



# Impregnation of task-specific ionic liquid into a solid support for removal of neodymium and gadolinium ions from aqueous solution



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## ABSTRACT

Impregnation of trihexyl(tetradecyl)phosphonium bis(2,4,4-trimethylpentyl)phosphinate, as an ionic liquid, into silica was carried out for removal of neodymium and gadolinium ions from aqueous solution. Silica and the impregnated silica (Cyphos@silica) were characterized using FT-IR, SEM, XRD and TGA. Comparative sorption behavior of silica and Cyphos@silica for sorption of neodymium and gadolinium ions was investigated. The experimental outcome revealed that the impregnation process enhanced the sorption behavior of silica, from 18 to 67% for neodymium ion and from 20 to 89.45% for gadolinium ion. From the kinetic studies, the sorption could be described well by pseudo-second-order model. Langmuir and Freundlich isotherms were performed to determine the best fit equation for the sorption process; the results indicated that the latter surpasses the former. Comparison of the sorption capacity of Cyphos@silica with other materials reported in the literature shows that the former has a comparatively high sorption capacity. Desorption percent of gadolinium and neodymium ions from Cyphos@silica using 1.0 mol/L of HNO<sub>3</sub> was found to be 97.74 and 86.33%, respectively. The desorption studies revealed the reusability of Cyphos@silica for further sorption.

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## 1. Introduction

Ionic liquids are ionic molecules that contain both organic cations and inorganic or organic anions [1]. They often exist in the liquid state at room temperature [2]. The ionic liquids have become extremely popular compounds in recent separation research due to their unique physicochemical properties. Compared with conventional organic compounds, ionic liquids are characterized by low volatility, low toxicity, high thermal and chemical stability, high polarity, high viscosity, high hydrophobicity and high surface activity due to the presence of both cation and anion species in the same molecule [3,4]. They can be modulated to suit the reaction conditions; therefore, earning the name task-specific ionic liquids [5]. The non-inflammability and non-volatility of ionic liquids provide environment-friendly advantages for using them instead of volatile organic compounds in solvent extraction.

Ionic liquids are used in several applications in different fields, such as waste recycling, nuclear fuel reprocessing, metal extraction, batteries, electrochemical capacitors and electrodeposition of metals [6]. Currently, some types of ionic liquids have been utilized in sorption of pollutants from water and have presented good performances [7,8]. trihexyl(tetradecyl)phosphonium

bis(2,4,4-trimethylpentyl)phosphinate, commercially known as Cyphos IL-104, is one of the phosphonium ionic liquids and classified as hydrophobic and highly viscous ionic liquid [9]. It has been used in various applications; it is utilized as carriers for extracting some metal ions such as; Zn<sup>2+</sup>, Fe<sup>3+</sup>, Cu<sup>2+</sup> and Pd<sup>2+</sup> from chloride media [10–12].

Even though ionic liquids are considered as efficient liquid extractants, several drawbacks such as large consumption, difficult recycling and tedious separation procedures have restricted their applications. To overcome these problems, impregnation of ionic liquids onto solid materials were carried out. Cyphos IL-104 was impregnated onto solid supports like Amberlite XAD-7 and then used for removal of Cr(VI) from potassium chromate aqueous solution [13]. Cyphos IL-104 was used in conjunction with imidazolium ionic liquids on a silica template for Y<sup>3+</sup> removal and separation of lanthanides, such as Ho<sup>3+</sup>, Er<sup>3+</sup> Dy<sup>3+</sup>, and Yb<sup>3+</sup> [14].

Separation and recovery of lanthanide elements from radioactive and industrial wastes are important both environmentally and economically. Radioisotope of neodymium is used as burn-up monitors to evaluate the performance of nuclear fuel [15]. Gadolinium oxide is used in the nuclear power as shielding and fluxing devices; it can be mixed directly with uranium to achieve a uniform neutron flux [16]. Further, the lanthanide elements are increasingly used in many electronic devices.

Toxicological investigations revealed that the lanthanides may have significant adverse effects on the living organisms [17]. Accumulation of

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lanthanides in soil, water and the food chain needs to be considered in environmental studies.

Several methods are available for sorption of lanthanide elements, including adsorption, ion exchange, chemical precipitation and evaporation. Impregnation technique is a type of ion selective exchanger based on the solvent extraction. Impregnation process has some potential advantage over solvent extraction such as minimal organic solvent utilization, less waste accumulation, simple operation and low capital cost. The concept of solvent impregnated resin is based on incorporation of extractant into a porous material matrix either by a physical impregnation technique, or introduction of extractant during synthesis of porous matrix. Some considerations must be taken in impregnation process such as; the solubility of extractant in water should be low to prevent the loss of impregnated extractant during the extraction process, the extractant should have an appropriate viscosity so that the extractant can easily penetrate into the pores of the matrix, and the impregnation process should not damage the chemical and physical structure of the support material. This work focused on removal of neodymium (light lanthanide element) and gadolinium (heavy lanthanide element) ions from aqueous solution using solvent impregnated resin, Cyphos IL-104 impregnated on silica.

## 2. Experimental

### 2.1. Materials

Phosphonium ionic liquid, trihexyl(tetradecyl)phosphonium bis(2,4,4-trimethylpentyl) phosphinate, commercially known as Cyphos IL-104, was purchased from Fluka. Gadolinium and neodymium nitrates ( $\text{Nd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  and  $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ ) were obtained from Merck. Silica gel, grade 62, (60–200) mesh,  $150 \text{ \AA}$ , was obtained from Sigma-Aldrich.  $\text{NH}_4\text{OH}$  and  $\text{HCl}$  were purchased from Winlab Company. All chemicals and reagents were of analytical grade and were used without further purification.

### 2.2. Impregnation of Cyphos IL-104 onto silica

Cyphos IL-104 was impregnated on silica in presence of ethanol as follow; 2 mL of Cyphos IL-104 was added to 4 g of silica to obtain 1:2 ratio, 50% loading percent, then few drops of ethanol were added to the mixture, stirring overnight to obtain a homogeneous mixture, the product, Cyphos@silica, was filtered and washed with distilled water to ensure complete elimination of solvent, then dried in an air oven at  $333 \pm 1 \text{ K}$  overnight.

### 2.3. Evidences of the impregnation process

The impregnation of Cyphos IL-104 on silica can be evidenced by two proofs; (1) silica was impregnated by different loading percent of Cyphos IL-104, and then weighed before and after the impregnation process. The experimental data illustrated that the weight of impregnated silica increased by increasing the loading percent. These results prove that Cyphos IL-104 was impregnated into silica. (2) Another evidence for the impregnation process is illustrated by testing silica, without extractant, for sorption of neodymium and gadolinium ions; the percent uptake was determined and found to be only 18–20%. While after the impregnation process, the percent uptake increased to be >67% for the two ions. This evidence also revealed that the impregnation process is successfully done.

### 2.4. Loss of the impregnated extractant

Cyphos IL-104 may be released into the liquid phase during the sorption process due to the mechanical force and the dissolution effect. Cyphos@silica was subjected to further washing by water to determine the loss of impregnated extractant. 1 g of Cyphos@silica was

consecutively rinsed with 20 mL of water in every washing step; the results revealed that after several times of rinsing, no significant loss of the impregnated Cyphos IL-104 was observed. This is due to the low solubility of Cyphos IL-104 in water.

### 2.5. Characterization of Cyphos@silica

Silica was characterized before and after the impregnation process using different analytical techniques such as; FT-IR, SEM, XRD and TGA.

IR spectra of the samples were recorded on a Model 2000 FT-IR, Perkin Elmer Co., USA. The sample was thoroughly mixed with KBr; then the mixture was ground and pressed with a hydraulic press at about 5 tons to obtain a disc of standard diameter. The spectrum was measured and recorded at a wavelength range of  $400\text{--}4000 \text{ cm}^{-1}$ .

The morphology of silica, Cyphos@silica and Cyphos@silica-Ln was investigated using Scanning Electron Microscopy, SEM, Philips XL 30 model, Netherlands. The sample was washed, dried and mounting on support.

X-ray diffraction spectra of silica before and after impregnation were made using X-ray powder diffractometer (Philips Analytical PW-1710) equipped with  $\text{Cu K}\alpha$  radiation at a scanning speed of  $2^\circ/\text{min}$  from  $10^\circ$  to  $90^\circ$  operated at voltage 40 kV and applied potential current 30 mA.

A thermogravimetric analysis of Cyphos@silica was carried out using a Shimadzu TGA thermal analyzer of type DTA-TGA-50, Japan, at a heating rate of  $20^\circ \text{C}/\text{min}$ , using 5–10 mg of sample. The experiments were performed under a 100 mL/min nitrogen flow.

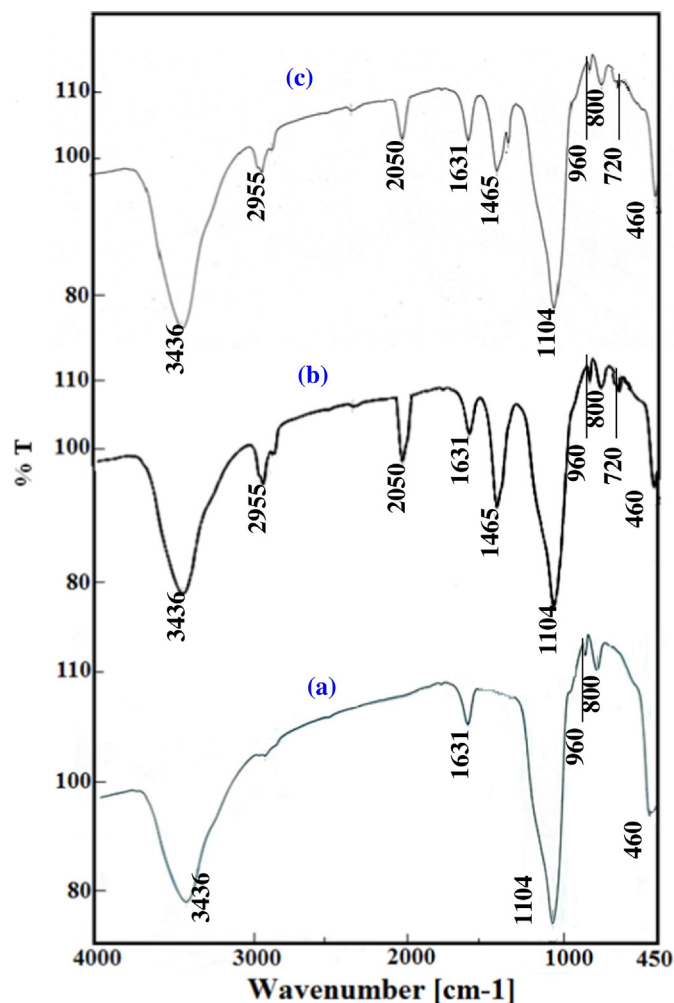


Fig. 1. FT-IR spectra of (a) silica (b) Cyphos@silica and (c) Cyphos@silica-Ln.

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