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Rapid Magnetic Dispersive solid phase extraction to preconcentration/determination of Cd and Pb in aqueous samples

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A new magnetic dispersive solid phase extraction (MDSPE) method and graphite furnace atomic absorption spectrometry (GFAAS) have been combined for the analysis of Cd and Pb in environmental samples. For the preconcentration, a shell structured Fe_3O_4@graphene oxide nanospheres was synthetized and characterized. The material was suspended in the ionic liquid 1-n-butyl-3-metilimidazolium tetrafluoroborate [BMIM][BF_4], the obtained stable colloidal suspension is named ferrofluid. GO presents excellent adsorbent properties for organic species due to the presence of the electronic π system. For this reason, the organic ligand [1,5-bis-(2-dipyridyl) methylene] thiocarbonohydrazide (DPTH) was used in order to form organic complexes of Cd and Pb. Once the DPTH ligand has been added to sample, the ferrofluid was injected and finely dispersed in the sample solution in order to extract the formed chelates (Fig. 1). The complete adsorption of the chelates took place within few seconds then, the solid was separated from the solution with the aid of a strong magnet. Cd and Pb ions were desorbed from the material with 1 mL of acid nitric 5% solution and quantified by GFAAS. All experimental and instrumental variables were optimized.

The analytical performances of the optimized method were: EF (Enrichment factor): 200 with LODs (detection limit): 0.005 and 0.004 μg L^{-1} and LOQs (determination limit): 0.017 and 0.013 μg L^{-1}, for Cd and Pb, respectively. The reliability of the developed procedure was tested by relative standard deviation (% RSD), which was found to be < 5%. The accuracy of the proposed method was verified using certified reference materials (SLRS-5, SPS-SW2, and BCR-723) and by determining the analyte content in spiked aqueous samples. Sea waters and tap water samples collected from Málaga (Spain) were also analysed. The determined values were in good agreement with the certified values and the recoveries for the spiked samples were around 100% in all cases.

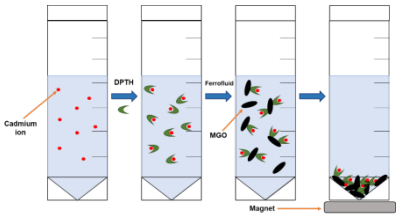

Fig. 1 Magnetic solid phase extraction process.

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