

# Radionuclide removal from aqueous solutions using potassium ferrate(VI)

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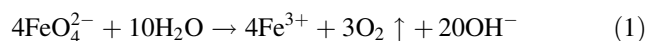
**Abstract** The paper describes the effectiveness of uranium(VI) and europium(III) removal from aqueous solutions using potassium ferrate(VI) at different pH values. The removal of a mixture of alpha- and beta-emitting radionuclides (<sup>137</sup>Cs(I), <sup>90</sup>Sr(II), <sup>152</sup>Eu(III), <sup>243</sup>Am(III), <sup>239</sup>Pu(IV), <sup>237+239</sup>Np(V), <sup>238+233</sup>U(VI)) from synthetic fresh water and simulated seawater has been checked as well. There is an indication that potassium ferrate(VI) could be used as an effective scavenger for almost all investigated radionuclides except cesium.

**Keywords** Potassium ferrate(VI) · Radionuclides removal · Radionuclides sorption · Water treatment

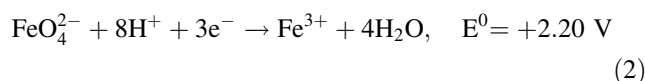
## Introduction

The acute problems of pollution of natural waters with radionuclides exist in many regions over the world [1–5]. These problems are extremely important for areas around acting and abandoned uranium ores mining (<sup>238</sup>U, <sup>226</sup>Ra, <sup>232</sup>Th etc.) and nuclear facilities after accidents (artificial radionuclides: <sup>137</sup>Cs, <sup>90</sup>Sr, <sup>239</sup>Pu etc.). One of the common ways to remove radionuclides and non-radioactive heavy metals from aqueous solutions is their sorption and/or co-

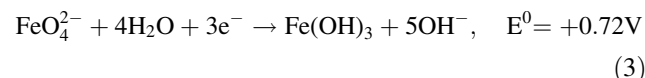
precipitation onto iron(III) oxy/hydroxides accompanying with subsequent coagulation and sedimentation [6–10]. This process requires addition of iron(III) salts to the solution and further increase of the pH value by sodium hydroxide or ammonia solutions [6, 7]. Potassium ferrate(VI), K<sub>2</sub>FeO<sub>4</sub>, interacts with water resulting in formation of iron(III) hydroxides without any additional reagents:



Under acidic conditions ( $E^0$  redox potential vs. SHE):



Under basic conditions:



Strong oxidizing action of iron(VI) and coagulation of its reduction products are the two main properties of the potassium ferrate(VI) which determine its possible application in drinking water treatment and decontamination of polluted groundwaters [11–13]. Ferrates(VI) can be used for the effective removal of metals [14–24]. Recently ferrates(VI) have been extensively studied for the organic pollutants oxidative destruction [25–36]. They have great potential for color removal (degradation of humic substances) and water disinfection (killing of bacteria). As far as heavy metals, including actinides, have tendency to form stable water soluble complexes with natural organic matter [37–39] and to be concentrated by microorganisms either due to sorption onto their membrane or due to uptake into cells [40–42], the aforementioned properties of ferrates(VI) can have a possible positive effect on removal of such

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metals. One of the advantages of the ferrates(VI) is the homogeneous distribution of an active agent (hexavalent iron) in the bulk of the solution and, thus, the efficient capture of contaminants by aggregated particles of ferrihydrite that are formed as a result of ferrate(VI) reduction [20].

Potassium ferrate(VI) can effectively remove europium, neptunium, plutonium and americium from specific aqueous solutions that simulate alkaline radioactive liquid waste [16, 17, 21]. In [20, 22] the removal of Sr(II), Th(IV), Np(V) and U(VI) was investigated in a broader pH range. It was shown that treatment with potassium ferrate(VI) works at least in the pH range from 4 to 8. The pH values of natural waters can vary significantly from about 3 (acid lakes usually located near volcanoes) to about 11 (alkaline lakes usually containing high amounts of dissolved salts, particularly sodium, calcium, magnesium carbonates and bicarbonates). To get fuller outlook on possible ferrate(VI) application, in this work we checked the effectiveness of uranium(VI), europium(III) and cesium(I) removal from aqueous solutions at pH values from 3 to 11. We also applied  $K_2FeO_4$  to remove the mixture of alpha- and beta-emitting radionuclides from synthetic fresh and sea waters as a model for drinking water treatment.

## Experimental

### Materials and chemicals

The electrochemical approach to the synthesis of the solid ferrates(VI) is considered to be the best way for the industrial production [43]. We synthesized  $K_2FeO_4$  by this approach according to the well-known procedure [44]. On the first step of the synthesis the concentrated solution of  $FeO_4^{2-}$  was obtained by anodic dissolution of metallic iron in 40 % aqueous solution of NaOH. After that, the solution was filtrated to remove the sediment of iron(III) compounds as well as the metallic shatters which could come off the electrode during the synthesis. Then, solid KOH was dissolved in the solution to precipitate  $K_2FeO_4$ . The precipitate of  $K_2FeO_4$  was separated by vacuum-filtration and washed on the filter gradually by ethanol and sulfuric ether. The last step was the drying in vacuum.

The synthesized potassium ferrate(VI) was the easily electrifiable fine crystalline powder of a black color. It gave typical for  $FeO_4^{2-}$ -solutions purple color when dissolved in water. The quality of the product was checked by room-temperature Mössbauer spectroscopy. Mössbauer spectra were measured on a Perseus spectrometer working at constant velocities, the control and the adjustment of the spectrometer-vibrator rate being performed by laser interferometer. A common Ritverc's  $\gamma$ -source of  $^{57}Co$  in metallic rhodium matrix [45] with the activity of 0.8 GBk

**Table 1** Composition of synthetic fresh and seawater used in this study

Moderately hard fresh water		Seawater	
Component	Concentration (mg L <sup>-1</sup> )	Component	Concentration (g L <sup>-1</sup> )
NaHCO <sub>3</sub>	96.0	NaCl	21.03
CaSO <sub>4</sub> ·2H <sub>2</sub> O	60.0	Na <sub>2</sub> SO <sub>4</sub>	3.52
MgSO <sub>4</sub>	60.0	KCl	0.61
KCl	4.0	KBr	0.088
		Na <sub>2</sub> B <sub>4</sub> O <sub>7</sub> ·10H <sub>2</sub> O	0.034
		CaCl <sub>2</sub> ·2H <sub>2</sub> O	1.32
		MgCl <sub>2</sub> ·6H <sub>2</sub> O	9.50
		SrCl <sub>2</sub> ·6H <sub>2</sub> O	0.02
		NaHCO <sub>3</sub>	0.17
	pH = 7.9		pH = 8.1

was employed. The constituent of  $K_2FeO_4$  (a single line with the isomer shift  $-0.90 \text{ mm}\cdot\text{s}^{-1}$  related to  $\alpha$ -iron [46]) comprised more than 90 % of the total spectrum area. Trivalent iron impurities are present as unresolved broadened constituents comprising less than 10 % of the spectrum area.

The following radionuclides were used in this study:  $^{238+233}U(VI)$ ,  $^{237+239}Np(V)$ ,  $^{239}Pu(IV)$ ,  $^{243}Am(III)$ ,  $^{152}Eu(III)$ ,  $^{90}Sr(II)$ ,  $^{137}Cs(I)$ . Concentration of all radionuclides was c.a.  $10^{-7}$  M. Due to low concentration and low specific activity of  $^{238}U$  ( $T_{1/2} = 4.47 \cdot 10^9$  a) and  $^{237}Np$  ( $T_{1/2} = 2.14 \cdot 10^6$  a) the relatively short-lived  $^{233}U$  ( $T_{1/2} = 1.59 \cdot 10^5$  a) and  $^{239}Np$  ( $T_{1/2} = 2.36$  days) tracers were added.  $^{239}Np$  was milked from the parent  $^{243}Am$  by solvent extraction with tri-octylamine in toluene. Solutions of other radionuclides ( $^{90}Sr$ ,  $^{137}Cs$ ,  $^{152}Eu$ ,  $^{239}Pu$  and  $^{243}Am$ ) were prepared from their stock solutions in nitric acid. All other commercial reagents were of analytical grade and used as received. All solutions were prepared with deionized MilliQ water.

The synthetic fresh (moderately hard) and sea waters were prepared according to [47] as presented in Table 1.

### Experimental procedures

All experiments were carried out at ambient atmospheric conditions.

Uranium, europium and cesium removal was studied in 0.1 M NaCl solutions at different pH values. Solutions were prepared with deionized water and pH values were adjusted with 0.1 M NaOH/NaCl and 0.1 M HCl/NaCl solutions prior to addition of potassium ferrate(VI). Uranium solutions with concentration of  $10^{-7}$  M were prepared by dilution of the uranyl nitrate stock solution with concentration of  $10^{-4}$  M.  $^{233}U$  was added as a tracer to measure uranium

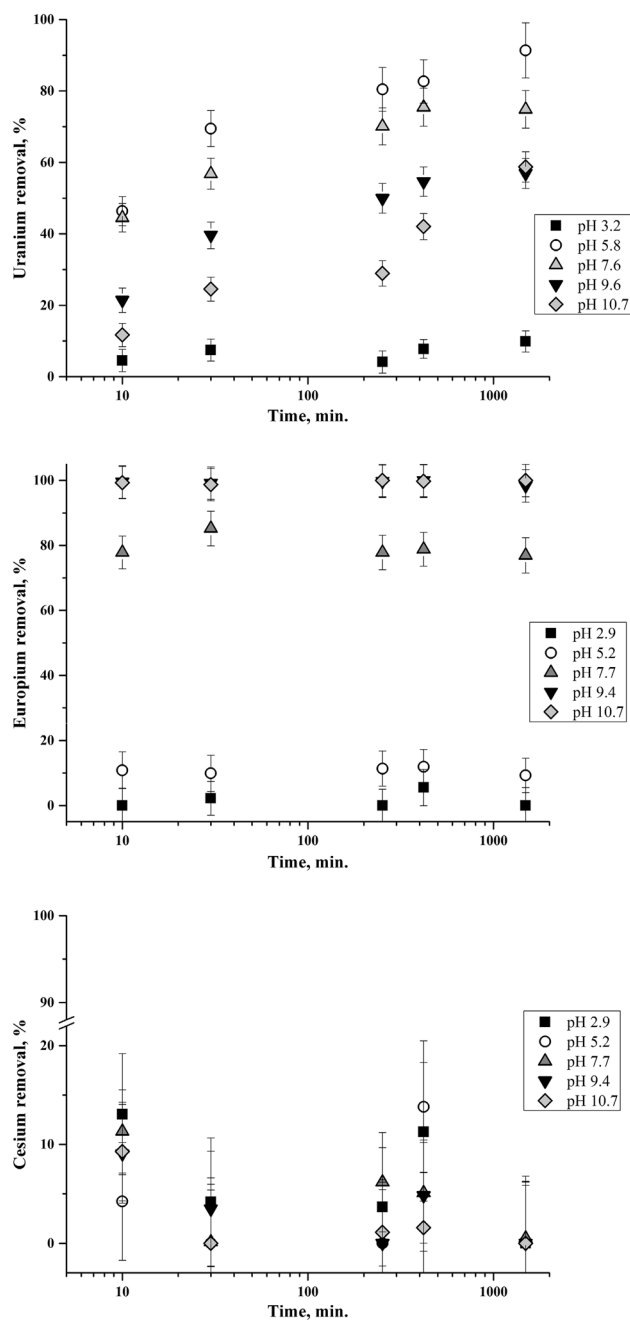
content by liquid-scintillation spectrometry (TriCarb 2500, Canberra/Packard). Europium and cesium solutions were prepared by dilution of  $\text{Eu}(\text{NO}_3)_3$  and  $\text{CsCl}$  solutions (natural content of stable isotopes).  $^{152}\text{Eu}$  ( $T_{1/2} = 13.54$  a) and  $^{137}\text{Cs}$  ( $T_{1/2} = 30.07$  a) were added as tracers to measure europium and cesium content by  $\gamma$ -ray spectrometry (high purity germanium detector, GC 3020, Canberra). The stock solution of 0.01 M potassium ferrate(VI),  $\text{K}_2\text{FeO}_4$ , was prepared by dissolving weighted amount of solid reagent in MilliQ water. The aliquots of  $\text{K}_2\text{FeO}_4$  stock solution were added to solutions containing uranium, europium and cesium. Then solutions were intensively shaken for few seconds and left to settle. The solutions were sampled periodically to determine the optimal equilibration time—i.e. time, when the steady-state is reached. All samples were centrifuged at  $40,000\times g$  within 10 min to remove particles of ferric hydroxide. Supernatants were then analyzed for the content of uranium, europium and cesium.

The second set of experiments included a mixture of  $^{137}\text{Cs}(\text{I})$ ,  $^{90}\text{Sr}(\text{II})$ ,  $^{152}\text{Eu}(\text{III})$ ,  $^{243}\text{Am}(\text{III})$ ,  $^{239}\text{Pu}(\text{IV})$ ,  $(^{237}+^{239})\text{Np}(\text{V})$  and  $(^{238}+^{233})\text{U}(\text{VI})$  in synthetic fresh water and seawater. The solutions were periodically sampled, solid particles were then separated and supernatant solutions were characterized. Concentration of  $^{90}\text{Sr}$  was determined by Cherenkov radiation after equilibration with  $^{90}\text{Y}$ .  $^{137}\text{Cs}$ ,  $^{152}\text{Eu}$ ,  $^{239}\text{Np}$  and  $^{243}\text{Am}$  content was determined by  $\gamma$ -ray spectrometry.  $^{233}\text{U}$  and  $^{239}\text{Pu}$  content was determined by alpha-spectrometry.

## Results and discussion

### U(VI), Eu(III) and Cs(I) removal from 0.1 M NaCl solutions

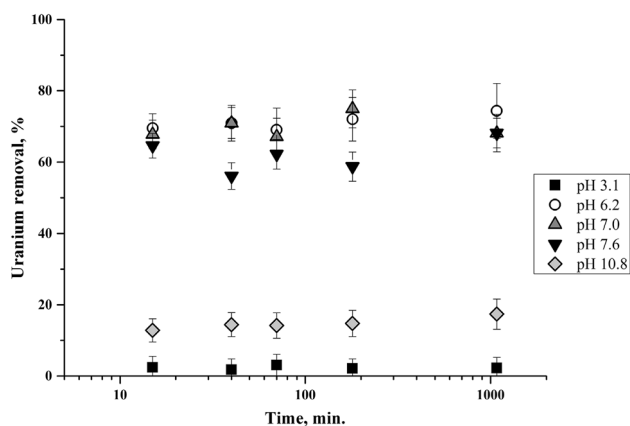
The dependence of the U(VI), Eu(III) and Cs(I) removal from 0.1 M NaCl solution at different initial pH values and initial concentration of  $\text{K}_2\text{FeO}_4$   $10^{-4}$  M versus time is shown in Fig. 1. The steady-state has been reached within 10 min of experiments in case of Eu(III) and within 7 h in case of U(VI). Removal of Cs(I) was negligible at any pH values and time intervals. The equilibrium fraction of the removed uranium(VI), europium(III) and cesium(I) is 91, 99 and  $<10\%$ , respectively, and remains constant within the 24 h. Precipitates forming due to ferrate(VI) decomposition were not specially studied. It is known from earlier work that they consist of nanosized particles of ferrihydrite [20]. The high values of the U(VI) and Eu(III) removal can be explained by their high tendency to sorb onto different mineral surfaces and formation of strong inner-sphere complexes with different surface hydroxyl groups (e.g.  $\equiv\text{Fe}-\text{OH}$ ) [48–50]. Cs(I), as other monovalent cations, has low sorption onto particle surfaces and reacts preferably via



**Fig. 1** Time-dependence of U(VI) *top*, Eu(III) *middle* and Cs(I) *bottom* removal from 0.1 M NaCl solutions at initial  $\text{K}_2\text{FeO}_4$  concentration of  $10^{-4}$  M and different initial pH values

cation exchange with clay and other minerals with layered structure [51–53] that is not inherent for ferrihydrite.

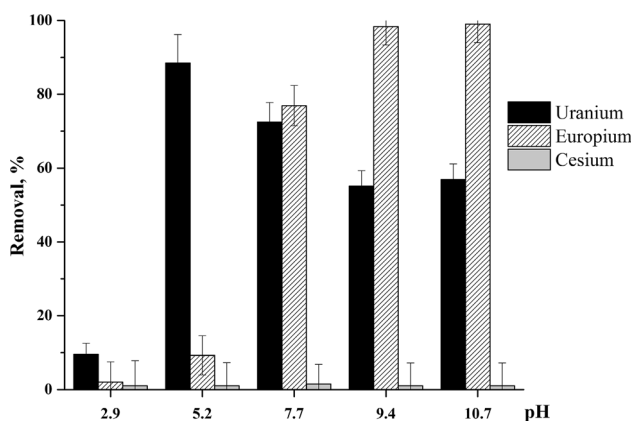
Removal of uranium(VI) at initial ferrate(VI) concentration of  $10^{-6}$  M is shown in Fig. 2. Lower concentration of  $\text{K}_2\text{FeO}_4$  results in lower uranium(VI) removal, but faster kinetic of the process: the steady-state is reached already in 10 min. This fact can be explained by partial dissolution of Fe(III) and low concentration of sorption sites at low initial



**Fig. 2** Time-dependence of uranium(VI) removal from 0.1 M NaCl solutions at initial  $K_2FeO_4$  concentration of  $10^{-6}$  M and different initial pH values

concentration of ferrate(VI). Thus, we used the concentration of  $K_2FeO_4$  as  $10^{-4}$  M for further experiments.

The removal of uranium(VI), europium(III) and cesium(I) from aqueous solutions at different initial pH values and initial concentration of potassium ferrate(VI) of  $10^{-4}$  M at steady-state is shown in Fig. 3. The complete recovery of U(VI) occurs at initial pH 6–8 and of Eu(III) at initial pH 8–11, while Cs(I) is not removed from solutions under all experimental conditions. Lanthanides(III) and actinides(III) have similar chemical properties and are usually considered as analogues. Their sorption onto iron(III) oxides becomes significant only at pH values higher than five [8, 54] that agrees with our observation on low removal of europium(III) at  $pH \leq 5.2$ . Uranium(VI) readily sorbs onto iron(III) oxides at  $pH > 4$  [8]. The hydrolysis and formation of neutral and negatively charged carbonate complexes decrease the U(VI) sorption [22]. These earlier findings are in agreement with presented here high sorption of uranium(VI) at initial pH 5–7 and its decline at higher pH values. Thus, the driving force of metals removal by



**Fig. 3** Removal (%) of uranium(VI), europium(III) and cesium(I) from 0.1 M NaCl solution at initial  $K_2FeO_4$  concentration of  $10^{-4}$  M and different initial pH values

**Table 2** Removal (%) of  $\alpha$ - and  $\beta$ -emitting radionuclides from synthetic fresh water and seawater

Moderately hard fresh water		Seawater	
Radionuclide	Removal (%)	Radionuclide	Removal (%)
<b><math>\alpha</math>-emitting radionuclides</b>			
$(^{238}+^{233})U$	$81 \pm 3$	$(^{238}+^{233})U$	$79 \pm 5$
$^{237}Np$	$66 \pm 3$	$^{237}Np$	$64 \pm 4$
$^{239}Pu$	$92 \pm 5$	$^{239}Pu$	$88 \pm 4$
$^{243}Am$	$79 \pm 4$	$^{243}Am$	$86 \pm 4$
<b><math>\beta</math>-emitting radionuclides</b>			
$^{90}Sr$	$83 \pm 4$		$75 \pm 3$
$^{152}Eu$	$78 \pm 2$		$71 \pm 1$
$^{137}Cs$	$2_{-2}^{+5}$		$1_{-1}^{+5}$

potassium ferrate(VI) is their sorption onto ferrihydrite and subsequent coagulation and sedimentation.

### Removal of $\alpha$ - and $\beta$ -emitting radionuclides from synthetic fresh water and seawater

The normative documentation for tap water usually contains requirements on maximum allowable levels of total  $\alpha$ - and  $\beta$ -activity. Here we present results on cleaning of the aqueous solutions containing a mixture of radionuclides. Removal of  $\alpha$ - and  $\beta$ -emitting radionuclides from their mixed solution in synthetic fresh water and seawater shows the same time-dependent trend—the steady-state is reached within 10 min. The initial pH value of both fresh and seawater is around 8. The results obtained for 0.1 M NaCl solutions indicate that the removal of uranium(VI) and trivalent actinides and lanthanides should be around 70–80 %. According to [20, 22] the Sr(II) and Np(V) removal can achieve 90–100 % under conditions of our experiment. The obtained experimental data are summarized in Table 2. The removal of almost all radionuclides under these conditions reaches 70–90 %. Not surprisingly, it was impossible to remove cesium(I). A bit lower removal in seawater for all radionuclides can be caused by competitive sorption of divalent cations ( $Ca^{2+}$ ,  $Sr^{2+}$ ,  $Mg^{2+}$ ) onto ferric hydroxide surface.

Thus, a simple treatment of either fresh or seawater with small amount of potassium ferrate(VI) ( $10^{-4}$  M;  $5.6 \text{ mg L}^{-1}$  of iron) can significantly lower the total  $\alpha$ - and  $\beta$ -activity.

### Conclusions

Potassium ferrate(VI) demonstrates high efficiency of radionuclide removal from aqueous solutions under different conditions. Steady-state is reached within 10–30 min after mixing the solutions containing radionuclides and

ferrate(VI). At average 80 % of all  $\alpha$ - and  $\beta$ -emitting radionuclides can be removed either from fresh water or seawater. Application of ferrates(VI) is ecologically friendly because it does not require any additional reagents, like ammonia or basics, to produce iron(III) hydroxides to sorb radionuclides. Homogeneous distribution of ferrate(VI) in solution volume at initial stage makes the removal of contaminants very efficient. These results along with oxidative and disinfecting properties of  $K_2FeO_4$  and low solubility of its reduction products meeting the criteria of maximum allowed concentration of iron in water show that potassium ferrate(VI) can be effectively used for the drinking water treatment and decontamination of polluted groundwater and liquid radioactive wastes.

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