Synthesis and characterization of magnetic Copper-Iron-Titanate and uptake studies of Americium from nuclear waste solutions

Krishnan Muthukumar^a, D. Shanthana Lakshmi^b*,Rajesh B. Gujar^c, A. B. Boricha^a, P. K. Mohapatra^{c,d**}, H.C.Bajaj^{a,***}

Electronic supportive information (ESI)

EXPERIMENTAL:

Sorption studies:

All the metal ion sorption studies were carried out by taking a known amount (ca. 10 mg) of the sorbent (TF or CTF nanomaterials) in a fixed volume of aqueous phase (usually 1 mL of pH 4 solution) and the melange was equilibrated in a thermostated water bath where samples were mounted on to a rotating plate which were subsequently rotated at a speed of 45 rpm.

²⁴¹Am was purified from the associated impurities (²³⁷Np) by an ion exchange method. Initially, hydroxylamine hydrochloride (5-10 mg) was added to the solution (ca. 2 mL of 7 M HNO₃) containing the radiotracer to adjust the oxidation state of Np to the +4 state. Subsequently, the mixture of ²⁴¹Am containing traces of ²³⁷Np was loaded to a DOWEX 1x4 resin, pre-conditioned with 7 M HNO₃. The washings were collected and used as ²⁴¹Am stock while ²³⁷Np was loaded on to the column. The purity of the tracer was confirmed by alpha-spectrometry.

²⁴¹Am and ^{152,154}Eu were assayed radiometrically using a well type NaI(Tl) scintillation counter which was interphased with a multi-channel analyzer. The counter was calibrated with known standards before the counting was done. Enough counts were collected (>10,000 counts) to neglect the counting statistics errors (<1%).



Fig-S1 PXRD of Pseudobrookite (Fe₂TiO₅) prepared at 200°C with different time

FTIR Studies:

The TF and CTF nanomaterials were analyzed by Fourier Transformation – Infrared Spectroscopy (FT-IR) using Perkin Elmer-Spectrum GX spectrometry from 400-4000cm⁻¹ with a resolution of 4cm⁻¹ using KBr pellets. As shown below, not much information was obtained for the metal-oxide bands.



Fig. S2: FT-IR spectra of the CTF and TF nanomaterials



Fig-S3 FE_SEM images of Iron precursor (IA) in methanol and TIA- iron precursor with Ti(IV) isopropoxide



Fig-S4 a, b, c and d FE-SEM of iron titanate prepared at different time interval



Fig-S5 HR-TEM image of Iron titanate prepared at different time interval



Fig-S6 Selective Area Electron Diffraction (SAED) of iron titanate prepared at different time interval

Isotherm	Linearized form	Plot	Parameters
Langmuir	$\frac{C_{eq}}{q_t} = \frac{[1]}{b \cdot q_{\max}} + \frac{C_{eq}}{q_{\max}}$	$\frac{C_{eq}}{q_t} vs \cdot C_{eq}$	$q_{max} = 1/\text{slope}$
			b = Slope/intercept
D-R	$\ln q_t = \ln X_m - \beta \varepsilon^2$	$\ln q_t \mathrm{vs} \boldsymbol{\varepsilon}^2$	$X_m = $ Intercept
			$E = 1/2\beta^{0.5}$ (slope)
Freundlich	$\log q_{eq} = \log K_f + \frac{1}{n} \log C_{eq}$	$\log q_e vs \cdot C_{eq}$	$K_f =$ Intercept
			n = 1/slope

Table-S1 Linearized form of various sorption isotherms

^a: q_{eq} and q_t are the concentration of Am(III) sorbed per gram of the nanomaterial at equilibrium and at time t, respectively; C_{eq} is the equilibrium concentration of metal ions in the aqueous phase; q_{max} and b are the maximum sorbed mass of Eu(III) at saturation and the sorption coefficient, respectively; X_m is maximum sorption capacity of the nanomaterial.

 Table-S2: Parameters of the Langmuir Model for the sorption of Eu(III) on iron

 titanate and copper doped iron titanate; Aqueous phase: pH 4; Temperature: 25°C

Nanomaterial Type	b (L·mg ⁻¹)	$q_{max} (mg \cdot g^{-1})$	R ²	Exp. q (mg·g ⁻¹)
Iron titanate	0.16 ± 0.03	5.9 ± 0.1	0.9910	6.1 ± 0.2
Copper - iron titanate	0.012 ± 0.002	20.4 ± 0.7	0.9963	21.3 ± 0.4

Table-S3: Parameters of the Freundlich Model for the sorption of Eu(III) on iron titanate and copper doped iron titanate; Aqueous phase: pH 4; Temperature: 25°C

Nanomaterial Type	K _f	R ²	1/n
Iron titanate	1.34 ± 0.02	0.8997	0.31 ± 0.03
Copper-iron titanate	2.09 ± 0.03	0.9977	0.33 ± 0.01

Leaching test:

The leaching test was carried out by contacting 1000 ppm nano material solution prepared and treated with different concentration nitric acid (0.1 M and 1 M solution). The results are included in the table S4.

Table S4: Leaching studies carried out by contacting the nanomaterials with 0.1 M and 1 M HNO₃. The aqueous phases after contacting with the nanomaterials were analyzed by ICP-OES method.

[HNO ₃] (nanomaterial)	% leaching of the components			Comment
	Cu	Fe	Ti	
0.1 M (CTF)	<1%	<1%	<1%	leaching is negligible
1 M (CTF)	~ 6.5%	>1%	>1%	Fe, Ti leaching is negligible
0.1 M (TF)	-	5%	~2%	Leaching is significant
1 M (TF)	-	6%	~2%	Leaching is significant