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12:27 **FO14 (P74)** ***Development of methodologies to evaluate odour retention capacity in textiles***

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12:31 **FO15 (P75)** ***Rapid determination of some of the most used pesticides in Northeast Portugal as emerging contaminants in rivers by SPME/GC-MS.***

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12:35 Closing Ceremony & "Best Presentation" Awards

13:15 Farewell Cocktail

FO15 (P75) Rapid determination of some of the most used pesticides in Northeast Portugal as emerging contaminants in rivers by SPME/GC-MS

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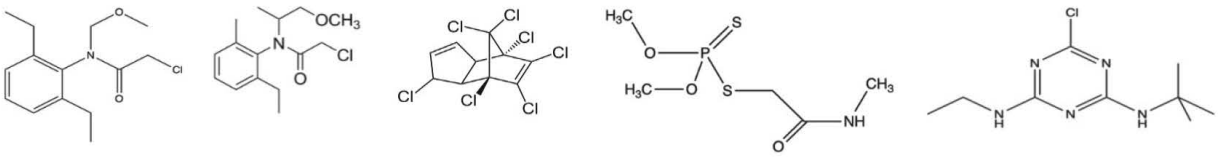
In this communication the development of an analytical methodology is presented for the monitoring of five emerging pollutants, namely, alachlor, metolachlor, heptachlor, dimethoate, and terbuthylazine, represented in Figure 1. These compounds are among the most used pesticides in the northeast region of Portugal and Tunisia[1,2]. The complete experimental methodology is optimized based on the simultaneous extraction and concentration of all five pesticides from aqueous matrices, by means of solid-phase micro-extraction (SPME) followed by detection and quantification using gas chromatography with mass spectrometry (GC-MS).



alachlor metolachlor heptachlor dimethoate terbuthylazine

Fig. 1 Chemical structure of the five pesticides.

The optimization of the extraction step is performed using a polydimethylsiloxane-divinylbenzene (PDMS-DVB) coated SPME fiber by direct immersion (DI-SPME) in the aqueous sample. Experimental conditions, such as extraction temperature and time, pH value, salt addition, and desorption time and temperature in the GC injector port were studied. The optimum value for each one of these parameters was selected based on the maximum total area value obtained in MS detector, using Full Scan Mode, for the mixture of five pesticides. The extraction optimized conditions were achieved by immersion of a PDMS-DVB fiber in the sample mixture with 10% NaCl, a pH value of 2, at 60 °C for 80 min. Desorption of the compounds from the fiber is done in the GC port at 250 °C during 4 min.

The Shimadzu GC-MS equipment, model QP2020, operating conditions were also studied, and the main separation and detection parameters were selected. Samples were analyzed using a Rxi-5MS Low Bleed capillary column from Restek and the following oven temperature program: initial temperature of 120 °C (held for 2 min), increased by 15 °C.min^{-1} to 190 °C (held for 4 min) and, finally, increased by 10 °C.min^{-1} to 227 °C and held for 1 min. The MS instrument operating in the Electron Ionization mode (EI) was used for a full scan. The acquisition was performed in the range of 35–450 (m/z). The ion source temperature was 200 °C and the interface temperature was 270 °C.

The identification and quantification were carried out using calibration curves obtained from the extraction of a standard mixture of the five selected pesticides for at least six concentration levels, in the same experimental conditions used for the real samples. Detection limits ranged from 4.2 to 6.6 ng/mL. For pesticides with low values of K_{OW}, like dimethoate, the use of a fiber of relatively non-polar nature would be more favourable. The developed experimental methodology was implemented by the analysis of different samples collected from the surface water of three rivers from Bragança, namely, Fervença, Sabor and Onor. All three rivers showed different types and levels of contamination

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