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Short Communication

Analysis of endocrine disruptors in water using magnetic nanoparticles in the extraction method

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ABSTRACT

The use of nanoparticles (NPs) of magnetite (Fe3O4) in the extraction process have been described as an interesting tool for the analysis of pollutants in liquid samples. These NPs have been successfully applied in the extraction by magnetic solid phase extraction (mSPE) or assisted dispersive liquid-liquid microextraction (DLLME) of endocrine disruptors from water samples. The sorbent is added directly to the solution, with or without with a solvent disperser, and the NPs containing the analytes are isolated by placing a magnet on the wall of the flask and discarding the solution. Finally, the target compounds can be eluted from the sorbent with a low quantity of an adequate organic solvent to be analyzed. Endocrine-disrupting compounds (EDCs) are naturally occurring or man-made substances that alter functions of the endocrine system and consequently cause adverse health effects in an intact organism, its progeny, or (sub) populations1. A wide range of substances, present in a high variety of everyday products, are thought to cause endocrine disruption at very low concentrations2. Therefore, the main objective was to develop quick, cheap and efficient methods for the simultaneous analysis of EDCs using Fe3O4 NPs. The applicability of mSPE and DLLME/D-μ-SPE (see Figure 1) for the extraction of five EDCs using Fe3O4 NPs with different coating was evaluated, and the best results were obtained by DLLME/D-μ-SPE using uncoated and Ol-coated NPs3. Very good results were obtained with the mSPE methods developed for the analysis of three steroid hormones4 and macrolides5 in water samples, respectively. The methods demonstrate their applicability for the determination of EDCs in different water samples with detection limits comparable to those reported as satisfactory for monitoring EDCs in environmental and biological samples.

Introduction:

A rapid extraction procedure is presented for the determination of five endocrine-disrupting compounds, estrone, ethinylestradiol, bisphenol A, triclosan, and 2-ethylhexylsalicylate, in water samples. The analysis involves a two-step extraction procedure that combines dispersive liquid-liquid microextraction (DLLME) with dispersive micro-solid phase extraction (D-μ-SPE), using magnetic nanoparticles, followed by in situ derivatization in the injection port of a gas chromatograph coupled to triple quadrupole mass spectrometry. The use of uncoated or oleate-coated Fe3O4 nanoparticles as sorbent in the extraction process was evaluated and compared. The main parameters involved in the extraction process were optimized applying experimental designs. Uncoated Fe3O4 nanoparticles were selected in order to simplify and make more cost-effective the procedure. DLLME was carried out at pH 3, during 2 min, followed by the addition of the nanoparticles for D-μ-SPE employing 1 min in the extraction. Analysis of spiked water samples of different sources gave satisfactory recovery results for all the compounds with detection limits ranging from 7 to 180 ng l-1. Finally, the procedure was applied in tap, well, and river water. Graphical abstract Diagram of the extraction method using magnetic nanoparticles (MNPs). In the present study, chitosan functionalized Fe3O4 magnetic microspheres coated with polyaniline were synthesized for the first time. The chitosan-functionalized magnetic microspheres were synthesized by a co-precipitation method, and then aniline was polymerized on the magnetic core. The obtained microspheres were spherical core-shell structure with uniform size at about 100nm with 20-30nm diameter core. The microspheres had a high saturation magnetization of 32emu g(-)(1), which was sufficient for magnetic separation. The obtained magnetic microspheres were applied as magnetic adsorbents for the extraction of aromatic compounds via π-π interaction between polyaniline shell and aromatic compounds.

Conclusion:

In this study, double functionalised magnetic nanoparticles for extraction of bisphenol A in an aqueous phase were designed and prepared. In the preparation of DFMNPs, amide and pyridine groups were simultaneously introduced into the surface of magnetic nanoparticles. A new dispersed solid-phase extraction method adopting DFMNPs as the adsorbents was developed for separating and enriching BPA from river water samples. This DSPE method showed fast magnetic response, high binding efficiency to target BPA, and short experimental time. The recovery of BPA in spiked river water was 94.4 % with the DSPE method, which was much higher than those with traditional solid-phase extraction methods. The high performance of DFMNPs on extraction of BPA from river water was attributed to the synergistic function of the amide and pyridine groups. The hydrophilic amide groups caused DFMNPs to disperse well in water, whereas the alkaline pyridine groups bound BPA effectively by ionic bonds. Our DSPE was particularly superior to conventional SPE in the pre-treatment of large-volume water samples as the time taken could be remarkably reduced.