Accepted Manuscript

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PII: S0021-9673(18)30011-6
DOI: https://doi.org/10.1016/j.chroma.2018.01.011
Reference: CHROMA 359137

To appear in: *Journal of Chromatography A*

Received date: 29-9-2017
Revised date: 2-1-2018
Accepted date: 3-1-2018

Please cite this article as: Asma'a Al-Rifai, Ahmad Aqel, Lamya Al Wahibi, Zeid A.ALOthman, Ahmed-Yacine Badjah-Hadj-Ahmed, Carbon nanotube-based benzyl polymethacrylate composite monolith as a solid phase extraction adsorbent and a stationary phase material for simultaneous extraction and analysis of polycyclic aromatic hydrocarbon in water, Journal of Chromatography A https://doi.org/10.1016/j.chroma.2018.01.011

Carbon nanotube-based benzyl polymethacrylate composite monolith as a solid phase extraction adsorbent and a stationary phase material for simultaneous extraction and analysis of polycyclic aromatic hydrocarbon in water

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Highlights

- Monolithic SPE and stationary phase with poly(benzyl methacrylate) and CNTs
- Extraction then separation of ten PAHs by nano-LC analysis
- Incorporation of CNTs to monolith improved the extraction performance up to 78%
- The presence of CNTs enhanced capillary column efficiency to over 41,000 plates/m

Abstract

A composite of multi-walled carbon nanotubes incorporated into a benzyl methacrylate-*co*-ethylene dimethacrylate porous monolith was prepared, characterized and used as solid phase adsorbent and as stationary phase for simultaneous extraction and separation of ten polycyclic aromatic hydrocarbons, followed by nano-liquid chromatography analysis. The extraction and chromatographic parameters were optimized with regard to the extraction efficiency and the quality of chromatographic analytes separation. Under the optimized conditions, all PAHs were separated in 13 min with suitable resolution values (Rs = 1.74–3.98). Addition of a small amount of carbon nanotubes (0.1% with respect to monomers) to the polymerization mixture increased the efficiency for the separation column to over 41,700 plates m^{-1} for chrysene at flow rate of 0.5 µL min^{-1}. The method showed a wide linear range (1–500 µg L^{-1} with R^2 more than 0.9938), acceptable extraction repeatability (RSDs<6.4%, n=3) and reproducibility (RSDs<12.6%, five parallel-made solid phase extraction cartridges) and satisfactory detection limits (0.02–0.22 µg L^{-1}). Finally, the proposed method was successfully applied to the detection of polycyclic aromatic hydrocarbons in environmental water samples. After a simple extraction procedure with preconcentration factor equal to 100, the average recovery values in ultra-pure, tap and sea water samples were found to be in the range 81.3–95.4% with %RSD less than 6.4. Again, the presence of carbon nanotubes (0.3% relatively to monomers) in native polymer enhanced the extraction performance for the solid phase adsorbent up to 78.4%. The application of the monoliths modified with CNTs in extraction and nano-scale liquid chromatography for analysis of environmental samples offered several advantages; it demonstrated an acceptable precision, low detection limits, good reproducibility, satisfying recoveries and wide dynamic linear ranges.

Keywords PAH pollutants; Polymer-based monolith; Benzyl methacrylate; Carbon nanotubes; Solid-phase extraction; Nano-liquid chromatography