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Thallium removal through adsorption onto ionic liquidimpregnated solid support: influence of the impregnation conditions

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Abstract This article presents the use of ionic liquid in the removal process of thallium ions using solid-liquid extraction by impregnating ionic liquid (1-n-hexyl-3methylimidazolium chloride) onto an inorganic solid support (Florisil). The influence of impregnation was studied in order to improve the adsorption capacity of the material obtained. Impregnation was realized with the help of a rotavapor. Stirring time and temperature used were varied. Ionic liquid-impregnated materials obtained at different conditions of impregnation were characterized using scanning electron microscopy, energy-dispersive X-ray analysis, and Fourier transform infrared spectroscopy. In order to determine the most efficient adsorbent material, the materials obtained were used in the removal process of thallium ions from aqueous solutions, varying the initial concentration of thallium ions and the stirring time used in the adsorption process. It was observed that for the improving the adsorption capacity of the obtained ionic liquid-impregnated material, it is not necessary to increase the stirring time of the impregnation process but to increase the temperature. The experimental data obtained in the adsorption process were fitted with the Langmuir isotherm. Adsorption of thallium ions onto Florisil impregnated with 1-n-hexyl-3-methylimidazolium chloride ionic liquid corresponds to a pseudo-second-order kinetic model.

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¹ Faculty of Industrial Chemistry and Environmental Engineering, Politehnica University of Timisoara, 6 V. Parvan Blvd., 300223 Timisoara, Romania Keywords Ionic liquid \cdot Impregnation \cdot Thallium \cdot Adsorption

Introduction

Ionic liquids (ILs) show unusual properties, such as extremely low vapor pressure, a wide range of liquid states, good chemical stability, high thermal stability, and excellent solvent characteristics. They have been investigated intensively in chemical reactions (Shiding et al. 2007).

As an alternative to conventional solvents, new environmentally friendly ionic liquids have recently been developed.

There are some limitations in the use of ionic liquids in industrial processes due to high costs of homogeneous catalysis. For the development of systems using SILC as catalysts, immobilization of an ILs as a suitable support is a very interesting alternative (Jouannin et al. 2012).

A composite material consisting of mesoporous silica and a functionalized copolymer (with embedded phosphonate groups) was synthesized. Then, it was characterized and evaluated in a cyclohexene oxidation reaction (Popa et al. 2008).

A variety of solid supports such as gels, polymers, and ionic changer/resins have been used for the separation of rare metals (Liu et al. 2009).

Ionic liquids at room temperature (RTILs) have attracted much attention as green solvents. They are promising for use in many areas of the chemical industry as reaction mediums for chemical synthesis in electrochemical applications and in separation processes, due to their stability and low vapor pressure, which make them ecologically suitable. RTILs are usually composed of organic cations and inorganic anions or organic anions (Wang et al. 2008).



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Due to these physicochemical properties, the cations and anions in RTILs are suitable for the desired application of removing metals from wastewater.

In literature are presented polymeric matrices incorporating ionic liquids in order to obtain polymer-electrolyte gel type solid state (ILGPEs). Combining the advantages of both ionic liquids and conventional polymer electrolyte gel (GPEs), ILGPEs have better properties than each individual component (Lu et al. 2008).

Water pollution, especially industrial wastewater containing metal ions and aromatic compounds, is a major environmental problem (Davidescu et al. 2014; Ardelean et al. 2012a, b). The metal ions are often hazardous and polluting, and their removal from water is an important area in environmental research (Davidescu et al. 2013; Salam 2013; Ciesielczyk et al. 2015).

Thallium ion is a very important element but at the same time presents high toxicity, so it must be removed from wastewater. Our researches present the process of removal of thallium ions from aqueous solutions.

The present paper shows the use of ionic liquid (1-nhexyl-3-methylimidazolium chloride) in the removal of thallium ions using the solid-liquid extraction. Then, the ionic liquid was impregnated onto an inorganic solid support (Florisil). Florisil was used as a solid support because in our previous study, it improved the adsorption capacity of the obtained adsorbent (Negrea et al. 2013). Through impregnation of the ionic liquid onto Florisil, adsorbent materials with good properties of Tl(I) ion removal from aqueous solutions can be obtained, due to the structural properties of Florisil (well-ordered pore structure and high specific surface area) and the advantages of the ionic liquid. Impregnation of ionic liquid onto Florisil was made using a rotavapor. This method requires a short time of impregnation and a short time of drying of the obtained material. We determined the influence of the impregnation conditions on the improvements in the adsorption capacity of the ionic liquid-impregnated solid support in the removal process of thallium ions from aqueous solutions.

Materials and methods

Materials

Ionic liquid 1-*n*-hexyl-3-methylimidazolium chloride and the solid support Florisil were provided from Sigma-Aldrich. For the preparation of thallium (I) ion solutions, a stock solution of 1 g/L (Merck standard solutions) was used. All chemicals used in the experiments were of analytical grade, and distilled water was used to prepare the solutions.



Preparing the ionic liquid-impregnated solid support

In order to impregnate the ionic liquid (1-n-hexyl-3methylimidazolium chloride) on the studied solid support (Florisil), the rotavapor (Heidolph) was used. The ratio of solid support to ionic liquid was 1:0.1 g. In order to avoid the drawbacks of the liquid-liquid extraction, a higher quantity of ionic liquids is not used. Using a smaller quantity of ionic liquid decreases the cost of adsorption. Other studies reported that increasing the quantity of ionic liquid impregnated onto the solid support did not have a significant influence on the adsorption capacity of the adsorbent (Lupa et al. 2013; Negrea et al. 2014). Furthermore, increase in the quantity of ionic liquid impregnated onto the solid support results in oily surfaces leading to the conglomeration of the adsorbent particles and in the decrease in the specific surface area, thus resulting in the decrease in adsorption capacity. In the first step, ionic liquid was dissolved in acetone. Conditions of impregnation such as stirring time (15, 30, 60, and 120 min) and temperatures of the solvent evaporation (20, 30, and 40 °C) were varied. This method is very advantageous because it requires a short time of impregnation and a short time of drying of the obtained material. In order to show that the Florisil was impregnated with the studied ionic liquid, the obtained materials at different impregnation conditions were characterized using Fourier transform infrared spectroscopy (FTIR), energy-dispersive X-ray analysis (EDX), and scanning electron microscopy (SEM) analysis. The FTIR spectra of the ionic liquid-impregnated solid support were registered using a Shimadzu Prestige-FTIR spectrophotometer 21 in the range 4000-400 cm⁻¹. For SEM and EDX analysis, the samples were fixed using carbon tape on aluminum-based pin stubs. After the samples were prepared, they were investigated by scanning electron microscopy (SEM) using a Quanta FEG 250 microscope equipped with EDAX analyzer with SDD Apollo X detector. The investigation was carried out in low vacuum mode to eliminate sample charging. The SEM images were recorded using a 5000× magnitude. The ratio of solid support to ionic liquid after impregnation was checked through difference between the quantity of resulted adsorbent and the quantity of the initial Florisil. The results were in the error range ≤ 0.003 g. This error can be explained by the fact that at the end of the drying process of the impregnated material, a small quantity of the resulted adsorbent could be sucked by the vacuum system.



Fig. 1 FTIR spectrum of a the studied ionic liquid; b Florisil before impregnation; c Florisil impregnated with the IL at different stirring times and 20 °C; d Florisil impregnated with IL at different temperatures and 15 min of stirring

Adsorption of Tl(I) ions onto the ionic liquidimpregnated solid support

The obtained materials were used in the removal process of Tl(I) ions from aqueous solutions in order to determine the influence of the different impregnation conditions used, with respect to the optimum conditions.

Adsorption of Tl(I) ions was realized in batch mode. Stirring was made using a MTA Kutesz shaker, with constant speed of stirring (200 rpm). Adsorption capacity of the obtained material was determined by treating 1 g of each material with 25 mL solution of various concentrations of Tl(I) ions (5, 7.5, 10, 15, and 20 mg/L). The samples were shaken for 2 h and filtrated; then, the residual concentration of Tl(I) ions in the resulted solution was determined by atomic emission spectrometry using an Varian SpectrAA 280 atomic adsorption spectrometer. In all the experiments, the initial pH of the Tl(I) solutions was around 3. After the adsorption process, the pH value of the solutions was $pH_i \pm 0.5$.

The adsorption capacity of the ionic liquid-impregnated solid support was determined using Eq. 1 (El-Kamash 2008; Hanafi 2010; Lupa et al. 2013; Negrea et al. 2013, 2014):

$$q_{\rm e} = \frac{(C_0 - C_{\rm e})V}{m} \tag{1}$$

where C_0 and C_e are the concentration of Tl(I) ions (mg/L) in the initial solution (t = 0) and at equilibrium, respectively, V represents the volume of Tl(I) ion solution (L), and m is the mass of the ionic liquid-impregnated material (g).

In order to determine the maximum adsorption capacities of the ionic liquid-impregnated materials obtained





Florisil before impregnation



120 min, 20°C

15 min, 30°C

15 min, 40°C

Fig. 2 SEM images of the Florisil before impregnation and impregnated with IL at different conditions of impregnation

using various impregnation conditions, the obtained experimental data were fitted with the well-known linearized isotherm of Langmuir (El-Kamash 2008; Hanafi 2010; Lupa et al. 2013; Negrea et al. 2013, 2014). These results will be useful to determine the optimum conditions of impregnation.

In the second step, dependence of the adsorption capacity of the obtained materials on the stirring time was determined. To do so, 0.1 g of the obtained materials was treated with 25 mL of Tl(I) ion solutions of concentration 10 mg/L using different stirring times (range 30–300 min).

Experimental data were fitted with the pseudo-secondorder reaction model in order to determine the mechanism of thallium adsorption onto Florisil impregnated with 1-*n*hexyl-3-methylimidazolium chloride ionic liquid using various impregnation conditions (Memon et al. 2008; John Peter and Viraraghavan 2008).

Results and discussion

Adsorbent characterization

The FTIR spectra of both 1-*n*-hexyl-3-methylimidazolium chloride ionic liquid and Florisil before and after impregnation with the studied ionic liquid using various conditions of impregnations are presented in Fig. 1.

In FTIR analysis, the presence of some characteristic peaks attributed to the studied ionic liquids confirms that Florisil was impregnated with 1-*n*-hexyl-3-methylimidazolium chloride:





Fig. 3 EDX spectra of the Florisil before impregnation and impregnated with IL at different conditions of impregnation

the intense bands between 2800 and 2960 cm⁻¹ are stretching vibration of CH₃ and CH₂, and the bands around 3100 and 3140 cm⁻¹ are attributed to the imidazolium CH stretching, both of which were not present in the Florisil FTIR spectrum before impregnation (Balaban et al. 1983).

The morphological structure of the ionic liquid-impregnated solid supports can be observed from the SEM images presented in Fig. 2. Onto smooth surface of Florisil from all the obtained materials have been observing the particles of impregnated ionic liquid. It can be noticed that stirring time does not influence the quantity of ionic liquid impregnated onto Florisil. On the other hand, increase in the temperature leads to a higher quantity of ionic liquid impregnated onto Florisil. Also, increase in the temperature leads to a shorter time of the solvent evaporation.

In EDX analysis, the presence of the characteristic peaks of the elements which are provided by the studied ionic





Fig. 4 Equilibrium isotherm of adsorption of thallium(I) ions onto ionic liquid-impregnated materials \mathbf{a} using various stirring times at impregnation and \mathbf{b} using various temperatures at impregnation

liquid also confirms that Florisil was impregnated with 1-*n*-hexyl-3-methylimidazolium chloride (Fig. 3).

Adsorption of Tl(I) ions from aqueous solutions

Ionic liquid-impregnated materials obtained using various impregnation conditions were used in the removal process of Tl(I) ions from aqueous solutions. The equilibrium isotherm of the samples is presented in Fig. 4. It is used to explain the adsorbent properties and affinity for the adsorbate and also can be used to compare the maximum adsorption capacity of various adsorbents (Negrea et al. 2014; Memon et al. 2008; John Peter and Viraraghavan 2008).

It can be observed that for all the studied materials, the increase in the initial concentration of Tl(I) ions increased the adsorption capacity of the studied materials, until a



Fig. 5 Langmuir isotherms of the obtained materials \mathbf{a} using various stirring times at impregnation and \mathbf{b} using various temperatures at impregnation

constant value was reached. The increase in the stirring time did not have a significant influence on the adsorption capacity of the obtained materials in the removal process of Tl(I) ions from aqueous solutions. Among all the cases, the maximum adsorption capacity obtained was 2.5 mg Tl(I)/g. Increase in the temperature led to an increase in the maximum adsorption capacity. Florisil impregnated with the ionic liquid using 15 min of impregnation at a temperature of 40 °C showed a maximum adsorption capacity of 2.9 mg Tl(I)/g.

In order to determine the maximum adsorption capacities of the studied materials, experimental data were fitted with the linear Langmuir isotherm. The Langmuir isotherms are presented in Fig. 5. The results of Langmuir parameters together with the correlation coefficients are presented in Table 1.



Table 1Parameters ofLangmuir isotherms for theadsorption of thallium(I) ionsonto the obtained impregnatedsolid supports

Adsorbent	$q_{\rm m}$, exp, mg/g	$K_{\rm L}, {\rm L/mg}$	$q_{\rm m}$, calc, mg/g	R^2
Florisil + IL obtained at 15 min and 20 °C	2.45	5.479	2.5	0.999
Florisil + IL obtained at 30 min and 20 °C	2.48	5.468	2.54	0.999
Florisil + IL obtained at 60 min and 20 °C	2.49	5.646	2.53	0.999
Florisil + IL obtained at 120 min and 20 °C	2.5	7.719	2.54	0.999
Florisil + IL obtained at 15 min and 30 °C	2.83	5.473	2.9	0.999
Florisil + IL obtained at 15 min and 40 °C	2.9	3.495	2.95	0.999



Fig. 6 Effect of contact time on the adsorption capacity of the obtained materials \mathbf{a} using various stirring times at impregnation and \mathbf{b} using various temperatures at impregnation

It can be noticed that the Langmuir model effectively describes the adsorption of studied radionuclides onto the obtained impregnated solid supports, because correlation coefficients close to 1 are achieved. For all the studied adsorbents, the maximum adsorption capacities obtained from the fitted equation are close to those determined experimentally. The increase in the stirring time used in the process of Florisil impregnation with IL did not have a



Fig. 7 Pseudo-second-order kinetic model for adsorption of thallium(I) ions onto the obtained materials \mathbf{a} using various stirring times at impregnation and \mathbf{b} using various temperatures at impregnation

significant influence on the adsorption capacity of the obtained materials in the removal process of TI(I) ions from aqueous solutions. Increase in the temperature led to an increase in the maximum adsorption capacity. Florisil impregnated with the studied ionic liquid using 15 min of stirring and 40 °C showed the highest adsorption efficiency (2.95 mg of TI(I)/g of adsorbent). These conclusions are in



 Table 2
 Kinetic parameters for adsorption of thallium(I) ions onto the studied materials

Adsorbents	$q_{\rm e, exp}$, mg/g	$q_{\rm e, \ calc}, {\rm mg/g}$	k_2 , min/(mg/g)	R^2
Florisil + IL obtained at 15 min and 20 °C	2.46	2.55	0.0186	0.999
Florisil + IL obtained at 30 min and 20 $^{\circ}C$	2.39	2.53	0.0268	0.999
Florisil + IL obtained at 60 min and 20 $^{\circ}\mathrm{C}$	2.47	2.57	0.0249	0.999
Florisil + IL obtained at 120 min and 20 $^{\circ}\mathrm{C}$	2.47	2.57	0.0314	0.999
Florisil + IL obtained at 15 min and 30 $^{\circ}\mathrm{C}$	2.47	2.58	0.0281	0.999
Florisil + IL obtained at 15 min and 40 $^{\circ}\mathrm{C}$	2.49	2.59	0.0340	0.999





accordance with the assumption resulted from the material characterization section. The calculated maximum adsorption capacity of Florisil impregnated with 1-*n*-hexyl-3-methylimidazolium chloride (2.95 mg/g), in the removal process of Tl(I) from aqueous solutions, is higher than other adsorbents studied in the literature such as modified Aspergillus niger biomass (Peter and Viraraghavan 2008) and polyacrylamide–aluminosilicate composite (Senol and Ulusoy 2010).

In the next step, the influence of the contact time used in the removal process of Tl(I) ions from aqueous solutions onto the adsorption capacity of the obtained materials was determined, determining in this way the time when the equilibrium between the adsorbent and adsorbate is achieved. The adsorption kinetics of Tl(I) ions onto the adsorbent materials is presented in Fig. 6.

From the experimental data, it can be observed that the increase in the contact time increases the adsorption capacity, achieving at constant value for a contact time higher than 180 min. It can be mentioned that the

equilibrium between the adsorbent and adsorbate is achieved in 180 min for all the studied adsorbent materials.

Pseudo-second-order reaction model was used in order to study the adsorption mechanism of thallium onto Florisil impregnated with 1-*n*-hexyl-3-methylimidazolium chloride. Parameters of the pseudo-second-order reaction model (q_i and K_2) are determined from the slope and intercept of (t/q_t) versus t (Fig. 7), and the obtained values are presented in Table 2 together with the correlation coefficients.

Table 2 shows that the degree of fit \mathbb{R}^2 for the pseudosecond-order is closer to unity; this suggests that the pseudo-second-order adsorption mechanism is predominant and that the overall rate of the Tl(I) ion adsorption process appeared to be controlled by the chemical process (Lupa et al. 2013; Negrea et al. 2013, 2014). Also, the q_e values calculated fitted the experimental data. Hence, it can be hypothesized that the removal of Tl(I) ions is realized by its binding to the alchil radical from the impregnated ionic liquid, forming the hypothetical complex [CH₃-Tl]. This conclusion is also based on the FTIR spectrum (Fig. 8) of



the exhausted adsorbent where the vibrational bands of the v_{sym} (Tl-C) bound at the wavelength of 452, 488, and 515 cm⁻¹ can be observed (Schwerdtfeger et al. 1990; Mendizabal and Olea-Azar 2007). Another hypothesis of Tl(I) adsorption is the possible interaction between Tl(I) ions and Cl⁻ ions from the ionic liquids, which play the role of an ion-pairing reagent, because the adsorption process occurred at a low pH value of the solution (Escudero et al. 2012; Rehman et al. 2012).

Conclusion

In this study, ionic liquid (1-n-hexyl-3-methylimidazolium chloride) was impregnated onto an inorganic solid support (Florisil). Both stirring time and the temperature used at the impregnation technique were varied in order to improve the adsorption capacity of the obtained material. The ionic liquid was used in the removal process of thallium ions using the solid-liquid extraction. Both the initial concentration of thallium ions and the contact time used in the adsorption process were varied. The obtained ionic liquidimpregnated materials at different conditions of impregnation were characterized using scanning electron microscopy (SEM), energy-dispersive X-ray analysis (EDX), and Fourier transform infrared spectroscopy (FTIR), which proved that Florisil was impregnated with the 1-n-hexyl-3methylimidazolium chloride. It was observed that for improving of the adsorption capacity of the obtained ionic liquid-impregnated material, it is not necessary to increase the stirring time of the impregnation process, but to increase the temperature. Experimental data obtained in the adsorption process were fitted with the Langmuir isotherm, and the studied adsorbent developed a maximum adsorption capacity of 2.95 mg Tl(I)/g. Adsorption of thallium ions onto Florisil impregnated with 1-n-hexyl-3methylimidazolium chloride ionic liquid corresponds to a pseudo-second-order kinetic model.

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