

MAGNESIUM POTASSIUM PHOSPHATE MATRIX FOR SOLIDIFICATION OF INTERMEDIATE LEVEL WASTE CONTAINING ACTINIDES AND AMMONIUM NITRATE

Vinokurov S. E.¹, Kulikova S. A.¹, Krupskaya V. V.², Myasoedov B. F.¹

¹Vernadsky Institute of Geochemistry and Analytical Chemistry of the Russian Academy of Sciences, Moscow, Russia

²Institute of Geology of Ore Deposits, Petrography, Mineralogy and Geochemistry of the Russian Academy of Sciences, Moscow, Russia

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The compound samples based on the mineral-like magnesium potassium phosphate (MPP) matrix were synthesized upon immobilization of the intermediate level waste (ILW) imitator containing actinides and ammonium nitrate. The mechanical strength of the prepared compound samples was 10–15 MPa, including after their irradiation with an electron beam and a source of Cs-137 (absorbed dose was 1 and 0.12 MGy respectively), after 30 cycles of freezing/thawing (–40 ...+40°C), and also after a 90-day immersion in water, which corresponds to the requirements of NP-019-15 for cement compound (≥ 5 MPa). High hydrolytic stability of the compounds in accordance with GOST R 52126-2003 was observed. It was shown that the leaching of the compound components occurs in accordance with different mechanisms, which is due to the formation of components with different solubility in the compound. Thus, the MPP matrix is promising for solidification of ILW, including high-salt actinide-containing waste of complex chemical composition.

Keywords: *magnesium potassium phosphate matrix, ammonium, cesium, strontium, plutonium, americium, intermediate level waste, immobilization.*

Introduction

Liquid radioactive waste (LRW) produced in large quantities in the nuclear fuel cycle (NFC) presents a substantial environmental hazard for the population and the environment. It is widely recognized that the future of nuclear power will depend on the effectiveness of LRW management solution. Current recommendations on LRW management suggest immobilization of LRW in solidified forms to reliably isolate the radionuclides for long periods of time and ensure radiological safety.

Implementation of the concept of safe LRW management at FSUE “PA “Mayak” covers management of waste of various composition.

At the same time, some types of waste cannot be solidified by the currently applied methods of cementation and vitrification due to non-compliance with the in-force regulatory requirements

NP-019-15 [1]. Among such cases are intermediate-level (ILW) liquid radioactive waste generated in regeneration of spent nuclear fuel at the stage of extraction affinage of plutonium and neptunium. Such ILW contain alpha-emitting nuclides with high specific activities (over 10^7 Bq/l), and ammonium nitrate, and therefore cannot be included in cement compounds in accordance with the requirements of [1]. At the same time, the waste contains large quantities of corrosion-active components (iron, sulphates) which negatively affect the quality of aluminum-phosphate glass.

Thus, there is a need for development of a new universal matrix for immobilization of LRW of complex chemical composition. A promising option is using mineral-like magnesium-potassium-phosphate (MPP) matrix ($\text{MgKPO}_4 \cdot 6\text{H}_2\text{O}$) produced

at room temperature, which is similar to natural K-struvite mineral [2]. Practical effectiveness of MPP matrix was demonstrated earlier in solidification of both simulants and real LRW of PA "Mayak" [3–5]. It was found that MPP matrix has substantial advantages over portland cement, first of all, radionuclide stability to leaching, as well as high value of solution/binder ratio (usually up to 0.8 l/kg) for high content of LRW salts (up to 30% of mass) and the capability to solidify waste in a wide pH range of 2–10. It should be noted that practical use of MPP matrix, in contrast to vitrification, does not require construction of massive electric furnaces or specialized smelters, which present a substantial radiological problem in terms of their decommissioning, which currently is not performed.

This paper presents the results of research on the capability of high-salt ammonium-containing ILW immobilization in a MPP matrix, and provides data on phase composition, structure, mechanical, thermal and radiation stability, and on hydrolytical stability of the obtained phosphate compounds and the mechanisms of their component leaching.

Experimental part

Highly acidic ILW simulant solution (solution 1 hereafter) with a density of 1.37 g/cm³ was prepared for the study. Radionuclide composition of the solution is given in Table 1. 1.

Table 1. Chemical and radionuclide composition of solution 1

| Chemical composition | Concentration, g/l | Radionuclide composition | Specific activity, Bq/l |
|-----------------------------------|--------------------|--------------------------|-------------------------|
| HNO ₃ | 300 | | |
| H ₂ SO ₄ | 150 | ¹³⁷ Cs | 7.0·10 ⁷ |
| NH ₄ NO ₃ | 265 | ⁹⁰ Sr | 9.8·10 ⁷ |
| Fe(NO ₃) ₃ | 64.8 | ²³⁹ Pu | 4.1·10 ⁸ |
| CsNO ₃ | 12.8 | ²⁴¹ Am | 5.7·10 ⁷ |
| Sr(NO ₃) ₂ | 9.6 | | |

Solution 1 was prepared for solidification by neutralization — sodium hydroxide solution (concentration (15.0±0.1) mol/l) was injected under the solution layer, followed by mixing until the pH level reached (2.0±0.1) in order to avoid ammonium excretion and iron deposition in process of hydrolysis. Ni(NO₃)₂ and K₄[Fe(CN)₆]·3H₂O were introduced to the solution for preliminary binding of cesium. The mass of reagents was preliminary calculated to obtain the required quantity of potassium-nickel ferrocyanide of 1% of the resulting compound. KH₂PO₄ with particle sizes of 0.15–0.25 mm and MgO preliminary annealed at 1300 °C for 3 hours were used for solidification of neutralized solution 1. Compound samples were prepared for solution/binder ratio of 0.68 l/kg: 0.371 kg of MgO and 1.113 kg of

KH₂PO₄ were used for solidification of 1 l of prepared solution 1. (1,3±0,1)% of mass of boric acid was added in order to reduce the binding rate of reaction mixture [6]. Filling of the resulting compound with salts of solution 1 was 20.8% of mass.

Similar method was used to synthesize compound samples in solidification of water solution containing 530 g/l of ammonium nitrate (solution 2 hereafter) with density of 1.15 g/cm³ in order to study behaviour of ammonium ions in process of ILW immobilization. Filling of the resulting compound with ammonium nitrate was 15.1% of mass.

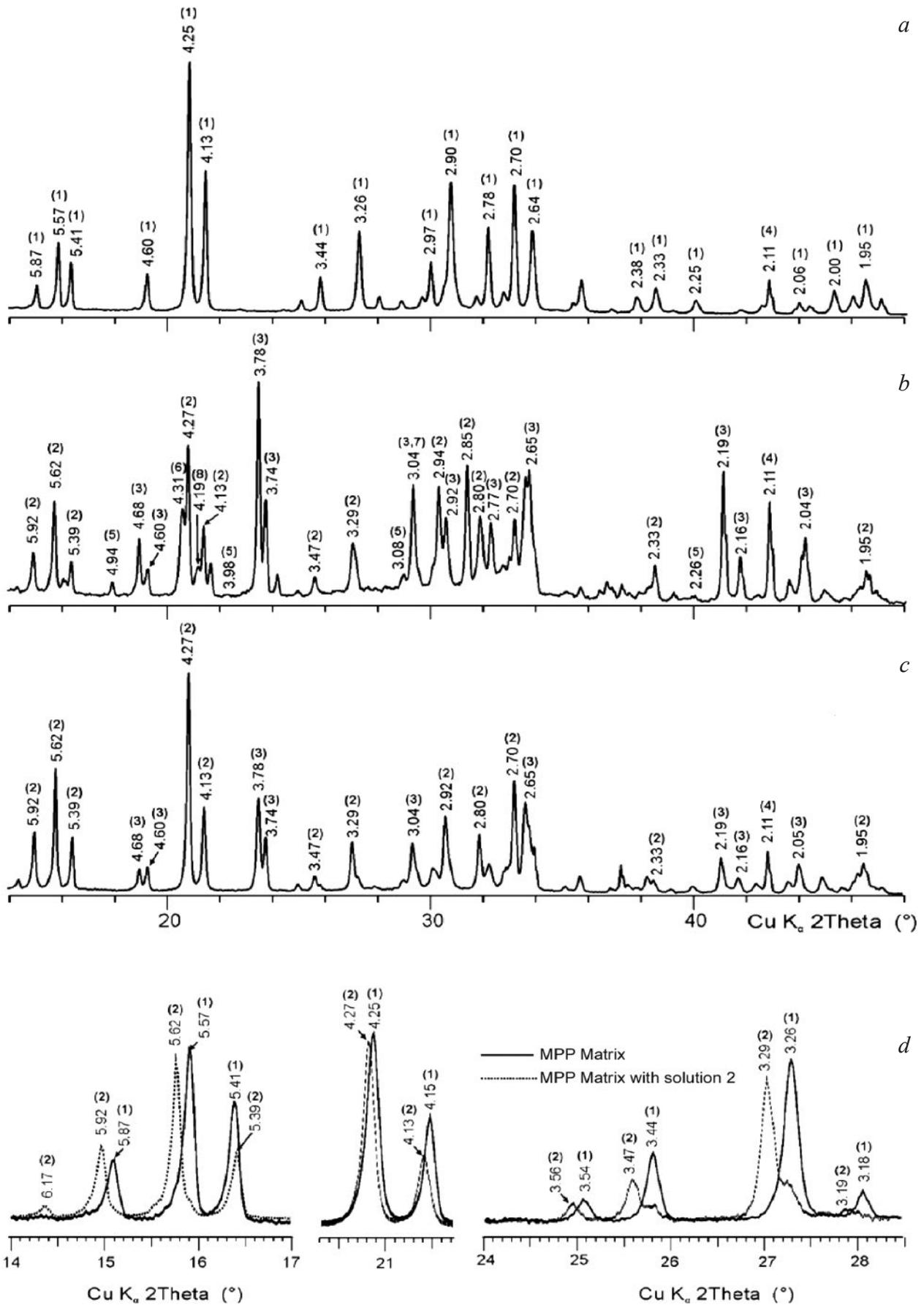
Cubic MPP compound samples with dimensions of 2×2×2 cm with density of (1.8±0.1) g/cm³ were synthesized in solidification of the studied solutions. Compound samples were left for 15 days for strengthening.

Phase composition of the resulting compounds was studied by x-ray diffraction (Ultima-IV, Rigaku); Jade 6.5 (MDI) software with powder database PDF-2 was used to interpret the obtained data. The sample structure was studied by scanning electronic microscopy using LEOSupra 50 VP (Carl Zeiss) electronic microscope and infrared (IR) spectrometry using Nicolet IR 200 (Thermo Scientific) IR spectrometer. X-ray spectral microanalysis of compounds was performed using energy-dispersive analyzer X-MAX 80 (Oxford Inst.).

Compression strength of the samples was measured in accordance with methods [7, 8]. The tests were also performed in process of study of samples stability to thermal cycles (30 freezing/thawing cycles in a temperature range (–40...+40 °C)) and radiation stability to irradiation by linear accelerator UELV-10-10-S70 with a vertical scanning electron beam (energy 8 MeV, IPCE RAN) and a ¹³⁷Cs source (MSU).

Hydrolytical stability of MPP compound with immobilized solution 1 to leaching of ILW radionuclides (¹³⁷Cs, ⁹⁰Sr, ²³⁹Pu and ²⁴¹Am) and other components was determined in accordance with half-dynamic test GOST R 52126-2003 [9]. Radionuclide quantities in the leaching solutions were measured by radiometric methods (gamma- and alpha-spectrometers, Canberra, and UMF 2000 installation). Other elements were determined by inductively coupled plasma emission spectrometry (spectrometer iCAP-6500 Duo, ThermoScientific). Ammonium quantity in leaching solutions was measured in accordance with GOST 33045-2014 [10]. Intensity of solutions colouring after introduction of Nessler reagents, which is proportional to ammonium ions concentration, was determined for peak wavelength in the range of 385–400 nm using spectrophotometer Unicam Visible Spectrometer UV 300.

Compound components leaching mechanism was studied in accordance with diffusion model [11]. The procedure of total elementary yield calculation (V, Bq/m² or mg/m²) from the compound in contact with water was described in [12]. The value of tangent of the angle of element yield logarithm



1 – MgKPO₄·6H₂O ((K)-struvite); 2 – MgNH₄PO₄·6H₂O (struvite); 3 – KNO₃ (niter); 4 – MgO (periclase);
 5 – NH₄NO₃ (nitramite); 6 – MgNaPO₄; 7 – NaNO₃ (nitratine); 8 – Na₃PO₄ (olympite)

Fig. 1. X-ray diffraction pictures of MPP matrix (a) and compounds obtained in solidification of solutions 1 and 2 (b and c respectively) and comparative data for characteristic peaks (d)

(logB) as a function of logarithm of time of its contact with water (logt) corresponds to the following mechanisms of element leaching from compound: > 0,65 – dissolution of the surface layer; 0,35–0,65 – diffusion; < 0,35 – washing out from the surface (including subsequent depletion of the surface layer).

Results and discussion

Phase composition and structure of MPP compounds. Fig. 1 shows x-ray diffraction pictures of the obtained MPP compounds, including data for $\text{MgKPO}_4 \cdot 6\text{H}_2\text{O}$ matrix given for comparison (Fig. 1a) [13].

It was demonstrated that the main crystal phase of compounds obtained in solidification of solutions 1 and 2 (Fig. 1b and 1c respectively) was a synthetic analogue of struvite mineral $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$, thus indicating that ammonium was included in the matrix structure.

Diffraction patterns of MPP matrix and a compound with immobilized ammonium in the ranges 4–17, 20.5–21.5 and 24–28.5° 2θ, where differences in peak positions are most evident, are given for comparative purposes (Fig. 1d).

It should be noted that no traces of NH_4NO_3 phase was found in the compound obtained in solidification of solution 2 (Fig. 1d), thus indicating that ammonium in the compound was present in the form of low-soluble compound with a general formula $\text{MgK}_{1-x}(\text{NH}_4)_x\text{PO}_4 \cdot 6\text{H}_2\text{O}$.

KNO_3 phase (niter) was also detected in the compound, formed by replacement of potassium with ammonium, and, possibly, sodium (for the immobilized solution 1 (MgNaPO_4 in Fig. 1b)) in the crystal lattice of MPP matrix $\text{MgKPO}_4 \cdot 6\text{H}_2\text{O}$. Another component found was MgO (periclase) due to excess of spent MgO in accordance with the method [6, 12]. The compound with immobilized solution 1 also included minor quantities of NH_4NO_3 (nitramite), NaNO_3 (nitratine) and Na_3PO_4 (olympite) (Fig. 1b). At the same time, no other crystal phases were reliably identified either due to their low content in the samples (e.g., the content of iron in the samples was 0.3% mass) or their weak crystallization.

SEM and x-ray spectral microanalysis data confirmed that the compound obtained in immobilization of solution 2, consisted of two main phases: phosphate compound with atomic ratio of potassium and ammonium up to 0.26 (corresponding to $\text{MgK}_{0,21}(\text{NH}_4)_{0,79}\text{PO}_4 \cdot 6\text{H}_2\text{O}$ composition), and potassium nitrate.

Comparison of compound IR spectra with immobilized solution 2 (Fig. 2a) and MMP matrix (Fig. 2b) in the interval of 900–1500 cm^{-1} demonstrates differences noted in [14] in process of study of natural minerals struvite and K-struvite, confirming the earlier conclusion on replacement of potassium with ammonium in $\text{MgKPO}_4 \cdot 6\text{H}_2\text{O}$ matrix. E.g. the compound spectrum (Fig. 2a) shows the band

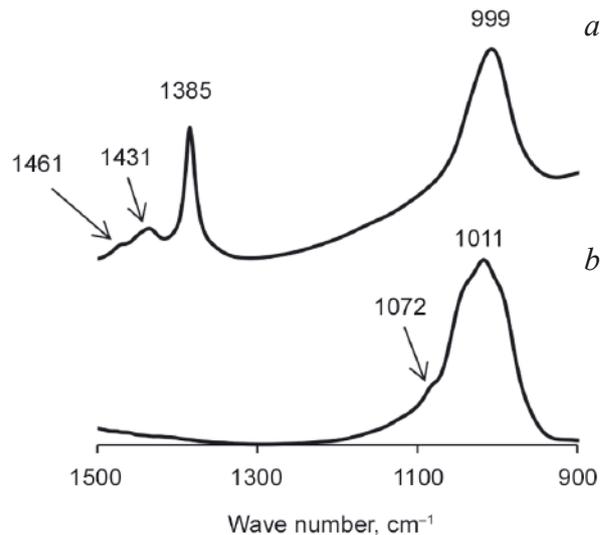


Fig. 2. IR spectra of MPP compound obtained in solidification of solutions 2 (a), and MPP matrix (b)

999 cm^{-1} , which in the case of MPP matrix was shifted to 1011 cm^{-1} , while the band 1072 cm^{-1} was absent. The spectrum also included bands 1431 and 1461 cm^{-1} , and the band 1385 cm^{-1} , characteristic for ammonium and nitrate ions respectively.

Physical and chemical and hydrolytical stability of MPP compounds. It was found that compression strength of compounds obtained in solidification of solutions 1 and 2 was (15.7 ± 3.7) and (12.2 ± 2.5) MPa respectively, which is substantially higher than the minimum allowed value for cement compound – 5 MPa [1]. At the same time the following compound parameters, also evaluated based on mechanical strength (at least 5 MPa), were proven to satisfy the requirements of [1]: radiation stability after irradiation by an electron beam or ^{137}Cs source (absorbed doses of 1 and 0.12 MGy respectively), stability to 30 thermal cycles ($-40 \dots +40^\circ\text{C}$), and water resistance after 90 days immersion in water.

Study of hydrolytical compound stability demonstrated that pH of solutions after leaching increases in the range of 7 to 10 depending on the duration of contact with water. The obtained data on compound component leaching rates is given in Fig. 3 and Table 2.

Table 2. Hydrolytical stability of MPP compound with immobilized solution 1 for 28th day of contact with water in accordance with GOST R 52126-2003 [9]

| Compound components | Leaching rate, g/($\text{cm}^2 \cdot \text{day}$); | |
|---------------------|--|---------------------|
| | differential | integral |
| ^{137}Cs | $1.6 \cdot 10^{-4}$ | $3.9 \cdot 10^{-4}$ |
| ^{90}Sr | $6.9 \cdot 10^{-6}$ | $4.9 \cdot 10^{-5}$ |
| ^{239}Pu | $4.3 \cdot 10^{-7}$ | $6.7 \cdot 10^{-6}$ |
| ^{241}Am | $1.8 \cdot 10^{-6}$ | $6.8 \cdot 10^{-6}$ |
| Na | $1.0 \cdot 10^{-3}$ | $1.4 \cdot 10^{-2}$ |
| NH_4 | $6.9 \cdot 10^{-4}$ | $1.3 \cdot 10^{-3}$ |
| Fe | $6.9 \cdot 10^{-5}$ | $8.7 \cdot 10^{-5}$ |

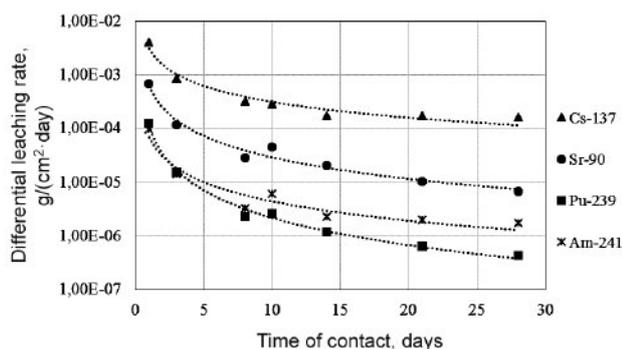


Fig. 3. Differential radionuclide leaching rate for MPP compound obtained in solidification of solution 1.

It was found that differential radionuclide leaching rate for MPP compound on 28 day of contact with water (Table 2) was, $g/(cm^2 \cdot day)$: $^{137}Cs - 1.6 \cdot 10^{-4}$; $^{90}Sr - 6.9 \cdot 10^{-6}$; $^{239}Pu - 4.3 \cdot 10^{-7}$; $^{241}Am - 1.8 \cdot 10^{-6}$, which is substantially lower than the data for cement compounds [15].

Integral ammonium ion leaching rate from solution 1 was $1.3 \cdot 10^{-3} g/(cm^2 \cdot day)$ (Table 2) for leaching rate of 5.9 mass %. Such a low value confirms the data on predominant binding of ammonium ions in a stable phosphate $MgK_{1-x}(NH_4)_xPO_4 \cdot 6H_2O$. At the same time, it is evident that yield of ammonium ions from the compound was mainly due to dissolution of unbound fraction of ammonium nitrate, which was recorded in minor quantities in Fig. 1b. Results on study of component leaching rates for MPP compound with solidified solution 1 are given in Fig. 4 and Table 3.

Table 3. Leaching mechanism of components of MPP compound obtained in solidification of solution 1

| Matrix components | Time of contact with water, days | Tangent of the angle of linear relation of $\log \beta$ and $\log t$ (Fig. 4) | Leaching mechanism |
|-------------------|----------------------------------|---|--------------------|
| ^{137}Cs | 0–7 | -0.03 | washing out |
| | 7–28 | 0.57 | diffusion |
| ^{90}Sr | 0–28 | -0.15 | washing out |
| ^{239}Pu | 0–7 | -0.68 | depletion |
| | 7–28 | -0.26 | washing out |
| ^{241}Am | 0–7 | -0.36 | washing out |
| | 7–28 | 0.43 | diffusion |
| Na | 0–7 | -0.32 | washing out |
| | 7–28 | -0.94 | depletion |
| NH_4 | 0–7 | -0.09 | washing out |
| | 7–28 | 1.41 | dissolution |
| Fe | 0–10 | 1.12 | dissolution |
| | 10–28 | 0.40 | diffusion |

It was demonstrated that in spite of similar monotonous reduction of radionuclide leaching rates from MPP compound with time of its contact with water (Fig. 3), the leaching mechanisms are different and may change in time in process of leaching,

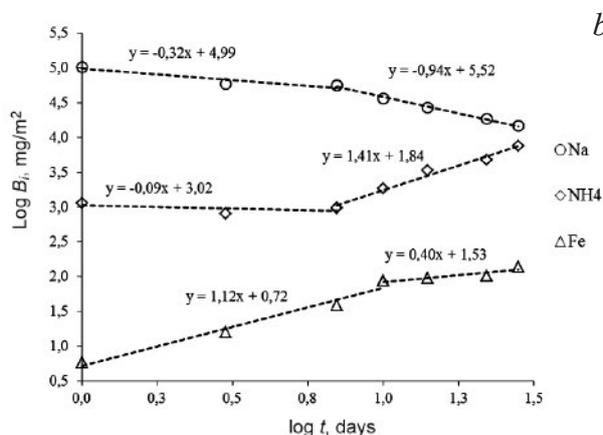
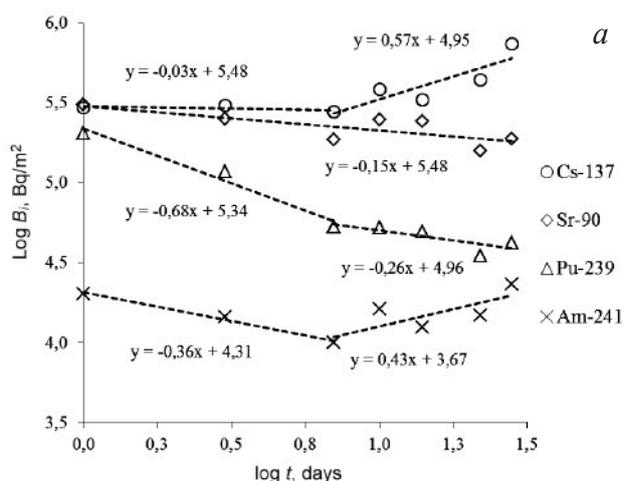


Fig. 4. Logarithm of ILW components release from MPP compound as a function of logarithm of time of its contact with water: a) Cs-137, Sr-90, Pu-239 and Am-241; b) ammonium, sodium and iron

similar to the leaching rate of chemical elements — components of solution 1 (Fig. 4 and Table 3). For example, ^{137}Cs (Fig. 4a), Na and NH_4 (Fig. 4b) leaching proceeds in a similar way during the first 7 days and is determined by their washing-out from compound surfaces (slope angle tangents: -0.03 , -0.32 and -0.09 , respectively). Then leaching of ^{137}Cs is determined by diffusion (0.57), evidently, from low-soluble ferrocyanide compound, while sodium leaching is determined by surface layer depletion (-0.94), and ammonium — by stepwise dissolution of surface layers (1.41), probably enriched in unbound ammonium nitrate. ^{239}Pu leaching rate in the first 7 days corresponds to surface layer depletion (-0.68), followed by washing out from compound surface (-0.26). Leaching behaviour of ^{90}Sr and ^{241}Am in the first 7 days is largely identical and determined by washing out from compound surface (-0.15 and -0.36 respectively). In continued contact with water the leaching mechanisms of these radionuclides become different: for ^{90}Sr it is continued washing out from the surface, while for ^{241}Am — leaching is due to diffusion from the internal layers of compound (0.43), consisting probably of more chemically strong orthophosphate $Am(III)$ — an analogue of monacite mineral. Iron leaching within

the first 10 days of sample contact with water occurs, probably, due to dissolution (1.12) of iron nitrate $\text{Fe}(\text{NO}_3)_3 \cdot n\text{H}_2\text{O}$, later followed by diffusion from low soluble phosphate $\text{Fe}(\text{III})$.

Conclusion

The study demonstrated, based on results obtained, that solidification of complex composition ILW containing actinides and ammonium nitrate using low temperature MPP matrix produces a compound with parameters complying with the requirements of NP-019-15 to cement compounds. There should also be noted the higher density of produced compound (about 1.8 g/cm^3) for optimal rate of its filling with LRW salts of about 20% mass, thus allowing reduction of solidified waste by up to 2 times compared to cementation.

One of the most important characteristics of compound used for LRW solidification is its stability to potential leaching by natural waters. The study demonstrated that ^{137}Cs and ^{90}Sr leaching rates were $1.6 \cdot 10^{-4}$ and $6.9 \cdot 10^{-6} \text{ g/(cm}^2 \cdot \text{day)}$ respectively, much lower than the data for cement compound. It should be specifically noted that cementation of LRW with specific activity of alpha-emitting nuclides above 10^7 Bq/l is not permitted and is not regulated. At the same time, we have measured the same low leaching rate of ^{239}Pu and ^{241}Am from MPP compound: $4.3 \cdot 10^{-7}$ and $1.8 \cdot 10^{-6} \text{ g/(cm}^2 \cdot \text{day)}$ respectively.

It was demonstrated that MPP compound has a high compression strength, about 15 MPa, and its strength is in compliance with the requirements of NP-019-15 (at least 5 MPa) following irradiation (dose 1 MGy), 30 thermal cycles ($-40 \dots +40 \text{ }^\circ\text{C}$), and 90 days immersion in water.

Thus, use of MPP matrix for solidification of LRW with complex chemical and radionuclide composition has a number of advantages over cementation and vitrification, and is a promising technology for industrial application.

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Information about the authors

Vinokurov Sergey Evgenevich, PhD, Head of Laboratory, Vernadsky Institute of Geochemistry and Analytical Chemistry of Russian Academy of Sciences (19, Kosygina st., Moscow, 119991). E-mail: vinokurov.geokhi@gmail.com

Kulikova Svetlana Anatolevna, Junior Research Associate, Vernadsky Institute of Geochemistry and Analytical Chemistry of Russian Academy of Sciences (19, Kosygina st., Moscow, 119991). E-mail: kulikova.sveta92@mail.ru

Krupskaya Viktoriya Valerevna, PhD, Senior Research Associate, Institute of Ore Deposits Geology, Petrography, Mineralogy, and Geochemistry of Russian Academy of Sciences (35, Staromonetny st., Moscow, 119017). E-mail: krupskaya@ruclay.com

Myasoedov Boris Fedorovich, Member of the Russian Academy of Sciences, Chief Research Associate, Vernadsky Institute of Geochemistry and Analytical Chemistry of Russian Academy of Sciences (19, Kosygina st., Moscow, 119991). E-mail: bfmyas@geokhi.ru

Bibliographic description

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