

LEACHING CHARACTERISTICS OF MAGNESIUM POTASSIUM PHOSPHATE CEMENT IN ALKALINE SOLUTION

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This work investigated the leaching characteristics of magnesium potassium phosphate cement (MKPC) in alkaline solution. Alkaline leaching tests were conducted on MKPC specimens according to the ANSI/ANS 16.1 test. Leachate compositions were determined using ICP-AES and cumulative concentrations, effective diffusion coefficients and leachability indices for Mg, K, P, and B were calculated from the data. X-ray diffraction/Rietveld (XRD) was used to analyze the phase composition changes of the MKPC surface layer after leaching. For all tested materials, the major elements Mg, K, P, and B showed limited leachability (leachability index ≥ 9) in an alkaline solution mimicking the internal waters of radioactive waste storage facilities.

INTRODUCTION

Magnesium potassium phosphate cement is a new cement material. It has wide application prospects in rapid road repair and solidification of hazardous materials due to its excellent characteristics such as fast hard and early strength, low shrinkage and high bonding strength [1–3].

Magnesium potassium phosphate cements (MKPCs) are also described as “chemically bonded phosphate ceramics (CBPCs)” [4] or by the trade name “Ceramicrete” and have been used to stabilize and solidify various low-level radioactive mixed wastes, Pu-contaminated ashes, and technetium-containing waste solutions at the Argonne National Laboratory, USA [1, 5, 6]. Studies have been conducted on the immobilization of simulated liquid high-level waste containing actinides, fission products, and corrosion products in MKPC matrices [7, 8].

Recently, MKPCs have been proposed for the stabilization of high-sodium waste streams [9], denitrated high-level liquid wastes [10], solidified ceramic forms [11], and radioactive concrete wastes generated by decommissioning of nuclear power plants [12].

MKPCs are also being considered as lower-pH binders for reactive metal wastes such as aluminium and beryllium from the nuclear industry. Reactive metals corrode in alkaline environments such as conventional Portland cement mixtures, releasing flammable hydrogen gas, which causes cracking of the cement mold. This means that the lower pH of MKPCs may be useful in reducing the corrosion of aluminium and beryllium [13, 14].

MKPCs are produced by the reaction between magnesia (MgO) and soluble potassium dihydrogen phosphate (KH_2PO_4) to form K-struvite ($\text{MgKPO}_4 \cdot 6\text{H}_2\text{O}$), a mineral with low solubility. The hardening (setting) of these cements is very rapid and the MgO must be calcined at high temperatures to minimize its high reactivity. Considering the high cost of hard-burned MgO, the use of inexpensive fillers becomes preferable. In addition, the addition of fillers

can help optimize the hydration process and improve the mechanical properties of the resulting product. Traditionally, fly ash [5], granulated blast furnace slag [15], and wollastonite (CaSiO_3) [16] are used as fillers in MKPC composites for nuclear applications.

Despite the potential advantages of MKPCs for radioactive waste stabilization, information on their long-term durability is still limited. A previous study on the microstructure and phase composition of MKPC specimens (fly ash as filler and a Mg/P molar ratio = 1) immersed in deionized water (pH = 6.8) and cementitious base mortars (pH = 13.3) for 6 months showed amorphization of MKPCs over time, which may be related to structural changes for the K-struvite phase [17]. Recently, Diaz Caselles et al. investigated the long-term durability of MKPCs by examining their leaching behavior [18, 19]. Semi-dynamic leaching tests were carried out on MKPC specimens (with fly ash as a filler) using demineralized water with pH adjusted to 7 and an alkaline solution (pH = 13.2). After leaching in demineralized water, three main zones were observed on the surface of the specimens: (i) a poorly cohesive residual layer where K-struvite was fully depleted, (ii) an intermediate zone where K-struvite coexisted with cattite ($\text{Mg}_3(\text{PO}_4)_2 \cdot 22\text{H}_2\text{O}$), and (iii) a third zone without any cattite. Leaching by the alkaline solution induced a decrease in the content of crystalline K-struvite, as well as the precipitation of calcium-deficient hydroxyapatite, brucite ($\text{Mg}(\text{OH})_2$) and possibly magnesium silicate hydrates, hydrotalcite-like phase (possibly meixnerite ($\text{Mg}_6\text{Al}_2(\text{OH})_{18} \cdot 4\text{H}_2\text{O}$)) or PO_4 -LDH [20].

The main objective of this work was to study the leaching behaviour of MKPC prepared with different fillers (blast furnace slag (BFS), fly ash (FA), or a mix of the two components) following ASTM ANS 16.1 procedure (American Nuclear Society Standard “Measurement of Leachability in Solidified Low-Level Radioactive Waste by the Short-Term Procedure”) [21]. An alkaline solution representative of the pore solution of conventional concrete placed in the near field of the

cemented waste packages was used as a leaching medium.

the Mariupol metallurgical plant (Mariupol, Ukraine) is shown in Table 2.

1. MATERIALS AND METHODS

1.1. MATERIALS

The MKPC compositions investigated in this study are summarized in Table 1.

Considering that BFS, along with FA, is a widely available and inexpensive material at metallurgical plants in Ukraine, the use of BFS as mineral additives in the production of MKPC compounds is very promising.

The chemical composition of FA from the Burshtyn thermal power plant (Burshtyn, Ukraine) and BFS from

Table 1
The MKPC compositions

Component	wt. %
MgO	6.47
KH ₂ PO ₄	21.85
Sand	28.33
3 options: FA, BFS and (½ FA+½ BFS)	28.33
Boric acid	0.57
Water	14.45

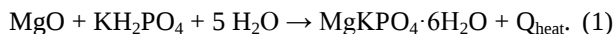
Table 2
Chemical composition of the fly ash and blast furnace slag

Industrial waste	Weight content, wt. %										
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	C	MnO	TiO ₂	(K,Na) ₂ O	Other
FA	46.12	18.00	22.17	4.09	1.46	0.21	2.50	0.14	1.78	2.10	1.43
BFS	38.20	4.60	1.65	48.5	4.3	—	—	0.81	—	0.82	1.12

The raw materials used to prepare the MKPC specimens had the following characteristics: calcined magnesium oxide (MgO > 97 %, particle size < 200 µm); potassium dihydrogen phosphate (KH₂PO₄ > 98 %, size < 630 µm); Sand (SiO₂ > 99.3 %, size < 400 µm); FA (size < 400 µm); BFS (size < 630 µm).

1.2. PREPARING SPECIMENS

The production of solid MKPC specimens is based on the formation reaction of magnesium potassium phosphate hexahydrate MgKPO₄·6H₂O (K-struvite):



The specimens were prepared as described in Sayenko, S. et al. [22], meaning that a MgO/KH₂PO₄ molar ratio equal to 1 and a water-to-cement (w/c) weight ratio of 0.51 were used. The MKPC specimens were prepared with different fillers: MKPC with FA filler (further referred as MKPC-FA), MKPC with BFS filler (MKPC-BFS) and MKPC with ½ FA+½ BFS filler (MKPC-FA-BFS). To manufacture these specimens, the dry mixtures were first prepared by mixing MgO, KH₂PO₄, sand and filler in a lab grinding mill machine (Fritch, single pulverising porcelain bowl of 200 g capacity, Ø2.0 cm aluminum oxide balls, speed 150 rpm, mixing time 10 min). Then, boric acid H₃BO₃, used as a reaction retarder, was dissolved in distilled water, and the mixture of dry components was added to this solution and mixed with a mechanical stirrer in a 250 ml polypropylene can for 5 min until a homogeneous paste was obtained. Finally, the mortar was poured into polypropylene moulds and covered with polyethylene film to prevent rapid drying. Cubic specimens with dimensions of 3×3×3 cm were demoulded after 1 day and kept for at least 28 days to cure in indoor ambient conditions.

1.3. LEACHING PROCEDURE

Leaching tests were performed according to ANS 16.1 by immersing the specimen for 90 days in distilled water contained in a 1-liter HDPE (High Density Polyethylene) vessel, ensuring that all sides of the specimen are in contact with the leachant.

Leaching tests were carried out at a temperature of 25 °C. The leachate was sampled, and the fresh leachant was replaced after increasing leaching periods, i.e., 1, 4, 14, 28, and 43 days. The test thus lasted 90 days (1+4+14+28+43 days). From the results of the leaching tests, the leaching characteristics of the different elements from the solidified MKPC specimens were evaluated based on their cumulative concentrations (CC) in the leachates. The effective diffusion coefficient (D_e) and leachability index (LI) were also calculated to quantitatively evaluate whether the MKPC matrix can be used for waste conditioning.

The concentrations (mg/l) of the major elements (Mg, K, P) and B leached from the MKPC specimens were analysed using an inductively coupled plasma atomic emission spectroscope (ICP–AES, iCAP 6300 Duo, Thermo Scientific Corporation). Also, the mineralogical and structural characteristics of the specimens before and after leaching were studied.

The measured element concentrations were used to plot cumulative concentrations versus time and calculate leachability indices. Leachability index for each element was determined based on the following expressions according to ANS-16.1:

$$LI = \frac{1}{5} \sum_{i=1}^5 [\log(\beta/D_e)]_n, \quad (2)$$

where β is a defined constant (1.0 cm²/s).

D_e can be calculated using the mass transport equation (i.e., Fick's second law) as follows:

$$D_e = \pi \left[\frac{a_n/A_0}{(\Delta t)_n} \right]^2 \left[\frac{V}{S} \right]^2 T, \quad (3)$$

where D_e is the effective diffusion coefficient, cm²/s; V is the volume of the specimen, cm³; S is the geometric surface area of the solid specimen, cm²; a_n is the amount of element released from the specimen for the n-th leaching interval; A₀ is the initial amount of the element in the specimen prior to leaching, Δt_n = t_n – t_{n-1} is the duration of the n-th leaching interval, and T is the leaching time representing the mean time (s) of the n-th leaching interval for a semi-infinite medium:

$$T = \left[\frac{1}{2} (\sqrt{t_n} + \sqrt{t_{n-1}}) \right]^2. \quad (4)$$

2. RESULTS AND DISCUSSION

2.1. LEACHING RESULTS

The purpose of the test is to assess the behaviour of MKPC matrix when leached by alkaline solution: “CEM I + silica fume” (without silica) synthetic water to mimic LILW repository. Leaching tests of MKPC specimens were carried out in “CEM I + silica fume” (without silica) synthetic water, prepared using the following recipe: for 1 l solution, add 0.9798 g Na_2SO_4 , 0.2666 g CaCl_2 , 15.2 ml 1M NaOH, 16.6 ml 1M KOH. Such solution had a pH of 12.6.

Leaching tests in alkaline solution were carried out according to the conditions of standard ANS 16.1. Concentration values for leached potassium were determined by subtracting the concentration in the original alkaline solution from the resulting concentrations in the leachates. Note that the solution initially contained K^+ at a concentration of 571.2, Na^+ – 665.0 and Ca^{2+} – 290.6 mg/l, measured by ICP-AES.

After leaching in an alkaline solution for 90 days, the specimens retained their integrity and shape (Fig. 1),

but limited precipitation of white salts was noticed on the surface of the specimens.



Fig. 1. MKPC-FA, MKPC-BFS and MKPC-FA-BFS specimens (from left to right) after leaching in an alkaline solution for 90 days

The concentrations of matrix elements released in the leachates for all specimens checked the following relationship: $\text{K} > \text{P} > \text{B} > \text{Mg}$ (Fig. 2).

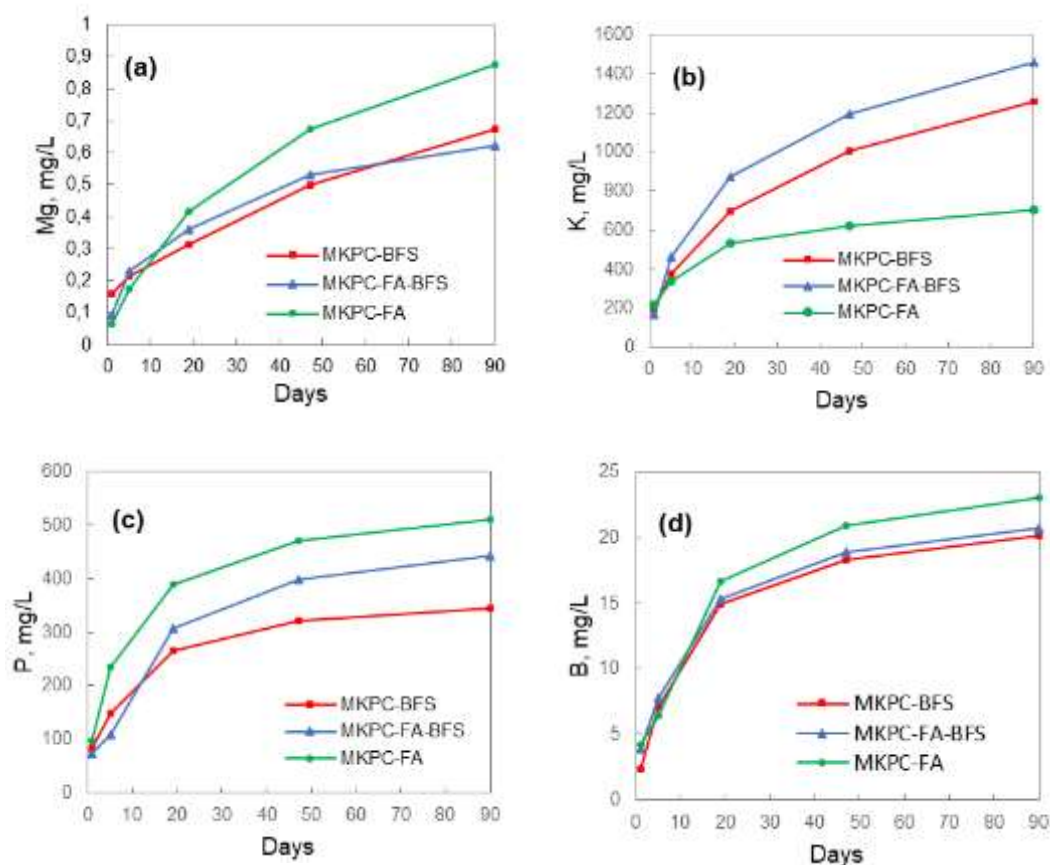


Fig. 2. Cumulative concentrations of leached elements in alkaline solution: a – Mg; b – K; c – P, and d – B

In contrast to distilled water (Fig. 3), the CC of Mg in the alkaline solution decreases significantly and becomes smaller than that of B (see Fig. 2,a,d). The highest release of K from MKPC matrix is observed with MKPC-FA-BFS specimen, whatever the leaching solution (alkaline solution or distilled water (see

Fig. 2,b; Fig. 3,b). However, the smallest release of K in the alkaline solution is observed with MKPC-FA specimen, instead of MKPC-BFS specimen in distilled water. Releases of P from MKPC matrices leached by distilled water or by the alkaline solution are almost of the same magnitude order (see Fig. 2,c; Fig. 3,c).

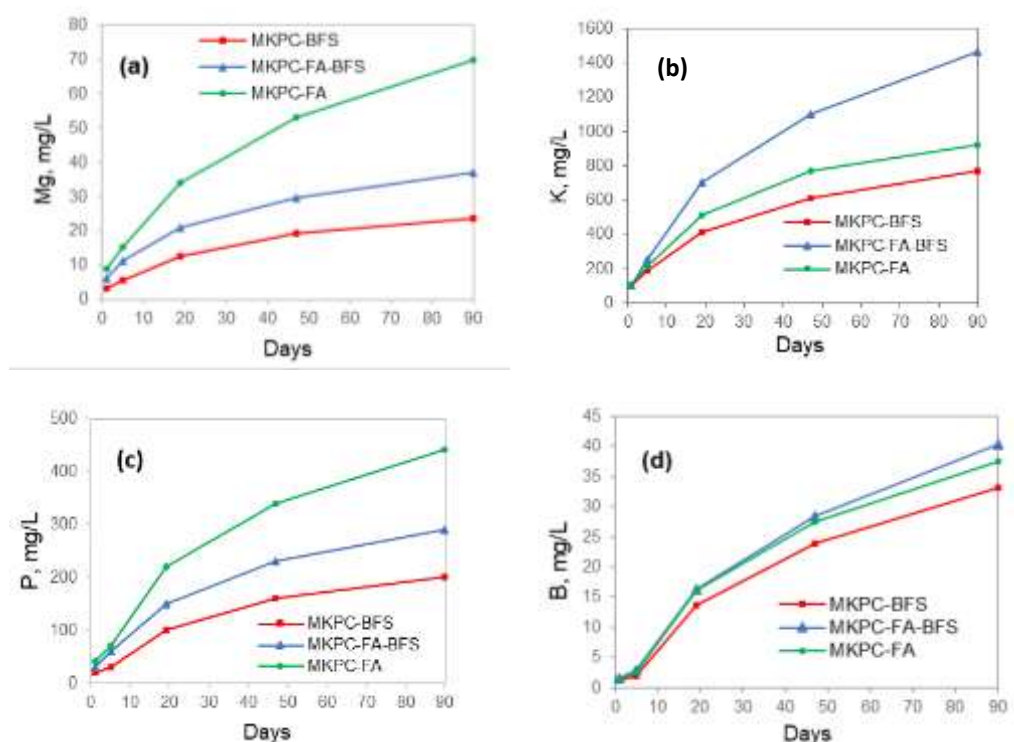


Fig. 3. Cumulative concentrations of leached elements in distilled water:
a – Mg; b – K; c – P, and d – B

Based on the measured element concentrations, effective diffusion coefficients and leachability indices were calculated (Table 3). These data are necessary to assess the behaviour of waste forms under conditions of constant water saturation, which simulate disposal conditions under the most conservative scenario.

Comparing the leachability indices for Mg, K, P, and B during leaching in an alkaline solution and in distilled

water, we can see that the LI values for Mg differ significantly. LI for Mg in the case of an alkaline solution increased by 25.0% (MKPC-BFS), 29.7% (MKPC-FA-BFS), and 33.7% (MKPC-FA). The highest leachability index values for K (LI=9.74) are characteristic of MKPC-FA, for Mg (LI=15.49), P (LI=10.61) and B (LI=9.29) – MKPC-BFS (Table 4).

Table 3

Leachability indices LI and effective diffusion coefficients D_e for elements released in alkaline solution and distilled water

Element	LI/ D_e , $\text{cm}^2 \cdot \text{s}^{-1}$					
	MKPC-BFS		MKPC-FA-BFS		MKPC-FA	
	alkaline solution	distilled water	alkaline solution	distilled water	alkaline solution	distilled water
Mg	15.49/7.98·10 ⁻¹⁶	12.39/8.29·10 ⁻¹³	15.45/1.56·10 ⁻¹⁵	11.91/3.41·10 ⁻¹²	15.19/1.27·10 ⁻¹⁵	11.36/8.26·10 ⁻¹²
K	9.17/1.59·10 ⁻⁹	9.61/5.31·10 ⁻¹⁰	9.06/3.29·10 ⁻⁹	9.15/1.47·10 ⁻⁹	9.74/9.49·10 ⁻¹⁰	9.50/6.78·10 ⁻¹⁰
P	10.15/1.19·10 ⁻¹⁰	10.61/3.86·10 ⁻¹¹	10.00/1.90·10 ⁻¹⁰	10.29/8.91·10 ⁻¹¹	9.8/3.51·10 ⁻⁹	9.97/1.65·10 ⁻¹⁰
B	9.29/1.34·10 ⁻⁹	9.60/5.21·10 ⁻¹⁰	9.27/2.28·10 ⁻⁹	8.99/1.76·10 ⁻⁹	9.24/2.70·10 ⁻⁹	9.06/1.42·10 ⁻⁹

The US Nuclear Regulatory Commission has defined a minimum value of six (6.0) for the leachability index as an acceptance criteria for radioactive waste forms to be disposed of at controlled sites [23], i.e., the leachability index, as calculated in accordance with ANSI 16.1, should be greater than 6. The leaching tests carried out in this work according to the ANSI/ANS 16.1 standard show that this requirement is checked for the main elements (Mg, K, P) and B of the MKPC matrices, regardless of the leaching solution (distilled water or alkaline solution). Indeed, the leachability indices are ≥ 9 (see Table 3). This means that it seems possible to use blast furnace slag, in addition or substitution to fly ash, as a filler in MKPC materials, which is extremely important for Ukrainian needs [22].

2.2. X-RAY ANALYSIS

The crystalline phase composition of all obtained MKPC based specimens cured for 28 days was studied by X-ray diffraction analysis (XRD) (DRON-4-07 device using copper Cu-K α radiation and a Ni selective absorbing filter). Crystalline phase quantification was performed using MAUD (Materials Analysis Using Diffraction) software to analyse diffraction data using the combined Rietveld method.

Rietveld quantification of MKPC-FA, MKPC-BFS, and MKPC-FA-BFS specimens shows that the content of K-struvite in the crystalline fraction of the three materials is almost the same and is equal to 58...60% (see Table 4).

Table 4

Quantitative phase composition of the initial MKPC specimen

Composition	Phase	%	Lattice parameters, Å
MKPC-FA	Quartz	38	a=4.912, c=5.398
	K-struvite	58	a=6.875, b=6.160, c=11.083
	Mullite	4	a=7.583, b=7.650, c=2.886
MKPC-BFS	Quartz	31	a=4.913, c=5.414
	K-struvite	60	a=6.897, b=6.188, c=11.109
	Akermanite	9	a=7.783, c=5.028
MKPC-FA-BFS	Quartz	34	a=4.913, c=5.402
	K-struvite	59	a=6.874, b=6.158, c=11.091
	Akermanite	7	a=7.773, c=5.020

MKPC-BFS specimen is characterized by the presence of K-struvite with higher crystal lattice parameters, as comparison to both MKPC-FA and MKPC-FA-BFS specimens. This indicates that, during the preparation of MKPC-BFS specimen, a more significant dissolution of the BFS particles occurs, releasing various elements that are included in the K-

struvite crystal structure with an increase in its crystal lattice parameters. The observation of the mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$) in MKPC-FA specimen (Fig. 4.a) is explained by its presence in initial FA powder. Similarly, akermanite ($\text{Ca}_2\text{MgSi}_2\text{O}_7$) observed in both MKPC-BFS (Fig. 4.b) and MKPC-FA-BFS (Fig. 4.c) specimens is a component of the BFS powder [22].

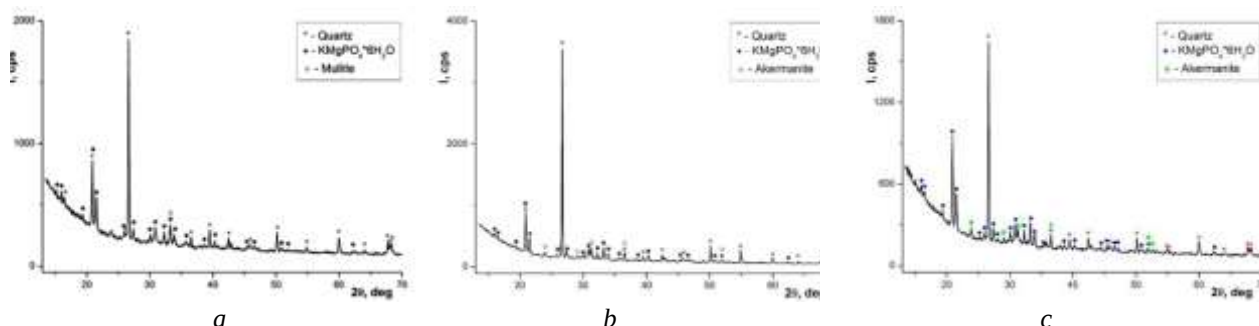


Fig. 4. Diffraction patterns of the initial specimens:
a – MKPC-FA; b – MKPC-BFS; c – MKPC-FA-BFS

After leaching for 90 days, X-ray analysis of the MKPC specimen surface was performed. The analysis was made on powder specimens, which were obtained by scratching the surface layer of the specimens. The thickness of the studied layer was 1...2 mm. Fig. 5 shows the XRD patterns of the surface of MKPC-FA, MKPC-BFS and MKPC-FA-BFS specimens. As can be

seen, K-struvite is absent from the surface layer of all specimens after leaching for 90 days. On the contrary, the presence of the hasenite ($\text{KNaMg}_2(\text{PO}_4)_2 \cdot 14\text{H}_2\text{O}$) phase is observed, which was deposited on the surface of the specimens as a result of K-struvite dissolution and the presence of Na^+ in alkaline solution.

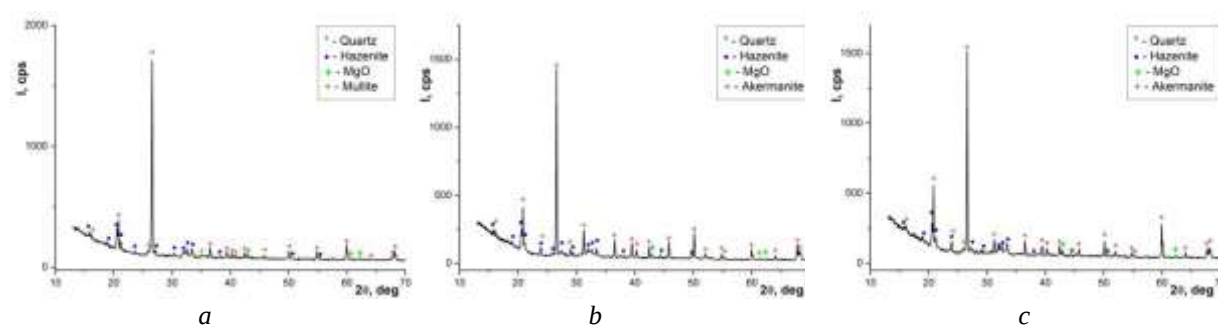


Fig. 5. Diffraction patterns of the specimens surface after leaching in alkaline solution:
a – MKPC-FA; b – MKPC-BFS; c – MKPC-FA-BFS

There is also a small amount of unreacted MgO (1.7...2.3%) and a significantly larger amount of quartz (58.3...60.7%) as a result of the addition of sand as a filler (Table 5). In addition to hasenite, MgO and quartz, mullite (7.5%) resulting from the fly ash addition was identified in the surface layer of the MKPC-FA specimen. The MKPC-BFS and MKPC-FA-BFS specimens are characterized by the presence of

akermanite (14.5 and 7.5%, respectively) coming from blast furnace slag.

In contrast to leaching of MKPC in alkaline solution, leaching in distilled water ($\text{pH} \sim 6.5$) results in the formation of bobierrite ($\text{Mg}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$) and dolomite ($\text{CaMg}(\text{CO}_3)_2$) in the near-surface layer of the specimens. Thus, after leaching tests in distilled water for 90 days according to the ANS 16.1 test, K-struvite,

quartz, akermanite, dolomite and bobierrite were found in the near-surface layer (thickness ~ 1 mm) of the MKPC-BFS and MKPC-FA-BFS specimens [24]. Formation of bobierrite and dolomite was previously noted in the layer on the surface of the MKPC specimen after leaching (ANS 16.1 test) in deionized water for 387 days [25].

Lahalle et al. studied the evolution of MKPC specimens (Mg/P molar ratio = 1) immersed in an alkaline solution simulating the pore solution of concrete prepared using Portland cement mixed with fly

ash and blast furnace slag over time [17]. The leaching solution had a pH close to 13.2 and contained K (133 mmol/l), Na (66 to 107 mmol/l) and Ca (1 mmol/l) ions, and in some cases chlorides (40 mmol/l). The authors suggested a possible replacement of K^+ by Na^+ in the K-struvite structure, leading to the formation of hazenite. Xu et al. showed that the main phase of K-struvite in MKPC is destabilized to bobierrite ($Mg_3(PO_4)_2 \cdot 8H_2O$) when exposed to solutions with $pH \leq 7$ and to brucite ($Mg(OH)_2$) and hazenite at $pH > 7$ [26].

Table 5

Quantitative phase composition of the specimens surface after leaching

Composition	Phase	%	Lattice parameters, Å
MKPC-FA	Quartz	58.7	a=4.921, c=5.411
	Hazenite	32.1	a=6.935, b=25.272, c=11.244
	MgO	1.7	a=4.213
	Mullite	7.5	a=7.57, b=7.69, c=2.89
MKPC-BFS	Quartz	60.7	a=4.916, c=5.412
	Hazenite	23.4	a=6.952, b=25.237, c=11.254
	MgO	1.7	a=4.214
	Akermanite	14.2	a=7.775, c=5.035
MKPC-FA-BFS	Quartz	58.3	a=4.919, c=5.412
	Hazenite	31.9	a=6.956, b=25.245, c=11.254
	MgO	2.3	a=4.215
	Akermanite	7.5	a=7.781, c=5.035

Comparison of these results with the sufficiently high values of leachability indices for Mg, K, P, and B suggests the formation of a protective layer on the surface of MKPC specimens, slowing down their release into distilled water or alkaline solution. In distilled water, the protective layer consists of bobierrite, dolomite, quartz and akermanite for MKPC specimens with the addition of blast-furnace slag. In the case of leaching in an alkaline solution, the protective layer is formed on the surface of MKPC specimens with the addition of fly ash and blast-furnace slag from hazenite, quartz and mullite and from hazenite, quartz and akermanite, respectively.

The leaching test conducting allows to evaluate the efficiency of using a specific matrix for radioactive waste stabilization. In this case, monolithic MKPC specimens were leached (ANS 16.1 test) in a periodically replaced alkaline solution. If we consider the results of leaching tests of magnesium-potassium-phosphate cement with various fillers, we can conclude that potassium is the most leached of the studied elements. Potassium is leached to the greatest extent from MKPC with blast furnace slag additives. Phosphorus is in second place in terms of leaching. Boron follows phosphorus. Magnesium is leached to the least extent. The highest leaching of P, B, and Mg is characteristic of MKPC with fly ash additives. At the same time, for all the main elements of the MKPC matrix both with blast furnace slag and fly ash additives, the obtained leaching index values are $LI \geq 9$. This fact indicates that MKPC matrices can function as highly effective waste forms for radioactive waste immobilization.

CONCLUSIONS

This study was aimed at understanding the behavior of MKPC over time when exposed to alkaline solution, which MKPC may encounter when used as a matrix material for immobilization of radioactive waste. Tests for leaching of MKPC specimens in alkaline solution were carried out in accordance with ANS 16.1.

XRD analysis showed the formation of hazenite ($KNaMg_2(PO_4)_2 \cdot 14H_2O$) and the disappearance of K-struvite in the surface layer of MKPC specimens with both fly ash and blast furnace slag additives after leaching in alkaline solution.

The highest leachability index LI values were obtained for MKPC specimens containing blast furnace slag (MKPC-BFS and MKPC-FA-BFS) for all investigated elements (Mg, K, P, B), except K. In this latter case, using fly ash as a filler seemed more advantageous.

For all tested materials, the major elements Mg, K, P, and B showed limited leachability ($LI \geq 9$) in both distilled water and an alkaline solution mimicking the internal waters of radioactive waste storage facilities.

The use of blast furnace slag as a filler makes it possible to design a MKPC-based material with a chemical resistance at least as good as that obtained with fly ash.

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ХАРАКТЕРИСТИКИ ВИЛУГОВУВАННЯ МАГНІЙ-КАЛІЙ-ФОСФАТНОГО ЦЕМЕНТУ В ЛУЖНОМУ РОЗЧИНІ

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Досліджували характеристики вилуговування магній-калій-фосфатного цементу (МКФЦ) у лужному розчині. Испити на вилуговування в лужному розчині проводили на зразках МКФЦ згідно з тестом ANSI/ANS 16.1. Склад розчинів після вилуговування був визначений за допомогою ICP-AES, і на основі отриманих даних розраховані кумулятивні концентрації, ефективні коефіцієнти дифузії та індекси вилуговування для Mg, K, P та B. За допомогою рентгенівської дифракції/аналізу Ритвельда проаналізовано зміну фазового складу поверхневого шару МКФЦ зразків після вилуговування. Для всіх досліджених матеріалів основні елементи Mg, K, P та B продемонстрували обмежену здатність до вилуговування (індекс вилуговування ≥ 9) у лужному розчині, що імітує внутрішні води сховищ радіоактивних відходів.