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A Multi-Family Offline SPE LC-MS/MS Analytical Method for Anionic, Cationic and Non-ionic Surfactants in Surface Water

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Abstract : Due to their production at high tonnages and their extensive use, surfactants are contaminants among those determined at the highest concentrations in wastewater. However, analytical methods and data regarding their occurrence in river water are scarce and concern only a few families, mainly anionic surfactants. The objective of this study was to develop an analytical method to extract and analyze a wide variety of surfactants in a minimum of steps, with a sensitivity compatible with the detection of ultra-traces in surface waters. 27 substances, from 12 families of surfactants, anionic, cationic and non-ionic were selected for method optimization. Different retention mechanisms for the extraction by solid phase extraction (SPE) were tested and compared in order to improve their detection by liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS). The best results were finally obtained with a C18 grafted silica LC column and a polymer cartridge with hydrophilic lipophilic balance (HLB), and the method developed allows the extraction of the three types of surfactants with satisfactory recoveries. The final analytical method comprised only one extraction and two LC injections. It was validated and applied for the quantification of surfactants in 36 river samples. The method's limits of quantification (LQ), intra- and inter-day precision and accuracy were evaluated, and good performances were obtained for the 27 substances. As these compounds have many areas of application, contaminations of instrument and method blanks were observed and considered for the determination of LQ. Nevertheless, with LQ between 15 and 485 ng/L, and accuracy of over 80%, this method was suitable for monitoring surfactants in surface waters. Application on French river samples revealed the presence of anionic, cationic and non-ionic surfactants with median concentrations ranging from 24 ng/L for octylphenol ethoxylates (OPEO) to 4.6 µg/L for linear alkylbenzenesulfonates (LAS). The analytical method developed in this work will therefore be useful for future monitoring of surfactants in waters. Moreover, this method, which shows good performances for anionic, non-ionic and cationic surfactants, may be easily adapted to other surfactants.

Keywords : anionic surfactant, cationic surfactant, LC-MS/MS, non-ionic surfactant, SPE, surface water
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