

CORROSION AND RADIATION RESISTANCE OF POTASSIUM MAGNESIUM PHOSPHATE MATRICES

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Corrosion and radiation resistances of potassium magnesium phosphate (PMP) matrices for hardening of liquid radioactive wastes of NPP were investigated. The high corrosion resistance of the PMP matrices to leaching of both basic components of matrix and cesium was shown. Results of performed work showed stability of physics and mechanical properties, as well as phase composition and microstructure of PMP after simulated γ -irradiation up to the absorbed dose 10^8 rad. It was determined that irradiation by high-energy electrons to the absorbed dose 10^{10} rad results in partial dehydration and amorphization of PMP.

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INTRODUCTION

At present, the problem of the radioactive waste safe handling is very challenging for further scale and dynamics of the nuclear power industry development [1]. It is known that in the process of exploitation of nuclear power plant, plenty of liquid radioactive wastes (LRW) of low and middle activity appears, gathers and kept. These radioactive wastes should not be used for the direct disposal, according to existent Ukrainian rules. One of the main methods of LRW solidification is the waste hardening by the addition of various binding components [2, 3]. The cementation is the simplest and most accessible method of LRW hardening. The main disadvantages of this method are low strength of the cement matrices, low filling of salts, low solidification speed, incomplete solidification of the liquid phase at a high water-cement ratio [4]. In addition, the salts included in LRW affect the basic processes of cement hydration, which leads to the degradation of the cement matrix over time [5].

As an alternative to the cement matrices, a potassium magnesium phosphate (PMP) matrices has been proposed for the immobilization of LRW [6]. The PMP matrix consists of a monophasic crystalline hexahydrate of magnesium and potassium double orthophosphate ($\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$). The PMP is an analog of natural phosphate minerals – monazite and apatite, which demonstrate high physic-chemical stability in the geological environment. PMP is classified as ceramic cement and has properties characteristic of both ceramics and hydraulic cements due to the uniqueness of its properties. The microstructure of PMP is similar to ceramics, with a strongly pronounced crystalline structure. However, in contrast to traditional ceramics, PMP is formed at room temperature and uses water at the beginning of chemical reactions, with subsequent solidification of the material, similar to the production of hydraulic concretes.

Examples of the PMP matrices application for solidification of Tc-containing wastes simulators, incorporation of Pu, immobilization of cesium and

simulators of liquid high-salt HLW are well known [7–10].

One of the main requirements for LRW solidification matrices selection is the radiation and corrosion resistance. The corrosion resistance of the matrices with respect to leaching in the water is the main criterion for their applicability for the environmentally safe storage of radioactive waste. The matrix with radioactive waste included will be subject to the action of β -particles and γ -radiation due to the decay contained in radionuclide waste. There can be changes in volume, microstructure, mechanical properties and resistance to leaching of solidified waste forms under the irradiation process.

The aim of the study was research of the radiation and corrosion resistance of PMP as promising materials for the solidification of LRW at nuclear power plants.

MATERIALS AND METHODS

For the synthesis of PMP $\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$, the following reactors were used:

- magnesium oxide MgO (grade h);
- potassium dihydrogen phosphate KH_2PO_4 (grade h);
- distilled water (pH = 5.5).

In the case of synthesizing cesium-containing PMP samples, cesium chloride CsCl was used as a starting reagent (grade hp).

Irradiation by electrons and bremsstrahlung γ -radiation of PMP samples was carried out at the KUT-1 accelerator of the NSC KIPT (electron energy $E = 10$ MeV). PMP samples were irradiated by electrons to a set of absorbed dose of 10^{10} rad (10^8 Gy) and bremsstrahlung γ -radiation – 10^8 rad (10^6 Gy).

The phase composition of the PMP materials was investigated by X-ray diffraction analysis (DRON-4-07 in copper Cu-K α -radiation using a Ni selectively absorbing filter). To identify the phases, the JCPDS diffraction data base was used. The density of the PMP samples was determined by hydrostatic weighing.

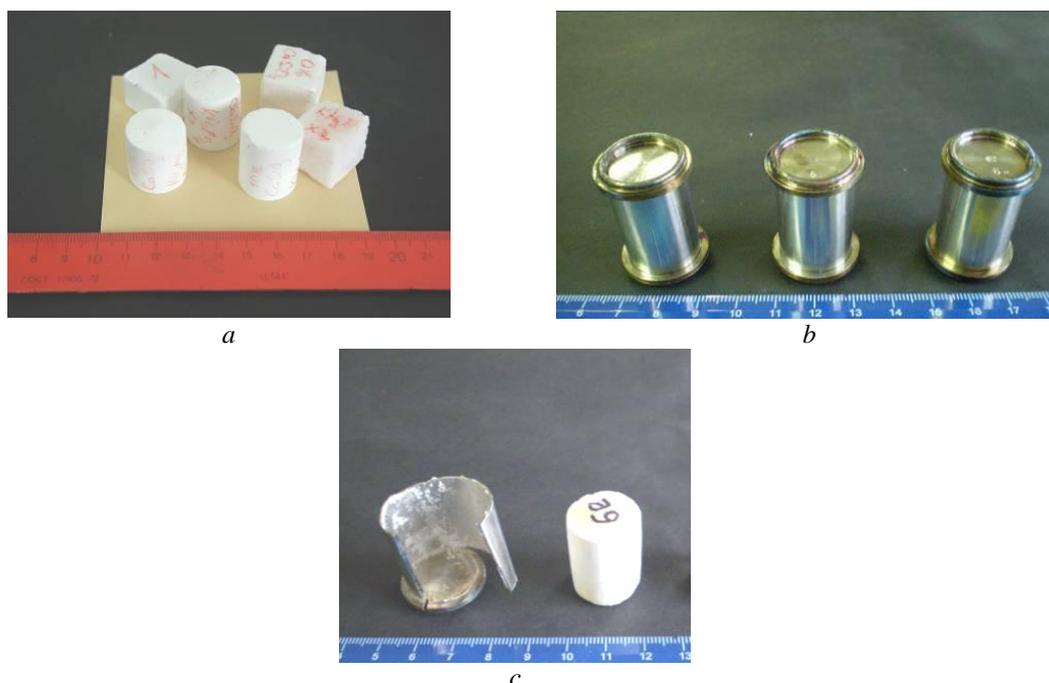


Fig. 1. PMP samples: a – view; b – in capsules before electron irradiation; c – after electron irradiation

Compression tests of PMP samples were carried out on an electromechanical press of the brand “ZD 10/90”, the maximum load – 10 tons.

To analyse the corrosion resistance of PMP matrices, leaching tests were carried out at a temperature of 90 °C for 7 days in accordance to the PCT test [11]. The test was made according to the procedure ASTM C1285 “Standard Test Methods for Deter-Mineral Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses: The Product Consistency Test (PCT)”.

Corrosion resistance of PMP matrices with respect to cesium leaching as the main radionuclide of LRW from NPP was determined in accordance to the ANS 16.1 test [12] in the long-term leaching of PMP samples containing 10 wt.% CsCl and 15 wt.% CsCl in bidistilled water at 25 °C. The concentration of K, Mg, P, Cs in the leachate was determined by atomic-emission spectroscopy with inductively coupled plasma ICP-AES (Spectrometer Scan Advantage, manufactured by Thermo Jarrell Ash, USA).

EXPERIMENTAL PART

PMP were obtained at room temperature as a result of an acid-alkaline reaction between magnesium oxide MgO and potassium dihydrogen phosphate KH_2PO_4 in water [9]. The dry mixture of MgO and KH_2PO_4 was thoroughly mixed, and then the required amount of water was added to this mixture. Further, the resulting mass was stirred for 20...30 min until a pasty state. The paste was placed in a plastic mold. The temperature of the paste rose to 45 °C within 10 min. To increase the reaction time, 1...2 wt.% of boric acid H_3BO_3 were added to the mixture. To increase the strength properties of the PMP samples, 10 wt.% of wollastonite CaSiO_3

was added to the mixture. After extraction from the plastic form of PMP, the samples were held at a temperature of 20 °C for 28 days. As a result, samples of the compositions $(\text{KMgPO}_4 \cdot 6\text{H}_2\text{O})$ and $(\text{KMgPO}_4 \cdot 6\text{H}_2\text{O} + 10...15 \text{ wt.}\% \text{CsCl})$ were obtained in the form of a cube with dimensions of 20x20x20 mm and cylindrical samples with a diameter of 19 mm and a height of 35 mm (Fig. 1,a).

Irradiation by electrons and bremsstrahlung γ -radiation of PMP samples was carried out on the accelerator KUT-1 up to a set of absorbed dose of 10^{10} rad and bremsstrahlung γ -radiation – 10^8 rad. The target, in which the samples were placed, exposed to electrons, was cooled with water. Therefore, to prevent the interaction of the PMP material with water samples during electron irradiation, the samples were placed in 0.3 mm stainless steel capsules (see Fig. 1,b). Containers with PMP samples were irradiated with electrons along the axis of the cylindrical capsules. PMP samples were irradiated with bremsstrahlung γ -radiation in an aluminum foil. Samples were irradiated with bremsstrahlung γ -radiation in air.

The amount of absorbed dose was chosen taking into account the requirements for the materials of containers for the packaging, transportation and storage of radioactive waste (including spent nuclear fuel, low – and high – active waste). According to the requirements, the material of the container must have radiation resistance of 10^8 rad or higher under irradiation conditions, in the case of necessity. The radiation dose of 10^8 rad is based on its equality to the total dose received in more than 300 years by radioactive waste containing ^{137}Cs or ^{90}Sr at a concentration of 10 Ci/ft³ [13].



a



b

Fig. 2. The appearance of the container with a sample for performing the leaching test: a – PCT; b – ANS 16.1

Corrosion resistance to the leaching of the main components of the PMP matrix (K, Mg, P) was determined in accordance with the PCT test. To carry out the corrosion resistance tests, a Teflon container was used. The starting PMP samples were ground and then sieved. The resulting powder was washed in distilled water and acetone in an ultrasonic bath. Next, the dried purified powder was milled in a Teflon container and water was added according to the ASTM C1285 standard (Fig. 2,a). The container was placed in a thermostat for 7 days at a temperature of (90 ± 2) °C. After the test, the resulting leach was filtered from the powder particles. The concentration of elements in the leachate was determined by ICP-AES.

The normalized leaching rate of the elements was calculated by the formula:

$$R = c_i V / (S f_i t), \quad (1)$$

where R is the normalized leaching rate of the element, $g/(m^2 \cdot day)$; c_i is the concentration of the i -th element in the solution after leaching, g/l ; V is the volume of the leaching water, l ; S is the specific surface area of the milled sample, m^2 ; f_i is the content of the i -th element in the matrix; t – leaching time, days.

Corrosion resistance of the resulting PMP matrices with respect to cesium leaching was determined in accordance with ANSI/ANS 16.1–1986 test “Measurement of the leach-ability of a solid-state low-level test procedure” (see Fig. 2,b). This test is usually used to characterize such forms of solidified low-level waste, such as bitumen, concrete and other cementing materials. The ANS 16.1 test provides for the long-term leaching of samples in water at 25 °C at the following time intervals: 2, 5, 17 hour, four 24-hour intervals followed by 14, 28, and 43-day intervals (total time 90 days). Determination of cesium content in the leach was carried out with ICP-AES. The obtained values were used to calculate the effective diffusion coefficient D and leaching indexes L_i :

$$D = n \left[\frac{a_n / A_0}{(\Delta t)_n} \right]^2 T \left[\frac{V}{S} \right]^2, \quad (2)$$

where D is the effective diffusion coefficient, cm^2/s ; V is the volume of the sample, cm^3 ; S is the geometric surface area of the sample, calculated from the measured sample parameters, cm^2 ; T is the average leaching time, s ; a_n – the amount of the element selected from the sample for the n -th interval; A_0 is the total amount of element in the sample prior to leaching; Δt_n is the duration of the n -th interval

$$L_i = \frac{1}{10} \sum_{i=1}^{10} \left[\log \left(\frac{\beta}{D_i} \right) \right]_n, \quad (3)$$

where L_i is the i -element leach index; β is a constant ($1.0 \text{ cm}^2/s$); D_i is the effective diffusion coefficient of the i -element.

RESULTS AND DISCUSSION IRRADIATION OF PMP SAMPLES BY BREMSSTRAHLUNG γ -RADIATION AND ELECTRONS

After irradiation of the PMP samples with bremsstrahlung γ -radiation up to a set of absorbed dose of 10^8 rad, no chemical, phase and noticeable microstructural changes have been detected. Fig. 3,a,b show the diffraction patterns of the sample composition ($KMgPO_4 \cdot 6H_2O + 10 \text{ wt.}\% CaSiO_3$) before and after γ -irradiation. Comparison of the diffractograms shows the complete coincidence of the main X-ray lines of potassium-magnesium phosphate before and after γ -irradiation.

In addition, according to IR spectroscopy, irradiation with bremsstrahlung γ -radiation with a maximum energy of 13.5 MeV to a dose of $1.35 \cdot 10^5$ Gy does not lead to a change in the main phase composition of the PMP samples material, since the number and position of all bands of the spectrum remains unchanged. Changes in the intensity of bands in the IR spectrum are associated with an increase in the degree of crystallinity of the material of the PMP sample after γ -irradiation [14].

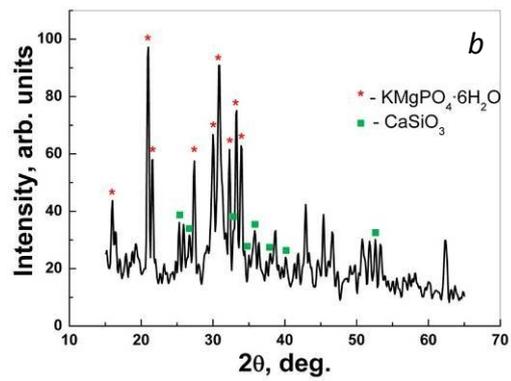
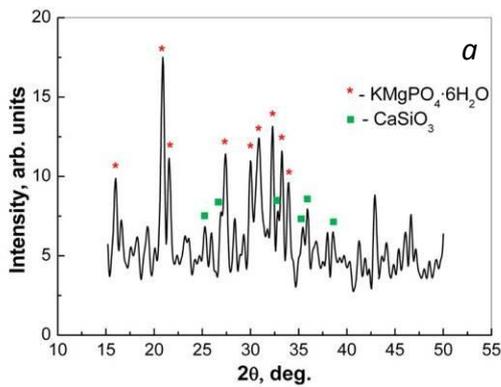


Fig. 3. XRD patterns of $\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$ + 10 wt.% CaSiO_3 : a – before γ -irradiation; b – after γ -irradiation

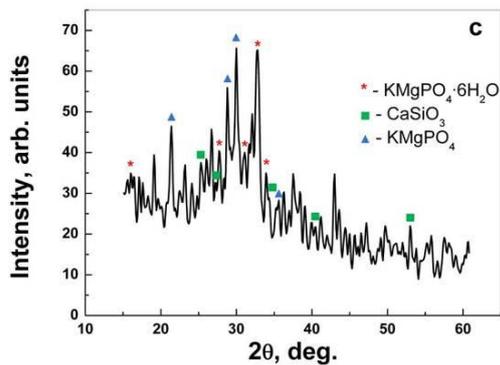


Fig. 4. XRD patterns of $\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$ + 10 wt.% CaSiO_3 after electron irradiation process

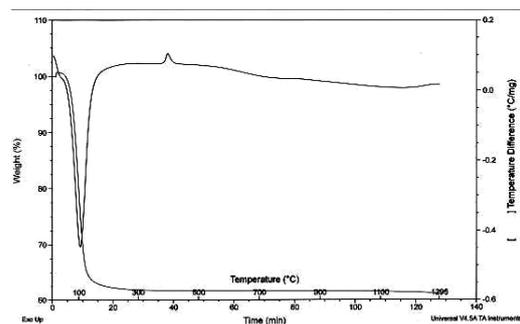


Fig. 5. DTA/TG analysis of PMP samples

In contrast to γ -irradiation, not only the $\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$ and wollastonite CaSiO_3 materials but also the anhydrous phosphate KMgPO_4 X-ray lines against the halo background at the angles of 20° – 2θ – 40° were observed on the diffractogram of the sample composition ($\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$ + 10 wt.% CaSiO_3) after electron irradiation with a set of absorbed dose of 10^{10} rad (Fig. 4). This fact indicates that after electron irradiation with high energies (10 MeV), partial dehydration and amorphization of the PMP samples took place. As it is known from the DTA/TG analysis [9] that the maximum endothermic peak, which corresponds to the intense dehydration of potassium-magnesium phosphate, is observed at a temperature of 120°C (Fig. 5). It was shown in [15] that when $\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$ is heated to a temperature of 200°C , it completely dehydrates and forms amorphous KMgPO_4 , which crystallizes at higher temperatures. The obtained results show that irradiation of PMP samples with high-energy electrons leads not only to partial amorphization of potassium magnesium phosphate, but also causes crystallization of amorphous KMgPO_4 at temperatures below 100°C .

Externally, PMP samples with wollastonite irradiated by γ -radiation and electrons did not differ from unirradiated samples. Any volume changes and visible damage of the samples were not observed (see Fig. 1,c). No changes in the microstructure of the PMP samples ($\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$ + 10 wt.% CaSiO_3) before and after the electron irradiation were detected by the SEM-method (Fig. 6). In both photographs, the well-bound microstructure of the samples $\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$ +

+10 wt.% CaSiO_3 , and elongated particles of wollastonite CaSiO_3 are seen.

However, irradiation with both electrons and γ -radiation leads to a slight change in the density and compression strength of PMP samples with wollastonite. Thus, a slight decrease in the density and compressive strength of $\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$ + 10 wt.% CaSiO_3 samples after γ -radiation is observed (Tabl. 1).

Table 1
Density and compressive strength of the samples $\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$ + 10 wt.% CaSiO_3 before and after electron and γ -irradiation

Conditions	Density, g/cm^3	Compressive strength, MPa
Before irradiation	1.67	12.7
After γ -irradiation	1.58	11.3
After electron irradiation	1.62	14.4

The decrease in the density and compressive strength after γ -irradiation can be explained on the basis that the energy of radiation can drive away part of the bound water from the samples and, thus, increase the porosity and reduce the strength. In the case of electron irradiation, a less noticeable decrease in the density of the $\text{KMgPO}_4 \cdot 6\text{H}_2\text{O}$ + 10 wt.% CaSiO_3 sample is observed. The increase in porosity of the PMP sample surface layer occurs under the radiative action, due to the small penetrating ability of electrons. The increase in the compressive strength of the test samples after irradiation with high-power electrons may be due to the formation of hardening calcium-phosphate inclusions in the process of irradiation.

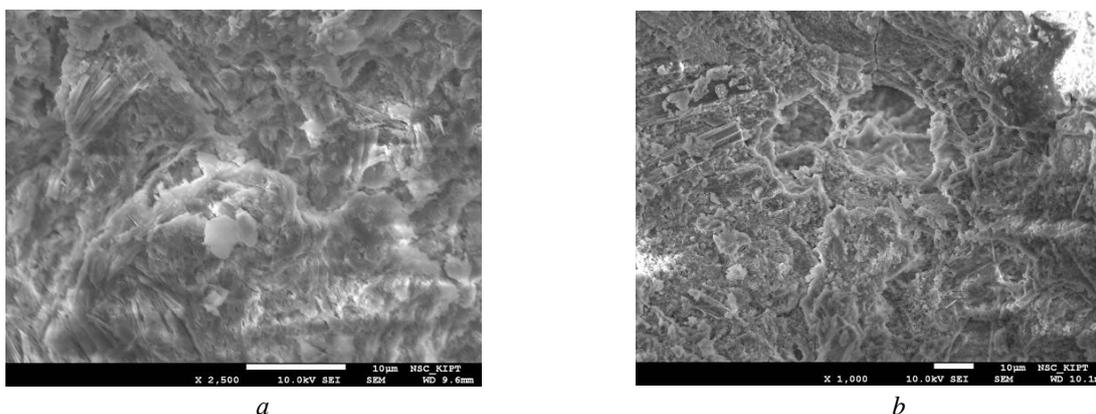


Fig. 6. Microstructure of the samples $KMgPO_4 \cdot 6H_2O + 10 \text{ wt.}\% CaSiO_3$:
a – before electron irradiation; *b* – after electron irradiation

Earlier, the authors of [16] noted the increase in compressive strength simultaneously with a decrease in the density of PMP samples with ash additives. Such samples behavior was explained by the formation of calcium-phosphate inclusions in the material, which leads to the strengthening of the material. The rate of such particles formation increases with increasing temperature of PMP material synthesis. In our case, the formation of such hardening calcium-phosphate inclusions can occur due to local heating of the PMP samples with wollastonite $CaSiO_3$ during irradiation with high-energy electrons. Obtained results shown that phase and structural changes in potassium-magnesium-phosphate materials after γ -irradiation were not detected. The density and compressive strength values of PMP materials before and after the γ -irradiation were practically not changed. This fact indicates the resistance of the PMP matrices to the radiation effect.

In addition, the behavior of the PMP samples obtained under the conditions of irradiation by high-energy electrons up to absorbed doses exceeding the doses that a protective matrix can collect in a real situation of long-term storage of radioactive waste was investigated.

After electron irradiation up to an absorbed dose of 10^{10} rad, any destruction of the PMP samples was not observed. The electron irradiation was not lead to significant changes in the physic-mechanical characteristics of the PMP materials.

LEACHING OF PMP SAMPLES WITH CESIUM CHLORIDE ADDITIONS

Corrosion properties of PMP matrices were determined by leaching in water samples of the compositions ($KMgPO_4 \cdot 6H_2O + 10 \dots 15 \text{ wt.}\% CsCl$), in accordance with the requirements of the PCT and ANS 16.1 tests [11, 12]. After conducting the PCT test using ICP-AES, the content of potassium, magnesium, phosphorus and cesium in the solution after leaching was determined. The values were used to calculate the leaching rates of these elements (Tabl. 2). Comparison of the reduced rates of leaching shows that an increase in the concentration of cesium does not lead to a significant change in the values. Low leaching rates at a temperature of $90 \text{ }^\circ C$ of the basic elements of the PMP matrix and cesium indicate a high hydrothermal resistance of the PMP matrices.

Table 2
 Leaching rate of basic elements of PMP matrix and cesium (PCT)

Element	Normalized Elemental Leach Rates, $g/(m^2 \cdot \text{day})$	
	$KMgPO_4 \cdot 6H_2O + 10 \text{ wt.}\% CsCl$	$KMgPO_4 \cdot 6H_2O + 15 \text{ wt.}\% CsCl$
K	$2.13 \cdot 10^{-3}$	$2.88 \cdot 10^{-3}$
Mg	$1.62 \cdot 10^{-6}$	$5.20 \cdot 10^{-6}$
P	$4.69 \cdot 10^{-4}$	$4.25 \cdot 10^{-4}$
Cs	$3.46 \cdot 10^{-5}$	$3.04 \cdot 10^{-5}$

The results of the ANS 16.1 test are presented in Tabl. 3. For cesium, despite the high content in the PMP matrix (10 wt.% CsCl and 15 wt.% CsCl), high values of the leaching indices L (11.5, 11.7), low values of the effective diffusion coefficients D ($8.23 \cdot 10^{-14}$, $1.19 \cdot 10^{-13} \text{ cm}^2/\text{s}$), and the rate of leaching R ($2.66 \cdot 10^{-5}$, $1.16 \cdot 10^{-5} \text{ g}/(\text{cm}^2 \cdot \text{day})$) with long-term (90 day) leaching at $25 \text{ }^\circ C$ is characteristic. It is known that the rates of cesium leaching from various types of glass used for radioactive waste solidification are $10^{-4} \dots 10^{-6} \text{ g}/(\text{cm}^2 \cdot \text{day})$, the rate of cesium leaching from cemented forms is $\leq 10^{-3} \text{ g}/(\text{cm}^2 \cdot \text{day})$, and from the ceramic Synroc $\sim 10^{-5} \text{ g}/(\text{cm}^2 \cdot \text{day})$ [17]. Thus, according to the level of corrosion resistance with respect to cesium leaching, potassium magnesium phosphates are not inferior to other matrices in use at present.

CONCLUSIONS

As a result of the acid-base reaction at room temperature, PMP $KMgPO_4 \cdot 6H_2O$ with the addition of wollastonite $CaSiO_3$ (10 wt.%) and cesium chloride CsCl (10 and 15 wt.%) were obtained at room temperature.

Simulated irradiation with electrons ($E - 10 \text{ MeV}$) and bremsstrahlung γ -radiation of PMP samples to a set of absorbed dose of 10^{10} and 10^8 rad, respectively, was carried out. The absence of phase and structural changes in the material of the PMP samples after irradiation processes was established, and also the value of the density and compressive strength was not practically changed. Irradiation with high-energy electrons of PMP samples leads to partial dehydration and amorphization of $KMgPO_4 \cdot 6H_2O$, as well as crystallization of amorphous $KMgPO_4$.

Effective Diffusion Coefficient, Leachability Index and Stabilized Leaching Rate of Cs (ANS 16.1)

Parameter	Sample	
	KMgPO ₄ ·6H ₂ O +10 wt.% CsCl	KMgPO ₄ ·6H ₂ O +15 wt.% CsCl
Effective Diffusion Coefficient D , cm ² /s	$8.23 \cdot 10^{-14}$	$1.19 \cdot 10^{-13}$
Leachability Index L	11.5	11.7
Stabilized Leaching Rate R , g/(cm ² ·day)	$2.66 \cdot 10^{-5}$	$1.16 \cdot 10^{-5}$

Corrosion properties of PMP matrices were determined by leaching in water samples of the compositions (KMgPO₄·6H₂O + 10...15 wt.% CsCl), in accordance with the requirements of the PCT and ANS 16.1 tests. Low leaching rates of the basic elements of the PMP matrix and cesium indicate the high hydrothermal stability of the PMP matrices (PCT). Based on the results of the ANS 16.1 test, the values of cesium leaching rates from PMP matrices are obtained that are at the level of the leaching rates from glasses and ceramics of Synroc.

The results of the studies of the radiation and corrosion resistance of the obtained PMP matrices indicate the prospects of potassium-magnesium-phosphate materials application for the solidification of LRW at nuclear power plants.

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КОРРОЗИОННАЯ И РАДИАЦИОННАЯ СТОЙКОСТЬ КАЛИЙ-МАГНИЙ-ФОСФАТНЫХ МАТРИЦ

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Исследована коррозионная и радиационная стойкость калий-магний-фосфатных (КМФ) матриц для отверждения жидких радиоактивных отходов АЭС. Показана высокая коррозионная стойкость КМФ-матриц к выщелачиванию как основных компонентов матрицы, так и цезия. Результаты выполненной работы показали устойчивость физических и механических свойств, а также фазового состава и микроструктуры КМФ после имитации γ -облучением до поглощенной дозы 10^8 рад. Было установлено, что облучение высокоэнергетическими электронами до поглощенной дозы 10^{10} рад приводит к частичной дегидратации и аморфизации КМФ.

КОРОЗІЙНА ТА РАДІАЦІЙНА СТІЙКІСТЬ КАЛІЙ-МАГНІЙ-ФОСФАТНИХ МАТРИЦЬ

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Досліджена корозійна та радіаційна стійкість калій-магній-фосфатних (КМФ) матриць для затвердіння рідких радіоактивних відходів АЕС. Показана висока корозійна стійкість КМФ-матриць до вилуговування як основних компонентів матриці, так і цезію. Результати виконаної роботи показали стійкість фізичних та механічних властивостей, а також фазового складу і микроструктури КМФ після імітації γ -опроміненням до поглиненої дози 10^8 рад. Було встановлено, що опромінення високоенергетичними електронами до поглиненої дози 10^{10} рад призводить до часткової дегідратації та аморфізації КМФ.