromatography A* **1000** (2003) 223-251
4. Z. Sosa Ferrera, C. Padrón Sanz, C. Mahugo Santana, J.J. Santana Rodríguez, *Trends in Analytical Chemistry,* **23** (7) (2004) 469-479.
5. V. Pino, J.H. Ayala, A.M. Afonso, V. González, *Analytica Chimica Acta* 477 (2003) 81-91
6. S. Millán, M.C. Sanpedro, N. Unceta, M.A. Goicolea, E. Rodríguez, R.J. Barrio, *Journal of Chromatography A,* **995** (2003) 135-142

ACKNOWLEDGEMENTS
This work was supported by funds provided by Ministry of Science and Technology (Spain) Research Proyect Nº PPQ 2002/04863