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Synthesis and characterization of silica microsphere and their application in removal of uranium and thorium from water

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Abstract Uranium (U) and thorium (Th) are known to cause acute toxicological effects in human, and their compounds are potential occupational carcinogens. During this work, silica microspheres were used to decontaminate the traces of U and Th in different water sources originated from groundwater and lake water. Ultra-filtration technique was used to isolate the microsphere from the water samples. Silica microspheres were synthesized based on a polyvinylpyrrolidone involved emulsion polymerization and subsequent sol-gel process. The microspheres were characterized for their size by using dynamic light scattering, and presence of silicate structure is verified by recording their attenuated total reflectance-Fourier transform infrared spectrum. The surface morphologies of the prepared silica microspheres were studied by using scanning electron microscope. Water samples collected from the groundwater and lake water were spiked with U and Th in the concentration range of $100-1,000 \text{ ng mL}^{-1}$. The results of batch sorption experiments suggest that silica microsphere is very effective at pH 3, and sorption is more than 99.9 %. Equilibrium sorption follows Langmuir isotherms and the maximum U, and Th uptake is 30 and 36 mg g^{-1} , respectively. Major physicochemical characteristics of the water were monitored before and after the decontamination process. Experimental results show no significant variations in any of the measured parameters.

Keywords Silica microsphere · Groundwater · Lake water · Uranium · Thorium · Decontamination

Introduction

Uranium and thorium are important elements not only in industrial application but also in energy and environmental problems. In view of the extensive usage of uranium and thorium for various industrial purposes and their sub trace level releases through liquid effluents which in turn reflects into the various aquatic medium (Gok and Aytas 2009; Basu et al. 2011; Singhal et al. 2011a, b). As per the recent works carried out in India (Singh et al. 1995; Singh et al. 2008), many location in the northern part of India (Punjab and Harvana) elevated levels of U was observed in groundwater. Although exact reasons of elevated level of U in groundwater is not certain but various probable reasons are either because of localized mineralization process in which uranium from the host rock is preferentially leaching in the aquatic subsurface environment and reflected in the groundwater or some other anthropogenic activity in that region like use of localized phosphate fertilizer etc.

Removal of pollutants from aquatic medium using adsorption phenomenon is a common practice, in which the pollutants accumulate on any solid surface. In water treatment, the pollutants and any solid phase are, respectively, called adsorbates and adsorbent. For a few decades, various adsorbents have been developed and used for water treatment using different processes (Gupta and Ali 2006; Ali 2010, 2012, 2014; Ali et al. 2012). A large number of studies have been carried out by using silica microspheres for removal of various pollutants from aquatic environments including dyes (Hench and West 1990; Frank et al. 1998; Guangyu et al. 2007). They are very effective in removal as they have large surface area, narrow distribution of the pore size, well-defined and adjustable pore structure. These spheres can be obtained from the synthesis of the core-shell particles. Most methods of fabrication of



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hollow silica spheres involve removing the core by calcination or dissolution into solvents. This can be achieved as one-step process, which meant that the formation of the inorganic shells and the dissolution of core particles occurred in the same medium or in two different steps, i.e., formation of composite particle, followed by removal of inner structure leaving behind the core with hollow space (Wang et al. 2010; Zhuang et al. 2009; Jal et al. 2004).Previous investigations have demonstrated that polymeric microparticles and inorganic cores can be coated with layers of various materials, including silica, yttrium basic carbonate, and zirconium hydrous oxide, either by controlled surface precipitation reactions on the core particles or by direct surface reactions. These core-shell particles often exhibit properties that are substantially different from those of the templated core (for example, different surface chemical compositions, increased stability, higher surface area, and different magnetic and optical properties), thus making them attractive from both a scientific and a technological viewpoint. Applications for such particles are diverse, including capsule agents for drug delivery, catalysis, coatings, composite materials, and protecting sensitive agents such as enzymes and proteins (Zou et al. 2008; Rosaria et al. 2011; Zhang et al. 2008; Naseem and Tahir 2001; Srivastava et al. 1989; Bektas et al. 2004). Zinc from aqueous solutions was successfully obtained by using magnetite silica core-shell Nanoparticles (Emadi et al. 2013).

Precise determination of U and Th in environmental and biological samples is a challenging task. Both of the metal ions are known to cause acute toxicological effects in human, and their compounds are potential occupational carcinogens. In case of uranium, World Health Organization (WHO 2008) had put up a drinking water guidelines and fixed the maximum uranium concentration in drinking waters to be less than 15 μ gL⁻¹(WHO 2008; Copenhaver et al. 1992; Banks et al. 1995; Berner and Berner 1996; Porcelli and Swarzenski 2003). This limit of uranium is put up considering its heavy metal toxicity rather than radiological. Depending upon the localized geo-chemical environment, groundwater has Th in the range of 10-20 picomolar (pM), whereas in seawater it is in 0.1-1 pM concentration. In the case of thorium, the toxicity is given in the form of radiological one as 0.5 Bq L^{-1} .

In this work, silica microspheres were synthesized by a polyvinylpyrrolidone (PVP) by using emulsion polymerization and subsequent sol-gel process. Decontamination of U and Th from groundwater and lake water in the concentration range of 100–1,000 μ g L⁻¹ were successfully carried out using silica microsphere. Effective separation of silica microspheres from the water samples was carried out by using ultra-filtration technique.

All the works discussed in this manuscript was carried out during December 2012 to October 2013 at Bhabha Atomic Research Centre, Trombay, Mumbai-400085, India.

Materials and methods

Chemicals and reagents

All chemicals used in this work were of analytical grade of Merck-Aldrich. Doubly distilled water (Millipore) was used throughout the experiment for the preparation of the reagents. Stock standard solutions $(1,000 \text{ mg L}^{-1})$ of uranium (VI) and thorium (IV) in HNO₃ medium were used for the preparation of the experimental solutions. The pH adjustment was made with 5 M nitric acid (for acidic pH values) or 5 M sodium hydroxide (for basic pH values). The glassware used were kept in 5 % nitric acid overnight and subsequently washed several times with double-distilled deionized water before using.

Preparation of polyvinylpyrrolidone (PVP) functionalized polystyrene (PS) latexes

The monodispersed PS particles were prepared by emulsion polymerization. Measured quantity of styrene 10 g, PVP (1.5 g) and water (90 mL) were charged into a 250-mL Teflon autoclave. The solution was stirred and deoxygenated by bubbling N2 at room temperature for 30 min. Then, the mixture was slowly heated to 70 °C, followed by addition of 10 g of aqueous solution containing potassium peroxodisulfate (0.1 %), and the reaction was carried out at 70 °C for 24 h in an autoclave (Hench and West 1990; Frank et al. 1998; Guangyu et al. 2007).

Formation of silica-coated PS particles and silica microspheres

The polyvinylpyrrolidone (PVP) functionalized polystyrene latexes were reacted with tetraethyl orthosilicate (TEOS) in a solution of ammonia in ethanol to yield silicacoated latex particles in a seeded growth process. In a typical procedure, NH₄OH solution and PS emulsion were added into ethanol under stirring, and the mixture was kept at 30 °C. Then, 10 mL solution of the TEOS in ethanol was added slowly to the suspension under stirring at 30 °C at a rate of 1 mL h⁻¹ using a peristaltic pump. The reaction mixture was stirred at 30 °C for an additional 5 h. After cooling to room temperature, the suspended particles were treated with toluene to dissolve the polystyrene and to make it porous and hollow (Frank et al. 1998).



Measurement of mean hydrodynamic diameter of particle distribution of silica microspheres

Size distribution and zeta potential of the silica microsphere in water samples were studied by using Malvern Zetasizer Nano ZS. Measurements were taken by dynamic light scattering using He–Ne laser with a power of 4.0 mW and wavelength 633 nm. The intensity of the scattered light was measured using an avalanche photodiode detector at room temperature (25 °C) having a quantum efficiency >50 % at 633 nm.

Preparation of contaminated potable water

Potable water samples having a pH 6.3 were spiked with uranium $[UO_2(NO_3)_2]$ and thorium $[Th(NO_3)_4]$ in the range of 100–1,000 ng mL⁻¹. pH of the samples was maintained at 3.0 \pm 0.2. Silica microspheres (10 mg L⁻¹) were added and stirred with the help of an electrical stirrer for 30 min. The process water samples were then passed through ultra-filtration membrane having 500 Dalton cutoff inside a pressurized cell to separate the microspheres from the process water. U and Th from the microsphere were desorbed using 4 M HNO₃.

Measurement of U and Th

Determination of uranium and thorium was carried out by using simultaneous solid-state detector inductively coupled plasma optical emission spectrometer (ICP-OES, model ACTIVA, from Horiba Jobin-Yvon SAS, France). Intensity of emission was measured at two different wavelengths, i.e., 398.58 and 367.01 nm in the case of uranium, whereas thorium emission was measured at 401.913 and 283.232 nm. The calibration of instrument was carried by using Aldrich standard solution of uranyl nitrate and thorium nitrate. ACTIVA utilizes a 2,048 × 512 pixel, ultra-low noise, high quantum efficiency charge-coupled device (CCD) solid-state detector. ACTIVA includes a unique optical design featuring a 0.64 m Czerny-Turner optical system and holographic gratings of 4,343 grooves per mm and 2,400 grooves per mm. Covering the full 6 mm height of the plasma, "Normal Analytical Zone Multi-WAV" acquisition mode provides complete sample fingerprinting of 75 elements in less than 30 s. It gives resolution up to 10 pm. Concentrations of uranium and thorium in different samples were also validated by using alpha spectrometry. Details of the same were discussed elsewhere (Singhal et al. 2011a, b).

Spectroscopic investigation

The Attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectrum of silica microsphere was recorded

between 500 and $4,000 \text{ cm}^{-1}$ by using Fourier transform spectrometer "Spectrum One (Bruker)" equipped with the universal ATR as an internal reflection accessory which have composite zinc selenide.

Scanning electron microscope (SEM)

The surface morphology of microsphere was studied using scanning electron microscopy coupled with energy dispersive X-ray fluorescence spectrometry (EDS) (Instrument Model No. VEGA MV 2300T). Before recording the surface morphology, gold coating was done on silica microsphere using mini sputter coater.

Results and discussion

The efficiency of separation of traces of uranium and thorium from the aquatic medium by using silica microsphere may severely be affected by the presence of others ions in medium and as well the size of the microsphere. Therefore, detailed characterization of the water sample was carried out.

Characterization of aquatic medium

Table 1 gives the concentration of various ions including U and Th along with other physicochemical characteristics of the water samples used in this work. At various stages of the experiments, concentration of these parameters was monitored to maintain the quality of the water to ensure its

 Table 1
 Variation in various physicochemical characteristics of the groundwater before and after the completion of the experiment

Parameters	Variations	
	Feed water	Processed water
pH	6.3–7.6	6.5–7.3
Conductance (µS)	230-290	240-280
Total hardness (mg L^{-1})	70–90	65-85
$\text{HCO}_3^- (\text{mg L}^{-1})$	55-70	55-70
Na^+ (mg L^{-1})	28-40	35–45
$\mathrm{Fe}^{3+} (\mathrm{mg} \ \mathrm{L}^{-1})$	0.5–4	0.4-3.4
$\operatorname{Ca}^{2+}(\operatorname{mg} L^{-1})$	39.26-50	44–54
$K^+ (mg L^{-1})$	0.7-1.2	0.6-1.3
Dissolved organic carbon [DOC] (mg L ⁻¹)	1.0–1.5	1.0–1.6
Total dissolved solid (mg L^{-1})	112-120	110-130
Turbidity (NTU)	3–5	3–6
DO [dissolved oxygen] (mg L^{-1})	6.5–7.2	6.4–7.2
Uranium (ng mL ⁻¹)	6–10	11–12
Thorium (Bq L ⁻¹)	BDL	BDL



suitability for the consumption by human being. From this table, it is clear that there is no domination of any particular geological system as there is no elevated level of any particular ion was observed.

Formation of silica microspheres

PVP is an amphiphilic, nonionic polymer soluble in water and many nonaqueous solvents which arises from the presence of a highly polar amide group within the pyrrolidone ring and a polar methylene and methine groups in the ring and along its backbone. PVP first participated in the polymerization reaction as a stabilizer or surfactant and then played a coupling agent role in the latter process. The PS particles were functionalized by PVP and incorporated



Fig. 1 Attenuated total reflectance-Fourier transform infra red (ATR-FTIR) spectrum of silica microspheres

hydrophilic PVP on their surfaces. The polystyrene particles were dissolved subsequently during the sol-gel coating process, followed by treatment with toluene to form silica microspheres. The details of the synthesis were discussed by Wang et al. 2010; Zhuang et al. 2009; Jal et al.2004. The size distribution of the silica microspheres as determined by Zetasizer nano (ZS) showed mean value of 220 ± 20 nm.

Characterization of silica microsphere

Figure 1 shows the attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectrum of the microspheres taken after separation by ultracentrifuge at 35,000 rpm [Model: Ultra 5.0, Make; Hanil, Korea]. Vibrational band at $1,100 \text{ cm}^{-1}$ in Fig. 1 indicates the presence of Si-O vibrational band. The surface morphology of the product was studied using scanning electron microscopy (SEM) coupled with energy dispersive X-ray fluorescence spectrometry (EDS) (Instrument Model No VEGA MV 2300T,). Two typical SEM images of the silica microsphere sample were shown in Fig. 2a, b. Image recorded after ultrasonication is shown in Fig. 2a from which it is clear that these microsphere are spherical nanoparticles, which are predominately agglomerated in aggregates with no specific shape as shown in Fig. 2b. Measurements in several SEM images obtained from distinct areas in the specimen showed that the average size of the SiO₂ particles is about 200–240 nm, with a very narrow size distribution. Chemical analysis conducted by the EDS



Fig. 2 SEM images of the silica microsphere (a, b) revealing the spherical morphology of the silica microsphere and their agglomeration





30

Volume (mL)

method confirmed that the particles are comprised of pure SiO_2 , with no indication of impurity elements (Frank et al. 1998; Guangyu et al. 2007).

0

10

20

pH optimization for sorption of U and Th by silica microsphere

In order to have effective sorption of U and Th by the silica microsphere, pH was optimized. The sorption profile of U and Th at concentration of 100 Bq L⁻¹ was observed at different pH. Experimental evidence clearly indicates that at pH 3 more than 99 % sorption of U and Th took place. In order to understand the various species of uranium present at pH 3, 5 and 6, work carried out by Wolery (1992) using computer program EQ3/6 was used. As per this uranium speciation model at 4×10^{-4} M, initial uranium solutions showed that the uncomplexed uranyl ion UO₂²⁺ dominates the U(VI) speciation at pH 3 and with the increase in pH, fraction of UO₂²⁺ decreases sharply (Meinrath 1996, 1997; Elliot et al. 1986; Erdem et al. 2004; Reza et al. 2009). There is no change in sorption

was observed with a change in pH. This is because chemical properties of Th are relatively simple. Th has only one stable oxidation state, IV, under all redox conditions in natural waters, and Th(IV) ions are extremely particle reactive. The reactions and equilibrium equations for sorption of Th on silica are very similar to the proton exchange (Östhols 1995; Osthols et al. 1997; Quigley et al. 1996).

40

50

60

Probable mechanism for thorium sorption on silica surface

$$2 \equiv SiOH + Th^{4+} + H_20 = (\equiv SiO)_2Th^{2+} + 2H^+$$

 $2 \equiv$ SiOH denotes a combination of two surface sites on the silica surface which are close enough to simultaneously chemically bind a sorbing Th⁴⁺ ion. However, at pH 8 and above, the prevailing inorganic species of Th in groundwater are hydroxo and carbonato complexes, in approximately equal proportions. In fact, in organic-free solutions, thorium, an A-type metal ion, will strongly hydrolyze to form polyhydroxy colloidal complexes (Donat et al. 2005; Barbier et al. 2000).



Fig. 4 a Model (Langumir) fitting of the experimental data for sorption of uranium at pH 3. b Model (Langumir) fitting of the experimental data for sorption of thorium at pH 3



Variation of sorption of U and Th on silica microsphere at different concentrations of U and Th

Figure 3a gives the sorption profile of uranium and thorium at pH 3 at six different concentrations $(100-1,000 \text{ ng mL}^{-1})$ of U and Th. All the experiment was carried out either with U or Th. From this Figure, it is evident that more than 99 % of U and Th got sorbed on the silica microspheres within 30 min.

Desorption of U and Th from silica microsphere

The desorption of U and Th from the silica microsphere was done by 30 mL of 3 M HNO₃. Figure 3b shows the elution curve. From this elution curve, it is clear that only 95 % sorbed U and 70 % sorbed Th ions will be extracted back from the silica microsphere. Lower desorption of Th is due to highly particle reactive nature of Th^{4+} , because of it Th is tightly bound within the silicate structure. After elution, the microsphere can be reused for the subsequent



sorption of U and Th. Microsphere was found suitable for 30 cycles for U and 20 cycles in case of Th. Thereafter, 25 % degradation in the sorption capacity of silica microsphere was observed.

Evaluation of the sorption capacity of the silica microsphere

Sorption capacity of the microsphere was evaluated by carrying out the U and Th sorption experiments in the batch mode at pH 3, and each time the level of uranium and thorium was increased with an increment of 5 mg L^{-1} of the sample was processed after initial equilibration time of 30 min. After increasing the concentrations of U and Th above 30 mg and 36 mg, respectively, elevated levels of U and Th were observed in the equilibrium solutions. Therefore, the sorption capacity evaluated as 0.12 and 0.16 mmol g⁻¹ (on wet basis) in case of uranium and thorium respectively. Experimentally observed sorption capacity of uranium and thorium are very close to the

Langumir constant Q_0 (see Fig. 4a, b), which is measure of the monolayer adsorption capacity, as obtained, 30 ± 0.15 and 36 ± 0.15 mg g⁻¹. As clear from the Fig. 4a, b and R^2 values, experimental data are very close to the Langmuirtype adsorption model. As per the work carried out, Yousefia and coworker the maximum sorption capacity of the modified MCM-41 was found to be 47 and 49 mg g⁻¹ for uranium (VI) and thorium (IV), respectively. Though these materials shows better sorption capability for uranium and thorium, but only disadvantage is elevated material cost, and the impact on drinking water quality was not tested in their work (Yousefia et al. 2009).

Monitoring of the drinking water quality

After having decontamination of U and Th from the water samples by using silica microsphere, various physicochemical characteristics of the process water were monitored. As its clear from Table 1 that there is only small variation in the parameters as compared to the feed solution. In view of this, it is inferred that effective decontamination was carried out without disturbing the water quality parameters.

Conclusion

Silica microspheres were synthesized using PVP and TEOS and applied as an effective sorbent for the removal of uranium and thorium from potable water in a batch process. The results of this study clearly showed the potential of this method to decontaminate various aquatic streams from U and Th without disturbing the water quality.

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