Determination of nonylphenol ethoxylates in wastewater samples with SPME/GC-MS

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ABSTRACT

Nonylphenol ethoxylates (NPnEOs) are non-ionic surfactants that have been widely used in the cleaning industry over the past decades. At the same time, they pose a major environmental threat due to their toxicity and estrogenic activity. During the wastewater treatment process, they degrade and form more persistent and toxic metabolites that are detected mainly in the plant effluent. In the present work headspace solid-phase microextraction (SPME) with in-sample derivatization was used with dimethyl sulfate as methylating agent in order to quantify nonylphenol ethoxylates in wastewater samples. The selection of the appropriate fiber coating material and the optimization of the desorption conditions were necessary in order to enhance the SPME performance. The analytes were successfully isolated using 65 μm PDMS/DVB fiber and they were separated by means of gas chromatography with mass spectrometry detector (GC/MS) with the injector at 270°C. The method was linear over a wide range of concentrations (0.05–480 μg/l depending on the compound). The limits of detection for the compounds of interest were found to be in the microgram per liter range. For nonylphenol the lower limit of detection was obtained (0.02 μg/l), while for NP1EO, NP2EO and NP1EC limits of detection were 0.61, 3.2 and 0.29 μg/l respectively. Samples from the municipal wastewater treatment plant of Chania (Crete, Greece) were collected from different stages of treatment and were analyzed for the presence of nonylphenol ethoxylates. Nonylphenol, which is considered to be the most abundant metabolite, was detected in all samples at low concentrations.

Keywords: SPME; Derivatization; Nonylphenol ethoxylates; Surfactants

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