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Research Article

Polyvinylimidazole/sol–gel composite as a novel solid-phase microextraction coating for the determination of halogenated benzenes from aqueous solutions

A polyvinylimidazole/sol–gel composite is proposed as a novel solid-phase microextraction fiber to extract five halobenzenes from the headspace of aqueous solutions in combination with gas chromatography with mass spectrometry. The prepared fiber was characterized by scanning electron microscopy and Fourier transform infrared spectroscopy. The obtained results showed that porous polyvinylimidazole/sol–gel composite was chemically deposited on fused silica fiber. The effect of important extraction parameters including extraction temperature, extraction time, and salt content were investigated. The optimum conditions were as follows: extraction temperature 25°C, extraction time 20 min, and salt concentration 30 w/v%. Detection limits and relative standard deviations of the developed method for halogenated benzenes were below 0.1 pg/mL and 15%, respectively. Repeatability of the proposed method, explained by relative standard deviation, varied between 5.48 and 9.15% ($n = 5$). The limits of detection (S/N = 3) ranged between 0.01 and 0.10 ng/L using gas chromatography with mass spectrometry with selected ion monitoring mode. For real sample analysis, three types of water samples with different matrices (ground, surface, and tap water) were studied. The optimized procedure was applied to extraction and method validation of halogenated benzenes in spiked water samples.

Keywords: Composite materials / Halogenated benzenes / Polyvinylimidazole / Sol–gel / Solid-phase microextraction
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1 Introduction

SPME developed in 1990 [1] is an adsorption/desorption technique combining sample extraction and concentration, which eliminates the need for solvents or complicated apparatus for concentrating volatile or nonvolatile compounds in liquid samples or headspace. In recent years, the SPME technique has been widely used in different fields including the environment, food, natural products, pharmaceuticals, biology, toxicology, and forensics [2]. SPME is based on distribution of analytes in a fiber coated with a stationary phase. It is believed that further developments in SPME, as a highly efficient extraction technique, will greatly depend on new breakthroughs in the development of new coating materials and new techniques of sorbent deposition for its fibers [3]. To date, several types of SPME fibers have been commercially available. Polydimethylsiloxane (PDMS) is a nonpolar phase that extracts nonpolar analytes, such as BTEX (benzene, toluene, ethylbenzene, and *o*-xylene) [4–6] and polycyclic aromatic hydrocarbons [7] as well. The polyacrylate (PA) fiber has a great affinity for polar compounds [8, 9]. PDMS and PA are liquid sorbents and extract analytes by way of absorption. Mixed-phase coatings, such as PDMS/divinylbenzene (DVB), Carboxen/PDMS, Carbowax/DVB, and Carbowax/template resin, extract analytes through adsorption because the fibers

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Abbreviations: AIBN, 2,2′-azobis(2-methylpropionitrile); **BBz**, bromobenzene; **BzCl**, benzyl chloride; **CBz**, chlorobenzene; **2CBzTF**, 2-chlorobenzotrifluoride; **1,2DCBz**, 1,2–dichlorobenzene; **DVB**, divinylbenzene; **EPA**, environmental protection agency; **HBz**, halobenzene; **HS**, headspace; **MSMA**, 3-(methacryloxy) propyl trimethoxysilane; **OH-TSO**, poly(dimethylsiloxane)hydroxy terminated; **PDMS**, polydimethylsiloxane; **PMHS**, poly (methylhydrosiloxane); **PVI**, polyvinylimidazole; **RSD**, relative standard deviation; **TFA**, trifluoroacetic; **THF**, tetrahydrofuran; **TMOS**, tetramethylorthosilicate; **VI**, vinylimidazole

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