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Scientific paper

Solid-Phase Extraction Method by Magnetic Nanoparticles Functionalized with Murexide for Trace U(VI) from Sea Water Prior to Spectrophotometric Determination

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Abstract

In this study, magnetic nanoparticles (Fe_3O_4/SiO_2/APTES) functionalized with murexide were used for the determination of uranium(VI) in sea water by spectrophotometric method in perchloric acid medium using Arsenazo-III as chromogenic reagent. The effects of some analytical parameters, such as pH, contact time, and eluent volume, on the recovery of uranium(VI) were examined in synthetic sea water. The optimum conditions were achieved with a 15 min adsorption time and 2 min elution time with 1 mL of 5 mol L^{-1} $HClO_4$ at pH of 6.5 and 25 mg of the magnetic sorbent. The linear range, detection limit, and precision (as RSD%) of the method were found to be 0.02–4.0 mg L^{-1}, 0.001 mg L^{-1} and 3.0%, respectively. The proposed method is simple, rapid, and cost-effective for the determination of U(VI) in sea water, with a total analysis time of approximately 30 min. The adsorption isotherm was well fitted to the Langmuir model, with a correlation coefficient of 0.9997 and Q_{max} value was found to be 77.51 mg g^{-1}. The magnetic sorbent was successfully used for the rapid determination of trace quantities of U(VI) ions in different sea waters, and satisfactory results were obtained.

Keywords: Magnetic nanoparticles; sea water; uranium(VI)

1. Introduction

In recent years, environmental pollution with toxic elements, such as uranium, has increased considerably. Uranium and its associated compounds are carcinogenic, dangerously toxic, and radioactive.[1–4] Furthermore, it can cause respiratory diseases, such as fibrosis and emphysema, and even cause irreversible effects in some tissues, such as the kidneys. Uranium is found in sea water at 3 μg L^{-1} and at approximately 0.0004% in the Earth's crust.[5] In many countries, the uranium concentration in drinking water is determined to be 0.03 mg L^{-1}, according to the United States Environmental Protection Agency.[6] Currently, the determination of uranium in environmental samples is crucial due to applications of uranium in areas, such as in the products of nuclear energy, catalysis, and nuclear weapons. The determination of trace uranium in complex samples and natural waters is a challenging task. Most instruments are not sensitive enough to allow for its determination at very low concentration levels in complex matrix such as sea water. For example the heavy salt matrix reduces sensitivity in direct determinations from sea water (ca. 3.5% salt). Therefore, a separation and preconcentration step is commonly applied before instrumental analysis.[1,6–9] Preconcentration/separation techniques, such as solid phase extraction (SPE),[3,10–13] liquid-liquid microextraction (LLME),[14] and cloud point extraction (CPE)[15–17] are used for the determination of uranium in various samples. SPE has commonly been used as a technique for preconcentration/separation due to its higher enrichment factor and practicality. In SPE, Amberlite-XAD, modified silica gels, mesoporous silica, and nanomaterials are commonly used as adsorbents.[18–21] Most of these sorbents have disadvantage such as low sorption capacities or efficiencies. Recent studies show that nanomaterials exhibit perfect sorption capacity. But the high dispersibility of nanomatereials in aqueous solutions makes it difficult to separate sorbents from aqueous phase after saturated sorption, which limits their real application in large volumes of waters.[22] Recently, nanosized iron oxide particles have become an important absorbent in SPE because they show

magnetic properties, as well as the general properties of nanomaterials. Furthermore, the use of magnetic nanoparticles in SPE has many advantages compared to other adsorbents. For example, magnetic nanoparticles are easily separated from solution with the use of a magnet, low toxicity, and the loss of adsorbent is minimal during the separation.[23–27] Aside from these advantages, raw Fe_3O_4 nanoparticles have several disadvantages, such as oxidation, aggregation tendencies, and low selectivity. However, magnetic nanoparticles can be modified by special ligands to overcome these problems. Magnetic nanoparticles modified with sulfur and nitrogen-containing ligands are preferred because heavy metals react with these ligands strongly and rapidly.[28–30] Murexide is one of these ligands.[18,31]

In this study, for the quantitative determination of uranium in seawater, a simple and rapid method was developed using an Fe_3O_4 nanoparticles modified with murexide. Several experimental parameters, such as pH, contact time, eluent concentration, and sorption capacity, were examined, and the developed method was then applied to real sea water samples.

2. Experimental

2. 1. Chemicals and Reagents

All chemicals used were of analytical reagent grade, and ultrapure water was used throughout the study. Tetraethyl orthosilicate (TEOS), 3-triethoxysilylpropylamine (APTES), iron(II) sulfate heptahydrate, methanol and ethanol were provided by Sigma-Aldrich. Ammonium hydroxide (25%), iron(III) chloride, hydrochloric acid, $UO_2(NO_3)_2 \cdot 6H_2O$, dimethyl sulfoxide (DMSO), and murexide were purchased from Merck. Arsenazo-III was obtained from Fluka.

2. 2. Apparatus

The UV–Vis spectra were recorded using a Shimadzu 3600 spectrophotometer. A Selecta brand pH metre was used for all pH measurements. A Biosan multi rotator was employed for the effective mixing of sorbent and solution.

2. 3. Synthesis of Murexide Functionalized Magnetic Nanoparticles

Fe_3O_4 nanoparticles (Fe_3O_4 NPs) were synthesized with an eco-friendly method, modified from Gautam et al. Briefly, $FeCl_3 \cdot 6H_2O$ (6.1 g) was dissolved in deionized water (100 mL), followed by the addition of a few drops of concentrated HCl to prevent $Fe(OH)_3$ precipitation. $FeSO_4 \cdot 7H_2O$ (4.2 g) was then added to the mixture and heated to 90 °C, followed by the rapid addition of NH_4OH (10 mL, 27%), with the solution kept at a pH of 10.0. The mixture was stirred at 90 °C for 30 min and cooled to room temperature. The resulting solid black substance was collected with a strong magnet and washed several times with ethanol and deionized water. The Fe_3O_4 NPs were then dried under vacuum at 60 °C.

To prepare core–shell nanoparticles (Fe_3O_4/SiO_2), the Fe_3O_4 nanoparticles (0.50 g) were dispersed in a solution of ethanol (80 mL) and deionized water (20 mL) by sonicating for 30 min. Then, ammonia solution (5 mL, 27 wt %) and TEOS (4 mL) were added sequentially. The mixture was stirred and allowed to react for 6 h at room temperature. The product, Fe_3O_4/SiO_2, was collected by a magnet, washed several times with deionized water, and dried under vacuum at 60 °C for 8 h. Fe_3O_4/SiO_2 nanoparticles (1 g) were dispersed in 50 mL of toluene in a flask. After 1 h, APTES (4 mL) was added to the mixture, stirred continuously, and refluxed at 125 °C for 12 h. The magnetic nanoparticles (Fe_3O_4/SiO_2/APTES) were separated with a strong magnet and washed several times with deionized water and ethanol, then dried at 70 °C for 8 h.

In the third step, Mu (0.1 g) was dissolved in DMSO (50 mL), and Fe_3O_4/SiO_2/APTES (1 g) was added to the reaction mixture and refluxed at 200 °C for 24 h. The resulting product was separated, washed several times with methanol, and dried at room temperature.

2. 4. Procedure

The method was tested with synthetic sea solutions prior to its application to real sea samples. For this purpose, the synthetic solutions containing the main components present in synthetic sea water (SSW) were prepared at the following concentrations: Na^+ = 10569 mg L^{-1}; Mg^{2+} = 1270 mg L^{-1}; K^+ = 379 mg L^{-1}; Ca^{2+} = 397 mg L^{-1}; BO_2^- = 18 mg L^{-1}; Cl^- = 18990 mg L^{-1}; HCO_3^- = 139 mg L^{-1}; SO_4^{2-} = 2648 mg L^{-1}; Br^- = 65.5 mg L^{-1}; and F^- = 14 mg L^{-1}.[13] Fe_3O_4/SiO_2/APTES (25 mg) was transferred to a 50-mL volumetric flask, and synthetic sea water solutions (40 mL) were added (U(VI): 0.05 mg L^{-1}). The pH was adjusted to 6.5 with 0.01 M CH_3COOH/NH_3. The solutions were shaken and allowed to stand for 15 min at room temperature. The magnetic sorbent was separated from the suspension using a powerful magnet and supernatant was decanted. 1.0 mL of 5 mol L^{-1} $HClO_4$ was added to the magnetic sorbent with shaking for 2 min to elute the U(VI) ion. The magnetic sorbent was separated from the eluent using a magnet.

U(VI) ion in eluent was determined spectrophotometrically in perchloric acid medium using Arsenazo-III as chromogenic reagent.[13] To this end, an Arsenazo-III solution (0.1 mL, 0.1%) was added to eluent solution, and the absorbance of the uranium(VI)–Arsenazo-III complex was measured spectrophotometrically (653 nm). Finally, the magnetic sorbent was washed with deionized water for reuse.

3. Results and Discussion

3. 1. Characterization of Fe_3O_4/SiO_2/APTES Functionalized with Murexide

Scanning electron microscopy (SEM) studies were performed on a Tescan Mira 3XMU with an Oxford EDS analysis system. As shown in Figure 1, the spherical structure of the Fe_3O_4 NPs changed after modification. The surface of Fe_3O_4/SiO_2/APTES functionalized with murexide had a rough morphology compared with Fe_3O_4. SEM images of Fe_3O_4 and Fe_3O_4/SiO_2/APTES functionalized with murexide are shown in Figure 1.

Elemental analysis showed the presence of C, N and Si in the structure of the modified magnetic nanosorbent. According to the EDS analysis, Fe_3O_4 modified with APTES and Mu contains C: 6.71%, N: 2.90%, Si: 4.06%, O: 49.17%, and Fe: 37.1% (Figure 2b).

Infrared absorption measurements of Fe_3O_4 and Fe_3O_4/SiO_2/APTES were carried out using a Fourier Transform Infrared (FTIR) spectrophotometer (Bruker Optics – Alpha). The FTIR spectra were obtained in the wavenumber range 500–4000 cm^{-1} using single bounce ATR with selenium crystal. The absorption peaks at 550 cm^{-1} (Fe-O) in the spectra of Fe_3O_4 NPs confirmed the synthesis of Fe_3O_4 nanoparticles.[32,33] On the other hand, the peaks observed at 1045 cm^{-1} (Si-O), 1450 cm^{-1} (C=N), 1530 cm^{-1} (C=C) and 1630 cm^{-1} (C=O) in the spectra of Fe_3O_4/SiO_2/APTES/Mu have shown the successful modification of Fe_3O_4 with silan agents and Mu.[33]

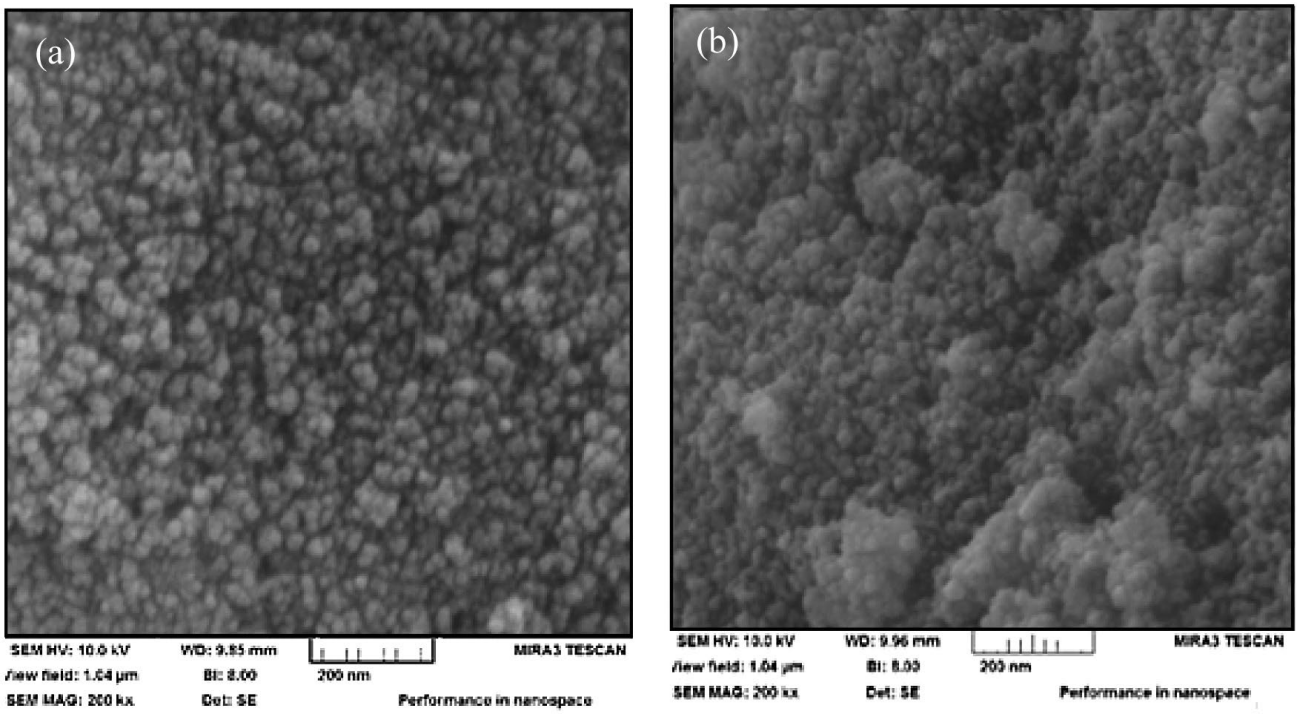

Fig. 1. The Scanning Electron Microscopy images of (a) Fe_3O_4 (b) Fe_3O_4/SiO_2/APTES functionalized with murexide.

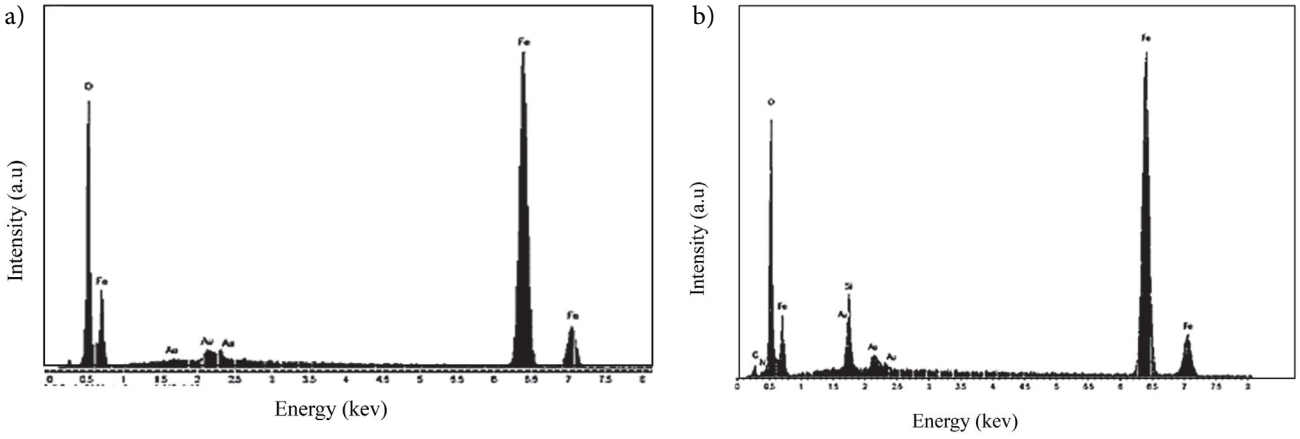

Fig. 2. Energy Dispersive X-Ray Spectroscopy analysis images of (a) Fe_3O_4 (b) Fe_3O_4/SiO_2/APTES functionalized with murexide.

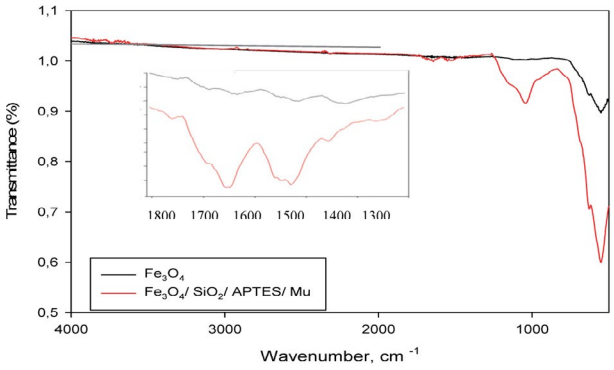

Fig. 3. The FTIR spectra of Fe_3O_4 and Fe_3O_4/SiO_2/APTES functionalized with murexide (the FTIR spectrum of wavenumber 1300–1800 cm^{-1} is shown ininner figure).

3. 2. Effect of pH

In the SPE, an important parameter for obtaining the quantitative adsorption and recovery of trace elements is pH. For this purpose, the adsorption of uranium ions on Mu-functionalized Fe_3O_4/SiO_2/APTES sorbent was studied as a function of pH. The pH of the model solutions (40 mL, SSW) containing 50 μg L^{-1} of U(VI), was adjusted to a pH range of 4–8 by the use of relevant buffer solutions; the retained uranium ions were eluted by $HClO_4$ (1 mL, 5 mol L^{-1}). The graph of retention as a function of pH is shown in Fig. 4. The quantitative recovery (≥ 95%) for the uranium ions studied was obtained at a pH of 6–7. Therefore, a pH of 6.5 was chosen as an optimum pH for subsequent experiments.

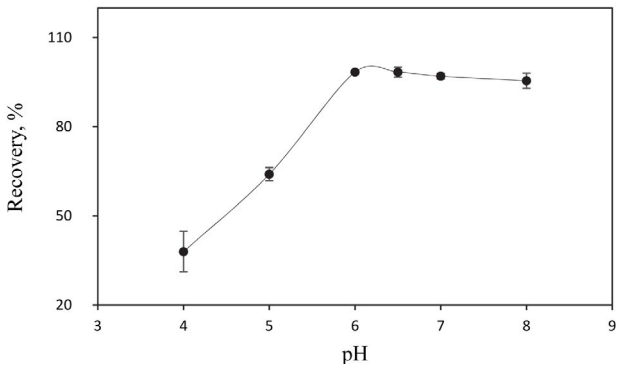

Fig. 4. Effect of pH on recovery % U(VI) with Fe_3O_4/SiO_2/APTES/Mu.

3. 3. Effect of Eluent Concentration and Volume

In this study the elution of uranium was studied to find the optimum amount of $HClO_4$ in the range of 2–5 M and volume of 0.5 to 2 mL. 1 mL of 5 M $HClO_4$ was found to be satisfactory for elution of uranium (recovery ≥ 95%). Therefore, 1 mL of 5 M $HClO_4$ as eluent was chosen for the following experiments.

3. 4. Effect of Matrix Components

The effects of matrix ions, which are found at high concentrations in real samples, on the recovery of metal ions were studied. Various concentrations of Fe^{3+}, Cd^{2+}, Pb^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Cr^{3+}, Al^{3+}, and Zn^{2+}, as their chloride, nitrate and sulfate salts, were added individually to a model solution of 50 mL containing 0.05 mg L^{-1} U(VI). The described method was applied under optimum conditions. The results are given in Table 1. The most significant interferences were found with 1 mg L^{-1} of Cr^{3+} and Ni^{2+} when determining the presence of uranium. These interferences were prevented by using 0.02 M EDTA. Besides, EDTA can be used as a masking agent for many elements such as Th, Zr, because EDTA forms stable complex with these elements, and unstable complex with U(VI).[34]

Table 1. Tolerance limits for interference ions on the determination of U(VI) (n = 3 0,05 mg L^{-1} U, 1 mg L^{-1} of metal ions)

Ion	Interference ion to metal ion/ratio	Recovery %, U(VI)
Zn^{2+}	20	101.4 ± 4.2
Cd^{2+}	20	97.5 ± 1.8
Pb^{2+}	20	99.2 ± 0.2
Fe^{3+}	20	100.2 ± 4.3
Al^{3+}	20	96.0 ± 2.3
Cr^{3+}	20	81.1 ± 0.7
Cr^{3+} + 0.02 M EDTA	20	96.2 ± 0,4
Ni^{2+}	20	86.8 ± 2.4
Ni^{+2} + 0.02 M EDTA	20	97.8 ± 0,5
Cu^{+2}	20	95.4 ± 1.2

3. 5. Effect of Adsorption and Elution Time

The rate of U(VI) adsorption by Fe_3O_4/SiO_2/APTES/Mu was studied (50 mL, 0.05 mg L^{-1}) with 25 mg of the sorbent over a series of varying shaking times (5–30 min). The results showed that the extraction percentage of U(VI) at 15 min was higher than 98%. The rate of elution of U(VI) by Fe_3O_4/SiO_2/APTES /Mu was studied (50 mL, 0.05 mgL^{-1}) with 25 mg of the sorbent and an adsorption of 15 min over a series of varying shaking times (1–5 min). Therefore, 15 min and 2 min, respectively, were used in all subsequent experiments for quantitative sorption and elution of U(VI).

3. 6. Sorption Capacity

The maximum sorption capacity of Fe_3O_4/SiO_2/APTES/Mu was obtained from the batch methods. A total of 25 mg of Fe_3O_4/SiO_2/APTES/Mu was added to a 40-mL

solution containing different amounts of U(VI) ions (0.8–8 mg) at pH 6.5. After shaking for 1 h, the mixture was separated with the use of a magnet. The supernatant solutions were then measured by UV-Vis spectrophotometry after dilution. Many isotherm models have been proposed to explain the adsorption equilibrium, such as the Langmuir and Freundlich isotherms, which are the most commonly used for the clarification of adsorption of molecules from the liquid phase. The Langmuir equation is given as follows:

$$\frac{C_e}{Q_e} = \frac{1}{Q_{max}} C_e + \frac{1}{KQ_m} \quad (1)$$

where Q_{max} (mg g^{-1}) is the maximum adsorption capacity; Q_e is the amount of solute adsorbed per unit weight of adsorbent (mg g^{-1}) at equilibrium; C_e is the equilibrium solute concentration (mg L^{-1}) in solution and K is the Langmuir constant (L mg^{-1}).

The Freundlich isotherm equation is given below:[35,36]

$$Q_e = K_f C_e^{\,1/n} \quad (2)$$

where Q_e is the amount of adsorbed U(VI) per mass of adsorbent, K_f is the Freundlich constant, C_e is the equilibrium U(VI) concentration and $1/n$ is a constant related to the adsorption intensity.[37]

As shown in Table 2, the adsorption mechanism was well-suited to the Langmuir model, with a correlation coefficient of 0.9997. The Q_{max} value was found to be 77.51 mg g^{-1}. The n value was 3.87, calculated from the Freundlich isotherm, which is higher than 1. The n value indicated the favourable adsorption of U(VI) on Fe_3O_4/SiO_2/APTES/Mu. The Langmuir and Freundlich isotherm parameters are shown in Table 2.

3. 7. Analytical Performance and Applications to Real Sea Water Sample

The limit of detection (LOD) study was performed by applying the described method to ten blank solutions of 40 mL. The limit of detection calculated as the ratio of the three standard deviations of the blank to the slope of plot was 0.001 mg L^{-1} with a preconcentration factor of 40. The relative standard deviation was calculated as 3.0% at 0.05 mg L^{-1} of U(VI) (n = 7) and the linear range in final eluate was 0.02–4.0 mg L^{-1} of uranium(VI).

The method was successfully applied to sea water. The accuracy of the developed method for sea water was tested by adding the known amounts of U(VI). After applying the separation/ preconcentration procedure, quantitative recovery (≥95%) was found for U(VI). The results of the analysis of sea water samples are shown in Table 3.

Table 3. The results for determination of U(VI) in sea water

Sample	Added (μg L^{-1})	Found (μg L^{-1})	Recovery, %
Sea water from	0	2.7±0.1	
the Aegean Sea	20	22.1±0.4	97.0±2.0
	40	44.2±0.5	104.0±1.2
Sea water from	0	≤DL	
the Mediterranean	20	19.3±1.0	96.7±5.3
	40	40.5±0.8	101.1±2.7

3. 8. Reusability of the Adsorbent

The reusability of the Fe_3O_4/SiO_2/APTES/Mu adsorbent was investigated by adsorption and desorption cycling experiments. The results have shown that the sorbent was stable up to 86 cycles without an obvious decrease in the recoveries. The mean recovery ± standard deviation from 86 runs was found to be 97.6 ± 3.6%. This result indicates that the adsorbent possessed a perfect reusability.

4. Conclusions

In this paper, Fe_3O_4/SiO_2/APTES/Mu was prepared. Then SPE procedure was developed by using these magnetic nanoparticles. The proposed SPE method is simple, fast, practical, and low-cost. The SPE method has a good potential for the extraction of uranium(VI) from sea water. Significant advantages of this method are a short analysis time and satisfactory results in sea water, which has a high salt concentration. In comparison to other SPE methods, the presented method has a low consumption of time, with a total analysis time of approximately 30 min, including the enrichment/separation procedure and the measurement by spectrophotometry. The adsorbent has considerable reusability. The initially synthesized Fe_3O_4/SiO_2/APTES/Mu was used for optimization studies and for a sample application. The adsorbent was reused for 86 cycles. The obtained results show that Fe_3O_4/SiO_2/APTES/Mu has a good adsorption capacity (77.51 mg g^{-1}). As a result, Fe_3O_4/SiO_2/APTES/Mu is indeed an efficient scav-

Table 2. Langmuir and Freundlich isotherm parameters

	Langmuir Parameters			Freundlich Parameters		
	Q_{max}(mg g^{-1})	K (L mg^{-1})	R^2	K_f (L mg^{-1})	n	R^2
U(VI)	77.51	0.896	0.9997	26.22	3.87	0.9455

enger for U(VI) in sea water in terms of its fast sorption time, large sorption capacity, selectivity, easy separation and good reusability of the material.

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Povzetek

V tej raziskavi smo uporabili magnetne nanodelce (Fe_3O_4/SiO_2/APTES), funkcionalizirane z mureksidom, za določanje urana(VI) v morski vodi s spektrofotometrično metodo v perklorno-kislinskem mediju z Arsenazo-III kot kromogenim reagentom. Vpliv nekaterih analiznih parametrov, kot so pH, kontaktni čas in volumen eluenta, na izkoristek ekstrakcije urana(VI) smo raziskovali v sintetski morski vodi. Optimalne pogoje smo dosegli z adsorpcijskim časom 15 min in elucijskim časom 2 min pri eluciji z 1 mL 5 mol L^{-1} $HClO_4$ pri pH 6,5 in s 25 mg magnetnega sorbenta. Linearno območje, meja zaznave in natančnost (kot RSD%) metode so bili: 0,02–4,0 mg L^{-1}, 0,001 mg L^{-1} in 3,0 %. Predlagana metoda je preprosta, hitra in cenovno ugodna za določanje U(VI) v morski vodi s skupnim časom analize približno 30 min. Adsorpcijska izoterma se je dobro prilegala Langmuirjevemu modelu s korelacijskim koeficientom 0,9997 in vrednostjo Q_{max} 77,51 mg g^{-1}. Magnetni sorbent smo uspešno uporabili za hitro določitev U(VI) ionov v sledovih v različnih vzorcih morske vode ter dobili zadovoljive rezultate.