

МАГНИЙФОСФАТНАЯ СВЯЗКА НА ОСНОВЕ БРУСИТА, ЕЕ АНАЛИЗ И ПРИМЕНЕНИЕ ДЛЯ СПЕКАНИЯ ПЕРИКЛАЗА

Н.Ф. Косенко, Н.В. Филатова, М.А. Глазков

Надежда Федоровна Косенко, Наталья Владимировна Филатова *, Максим Андреевич Глазков

Кафедра технологии керамики и наноматериалов, Ивановский государственный химико-технологический университет, пр. Шереметевский, 7, Иваново, Российская Федерация, 153000

E-mail: nfkosenko@gmail.com, zyanata@mail.ru *, bellechik.super@yandex.ru

Представлены результаты синтеза и физико-химического исследования магний-фосфатной связки, исходя из природного брусита Кульдурского месторождения, состоящего в основном из гидроксида магния $Mg(OH)_2$, и 60 % раствора H_3PO_4 . По данным термогравиметрии установили, что все потери массы относились к многоступенчатому удалению воды. До 140 °С удалялась кристаллогидратная влага. В дальнейшем однозамещенный гидрофосфат магния превращался в $MgH_2P_2O_7$ и $Mg_2P_4O_{12}$, что не противоречит литературным данным. При нагревании бруситфосфатной связки (БФС) дегидратация сопровождалась эндотермическими эффектами. В интервале 450–575 °С наблюдался слабый экзотермический эффект, который мог быть связан с перестройкой структуры (циклизацией) первичных метафосфатов магния и/или кристаллизацией безводных циклофосфатов из первично выделившейся аморфной фазы, несвязанной с изменением массы. При 500–1000 °С фазовый состав оставался неизменным, что хорошо согласуется с данными, полученными другими методами. С помощью ИК спектроскопии подтверждено наличие в структуре циклов из фосфор-кислородных тетраэдров. БФС достаточно устойчива; время живучести не менее года. Для определения кинетических параметров спекания периклаза с БФС была использована модель, учитывающая роль физического уплотнения и химического связывания в присутствии связующего в процессе нагревания. Анализ параметров показал, что прочность конгломерата обеспечивалась как за счет собственно спекания, так и за счет действия связующего; при этом предполагаемый вклад химического фактора максимален при низких температурах. При дальнейшем нагревании диффузионные процессы активизируются, и собственно спекание активизируется. Определили эффективную энергию активации спекания периклаза: $E_a=255\pm 8$ кДж/моль, которая совпадает с энергией активации диффузии кислорода в MgO (252,05 кДж/моль).

Ключевые слова: магнийфосфатная связка, бруситфосфатная связка, брусит, фосфаты магния, термический анализ, периклаз, спекание, кинетика

BRUCITE-BASED MAGNESIUM PHOSPHATE BONDING AGENT, ITS ANALYSIS AND APPLICATION FOR PERICLASE SINTERING

N.F. Kosenko, N.V. Filatova, M.A. Glazkov

Nadezhda F. Kosenko, Natalya V. Filatova *, Maxim A. Glazkov

Department of Technology of Ceramic and Nanomaterials, Ivanovo State University of Chemistry and Technology, Sheremetevskiy ave., 7, Ivanovo, 153000, Russia

E-mail: nfkosenko@gmail.com, zyanata@mail.ru *, bellechik.super@yandex.ru

Some results on magnesium phosphate binder obtained from natural brucite (mainly $Mg(OH)_2$) and H_3PO_4 were discussed. Hereafter, it was named brucite phosphate (BPB). This binder then was used to the sintering of MgO (periclase). MgO is one of the most high-refractory materials and chemically stable to various aggressive mediums, so the aim was presented as actual. To determine the phase composition data on sample weight loss in different temperature intervals

were compared. All weight losses were related to the multistage water moving away. Up to 140 °C it was the removal of crystalline hydrate water. Then monosubstituted magnesium hydrophosphate transformed into $MgH_2P_2O_7$ and $Mg_2P_4O_{12}$, that in principle was in accordance with literature data. Dehydration was accompanied with appropriate endothermal effects. There was a slight exothermal effect which could relate to a structure rearrangement (cyclization) of primary magnesium metaphosphates and/or some crystallization of anhydrous cyclophosphates from primarily obtained amorphous phase non-connected with a weight loss in the interval of 450-575 °C. At 500–1000 °C the phase composition remained constant that was in a good accordance with data obtained by other methods. By means of IR-spectroscopy, it was confirmed the formation of structure cycles composed from oxygen-phosphor tetrahedrons. To determine kinetics parameters of periclase sintering with BPB it was used a model being proposed that took the role of a physical consolidation and a chemical binding in the presence of binder into account under heating. The obtained parameters analysis showed that the forming conglomerate strength was provided with the sintering properly as well as with binder action; an estimated chemical factor contribution was maximal at low temperatures (1100-1200 °C). The activation energy value was practically identical to the activation energy of oxygen diffusion in MgO.

Key words: magnesium phosphate binder, brucite phosphate binder, brucite, magnesium phosphates, thermal analysis, periclase, sintering, kinetics

Для цитирования:

Косенко Н.Ф., Филатова Н.В., Глазков М.А. Магнийфосфатная связка на основе брусита, ее анализ и применение для спекания периклаза. *Изв. вузов. Химия и хим. технология*. 2019. Т. 62. Вып. 12. С. 119–124

For citation:

Kosenko N.F., Filatova N.V., Glazkov M.A. Brucite-based magnesium phosphate bonding agent, its analysis and application for periclase sintering. *Izv. Vyssh. Uchebn. Zaved. Khim. Khim. Tekhnol.* 2019. V. 62. N 12. P. 119–124

INTRODUCTION

The bonding properties of metal phosphates are recognized for numerous years [1]. Phosphate substances are of interest due to high melting points, hardening of many compositions at ambient temperatures and considerable strength. Phosphate systems are exploited as dental cements [1], refractories [2], coatings [3], etc. Early works on phosphate materials were reviewed [4]. Many compositions of cold and thermal setting and hardening have been worked out. In recent years the interest to phosphate cements and binder became stronger because of application field enhancement. One of key position among them is taken by magnesium phosphates-based materials. They are the most readily available, harden rapidly, have a little shrinkage, high early strength at ambient temperatures that considerably increases under heating. These advantages have rendered magnesium phosphates cements (MPCs) popular in various applications including fast-repair of civil structures [5], in refractories and coatings [6,7]. Several cements are used as biological material including orthopedic [8], dental [9], antibacterial preparations [10], for porous and foamed concretes fabrication [11]. The important direction of MPCs applications is stabilization/solidification of hazardous substances, nuclear and wood wastes [12,13]. Recently, some new ways of MPCs use have been formed, such as thermal energy storage [14], 3D printing [15], etc.

At present, magnesia is often mixed with one of hydrogen salts: ammonium $NH_4H_2PO_4$ [16] or potassium KH_2PO_4 [17, 18] instead of phosphoric acid. It allows decreasing the intensity of components interaction and in some cases avoiding the necessity of MgO activity reduction to diminish heat release that hinders the strength structure forming. It is not a problem under a liquid binder preparation, as a binder is prepared in advance and used in the form of metastable solution of acid salts [1, 19]. Besides, an exothermic reaction allows obtaining of a binder without heat.

Whereas compositions, structure and properties of MPCs depending on many factors have been elaborated, it would not say about magnesium phosphate binders (MPBs). Since MPBs for the most part are used in refractories and high-temperature coatings, the presence of NH_4^+ , K^+ , Na^+ ions is not desirable. An ammonium salt decomposes under heating and releases unpleasant ammonia which aerates a green product and restricts its outdoor applications. Alkaline ions are active fusing agents that reduce the refractoriness of a sintered material.

Earlier thermal transformations of several phosphates have been investigated [20,21]. MgO is also of interest from the point of view of its reactivity [22].

MPBS are obtained on the base of reactive MgO or a technical product: caustic magnesite or periclase. However, it is known [1], that binders could be also synthesized using any raw material of a natural origin (talc, mica, kaolin, etc.).

In the present work, we discuss some results on magnesium phosphate binder obtained on the base of natural brucite (mainly $Mg(OH)_2$) and phosphoric acid. Hereafter, it was named brucite phosphate (BPB). This binder then was used to the sintering of MgO (periclase). As MgO is one of the most high-refractory materials and stable to various aggressive mediums, the aim was presented as actual.

EXPERIMENTAL PART

A commercially available brucite of Kuldur mine (Jewish Autonomous Region) was used. Its chemical analysis reported MgO 96.7 wt%, CaO 1.95%, SiO_2 0.80%, Fe_2O_3 0.10%, loss on ignition 32.4. When brucite powder was added to H_3PO_4 , an exothermic reaction was observed. The brucite powder was stirred in a 60 wt% thermal phosphoric acid (Russian National State Standard 10678) solution. The weight ratio of MgO and H_3PO_4 ensured the acid neutralization to monosubstituted $Mg(H_2PO_4)_2 \cdot nH_2O$ formation (with 50% excess of H_3PO_4):



Magnesium dihydrophosphate is a water-soluble compound. The obtained binder contained undissolved suspended solids was filtered through a ceramic filter, and then was kept in a tightly closing vessel.

Differential thermal analysis (DTA) and thermogravimetry (TG) were recorded using a METTLER TOLEDO STAR^e System TGA/SDTA851e/LF/1600. In these tests, samples were heated up to 900 °C at a rate of 5 °C/min under an atmosphere of flowing air (50 mL/min) with α -alumina as reference substance. Infrared (IR) spectroscopy was conducted on KBr using Avatar 360-FT-IR.

Sintered periclase (China) for high-temperature treatment with BPB was refractory grade; 70% of particles had a particle size 15-50 μm . Samples were cold-pressed under 200 MPa into tablets and were submitted to a thermal treatment 15-150 min at 1100-1600 °C in electric furnace in air atmosphere.

RESULTS AND DISCUSSION

TG and DTA were conducted for BPB samples partially pre-dried in a vacuum drying chamber (Fig. 1). To determine the phase composition experimental and calculated data on sample weight loss in different temperature intervals were compared (Table 1). All weight losses were related to the multistage water moving away. Up to 140 °C it was the removal of crystalline hydrate water.

Then monosubstituted magnesium hydrophosphate transformed into $MgH_2P_2O_7$ and $Mg_2P_4O_{12}$. Dehydration was accompanied with appropriate endothermal effects. There was a slight exothermal effect which

could relate to a structure rearrangement (cyclization) of primary magnesium metaphosphates and/or some crystallization anhydrous cyclophosphates from primarily obtained amorphous phase non-connected with a weight loss in the interval of 450-575 °C. At 500-1000 °C the phase composition remained constant that was in a good accordance with data obtained by other methods. It is well-known [20], that cyclotetraphosphates (CTPs) of different metals are stable in a wide temperature interval. At high temperature (> 1000 °C) P_2O_5 gradually removed, and magnesium pyro- and orthophosphate were formed.

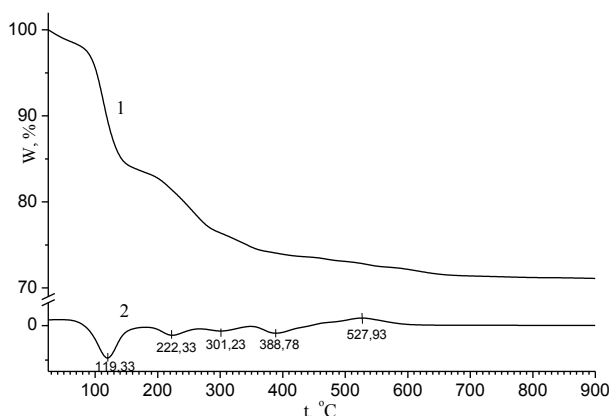


Fig. 1. TG (1) and DTA (2) curves of brucite phosphate binder
Рис. 1 Кривые ТГ (1) и ДТА (2) для бруситфосфатной связки

Table 1

Phase transformations of BPB under heating
Таблица 1. Фазовые превращения БФС при нагревании

Temperature interval, °C	Decomposition stage	Weight loss, %	
		Experimental	Calculated
< 140	$Mg(H_2PO_4)_2 \cdot 2H_2O \rightarrow Mg(H_2PO_4)_2 + 2H_2O$	13.8	14.2
180-260	$Mg(H_2PO_4)_2 \rightarrow MgH_2P_2O_7 + H_2O$	7.9	7.1
260-330	$MgH_2P_2O_7 \rightarrow Mg_2P_4O_{12} \cdot H_2O + H_2O$	3.9	3.5
360-450	$Mg_2P_4O_{12} \cdot H_2O \rightarrow Mg_2P_4O_{12} + H_2O$	3.5	3.5
	Sum	29.1	28.3

As an amorphous phase was presented in all temperature interval, and it was predominant at high and low temperatures, X-ray diffraction was not an infallible method for the phase constitution definition. So, X-ray pictures for the binder were not presented. We used IR-spectroscopy to confirm the BPB composition. After BPB burning at 900 °C and 1000 °C spectra were practically identical, and magnesium CTP presented the only phase (Fig. 2).

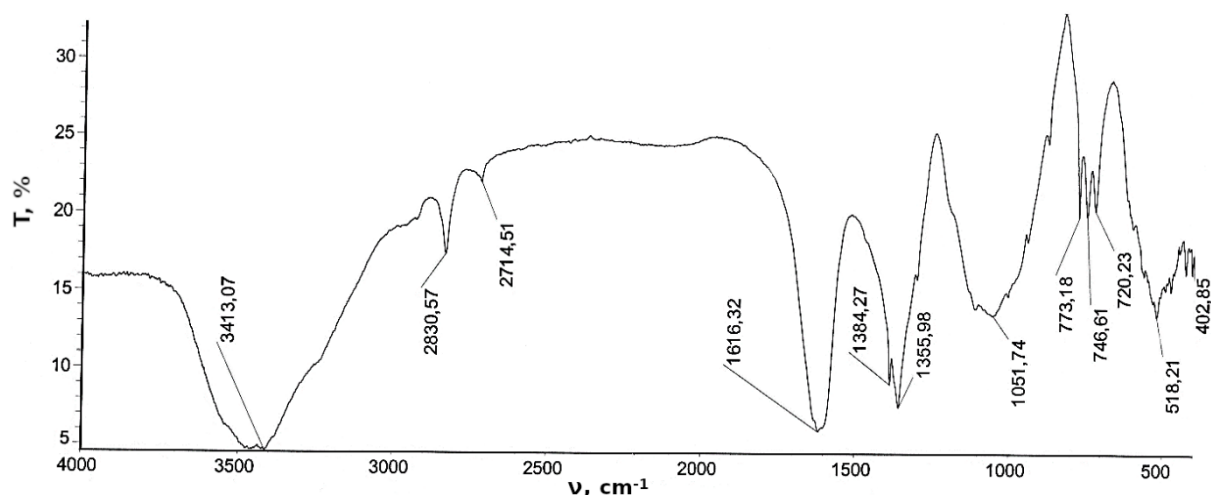


Fig. 2. IR spectrum of brucite phosphate binder burned at 900 °C
 Рис. 2. ИК-спектр бруситфосфатной связки, обожженной при 900 °C

In the wave number field of 1000-1200 and 400-550 cm^{-1} a strong absorption was observed, besides, bands had sufficiently complicated contour. Many CTP bands relate to the structure complexity [23]. Each of four valence vibration groups (ν_{as} и ν_{s} O^-PO^- and ν_{as} и ν_{s} POP) give two vibrations that are active in IR-spectra: ν_{as} O^-PO^- – three bands in the interval of 1300-1400 cm^{-1} , ν_{s} O^-PO^- – several joined bands at 1000-1200 cm^{-1} , ν_{s} POP – three bands in the field of 700-780 cm^{-1} . Besides, cycles P_4O_{12} have the symmetry S_4 in crystal; it complicates additionally the character of particle vibrations [23]. The characteristic bands at 700-770 cm^{-1} related to structure cycles composed from bounded oxygen-phosphor tetrahedrons.

Brucite phosphate binder was rather sable; its time of liveness was not less than 1 year, that was convenient for its use.

Further, periclase sintering with BPB was studied. Earlier [24] a model for corundum materials

sintering kinetics had been proposed; it took the role of a physical consolidation and a chemical binding in the presence of alumina-boron-phosphate binder into account under heating:

$$\sigma_{\text{destr}} = k\tau^n + \chi\tau^m,$$

where σ_{destr} is destruction strength (ultimate compressive strength); k is an effective sintering rate constant, τ is time, n is coefficient characterizing the sintering mechanism; χ и m are coefficients, defining a chemical factor contribution into strengthening of sintering material (not are constants of chemical reaction).

In this work, an attempt of treatment of experimental data obtained for periclase strengthening in the presence of BPB using this model has been done. Computer simulation was carried out by means of program package MathCad. Experimental and calculated parameters are given in Table 2.

Table 2

Sintering parameters of periclase with BPS
 Таблица 2. Параметры спекания периклаза с БФС

Strength and kinetics parameters	Burning temperature, °C					
	1100	1200	1300	1400	1500	1600
Compressive strength after burning (150 min) σ_{150} , МПа	18.9 ± 0.3	36.6 ± 0.6	53.9 ± 0.7	70.8 ± 0.9	94.9 ± 0.3	106 ± 2
Effective rate constant k	4.06 · 10 ⁻⁵	1.87 · 10 ⁻⁴	5.78 · 10 ⁻⁴	1.49 · 10 ⁻³	5.17 · 10 ⁻³	1.02 · 10 ⁻²
Chemical factor contribution $\chi\tau^m$, %	70.2	35.5	21.7	11.0	7.3	5.1

The obtained parameters analysis showed that the forming conglomerate strength was provided with the sintering properly as well as with binder action; an estimated chemical factor contribution was maximal at low temperatures (1100-1200 °C), as it could be expected. Under further heating, diffusive processes were activated, and sintering properly intensified. In MgO,

diffusion coefficient of oxygen ions is considerably less than that for magnesium ions, so their diffusion was the limiting stage of the sintering. Arrhenius plot (Fig. 3) of periclase sintering with brucite phosphate binder (coefficient of linear approximation 0.997) allowed determination of the effective activation energy, that was equal to (255±8) kJ/mole.

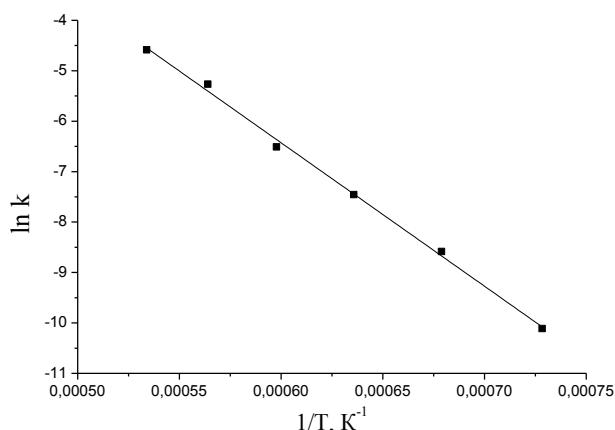


Fig. 3. Arrhenius plot of periclase sintering with brucite phosphate binder

Рис. 3. Зависимость в координатах уравнения Аррениуса для спекания периклаза с бруситфосфатной связкой

This value was practically identical to the activation energy of oxygen diffusion in MgO (252,05 kJ/mole [25]). Comparing obtained activation energy values, the MgO sintering could be considered from the position of dislocation-viscous flow [26].

ЛИТЕРАТУРА

1. **Судакас Л.Г.** Фосфатные вяжущие системы. СПб: РИА "Квинтет". 2006. 260 с.
2. **Lopes S.J.S., Luz A.P., Gomes D.T., Pandolfelli V.C.** Self-flowing high-alumina phosphate-bonded refractory castables. *Ceram. Int.* 2017. V. 43. N 8. P. 6239-6249. DOI: 10.1016/j.ceramint.2017.02.023.
3. **Chen C., Feng B., Hu S., Zhang Y., Li S., Gao L., Zhang X., Yu K.** Control of aluminum phosphate coating on mullite fibers by surface modification with polyethylenimine. *Ceram. Int.* 2018. V. 44. N 1. P. 216-224. DOI: 10.1016/j.ceramint.2017.09.161.
4. **Walling S.A., Provis J.L.** Magnesia-based cements: a journey of 150 years, and cements for the future? *Chem. Rev.* 2016. V. 116. P. 4170-4204. DOI: 10.1021/acs.chemrev.5b00463.
5. **Li J.S., Zhang W.B., Cao Y.** Laboratory evaluation of magnesium phosphate cement paste and mortar for rapid repair of cement concrete pavement. *Constr. Build. Mater.* 2014. V. 58. P. 122-128. DOI: 10.1016/j.conbuildmat.2014.02.015.
6. **Fang Y., Cui P., Ding Z., Zhu J.-X.** Properties of a magnesium phosphate cement-based fire-retardant coating containing glass fiber or glass fiber powder. *Constr. Build. Mater.* 2018. V. 162. P. 553-560. DOI: 10.1016/j.conbuildmat.2017.12.059.
7. **Ren Y., Babaie E., Lin B., Bhaduri S.B.** Microwave-assisted magnesium phosphate coating on the AZ31 magnesium alloy. *Biomed. Mater.* 2017. V. 12. P. 045026. DOI: 10.1088/1748-605X/aa78c0.
8. **Nabiyouni M., Brückner T., Zhou H., Gbureck U., Bhaduri S.B.** Magnesium-based bioceramics in orthopedic applications. *Acta Biomater.* 2018. V. 66. P. 23-43. DOI: 10.1016/j.actbio.2017.11.033.
9. **Mestres G., Aguilera F.S., Manzanares N., Sauro S., Osorio R., Toledano M., Ginebra M.P.** Magnesium phosphate cements for endodontic applications with improved long-term sealing ability. *Int. Endod. J.* 2014. V. 47. P. 127-139. DOI: 10.1111/iej.12123.
10. **Mestres G., Ginebra M.-P.** Novel magnesium phosphate cements with high early strength and antibacterial properties. *Acta Biomater.* 2011. V. 7. N 4. P. 1853-1861. DOI: j.actbio.2010.12.008.
11. **Ma C., Chen B.** Experimental study on the preparation and properties of a novel foamed concrete based on magnesium phosphate

Thus, the selected mathematic model described adequately processes under periclase sintering with brucite-phosphate binder.

CONCLUSION

Magnesium phosphate bonding agent based on a natural brucite (mainly Mg(OH)₂) was synthesized by means of the brucite dissolution in 60% phosphoric acid solution. Obtained binder had a time of liveness was not less than 1 year.

A physicochemical investigation (thermal analysis, IR spectroscopy) allowed to determine the phase composition based on sample weight loss in different temperature intervals. Some kinetics parameters (effective rate constants, activation energy) on periclase sintering with brucite phosphate binder have been obtained.

ACKNOWLEDGEMENTS

The research was performed using resources of the Center for Collective Use, Ivanovo State University of Chemical Technology "ISUCT".

REFERENCES

1. **Sudakas L.G.** Phosphate binding systems. SPb.: RIA "Kvintet". 2006. 260 p. (in Russian).
2. **Lopes S.J.S., Luz A.P., Gomes D.T., Pandolfelli V.C.** Self-flowing high-alumina phosphate-bonded refractory castables. *Ceram. Int.* 2017. V. 43. N 8. P. 6239-6249. DOI: 10.1016/j.ceramint.2017.02.023.
3. **Chen C., Feng B., Hu S., Zhang Y., Li S., Gao L., Zhang X., Yu K.** Control of aluminum phosphate coating on mullite fibers by surface modification with polyethylenimine. *Ceram. Int.* 2018. V. 44. N 1. P. 216-224. DOI: 10.1016/j.ceramint.2017.09.161.
4. **Walling S.A., Provis J.L.** Magnesia-based cements: a journey of 150 years, and cements for the future? *Chem. Rev.* 2016. V. 116. P. 4170-4204. DOI: 10.1021/acs.chemrev.5b00463.
5. **Li J.S., Zhang W.B., Cao Y.** Laboratory evaluation of magnesium phosphate cement paste and mortar for rapid repair of cement concrete pavement. *Constr. Build. Mater.* 2014. V. 58. P. 122-128. DOI: 10.1016/j.conbuildmat.2014.02.015.
6. **Fang Y., Cui P., Ding Z., Zhu J.-X.** Properties of a magnesium phosphate cement-based fire-retardant coating containing glass fiber or glass fiber powder. *Constr. Build. Mater.* 2018. V. 162. P. 553-560. DOI: 10.1016/j.conbuildmat.2017.12.059.
7. **Ren Y., Babaie E., Lin B., Bhaduri S.B.** Microwave-assisted magnesium phosphate coating on the AZ31 magnesium alloy. *Biomed. Mater.* 2017. V. 12. P. 045026. DOI: 10.1088/1748-605X/aa78c0.
8. **Nabiyouni M., Brückner T., Zhou H., Gbureck U., Bhaduri S.B.** Magnesium-based bioceramics in orthopedic applications. *Acta Biomater.* 2018. V. 66. P. 23-43. DOI: 10.1016/j.actbio.2017.11.033.
9. **Mestres G., Aguilera F.S., Manzanares N., Sauro S., Osorio R., Toledano M., Ginebra M.P.** Magnesium phosphate cements for endodontic applications with improved long-term sealing ability. *Int. Endod. J.* 2014. V. 47. P. 127-139. DOI: 10.1111/iej.12123.
10. **Mestres G., Ginebra M.-P.** Novel magnesium phosphate cements with high early strength and antibacterial properties. *Acta Biomater.* 2011. V. 7. N 4. P. 1853-1861. DOI: j.actbio.2010.12.008.
11. **Ma C., Chen B.** Experimental study on the preparation and properties of a novel foamed concrete based on magnesium phosphate cement. *Constr. Build. Mater.* 2017. V. 137. P. 160-168. DOI: 10.1016/j.conbuildmat.2017.01.092.

- cement. *Constr. Build. Mater.* 2017. V. 137. P. 160–168. DOI: 10.1016/j.conbuildmat.2017.01.092.
12. **Wagh A.S., Sayenko S.Y., Shkuropatenko V.A., Tarasov R.V., Dykiy M.P., Svitlychniy Y.O., Virych V.D., Ulybkina E.A.** Experimental study on cesium immobilization in struvite structures. *J. Hazard. Mater.* 2016. V. 302. P. 241–249. DOI: j.jhazmat.2015.09.049.
 13. **Wang L., Yu I.K.M., Tsang D., Li S., Poon C.S.** Mixture design and reaction sequence for recycling construction wood waste into rapid-shaping magnesia-phosphate cement particleboard. *Ind. Eng. Chem. Res.* 2017. V. 56. P. 6645–6654. DOI: 10.1021/acs.iecr.7b01175.
 14. **Maldonado-Alameda A., Lacasta A.M., Giro-Paloma J., Chimenos J.M., Haurie L., Formosa J.** Magnesium phosphate cements formulated with low grade magnesium oxide incorporating phase change materials for thermal energy storage. *Constr. Build. Mater.* 2017. V. 155. P. 209–216. DOI: 10.1016/j.conbuildmat.2017.07.227.
 15. **Klammert U., Vorndran E., Reuther T., Müller F.A., Zorn K., Gbureck U.** Low temperature fabrication of magnesium phosphate cement scaffolds by 3D powder printing. *J. Mater. Sci. Mater. Med.* 2010. V. 21. N 11. P. 2947–2953. DOI: 10.1007/s10856-010-4148-8.
 16. **Fan S., Chen B.** Experimental study of phosphate salts influencing properties of magnesium phosphate cement. *Constr. Build. Mater.* 2014. V. 65. P. 480–486. DOI: 10.1016/j.conbuildmat.2014.05.021.
 17. **Xing F., Ding Z., Li Z.-J.** Study of potassium-based magnesium phosphate cement. *Adv. Cement Res.* 2011. V. 23. N 2. P. 81–87. DOI: 10.1680/adcr.9.00030.
 18. **Gardner L.J., Lejeune V., Corkhill C.L., Bernal S.A., Provis J.L., Stennett M.C., Hyatt N.C.** Evolution of phase assemblage of blended magnesium potassium phosphate cement binders at 200° and 1000 °C. *Adv. Appl. Ceram.* 2015. V. 114. N 7. P. 386–392. DOI: 10.1179/1743676115Y.0000000064.
 19. **Abyzov V.A.** Lightweight refractory concrete based on aluminum-magnesium-phosphate binder. *Proc. Eng.* 2016. V. 150. P. 1440–1445. DOI: 10.1016/j.proeng.2016.07.077.
 20. **Косенко Н.Ф., Филатова Н.Ф., Фукина Т.А.** Термические превращения алюмоборфосфатного связующего. *Неорган. матер.* 2004. Т. 40. № 10. С. 1276–1280.
 21. **Tricot G., Coilot D., Creton E., Montagne L.** New insights into the thermal evolution of aluminophosphate solutions: A complementary XRD and solid state NMR study. *J. Eur. Cer. Soc.* 2008. V. 28. P. 1135–1141 DOI: 10.1016/j.jeurceramsoc.2007.09.046.
 22. **Косенко Н.Ф., Филатова Н.В.** Регулирование активности связующих материалов механико-химическими методами. *Изв. вузов. Химия и хим. технология.* 2018. Т. 61. Вып. 1. С. 66–71. DOI: 10.6060/tcct.20186101.5664.
 23. **Печковский В.В., Дзюба Е.Д.** Атлас инфракрасных спектров фосфатов. Конденсированные фосфаты. М.: Наука. 1985. 240 с.
 24. **Косенко Н.Ф., Филатова Н.В., Денисова О.П.** Моделирование процесса изотермического спекания корундовых материалов на химической связке. *Изв. вузов. Химия и хим. технология.* 2004. Т. 47. Вып. 7. С. 113–116.
 25. **Физико-химические свойства окислов:** Справочник. Под ред. Г.В. Самсонова. М.: Metallurgy. 1978. 472 с.
 26. **Синельников С.В., Гропянов В.М., Абакумов В.Г.** Кинетика неизотермического спекания оксида магния. *Журн. прикл. химии.* 1982. Т. 55. № 4. С. 765–769.
 12. **Wagh A.S., Sayenko S.Y., Shkuropatenko V.A., Tarasov R.V., Dykiy M.P., Svitlychniy Y.O., Virych V.D., Ulybkina E.A.** Experimental study on cesium immobilization in struvite structures. *J. Hazard. Mater.* 2016. V. 302. P. 241–249. DOI: j.jhazmat.2015.09.049.
 13. **Wang L., Yu I.K.M., Tsang D., Li S., Poon C.S.** Mixture design and reaction sequence for recycling construction wood waste into rapid-shaping magnesia-phosphate cement particleboard. *Ind. Eng. Chem. Res.* 2017. V. 56. P. 6645–6654. DOI: 10.1021/acs.iecr.7b01175.
 14. **Maldonado-Alameda A., Lacasta A.M., Giro-Paloma J., Chimenos J.M., Haurie L., Formosa J.** Magnesium phosphate cements formulated with low grade magnesium oxide incorporating phase change materials for thermal energy storage. *Constr. Build. Mater.* 2017. V. 155. P. 209–216. DOI: 10.1016/j.conbuildmat.2017.07.227.
 15. **Klammert U., Vorndran E., Reuther T., Müller F.A., Zorn K., Gbureck U.** Low temperature fabrication of magnesium phosphate cement scaffolds by 3D powder printing. *J. Mater. Sci. Mater. Med.* 2010. V. 21. N 11. P. 2947–2953. DOI: 10.1007/s10856-010-4148-8.
 16. **Fan S., Chen B.** Experimental study of phosphate salts influencing properties of magnesium phosphate cement. *Constr. Build. Mater.* 2014. V. 65. P. 480–486. DOI: 10.1016/j.conbuildmat.2014.05.021.
 17. **Xing F., Ding Z., Li Z.-J.** Study of potassium-based magnesium phosphate cement. *Adv. Cement Res.* 2011. V. 23. N 2. P. 81–87. DOI: 10.1680/adcr.9.00030.
 18. **Gardner L.J., Lejeune V., Corkhill C.L., Bernal S.A., Provis J.L., Stennett M.C., Hyatt N.C.** Evolution of phase assemblage of blended magnesium potassium phosphate cement binders at 200° and 1000 °C. *Adv. Appl. Ceram.* 2015. V. 114. N 7. P. 386–392. DOI: 10.1179/1743676115Y.0000000064.
 19. **Abyzov V.A.** Lightweight refractory concrete based on aluminum-magnesium-phosphate binder. *Proc. Eng.* 2016. V. 150. P. 1440–1445. DOI: 10.1016/j.proeng.2016.07.077.
 20. **Kosenko N.F., Filatova N.V., Fukina T.A.** Thermal transformations of alumina-borophosphate binder. *Neorg.Mater.* 2004. V. 40. N 10. P. 1276–1280 (in Russian).
 21. **Tricot G., Coilot D., Creton E., Montagne L.** New insights into the thermal evolution of aluminophosphate solutions: A complementary XRD and solid state NMR study. *J. Eur. Cer. Soc.* 2008. V. 28. P. 1135–1141 DOI: 10.1016/j.jeurceramsoc.2007.09.046.
 22. **Kosenko N.F., Filatova N.V.** Binding materials activity regulating by mechanical chemical methods. *Izv. Vyssh. Uchebn. Zaved. Khim. Khim. Tekhnol.* 2018. V. 61. N 1. P. 66–71. DOI: 10.6060/tcct.20186101.5664.
 23. **Pechkovsky V.V., Dzyuba E.D.** Atlas of phosphate infra-red spectra. Condensed phosphates. M.: Nauka. 1985. 240 p. (in Russian).
 24. **Kosenko N.F., Filatova N.V., Denisova O.P.** Simulation of the isothermal sintering of corundum materials with a chemical binder. *Izv. Vyssh. Uchebn. Zaved. Khim. Khim. Tekhnol.* 2004. V. 47. N 7. P. 113–116 (in Russian).
 25. **Physicochemical properties of oxides:** Handbook. Ed. by G.V. Samsonov. M.: Metallurgy. 1978. 472 p. (in Russian)
 26. **Sinelnikov S.V., Gropyanov V.M., Abakumov V.G.** Kinetics of non-isothermal MgO sintering. *Zhurn. Prikl. Khim.* 1982. V. 55. N 4. P. 765–769 (in Russian).

Поступила в редакцию (Received) 13.03.2019
 Принята к опубликованию (Accepted) 16.09.2019