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Abstract

In the present study, a new cloud point extraction methodology based on the selective preconcentration and the extraction of stable lead in acidic conditions with 4',4"(5")-di-tert-butyldicyclohexano-18-crown-6 as a chelating agent was developed, optimized and validated. A mixture of Triton X-114 as non-ionic surfactant and CTAB as cationic surfactant was used to produce micellar structures that incorporate the chelating agent. Phase separation, induced by coacervation, was achieved by increasing the temperature of the system above the cloud point temperature. Pb extraction efficiency was maximized through an optimization process where the effect of each parameter (i.e. non-ionic and ionic surfactant concentrations, pH, chelating agent concentration and cloud point temperature) on the chemical recoveries of Pb was assessed. Under optimum experimental conditions, the method reaches recoveries greater than 67% for Pb in a variety of complex matrices. In order to facilitate the quantification of Pb by plasma based instrumentations, a back-extraction procedure using aqueous solution of ammonium citrate were performed on the surfactant rich phase in order to reduce the effects on sample introduction and non-spectral interferences. LOD and LOQ of 0.8 µg L^{-1} and 2.6 µg L^{-1}, respectively, were determined by ICP-OES for the complete procedure. Using the back-extraction approach, a preconcentration factor of 39 was achieved for an initial sample volume of 195 mL. The ruggedness of the methodology was validated by determining Pb concentration in various environmental and biological samples.

Graphical abstract