

INTRODUCTION

A mix of non-ionic surfactant solutions coupled with microwave assisted extraction has been used for the extraction of several pesticides from agricultural soils.

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	Matter Organic (%)	% clay
<i>Valleseco Type I</i>	Potatoes	3,91	6,21	47,29
<i>San Roque Type II</i>	Bananas	7,86	12,54	14,6
<i>Tafira Type III</i>	Garden	8,3	4,8	23,8
<i>Sta Brígida Type IV</i>	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
- 6.- aldrin

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 and aldrin). For the extraction was added to the soils a micellar solution of a mix of 2 non-ionic surfactants (70% Polyoxyethylene 10 lauryl ether and 30% Polyoxyethylene 10 tridecyl ether).

Optimized Variables

For the variables optimization was used a multiparametric analysis.

Surfactant Concentration

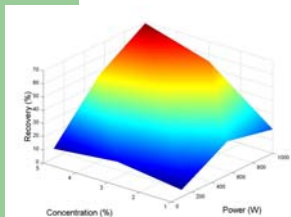


Figure 1. Response Surface Diagram where is represented the recovery of the compound 4,4'-DDT versus Power and Surfactant Concentration

Microwave's Power and Time

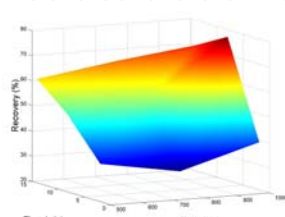


Figure 2. Response Surface Diagram where is represented the recovery of the compound 4,4'-DDT versus Power and Time

RESULTS AND DISCUSSION

Optimum Conditions

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

Results	Mean Recovery (%)	R.S.D. (n=6)	Detection Limit (ng·g ⁻¹)
4,4'-DDD	71.52	6.07	25
Dieldrin	67.71	4.10	173
4,4'-DDT	66.02	7.22	51
2,4'-DDT	55.57	8.78	54
4,4'-DDE	64.37	6.41	23
Aldrin	49.88	8.55	149

ANALYTICAL APPLICATIONS

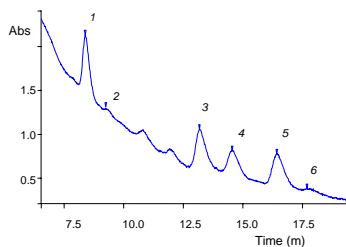


Figure 3. Chromatogram of an extract of six organochlorine pesticides at $\lambda_{obs} = 238$ nm for compounds 1, 3, 4 and 5 and $\lambda_{obs} = 220$ nm for compounds 2 and 6.

Comp.	Certif. Concen.	S.D.	Predict. Interval	Obtained Concn.
4,4'-DDD	1.531	0.476	0.49-2.562	1.28 ± 0.010
Dieldrin	1.863	0.655	0.44-3.288	1.79 ± 0.040
4,4'-DDT	1.060	0.275	0.63-2.407	0.79 ± 0.019
4,4'-DDE	1.520	0.410	0.47-1.655	1.11 ± 0.040

Table 1. Results of the extraction of a certified soil. Concentrations expressed in $\mu\text{g}\cdot\text{g}^{-1}$

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.

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

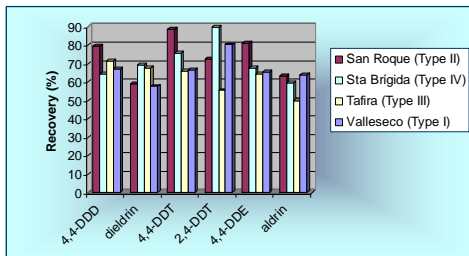


Figure 4. Recoveries of the mix of 6 organochlorine pesticides from 4 different types fresh samples

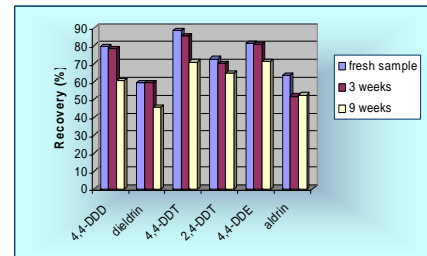


Figure 5. Recoveries of the 6 pesticides from San Roque soil for a 24 hours (fresh sample), 3 weeks and 9 weeks spiked soil under the optimum conditions

References

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