



Solid phase extraction of Pb(II), Cu(II), Cd(II) and Cr(III) with syringe technique using novel silica-supported *bis*(diazimine) ligands



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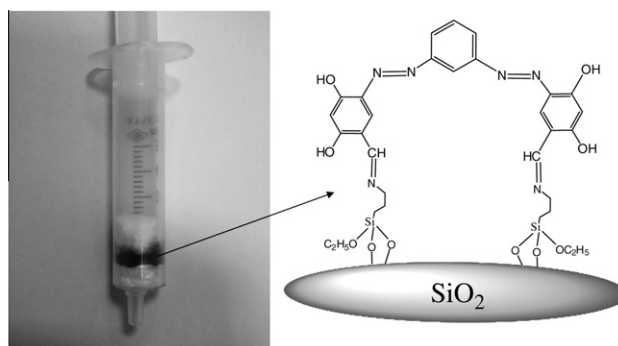
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HIGHLIGHTS

- Synthesis of novel *bis*(dialdehyde)s and silica-supported *bis*(diazimine) ligands.
- Characterization and electrochemical properties of the novel compounds.
- Multidentate *bis*(diazimine) ligands exhibit highly efficient solid phase extraction properties.

GRAPHICAL ABSTRACT



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ABSTRACT

Novel multidentate silica-supported *bis*(diazimine) ligands were synthesized and characterized by elemental analysis, solid phase NMR (²⁹Si and ¹³C CP MAS), FT-IR, TG/DTA, SEM and EDX techniques. In the synthesis, diazotized electrophiles of 1,4-diaminobenzene and 1,3-diaminobenzene coupled with 2,4-dihydroxybenzaldehyde to give *bis*(diazocarbonyl) compounds; (A₁: 5,5'-(1,4-phenylenebis(diazene-2,1-diyl))bis(2,4-dihydroxybenzaldehyde and A₂: 5,5'-1,3-phenylenebis(diazene-2,1-diyl))bis(2,4-dihydroxybenzaldehyde) which were chemically supported with amino-modified silica-gel (L1 and L2). The electrochemical properties of *bis*(diazocarbonyl) compounds (A1 and A2) were investigated by cyclic voltammetry. Optimum solid phase extraction parameters of the synthesized sorbents for syringe and batch techniques were determined by using ICP-OES. At optimum conditions, the recoveries of Pb(II), Cu(II), Cd(II) and Cr(III) were 95–99% ± 2–3%. All the multifunctional multidentate silica-supported ligands showed highly effective solid phase extraction properties.

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1. Introduction

The Schiff bases and diazo compounds are the areas of increasing interest due to the fact that they are very important intermediates in the synthesis of numerous organic and inorganic compounds. Metal complexes of Schiff bases or diazo compounds

have attracted considerable attention due to their properties which can be used for many different purposes. These ligands form stable complexes with metal ions and provide the opportunity to design new systems selective to specific metallic ions [1–3]. The multifunctional ligands having imine or diazo groups have considerably interest due to the fact that their metal complexes are relatively robust and chemically stable in severe catalytic reaction mediums [4,5]. Solid phase extraction and pre-concentration properties of chemically modified solid supported ligands can be determined for heavy metal ions in water samples by using different methods, such as, batch, syringe and column [6–10]. Many studies have been

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