Optimization of desorption conditions of poly-dimethylsiloxane rods for the determination of pesticides in water samples

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Introduction

The passive Stir Bar Sorptive Extraction (SBSE) is developed in the laboratory as a passive sampling technique for the determination of pesticides in surface waters. The SBSE stir bars, coated with an absorption phase in polydimethylsiloxane (PDMS), are immersed in the water and accumulate the pesticides in situ. The SBSE stir bars are then brought back to the laboratory for liquid desorption and chemical analysis by LC-MS/MS. These SBSE stir bars are expensive and could be damaged when used in the river. Therefore, small PDMS rods were developed in the laboratory to replace the SBSE stir bars. This material is an organic polymer and is used to absorb hydrophobic contaminants.
Previous work in the laboratory obtained encouraging results for the extraction of pesticides by these PDMS rods. However, the amounts of pesticides adsorbed by the PDMS rods were lower than those found on the SBSE stir bars. It is possible that the desorption protocol, optimized for SBSE stir bars, was not adapted to PDMS rods. One objective of my internship was to increase the quantities of pesticides found on PDMS rods by optimizing the desorption conditions.

Experimental conditions

In order to assess the absorption capacity of the PDMS rods, extractions of water samples were made in the laboratory. The water samples were spiked with a mixture of seven pesticides, at concentrations ranging from 0,4 µg/L to 500 µg/L. The extraction lasted three hours with magnetic stirring (at 800 rpm),
According to the SBSE method, PDMS rods were desorbed in 200 µL of acetonitrile / methanol 50:50 (v:v) by sonication for 15 min.
An experimental design was used to assess the impact of desorption conditions on the pesticide quantities recovered from the PDMS rods. This plan, with two factors at two levels (full factorial), was replicated twice. 12 experiments were performed, the factors were the volume of the solvent and the desorption time, and the response was the mass of pesticide determined. The data were processed using the software Statgraphics®. (a)

Results and discussion

The reliability of the calculation models (R^2) varied between 74% and 89%, except for spiroxamine with R^2 = 11. Some pesticides were more affected than others by the desorption conditions. For example, for the fenitrothion the effect of solvent volume was not significant and negative, whereas for procymidone, it was significant and negative.
Generally, the desorption time had a significant and positive effect and the volume had an insignificant and negative effect on the quantities of absorbed pesticides.

Conclusion

The analysis of multi-response experimental design determined the optimal conditions for the desorption of the pesticides studied (b). That is to say erefore, the desorption of PDMS rods must be made with a low volume (200µL) and for a long time (45min).

Experimental design (a) and multi-response surface depending on volume and time of desorption (b).

(a).

Times (min)	Volume (µL)	Quantity of FNT absorb(ng)	Quantity of DFF absorb(ng)	Quantity of DFF absorb(ng)	Quantity of DCA absorb(ng)	Quantity of SPX absorb(ng)	Quantity of PCM absorb(ng)
45.0	500.0	350	130	142	7	5	43
45.0	200.0	405	123	162	8	6	66
15.0	200.0	350	110	149	7	8	62
15.0	500.0	297	99	120	5	5	36
45.0	500.0	435	116	166	8	7	49
45.0	200.0	365	123	152	8	6	64
15.0	200.0	340	101	129	6	4	42
15.0	500.0	303	99	120	6	7	35
45.0	500.0	332	127	155	7	5	48
45.0	200.0	390	113	166	7	8	60
15.0	200.0	274	98	129	7	6	57
15.0	500.0	266	78	117	6	5	34

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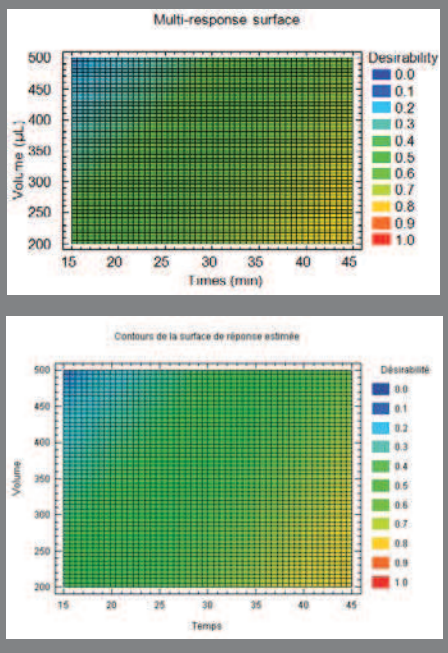
(b).



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