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Physicochemical structure features of refractory compositions with inorganic binders

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The article presents the study results of new inorganic binders and the physicochemical processes of their formation. The main purpose of the created materials is to make molds and cores for foundry production. Creating environmentally-friendly binders with a set of functional properties for foundry production is relevant worldwide. Therefore, scientists from different countries are paying special attention to the study of silicate and phosphate binders. The study analyzes the kinetics of the orthophosphoric acid interaction with several inorganic materials – pulverent pyrophyllite, disthene-sillimanite, a by-product of electrocorundum production, and sodium chloride. The phase and chemical composition of all formed binders have been established. In aluminum-containing compositions, those are represented by aluminum orthophosphates in crystalline and amorphous forms. Sodium metaphosphate is formed in the composition with sodium chloride.

Peculiarities of the structuring compositions physical process with liquid glass and granular quartz filler due to steam-microwave treatment are determined. It is shown that structuring occurs due to dehydration, which is completed within 4…12 min, which allows reducing the liquid glass content in the composition to 1.5% while ensuring a high level of strength. The properties of structured compositions with the developed binding components are researched, and it is shown that all of them are competitive. Recommendations for their possible application were created.

Keywords: disthene-sillimanite, binder, metaphosphate, compressive strength, orthophosphate, orthophosphoric acid, vapor-microwave treatment, pyrophyillite, liquid glass, composition structuring.

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Introduction

Binders are common in various fields of technology. Most of them are used by the construction industry, metallurgy, and foundry. In the latter two sectors, binders operate in extreme conditions exposed to high temperatures and chemically aggressive environments, such as metal melts.

About 500 thousand tons of cast parts are produced annually in Ukraine. Considering economically justified trends in local industry development, a significant increase in these indicators is projected in the coming years, at least to the level of 1991, i.e., 2 million tons. In the foundry industry, the largest amount of materials is spent on manufacturing molds and cores. The most important group of materials that determines the set of properties of molding and core compositions are binders [1, 2]. These include both inorganic (liquid glass, phosphates and others [3, 4], as well as products of organic origin [5].

The most common synthetic resins used in the foundries are binders, which are extremely pure and environmentally hazardous substances. Their composition and destruction products include toxic and carcinogenic components (phenol, formaldehyde, benzene, toluene, isocyanates, furfural, etc.) [3, 5]. Equally dangerous are resin binders, which are used for cold-hardening and for heat-hardening mixtures [6, 7]. Therefore, most researchers agree that creation of new environmentally-friendly binders and research and developing new ways of structuring compositions of granular refractories with

environmentally friendly binders is an urgent problem .[8, 9].

For the successful implementation of making molds and cores in foundries, especially when it comes to the introduction of new materials, it is also necessary to clearly understand the nature of physicochemical processes that occur during binder hardening in structuring the composition. That provides a basis for establishing effective methods of influencing those processes to predict the compositions' properties and control them and, consequently, improve the casting quality.

According to the binders hardening processes, presented in the fundamental papers [5, 10], as well as based on previous own theoretical and experimental studies [2], it is determined that the compositions structuring is caused by the two groups of processes:

Physical processes to which they belong:

– dehydration: binder is a material that, in combination with water, forms a paste, liquid, or suspension capable of forming adhesive bonds (cuffs) with filler particles;

– crystallization from solution: binder is a solution of a certain material. The curing process includesthis material separation from the solution, subsequent splicing, and strengthening of binder films [2, 11];

– hydration: binder is a material that forms crystal hydrates when interacting with water. Crystal hydrates grow and form adhesive bonds with the filler and cohesion bonds with each other. The composition solidifies at a normal temperature [1, 2]. That curing scheme is typical for types of cement and gypsum;

– gelation: binder is a monomer or sol that spontaneously turns into a solid-state (gel) under physical factors. Use solid, liquid [2, 5] and gaseous hardening [12, 13], thermal, infrared, and even microwave drying [14].

Chemical processes to which they belong:

– polymerization and polycondensation: binder in the initial state is a monomer or oligomer. Due to a certain external influence, there is an irreversible polymerization process accompanied by the possible formation of by-products;

– chemical synthesis: there is no binder in the composition at the preparation stage. It is formed due to a chemical process between the composition components.

The scheme of chemical synthesis is implemented in compositions with several phosphate binders. Phosphate binders are widely used in various technology fields, but given the wide range of possible materials and schemes for their synthesis, they are studied only in fragments. The series of metal oxides activity to H_3PO_4 has the following form (in increasing activity) [11, 15, 16]:

$$SiO_2, TiO_2, Al_2O_3\ , Cr_2O_3, ZnO, Fe_2O_3, CuO, FeO, MgO, CaO, BaO, Na_2O, Ka_2O$$

Almost all of the listed metal oxides are used to create phosphate binders. It is known that the interaction of the first three oxides with orthophosphoric acid occurs only when heated to 200...300°C. Oxides of zinc, iron, copper, and magnesium form orthophosphoric acid compositions that harden at normal temperatures [15, 17, 18]. But the research results were conducted during the '80-the '90s of the twentieth century on the creation of cold-hardening compositions with pulverulent by-products of various industries are now obsolete. There are no data on the interaction kinetics of orthophosphoric acid with by-products of modern Ukrainian enterprises.

In addition, it is believed that phosphates of more active metals have no binding capacity as their formation occurs at a significant rate not allowing structuring the composition with a refractory filler. The chemical synthesis processes of sodium phosphates and their stable forms are known [19, 20], but their use is far from inorganic binders.

Unlike metallophosphates, binder in the sand-liquid-glass (SLG) composition forms due to liquid glass dehydration (LGD) that leads to changes in its primary chemical composition and, accordingly, its polymerization degree. In particular, according to [21], the polymerization degree in LGD shows that there are α-SiO_2-monomeric silica, chain and cyclic oligomers with a degree polymeric not exceeding 8, β-SiO_2-hydrated polymers of silica, γ-SiO_2-silica, similar in structure to quartz.

The LGD polymerization in the process of its dehydration begins with the individual silica particles aggregation and occurs according to the following acid-base reactions schemes [22]:

$$\equiv SiOH^- + OH^- \leftrightarrow \equiv SiO^- + H_2O, \quad (1)$$

$$\equiv SiOH^- + \equiv SiO^- \leftrightarrow \equiv Si - O - Si + OH^- \quad (2)$$

Reaction (1) leads to the occurrence of ionic forms of silica in LGD, reaction (2) – to polymerization (polysilicates formation). In this case, both reactions (1) and (2) occur regardless of the method and intensity of SLG heating. Today, a common method of drying SLG is a radiation-convective method, which, when the SLG is kept with 5% LGD for 2 hours at 200°C, provides the composition with compressive strength at the level of 2.0...3.0 MPa. At the same time, the change in SLG compressive strength with less LGD during its processing in a steam-microwave environment has not been studied.

Obviously, the SLG strength, other things being equal, will depend on the level of silica polymerization. The mass content of water can estimate the degree of silica polymerization for practical purposes in the structured SLG. We can assume that the strength of the structured SLG by the steam-microwave hardening (SMH) directly depends on the residual water content in its LGD, which data is lacking, i.e., the time of exposure to SLG microwave radiation.

Thus, the urgent task is to study the formation processes of phosphate and liquid glass BC and the possibility of their use in the molding and core compositions for foundry production.

I. The purpose and objectives of the study

The paper aims to create inorganic binders of phosphate and silicate nature and study the physical and chemical processes of structuring compositions based on granular quartz fillers.

Tasks:

1. To study the interaction kinetics of aluminosilicate materials (pyrophyllite and disthene-sillimanite) with orthophosphoric acid.
2. To study the interaction kinetics of pulverulent by-products of metallurgical production with orthophosphoric acid.
3. To study the interaction kinetics of sodium chloride, as the salts'representative of active metals, with orthophosphoric acid.
4. Determine the phase and chemical composition of inorganic binders formed in the studied systems.
5. To establish the influence of the final mass amount of water in a liquid glass on the strength of sand-liquid-glass compositions when drying in a steam-microwave environment.
6. Determine the strength of compositions based on granular quartz filler with developed inorganic binders and provide recommendations for their possible use.

II. Materials and methods of research

The studies used orthophosphoric acid technical thermal GOST 10678-76, concentration from 60% to 85%, China made, sodium chloride of technical purity, sodium liquid glass with a silicate modulus of 2.8...3.0 and an apparent density of 1,44 g/cm^3, quartz sand with the clay component up to 0.5% and an average particle size of 0.20...0.25 mm.

A natural mineral is used – a refractory filler containing pyrophyllite, extracted in Korosten, Zhytomyr region, Ukraine. According to the phase analysis, the mineral has 45% pyrophyllite $Al_2(OH)_2[Si_4O_{10}]$, 50% quartz, and 5% kaolinite.

As the second aluminosilicate material, powdered disthene-sillimanite GOST 10772-78 was used.

In addition to those materials, the by-product of metallurgical production was investigated. The gas purification dust production of brown aluminium oxide (GPDBAO), the by-product of the PJSC "Zaporizhzhya Abrasive Plant" enterprise, Zaporizhia, Ukraine is dispersed gray material with a moisture content of about 1.8% (by weight). GPDBAO particles are predominantly spherical, but angular grains are also available [23]. This material is a heterogeneous polydisperse heterogeneous system with particle sizes of 1.5...25.0 μm [24, 25]. Table 1 provides the GPDBAO chemical composition.

X-ray phase qualitative and quantitative analysis was performed on "Rigaku Ultima IV" and "DRON-2.0".

Differential thermal analysis was performed on the "STA 449 C Jupiter" synchronous thermal analyzer.

The SLG structuring was performed by steam-microwave hardening (SMH process) in microwave radiation with a frequency of 2.45 GHz and a magnetron power of 700 W for 2...12 minutes. For structuring, quartz sand was used, which was pre-plated with 1.5% LGD and dried to a final water content of 20...22%. Quartz sand impregnated with 1 g of water was used as a water charge. The SLG structuring samples weighing 700 g were performed in cardboard containers with a working cavity diameter of 50 mm. The mass was determined on electronic scales with an accuracy of 0.1 g. The time was recorded on the stopwatch readings with an accuracy of 1 s.

The mass fraction of water in the structured SLG was determined by weighing the samples after their tests for compressive strength and after calcination at 700°C for 2 hours.

The strength of structured compositions was determined on standard cylindrical samples with a diameter of 50 mm and a height of (50.0 $\pm$ 0.8) mm according to GOST 23409.7-78 on the US-700 universal installation model has a measurement limit of up to 3.5 MPa.

III. Results and discussion

Many natural minerals and industrial products contain aluminum, which phosphates with high binding potential, and thermal and chemical resistance.

To establish the kinetics of the interaction with orthophosphoric acid, samples of pyrophyllite, disthene-sillimanite, and GPDBAO were mixed with it and held for 3 hours. As established visually, the composition of orthophosphoric acid with GPDBAO hardening occurred, while in the pyrophyllite and disthene-sillimanite compositions there was no hardening.

To establish the temperatures and the nature of the orthophosphoric acid interaction with pyrophyllite and disthene-sillimanite, experiments were conducted, which essence was to prepare and hold compositions at different temperatures consisting of 3 mass fraction (m.f.) of acid and 7 m.f. of pulverulent refractory materials.

The compositions made 15…20 samples weighing 3…5 g, which were placed in ceramic containers. They were further put into a drying oven, which temperature was gradually changed from 120 to 360 °C by a range step

Table 1

The GPDBAO chemical composition.

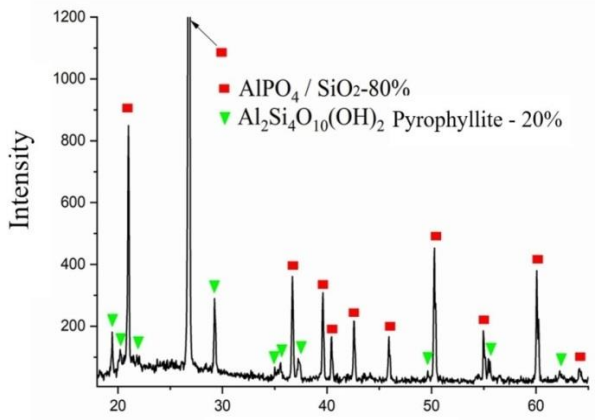
GPDBAO	Mass content, %								
	K_2O	Na_2O	CaO	MgO	SiO_2	Fe_2O_3	C	Al_2O_3	TiO_2
E1*	0.5	0.3	0.35	0.15	4.9	10.9	48.1	30.0	2.3
E2	1.5	0.7	0.3	0.9	14.1	8.4	38.6	33.1	1.1
E3	3.7	1.4	0.22	1.75	23.1	8.1	13.0	46.0	0.4
E4	7.0	2.0	0.15	2.16	33.0	7.5	11.8	34.9	0.36

Note: * E1 – prechamber; E2 – field 1; E3 – field 2; E4 – field 3

of 20 °C. Each temperature range was held for 15 minutes. After each exposure, one portion was removed from the oven. After cooling, the hardening was monitored by a one-millimeter-diameter needle.

As a result, it was found that the compositions hardening with pulverulent pyrophyllite and disthene-sillimanite begins at 300 °C.

Fig. 1 depicts the diffraction pattern of the pyrophyllite composition with orthophosphoric acid.



Fig, 1. The filler composition diffractogram based on pyrophyllite (5 m.f.) with orthophosphoric acid (1 m.f.), treated at a temperature of 300 °C(experimental data from the Rigaku Ultima IV installation).

The sample identified pyrophyllite and lines that may belong to two phases – aluminum orthophosphate and quartz. Silicon phosphates in crystalline forms were not detected due to the higher activity of the alumina component of pyrophyllite (Al_2O_3) to acid compared to silica (SiO_2), according to the introduction of several chemical activities.

The following reaction describes the chemical transformations that occured in this composition:

$$Al_2O_3 \cdot 4SiO_2 \cdot 2H_3PO_4 \rightarrow 2AlPO_4 + 4SiO_2 + 4H_2O (3)$$

Earlier, the authors [11, 15] in studies of pyrophyllite and kaolinite systems with orthophosphoric acid indicated that only amorphous products are formed in such scenarios. On the contrary, no amorphous phase was detected in our study, and all phosphates are contained in crystalline form.

Fig. 2 illustrates the diffraction pattern of the disthene-sillimanite composition with orthophosphoric acid.

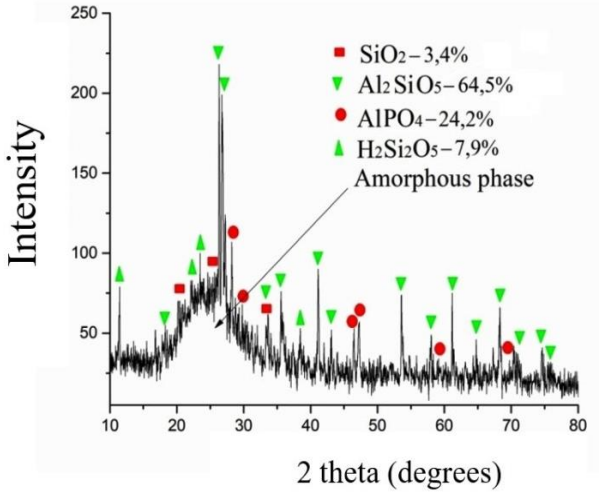


Fig. 2. The disthene-sillimanite composition diffractogram (7 m.f.) with orthophosphoric acid (3 m. f.), treated at a temperature of 300°C (experimental data from the Rigaku Ultima IV installation).

The presence of four crystalline phases was established – sillimanite (65.4%), aluminum orthophosphate (17.8%), quartz (9.8%), silicic acid (7.9%). Part of the sample is amorphous.

In the system of disthene-sillimanite with orthophosphoric acid, as confirmed by phase analysis, the following reaction occurs:

$$3Al_2SiO_5 + 6H_3PO_4 \rightarrow 6AlPO_4 + SiO_2 + H_2Si_2O_5 + 8H_2O \quad (4)$$

As in the authors' studies [11, 15], the formation of amorphous aluminum phosphates is observed in this system, but most of them are still in the crystalline state.

Binders formed in orthophosphoric acid systems with pyrophyllite and disthene-sillimanite can be used for foundry molds and cores made by heat treatment, or for the manufacture of refractory products that are subjected to high-temperature annealing.

Fig. 3 represents the GPDBAO composition diffraction pattern with orthophosphoric acid.

The Fig. 3 analysis shows that after interaction with orthophosphoric acid, the sample structure is completely crystalline; there are phases: SiO_2 – quartz (a small amount of cristobalite possible), C – graphite, $CaCO_3$ (calcite), $AlPO_4$, α-Al_2O_3, α-Fe_2O_3.

Based on this, the most probable GPDBAO chemically active components relative to phosphoric acid may be their crystalline phases of SiO_2, Al_2O_3,and Fe_2O_3. That indicates that small particles of aluminum and silicon oxides smaller than 25 μm are active against orthophosphoric acid at normal temperature. That effect reveals the possibility of creating cold-hardening compositions.

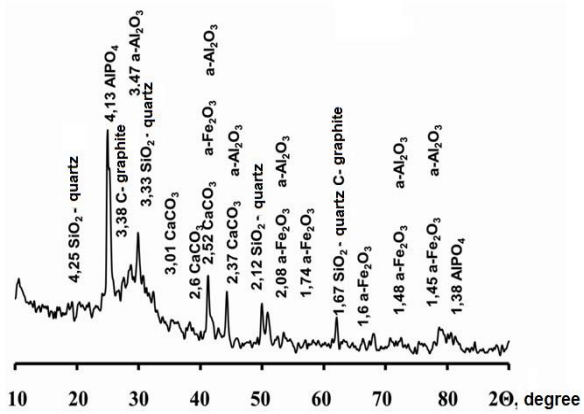


Fig. 3. The GPDBAO sample diffractogram after interaction with orthophosphoric acid (experimental data from the DRON-2.0 installation).

Therefore, in this material, the most probable and decisive is the chemical interaction of H_3PO_4 with Al_2O_3 as the Al_2O_3 content in those products exceeds the content of other oxides [15, 26]. The binder, formed from orthophosphoric acid and the GPDBAO pulverulent product is promising for cold-hardening compositions for molds and cores.

To develop a controlled chemical synthesis process of sodium phosphates, giving them binding properties and ensuring the composition structuring with quartz filler for interaction with orthophosphoric acid, a chemically inert material was selected – sodium chloride. NaCl is a salt of hydrochloric acid, which is much stronger than orthophosphoric acid. Therefore, the interaction at normal temperature in this system is impossible. But, as established, such an interaction becomes possible when heated to 300°C.

Fig. 4 presents an X-ray phase analysis of the NaCl sample with orthophosphoric acid, treated at a temperature of 300°C.

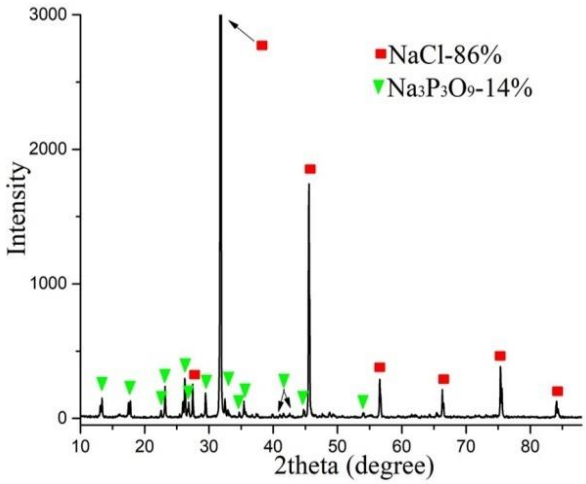


Fig. 4. The sodium chloride composition diffractogram (8 m.f.) and orthophosphoric acid (3 m.f.) after exposure at 300 °C (experimental data from the Rigaku Ultima IVinstallation).

Phase analysis showed that there is a chemical interaction between the components in this system, which results in the formation of one of the forms of sodium metaphosphates. Therefore, the formation of phosphate binder in this system is a chemical reaction:

$$3NaCl + 3H_3PO_4 \rightarrow Na_3P_3O_9 + 3HCl + 3H_2O \qquad (5)$$

The sodium metaphosphate formation is explained by the fact that the chemical interaction occurs in the temperature range of 250… 300 ℃. It is known that at temperatures below 215 ℃ the most stable is orthophosphoric acid H_3PO_4, from 215 to 300 ℃ – pyrophosphoric acid $H_4P_2O_7$, and at 300 ℃ and more – metaphosphoric acid HPO_3 [11]. Therefore, in the sample made at 300 ℃, the sodium metaphosphate formation is logical.

The binder formed in the system of orthophosphoric acid with sodium chloride can be used to manufacture ащгтвкн сщкуі by heat treatment at 300°C.

As a result of the conducted experiments, it is established that in orthophosphoric acid systems with all investigated materials, phosphate binders are formed by a purely chemical mechanism. The difference is in the temperature limits of the chemical synthesis processes. Thus, new data on the creation of cold-hardening phosphate binders and thermal hardening of binders were obtained.

The results of testing samples with SLG demonstrated that with decreasing residual water content in the LGD structure, the strength of the compositions structured by the SMH-process, increases, which follows from the curve in Fig. 5 analysis.

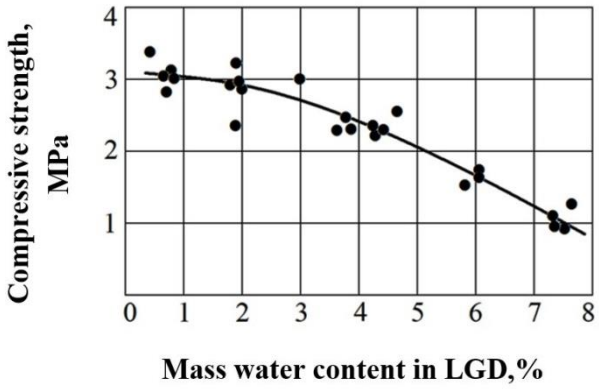


Fig. 5. The compressive strength dependence of SMH with 1.5% LGD from the mass content of water in it.

The results show that for the molds and rods manufacture from SLG, which SMS-process structures, the amount of residual water in the LGDshould not exceed 5%, which will provide structured compositions strength of at least 2 MPa and absolute humidity less than 0.1% by weight.

New inorganic BC were created, and influence regularities of residual water content in LGD were revealed, which solidified in SLG under microwave radiation, tested in laboratory conditions for structuring compositions with quartz fillers. After processing the samplescorresponding to the structuring mode of each of the combinations, their strength was determined. Fig. 6 presents the results.

Fig. 6 data show that cold-hardening compositions with pulverulent by-products have less strength, but it is more than enough to manufacture molds. The strength of combinationsthat are strengthened by heating or under microwave radiation reaches 2.0…3.0 MPa, which is sufficient for the foundry cores manufacture.

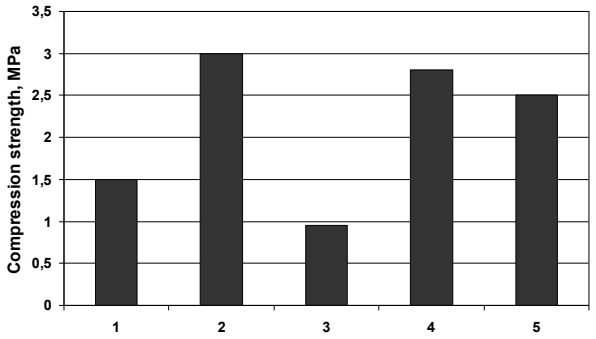


Fig. 6. Strength of compositions samples with inorganic binders:
1 – 5% pyrophyllite, 3% orthophosphoric acid (85%concentration), hardening 1hour at 300 ºC;
2 – 3% disthene-sillimanite, 3% orthophosphoric acid (85%concentration), hardening 1hour at 300 ºC;
3 – 5% GPDBAO, 4% orthophosphoric acid (60%concentration), hardening 24hour at normal temperature;
4 – 8% NaCl, 3% orthophosphoric acid (85%concentration), hardening 1hour at 300 ºC;
5 – 1.5% liquid glass, strengthening in a microwave field with a frequency of 2.45 GHz and a magnetron power of 700 W.

Conclusions

1. It is established that there is a chemical interaction in the system of orthophosphoric acid H_3PO_4 with pyrophyllite $Al_2(OH)_2[Si_4O_{10}]$ when heated to 250…300 ºC, which results in the formation of crystalline aluminum orthophosphate – berlinite $AlPO_4$. It has a bonding ability and, combined with a granular quartz filler, provides sufficient compressive strength to make molds and cores.

2. In the system of orthophosphoric acid H_3PO_4 with disthene-sillimanite Al_2SiO_5 when heated to 300 ºC also occurs chemical interaction, which leads to the formation of two forms of aluminum orthophosphate – crystalline and amorphous. Those chemicals have binding properties and, combined with a granular quartz filler, provide sufficient compressive strength to make molds and cores.

3. It was found that the chemical interaction of orthophosphoric acid H_3PO_4 with a dusty product of metallurgical production – gas purification dust of brown aluminium oxide (GPDBAO) – occurs at normal temperature, resulting in binders represented by crystalline phosphates of aluminum and iron. This indicates that the particles of alumina of particularly small size (1.5…25.0 μm) are capable of chemical interaction with the acid without heating. The composition with such phosphate binder is cold-hardening.

4. It was found that molds and cores of clad sand 1.5% LGD after 4...12 minutes of structuring by SMS-process (in the steam-microwave environment) due to water loss in LGD require no additional drying as their humidity and strength corresponds to similar SLG indicators, which have undergone heat drying at 200 °C for 2 hours. That is, molds and SLG cores, structured by SMH-process, can be used for pouring melt immediately after their removal from the equipment, which reduces the time of their preparation for pouring and the energy consumption of their manufacture.

5. A new binder is formed when heated to 300 ºC due to the chemical interaction between orthophosphoric acid H_3PO_4 and sodium chloride NaCl. This binder is presented in the form of crystalline sodium metaphosphate $Na_3P_3O_9$. In combination with granular quartz filler, it provides compressive strength sufficient for manufacturing molds and cores.

6. Developed inorganic binders, physical and chemical schemes of their hardening are recommended for use in the following areas: compositions with liquid glass and microwave treatment, as well as compositions with orthophosphoric acid and sodium chloride – for the foundry cores manufacture; compositions with orthophosphoric acid and GPDBAO – for the manufacture of foundry molds and cores; compositions with orthophosphoric acid and pyrophyllite or disthene-sillimanite – for the manufacture of molds, cores, and refractory products.

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Фізико-хімічні особливості структурування вогнетривких композицій з неорганічними зв'язувальними компонентами

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У статті представлено результати дослідження нових неорганічних зв'язувальних компонентів та фізико-хімічних процесів їх утворення. Основне призначення створених матеріалів – виготовлення форм і стрижнів для ливарного виробництва. Проблема створення екологічних зв'язувальних матеріалів з комплексом функціональних властивостей для потреб ливарного виробництва є актуальною у світовому масштабі. Тому науковці різних країн привертають особливу увагу дослідженням силікатних та фосфатних зв'язувальних матеріалів. У дослідженні проаналізовано кінетику взаємодії ортофосфорної кислоти із рядом неорганічних матеріалів – пилоподібними пірофілітом, дистен-силіманітом, побічним продуктом виробництва електрокорунду та хлоридом натрію. Установлено фазовий та хімічний склад усіх утворених ЗК. У композиціях з алюмовмісними сполуками вони представлені ортофосфатами алюмінію в кристалічній та аморфній формах. У композиції з хлоридом натрію утворюється метафосфат натрію.

Визначено особливості фізичного процесу структурування сумішей із рідким склом та зернистим кварцовим наповнювачем внаслідок паро-мікрохвильової обробки. Показано, що структурування таких сумішей відбувається внаслідок дегідратації рідкого скла, яка завершується впродовж 4…12 хв обробки, що дає змогу зменшити вміст рідкого скла в суміші до 1,5% при забезпеченні сумішам високого рівня міцності. Досліджено властивості структурованих сумішей із розробленими ЗК та показано, що всі вони є конкурентноздатними. Розроблено рекомендації щодо їх можливого використання.

Ключові слова: дистен-силіманіт, зв'язувальний компонент, метафосфат, міцність при стисканні, ортофосфат, ортофосфорна кислота, паро-мікрохвильове оброблення, пірофіліт, рідке скло, структурування суміші.