TECHNICAL COMMUNICATIONS

Optimization of Solid-Phase Microextraction for the Gas Chromatography/Mass Spectrometry Analysis of Persistent Organic Pollutants

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In this work, solid-phase microextraction (SPME) has been applied as an alternative for the selective extraction of 3 polybrominated diphenyl ethers (PBDEs), 2,2′,4,4′-tetrabromodiphenyl ether (PBDE-47); 2,2′,4,4′,5-pentabromodiphenyl ether (PBDE-99); and 2,2′,4,4′,6-pentabromodiphenyl ether (PBDE-100), and 2 alkylphenols, 4-*tert*-OP and 4-NP, prior to their analysis by gas chromatography/mass spectrometry (GC/MS). The advantages of this technique are mainly its simplicity, cost-effectiveness, and time-saving sample preparation, as well as being a solvent-free technique. With the aim of optimizing the conditions for an efficient extraction of the studied compounds, different fiber coatings and the main parameters affecting the extraction procedure have been evaluated. The results obtained showed a good linearity in the range of concentrations investigated, and adequate relative standard deviation values were found according to the range accepted for SPME. Recovery values were in the range of 78–108%, and good detection and quantitation limits at ppt levels were obtained for both methods, allowing the determination of the selected compounds in samples at trace levels. The results obtained clearly show the potential of SPME for efficient concentration of the target compounds and also demonstrate the reliability of this extraction technique for their GC/MS analysis.

The increase of pollutants due to the growth of technology and chemical industries is an important concern that is recognized worldwide. Persistent organic pollutants (POPs) are very stable compounds that remain unaltered in the environment and bioaccumulate along the food chain, representing a potential hazard for both humans and wildlife. In this work, we focused on 2 groups of POPs: polybrominated diphenyl ethers (PBDEs) and alkylphenols.

PBDEs are ubiquitous environmental pollutants used as flame retardants in many common materials such as plastics, textiles, or electronic devices (1). These brominated flame retardants are additives and are not chemically bound to the material and thus can be released from products and enter the environment (2). Due to their persistence and bioaccumulation potential, they are found in the environment and consequently in humans (3). At present, the toxicity of PBDEs in humans is not fully understood. Nevertheless, recent studies show concerns regarding to their estrogenicity (4). Furthermore, it has been reported that brominated dibenzo-*p*-dioxins (PBDDs) and brominated dibenzofurans (PBDFs), which have toxicological effects similar to chlorinated dioxins, (5) are formed via thermolysis (e.g., during manufacture of plastics) of some brominated flame retardants.

Alkylphenols are degradation products of their respective alkylphenol ethoxylates (APEs), which are nonionic surfactants with a wide variety of industrial, agricultural, and household applications (6–8). APEs are biodegraded under anaerobic conditions by removal of ethoxy groups, with the formation and further accumulation of alkylphenols (9–11). Nonylphenols and *tert*-octylphenols, the main alkylphenols produced by this process, are more toxic and bioaccumulative in aquatic organisms than in parent compounds (11–14), entering into the aquatic environment via industrial and municipal wastewater effluents (15–18). These compounds have been considered as endocrine disruptors because of their ability to mimic natural hormones by interacting with the estrogen receptor and causing a variety of adverse effects in both humans and wildlife (19–25).

The efficiency of the analysis of organic contaminants, such as the ones described in this work, is always compromised by the efficiency of the sample pretreatment, the

Received July 9, 2003. Accepted by JS January 6, 2004.
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