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# Supported hydrophobic ionic liquid on nano-silica for adsorption of lead

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## ABSTRACT

The hydrophobic character incorporated into 1-methyl-3-octylimidazolium bis(trifluoromethylsulfonyl)imide [OMIM<sup>+</sup>Tf<sub>2</sub>N<sup>-</sup>] based on both the anion and cation natures was employed to develop a novel solid phase extraction medium based on physical adsorption approach. Two modified nanosilica sorbents were synthesized via direct immobilization of [OMIM<sup>+</sup>Tf<sub>2</sub>N<sup>-</sup>] on the surface of active nano-silica [NSi-OH] and amino nano-silica [NSi-NH<sub>2</sub>] for the formation of [NSi-OH-OMIM<sup>+</sup>Tf<sub>2</sub>N<sup>-</sup>] and [NSi-NH<sub>2</sub>-OMIM<sup>+</sup>Tf<sub>2</sub>N<sup>-</sup>], respectively. Surface characterization was confirmed by TGA, FT-IR and SEM. The surface coverage values were identified in the range of 1.223–1.272 mmol g<sup>-1</sup>. Modified sorbents were extensively studied and characterized by their high similarity and affinity for lead extraction without the need for chelating intermediate. Excellent sorption capacity values (0.5–0.9 and 0.8–1.3 mmol g<sup>-1</sup>) of lead were determined in pH 1–7. Other factors such as reaction contact time, sorbent dose, initial metal ion concentration and interfering ions were studied to spot more light on the sorption capability of loaded hydrophobic ionic liquid for application in the solid phase extraction mode. These sorbents were successfully implemented for removal and preconcentration of lead from various water samples via application of a multi-stage micro-column giving rise to percentage recovery values of lead in the range of 98.0–100.0 ± 3.0–4%.

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#### 1. Introduction

In recent years, ionic liquids (ILs) have been widely used and extremely found high interests in almost all application fields of chemistry such as organic and inorganic syntheses, catalysis, electrochemistry, chromatography, separation and extraction techniques as well as others owing to their unique and favorable characteristics compared to other conventional organic solvents and compounds. These expanding interests have led to a number of research reports devoted to study and evaluate physico-chemical properties, design of new families of ionic liquids, chemical engineering and wide range of arrangements in which ILs have been utilized in industrial developments [1,2]. Thus, these types of available ILs have been extended to cover some new families and generations with some incorporated and aimed properties. The manipulation of ionic liquids in various fields may be categorized and termed as task-specific compounds such as multifunctional ILs, deep eutectic solvents, protic ILs, polarizable ILs, amphiphil ILs, supported ILs, switchable polarity solvents, metal salts ILs, chiral ILs, and bio ILs [3].

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Applications of ILs in analytical chemistry are mainly focused on three major areas including (i) liquid-phase microextraction (LPME) methodologies, (ii) liquid-liquid extraction techniques (LLE) and (iii) modified solid phases in chromatographic applications. Liquid-phase microextraction techniques including singledrop microextraction (SDME) have been recently developed to overcome some of the limitations in manipulation of conventional liquid-liquid extraction techniques. SDME is characterized by several advantages including simplicity, significant reduction in the amount of organic solvent, cost-effectiveness, high sample throughput and combination of the extraction, preconcentration and sample introduction in one step. The applications of ionic liquids in single-drop microextraction techniques for metal ions separation from various systems were numerous as recently reported [4-10]. An improved single-drop microextraction procedure was developed and reported for the preconcentration of lead prior to determination by electrothermal atomic absorption spectrometry by the use of ionic liquid, 1-butyl-3-methylimidazolium hexafluorophosphate [C4MIM][PF6] [4]. An efficient single-drop microextraction procedure using a low-cost room temperature ionic liquid (RTIL), tetradecyl(trihexyl)phosphonium chloride for Pb determination at trace levels in real water samples was studied and discussed [5]. A new liquid-phase microextraction system was developed and described for the pre-concentration of lead via lead transfer into its complex using dithizone as a chelating agent followed by partition into the infinite ionic liquid drops

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