Determination of polybrominated diphenyl ethers in sewage sludge by hollow-fiber microporous membrane liquid-liquid extraction and gas chromatography

Polybrominated diphenyl ethers (PBDEs) are a group of persistent organic pollutants, which are used as flame retardants in plastics in consumer products such as computers and TV sets. The rapid increase in PBDE levels in human and biota samples as well as their adverse health effects on animals have raised concerns over potential health effects of PBDEs in human and many studies have been conducted to determine these persistent pollutants in environmental samples such as sewage sludge which is a major source of contamination by PBDEs.

Conventional methods for extraction of PBDEs from sewage sludge consume a lot of time and extraction solvent, which leads to a significant expense. An alternative technique that can be used for extraction of contamination from different types of matrices is hollow fiber microporous membrane liquid-liquid extraction (HF-MMLLE). The advantages of this technique are simplicity without requiring special expensive instrument and reduction in solvent consumption that make it economic for analytical procedure.

Depending on the analyte hydrophobicity, a two or three phase extraction system can be applied using hollow fiber. In two-phase (HF-MMLLE) the organic solvent filled in the pores and inside (lumen) the hollow fiber act as acceptor phase and is used to extract more hydrophobic analytes from an aqueous sample which is donor phase. The three phase system (aq/org/aq) involves extraction of polar compounds from an aqueous sample matrix, through an organic phase in the pores of the hollow fiber into a new aqueous phase inside the hollow fiber.

In this study hollow fibers with 5.5 cm effective length were filled with small amount (µl ranges) of n-undecane as organic solvent and then they were immersed in 100 ml of aqueous samples containing 1 gr of wet sludge which were added (spiked) with the PBDEs at ng/l level. Extracts were analyzed by gas chromatography with electron capture detector. Parameters such as pH of donor phase, salt concentration, stirring speed, and extraction time were optimized based on extraction efficiency, Ee that is the ratio of the number of moles of analyte in the acceptor phase to that in the donor phase.

The optimized factors (pH 6.6, 10% NaCl, stirring speed 10 and extraction time of 1 hour) were then applied to the determination of PBDEs in sewage sludge from Källby sewage treatment plant in Lund. The optimized method allowed detection of 1.8, 0.11 and 0.38 µg/kg of BDE_47, 99 and 183 respectively from dried sludge. The findings were compatible with the results from recent research on PBDEs level in Sweden although PBDE 209 was not detected. Limit of detection, photodegradation or/and biodegradation of BDE-209 during experiment or treatment in sewage treatment plant are major reasons.

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Determination of polybrominated diphenyl ethers at trace levels in sewage sludge by hollow-fiber microporous membrane liquid-liquid extraction and gas chromatography

Polybrominated diphenyl ethers (PBDEs) are the most common brominated flame retardants, used in plastics, resins and textiles. They can bioaccumulate in the environment and in biota. Due to concerns over potential health effects of these chemicals, environmental laboratories analyze thousands of samples annually. In order to decrease costs for laboratories of environmental analysis, the idea of developing a simple method that use small amount of organic solvent for PBDEs in complex matrices such as sludge, as a major source of PBDEs, was proposed.

In the present work a two-phase hollow-fiber (HF) liquid-phase microextraction (LPME) method followed by gas chromatography was developed for quantification of 8 major polybrominated diphenyl ethers at trace level in sewage sludge. In this method the porous polypropylene hollow fibers filled with a very small volume (micro liter ranges) of organic solvent, were immersed in aqueous samples of wet sludge which were spiked with the PBDEs at ng/l level. Parameters such as pH of donor phase, salt concentration, stirring speed, and extraction time were optimized and the optimum extraction conditions were then applied to determination of PBDEs in sewage sludge from Källby sewage treatment plant in Lund.

The optimized method allowed detection of 1.8, 0.11 and 0.38 µg/kg of BDE_47, 99 and 183 respectively from dried sludge. The findings were compatible with the results from recent research on PBDEs level in sewage sludge samples from Sweden although PBDE 209, which was expected to have the highest level, was not detected. Limit of detection, photodegradation or/biodegradation of BDE-209 during treatment or experiment are major reasons.

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