



Solid phase extraction using silica gel modified with murexide for preconcentration of uranium (VI) ions from water samples

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ABSTRACT

Murexide was chemically bonded to silica gel surface immobilized 3-aminopropyl trimethoxysilane (APMS) to produce the new sorbent. A solid phase extraction method using the new sorbent has been developed to separate and concentrate trace amount of uranium (VI) from aqueous samples for the measurement by spectrophotometry method using Arsenazo III reagent. The influences of some analytical parameters on the quantitative recoveries of the analyte were investigated both in batch and column methods. Quantitative recovery of U(VI) was achieved by stripping with 0.1 mol L^{-1} HCl. The maximum sorption capacity of the modified silica gel was 1.13 mmol g^{-1} U(VI). A high preconcentration factor value of 400 with a lower limit of detection of $1 \mu\text{g L}^{-1}$ was obtained for U(VI). The practical applicability of the developed sorbent was examined using synthetic and real samples such as sea/ground water samples.

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1. Introduction

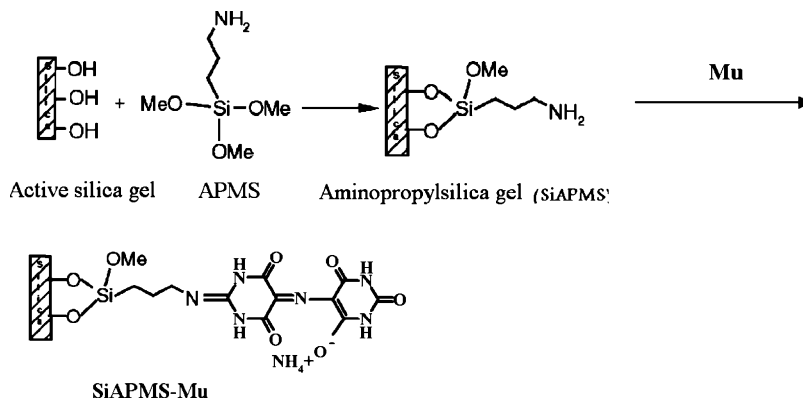
There is high interest for new separation techniques which selectively extract metal ions from dilute water and waste water samples. Although a variety of methods such as precipitation, solvent extraction, electrolysis and ion exchange can be used to remove dissolved metals from aqueous samples, most of them have disadvantages of non economic, poor removal efficiency, high cost, generation of secondary pollution and ineffectiveness for low metal concentrations [1].

Solid phase extraction (SPE) has commonly been used as a technique for preconcentration/ separation of various inorganic and organic species. SPE is used to enhance the selectivity and sensitivity of the method as it allows for discriminatory binding of analyte to a solid support where it will be accumulated and subsequently eluted with a small volume of solvent. This technique has advantages of higher enrichment factor, absence of emulsion, safety with respect to hazardous samples, minimal costs due to low consumption of reagent, environment friendly, flexibility and easier incorporation into automated analytical techniques [2–5]. Selectivity of the solid phase sorbent towards an analyte depends on the structure of the immobilized organic ligands.

Uranium is extensively used in the nuclear industry and is highly radioactive. It is present in low quantities in wash streams

coming out of nuclear reactors both in aqueous as well as non aqueous medium and monitoring of these streams for the presence of uranium in high activity content is essential. The maximum uranium concentration in drinking water and seawater also is reported less than $9 \mu\text{g L}^{-1}$ and $1\text{--}3 \text{ ng mL}^{-1}$, respectively [6]. This extreme dilution in the presence of relatively high concentration of other ions makes it difficult to determine directly uranium ions and refined analytical methods must be employed to detect small concentrations. For this purpose, new sorbent materials such as polymeric resins, activated carbon, naphthalene and silica gel have been developed for more effective extraction. However, some of the sorbents suffer from a number of drawbacks such as long preconcentration time, low mechanical stability of the sorbent, slow kinetics, irreversible adsorption of target and swelling [7–17]. So, there is still a need for developing good sorbent for U(VI) ions. Silica gels modified with both inorganic and organic functionalities have been most commonly used in various areas, notably in separation and preconcentration of trace metal ions from aqueous systems since immobilization reactions on silica are relatively simple and show fast kinetics in metal ions uptake [18–24]. Recently, a number of silica gel sorbents functionalized with a variety of chelating ligands have been synthesized for preconcentration of U(VI) from aqueous solutions [25–29]. Some of the reported chemically modified sorbents are those containing ligands with oxygen and nitrogen donors involved in U(VI) chelation forming for selective extraction of U(VI) from various matrix components. Murexide (Mu, the ammonium salt of purpuric acid) is a dark red reagent, and soluble in water which is used as an indicator in

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Scheme 1. Proposed route for preparation of SiAPMS–Mu adsorbent.

complexometric titrations. Complexation of Mu with the alkaline, alkaline earth and transition cations had been studied [30–32]. To the best of our knowledge, there is no report on complexation of Mu with U(VI) ions or immobilization on the surface of silica gel. Therefore, this work is devoted to the preparation and the evaluation of the sorption properties of silica gel modified with murexide (SiAPMS–Mu) as a new solid phase extractor for preconcentration of U(VI) from aqueous samples.

2. Experimental

2.1. Chemicals and reagents

Reagent grade of uranyl acetate, $\text{UO}_2(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$, was obtained from Fluka. Extra pure silica gel with particle size 70–230 mesh and 60 Å pore diameter purchased from Merck. All reagents and solvents used were of analytical reagent grade and were provided from Merck. Buffer solutions were prepared by using 0.01 mol L⁻¹ sodium acetate–acetic acid (pH 3–6) or ammonium acetate (pH 7–8) and the pH of the resulting solutions was adjusted by the use of pH meter. Stock solution of U(VI) was prepared by dissolving an appropriate amount of uranyl acetate in buffer. All aqueous solutions were prepared in doubly distilled water.

2.2. Apparatus

The pH measurements were conducted by a corning 125 pH-meter. The UV–vis spectra were recorded using Shimadzu 2501 PC spectrophotometer. The IR spectra were obtained using KBr pellets by PerkinElmer 781 IR-spectrophotometer. A Shimadzu flame atomic absorption spectrophotometer (FAAS) model 6300 was used for determination of some transition metals as interfering ions.

2.3. Preparation of the murexide modified silica gel

The procedure for preparation of the new sorbent includes three steps as follows: In the first step, activation of 50.0 g silica gel surface was accomplished by reflux in 6.0 mol L⁻¹ hydrochloric acid for 24 h, filtered and repeatedly washed with doubly distilled water till acid free filtrate and finally dried in an oven at 160 °C for 8 h.

In the second step, 40.0 g of the activated silica gel was suspended in 400 mL of dry toluene containing 45 mL of 3-aminopropyltrimethoxy silane (APMS) as silylating reagent and refluxed for 12 h. At the end of this period, the modified silica gel (SiAPMS) was filtered off, washed with toluene, ethanol and diethyl ether, respectively, to eliminate possible trace amounts of the reagent and dried at 70 °C for 6 h.

In the last step, silica gel bounded murexide was prepared by mixing 30 g of dry SiAPMS with 30 mmol of Mu already dissolved in 300 mL of dimethyl sulfoxide (DMSO) and the reaction mixture was refluxed for 24 h. The resulting product was filtered, washed with DMSO and water several times until the filtrate showed no trace color of adsorbed Mu. The resulting brown sorbent (SiAPMS–Mu) was then dried at 80 °C for 6 h. The preparation route to SiAPMS–Mu can be proposed in Scheme 1.

2.4. Analytical procedure

2.4.1. Batch method

The stability of the SiAPMS–Mu in different buffer solutions (pH 2–8) was investigated by the batch equilibrium technique in order to identify the degree of hydrolysis or possible leaching of the sorbent. In this procedure, 500 mg of the SiAPMS–Mu was mixed with 100 ml of the selected buffer solution (pH 2–8) and automatically shaken for 2 h. The mixture was filtered off, washed with the same buffer and doubly distilled water, respectively, and dried in an oven at 70 °C. To determine the uptake of U(VI), 100 mg of the treated SiAPMS–Mu was added to a solution of 100 ml of 135 μg mL⁻¹ of U(VI) at pH 5.5. The percentage of hydrolysis of SiAPMS–Mu at various pH was calculated from μmol g⁻¹ value of treated sorbent.

For sorption study, a sample solution containing 2.7 μg mL⁻¹ of U(VI) in a volume of 10–100 mL was taken and its pH was adjusted to 5.5 by acetate buffer. Then, 100 mg of the SiAPMS–Mu was added to the above solution in a polyethylene bottle and was shaken mechanically for 15 min. The amount of U(VI) adsorbed was estimated by the difference between the initial concentration in aqueous solution and that found in the supernatant spectrophotometer.

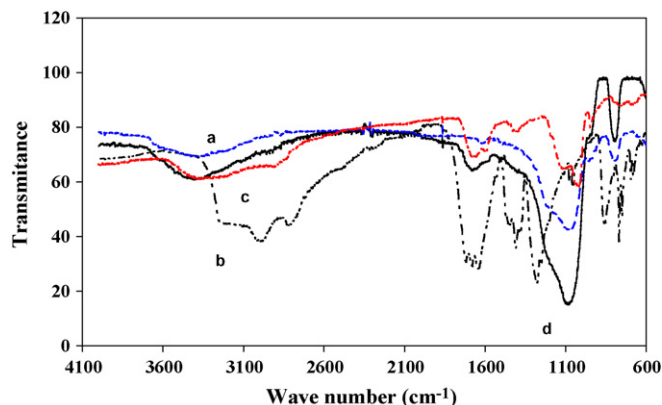


Fig. 1. IR spectra of silica gel (a), Mu (b), APMS–Mu (c) and SiAPMS–Mu (d).

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