

Eco-friendly construction materials: Modified gypsum-containing bind

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Abstract. The use of gypsum-containing binders significantly expands both the raw material base and the possibilities of application of materials based on them. Clay-gypsum belongs to this group of binders. Clay-gypsum raw material and binder on its basis are local building materials. Binders based on clay gypsum have advantages under the following conditions: the remoteness of deposits of natural gypsum raw materials and the proximity of production from consumers. Clay gypsum is a natural formation of fine-grained crystalline gypsum, evenly mixed with clay or loam. The amount of gypsum varies from 50 to 95%. The aim of the research was the formation of scientific bases for obtaining clay-gypsum binder stabilized by its properties, as well as the development of optimized parameters for the production of this binder. As a result of research, the technological parameters of receiving clay-no-gypsum binder and modes of its thermal processing (firing) are optimized, and also the influence of technologies on the properties of clay-no-gypsum binder, which can be considered an analogue of binder received from natural gypsum raw material, is studied. It is established that at the age of 28 days of hardening, the compressive strength of clay-gypsum binder fired at the temperature of 250 °C reached 8.8–8.6 MPa, which at the accepted level of security allowed to obtain a binder of G-8 grade. The formation of soluble anhydrite did not exceed 1.5%.

1 Introduction

Gypsum is widely used in the construction and production of building materials, products, and parts, as well as for the preparation of plaster mortars, the production of gypsum, gypsum-slag, and gypsum-fiber boards and panels for partitions, gypsum board and gypsum-

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fiber sheets, architectural details, and artificial marble as bases for self-leveling and floating floors [1-3].

One of the features of pure gypsum binder is its low water resistance. Increasing the water resistance of gypsum, and consequently products based on it, is realized by the introduction of mineral or synthetic additives [4-6]. A positive result, for example, is achieved by introducing Portland cement and pozzolans, as well as by using thermosetting resins or other curing polymers [7-9].

In the production of building materials (in particular, plaster building mixtures), along with high-quality gypsum binder, gypsum compounds containing various mineral components are used. As a rule, these are by-products of other industries, less often natural mixtures of natural genesis [10-13].

Phosphogypsum, a by-product of phosphate fertilizer production, can be distinguished by the volume of theoretical and experimental studies and scientific interest in the material [14–17]. Phosphogypsum is distinguished as dihydrate, semihydrate (passivated), and anhydrite, which is caused by the peculiarities of the technology of phosphoric acid production. When phosphogypsum is stored, the soluble compounds of phosphorus, fluorine, alkali, etc. contained in it gradually penetrate into the soil and nearby water bodies, which significantly affects the sanitary condition of the environment [18-21].

Gypsum-containing wastes also include products of desulphurization of flue gases of thermal units, in particular, power plants. Flue gases from fuel combustion containing inert sulfur oxides are treated with lime sludge to produce calcium sulfide. Calcium sulfide, during oxidation in the reactor in the presence of oxygen, transforms into calcium sulfate dihydrate [22, 23].

Anhydrite, a waste product of hydrofluoric acid production, is formed as a result of hydrofluoric feldspar processing. In the process of production of organic and inorganic acids (hydrofluoric, boric, citric, oxalic, etc.), chemical gypsum wastes are also produced, which distinguish them from phosphogypsum [24-26].

The peculiarities of chemical gypsums are the instability of chemical composition and the difficulty of removing impurities. Therefore, the simplest (and reliable) methods of utilization are filling of mined-out spaces of mines, use (granulate) for soil reclamation, and so on. At the same time, there is experience in using (primarily phosphogypsum) as a setting regulator in cement clinker grinding, as well as experience in processing phosphogypsum into gypsum binder [27, 28]. It is considered that gypsum binders based on chemical gypsum have advantages under the following conditions: remoteness of deposits of natural gypsum raw materials; proximity of production location from consumers [29, 30].

Due to such a wide application of construction gypsum in the production of construction materials, it is subject to various requirements, the most important of which is high strength and the ability to adjust the setting time within wide limits. Setting time is of great importance for the practice of using construction gypsum in construction for the production and use of dry building mixtures.

Clay gypsum (CG), clay gypsum binder (CGB), and stabilized clay gypsum binder (CGBBM) by their properties and material composition are representatives of this group of gypsum-containing materials. Since the basis of these materials is semi-aqueous gypsum, theories of its hardening are a determining factor in the formation of the properties of all materials in this group.

In the historical practice of construction works, alumina gypsum was used as an ornamental material and as a binder, which was especially characteristic for regions with hot climates. This material was well known in Central and West Asia, Transcaucasia, and Shemakha (modern Azerbaijan). It is called differently: gazhey, gan-cham, arzlyk. In the steppe regions of south-eastern Russia, it is called mica clay, and in the Volgograd, Stavropol, and Rostov regions, it is called clay-hypsum. Experience in the use of clay-hypsum binder is

limited to its artisanal production for local needs, the manufacture of plaster compositions or slab products [31].

Clay-gypsum binder is of particular interest for regions where there is no production of mineral binders and the existing deficit is covered by imports. The Republic of Karakalpakstan, for example, uses imported gypsum binder [32-33].

Large reserves of gypsum-containing rocks explored on the territory of the republic remain unused due to the low concentration of gypsum. This situation is typical for many regions, especially in countries with developing economies. Therefore, it is of interest to create technologies of production and use of low-grade gypsum with gypsum content of 50–80% to obtain local binders.

Clay gypsum is a sedimentary rock of chemical genesis and is of great interest as a local raw material for the production of air-curing binders. By its properties, this material is close to binders of the gypsum group, including industrial gypsum-containing wastes. At the same time, properties and possibilities of clay-gypsum binder are practically not studied, and there is no theoretical basis for the formation of properties of this material.

The purpose of research was the formation of scientific bases for obtaining clay-gypsum binder stabilized in its properties and to develop optimized parameters of heat treatment and compositions of this binder.

2 Materials and methods

The basis of the research was the scientific hypothesis that obtaining clay-hypsum binder is possible as a result of complex influence on clay-hypsum of factors determining the parameters of heat treatment, as well as the use of modifying additives.

Clay gypsum differs from gypsum, first of all, by the amount of impurities, which are from 5% and higher. It is a natural formation of fine-grained crystalline gypsum, evenly mixed with clay or loam. The amount of gypsum varies from 50 to 95%. Clay gypsum is a mechanical mixture consisting mainly of sulfuric acid substances. Usually it is a loose, earthy formation mixed with fine-grained crystals. Organic impurities are also present in the clay gypsum rock. In terms of color, clay gypsum is gray, with a brownish-yellowish tinge, lean, and dry to the touch.

The technology of clay-gypsum processing was formed in such a way that clay-gypsum binder meets the characteristics of GOST 125-79. Gypsum binders. Regulation of setting time, hardening process, and structure formation was carried out by various additives, which were introduced when mixing the binders with water. Citric acid as the most effective retarder was used in the research.

The experimental block consisted of two parts: the study of technology and material properties aimed at obtaining stabilized clay-hypsum binder and the development of the concept of interaction of components of clay-hypsum.

The development of the concept of interaction of components of clay-hypsum was based on the technological principles of obtaining stabilized clay-hypsum binder and applied conclusions from the chemistry of colloidal clay structures and mechanisms of hydration hardening of gypsum and its modifications, as well as the study of properties of model compounds of clay-hypsum binder.

The solution of scientific problems is considered from two perspectives: the formation of conditions for the optimal use of heat-insulating material, the development of principles of its production, and the optimization of technological processes.

3 Experiment and its results

Clay gypsum (gaja, ganch, white clay) is an earthy rock consisting of a mixture of gypsum with sandy-clay and lime-clay admixtures. Clay-gypsum contains from 50 to 95% of bi-hydrous sulfuric acid calcium. Clay-gypsum has the following phase composition, % wt: bi-aqueous gypsum - 1.8; semi-aqueous gypsum - 60-70; quartz - 11.1; albite - 8.6; calcite - 6.1; kaolinite - 2.1. There is no anhydrite II in the composition, which should have formed, and there is bi-aqueous gypsum present, which should not be in the system. Presumably clay envelops the bi-hydrous gypsum and does not allow the material to dehydrate fully during firing.

Obtaining binders based on clay gypsum is associated with its thermal treatment. Processes during thermal treatment of clay-hypsum (gypsum-clay-sand) clay minerals significantly complicate the influence on dehydration. Processes of dehydration of clay gypsum raw materials (Table 1), depending on their composition, differ from the dehydration of pure gypsum, especially in the temperature range of 100-120 °C some slowing down and the absence of a break in the temperature curves associated with the transition of dihydrate to semihydrate.

This is explained by the influence of impurities, in particular montmorillonite, which is also capable of dehydration in this region. The content of residual water as a result of heat treatment at different temperatures is presented in Table 1. The results of monitoring of kinetics of hardening and changes in strength of clay-hypsum binder are given in Table 2. The properties of clay-gypsum binder obtained at different firing temperatures are presented in Table 3.

Table 1. Firing temperature of clay gypsum in the interval of 140–160 °C

No. of items	Firing temperature	Residual water content, %	
		post-firing	estimated
1	140	3.36	3.61
2	140	3.67	3.47
3	160	3.60	3.10
4	160	3.41	3.67

Table 2. Hardening kinetics of clay-gypsum binder

Sample	Normal density %	Setting time, min		Strength, MPa					
		start	end	2 hours		3 days		7 days	
				FS	CS.	FS	CS.	FS	CS.
Clay gypsum	57	13	18	1.7	3.8	1.8	3.9	1.9	4.5

FS: flexural strength; CS.: compressive strength

Table 3. Results of physical and mechanical tests of clay-gypsum binder obtained as a result of thermal treatment at 140-160 °C.

No. of items	Fineness of grind		Normal density, %	Setting time, min		Compressive strength, MPa		Tensile strength, MPa	
	Residue on sieve No. 02, %	PSX device		Starting	Ending	Two hours later.	Dried to constant weight	After 1 day	After 7 days
1	8	2632	0.53	3	5	2.1	3.2	3.2	4.2
2	6	2875	0.55	3	5	2.3	3.3	3.2	4.1
3	1.8	4256	0.59	4	7	2.5	4.7	4.1	5.3
4	7.6	2712	0.54	4	7	2.3	5.2	4.0	5.0

The obtained products were subjected to physical and mechanical tests according to GOST 125-79. At 40 min of grinding, the specific surface area is 250-280 m²/kg. Normal dough density is in the range of 0,46-0,59. Setting time beginning: 3-4 min, end: 5-7 min, which meets the requirements.

Physico-mechanical compressive strength after 2 hours did not exceed 2.5 MPa (G-2), and dried to a constant mass was not the main, but considered a control result. Those dried to constant mass had strengths from 4-5 MPa (G-5). The test results (see Table 3) allow us to conclude that it is possible to obtain clay-no-gypsum binder of G4-G8 grades.

Reduction of setting time is explained by the difference in chemical and mineralogical composition of clay-gypsum rock of different deposits. To obtain optimally strong clay gypsum requires a temperature of heat treatment from 200-300 °C.

Properties of the material depending on temperature and time of thermal treatment are presented in tables 4 and 5. Tests of samples for strength were carried out according to the standard method on samples 40×40×160 mm dried to constant weight for 7 days.

Table 4. Properties of binder based on clay gypsum fired at different temperatures for 2 hours

Firing temperature, °C	N.g. %.	Setting time, min		Tensile strength, MPa				Softening factor	Density, kg/m ³
		start	finish	2 hours		7 days			
				FS	CS	FS	CS		
180	0.57	5.3	7.5	1.7	2.5	1.9	4.5	0.32	1210
250	0.53	12.4	17.2	2.1	4.4	3.4	8.5	0.4	1213
280	0.5	14.2	16.3	–	3.6	–	8.26	0.31	1321
300	0.49	15.1	18.2	–	2.9	–	8.0	0.5	1313

FS. flexural strength; CS.: compressive strength

During the research (in the process of heat treatment), there was a lag in the temperature of heating of the fired raw material from the temperature of heating of the drying cabinet. At the temperature of the drying cabinet at 180°C, the temperature of the material was 150°C, at 250°C and, respectively, 240°C. Further, the difference in temperatures approached zero. This technological feature associated with the conditions of heat exchange was taken into account in all subsequent experiments.

At the age of 28 days of curing, the compressive strength of clay-gypsum binder fired at temperatures 280-300 °C reached 8.8–8.6 MPa, which at the accepted level of assurance allowed to obtain a binder grade of G-8. The formation of soluble anhydrite does not exceed 1.5%.

Kinetics of hardening and setting time were studied according to the standard method with the help of Vick's device. According to the results of the obtained data on the influence of firing temperature on the properties of clay gypsum binder, it can be noted that obtaining clay gypsum binder really differs from the construction gypsum; at a temperature of 250 °C, slowly setting and obtaining high strength compared to the binder at 180 °C.

Table 5. Content of different forms of CaSO₄ at different heat treatment temperatures

Temperature, °C	Content by weight, %		
	CaSO ₄ ·2H ₂ O	CaSO ₄ ·0,5H ₂ O	CaSO ₄
160	54.95	26.85	0
200	18.40	62.36	0
250	1.8	70.3	0
300	0	72.8	1.51
350	0	50.55	27.75
400	0	0	~100

The evolution of the properties of clay-hypsum binder is determined to the greatest extent by the temperature of thermal treatment, as a result of which the mineralogical composition of fired clay-hypsum changes (Table 6, Fig. 4). The performed analysis of mineralogical composition of clay-hypsum shows that in the temperature range of 250-300 oC the maximum yield of semi-aqueous gypsum is reached, which determines the optimal properties of clay-hypsum binder.

Comparative studies of the properties of clay gypsum fired at different temperatures have shown the following: Water consumption of the binder at a temperature of 140 °C, compared to 180 °C and 250 °C is higher by almost 8–9%. When increasing the temperature of thermal treatment up to 250-300 oC, the strength increases almost 2-2.5 times. The second technological factor determining the efficiency of heat treatment is the firing time, which varies in the experiments from 1.5 to 4 hours.

Table 6. Properties of binder based on clay gypsum fired at 250 oC as a function of firing time.

Firing time, h.	N.g. %.	Setting time, min		Tensile strength, MPa				Kp 7 days	Density, kg/m ³
		start	end	2 hour's		7 days			
				FS.	CS.	FS.	CS.		
1.5	0.55	12.4	14.15	1.8	3.4	3.5	8.0	0.3	1209
2.0	0.53	12.4	17.2	2.1	4.4	3.4	8.5	0.32	1210
3.0	0.54	15.4	17.2	1.9	4.3	4.9	10.1	0.43	1217
4.0	0.52	12.3	17.06	1.8	3.8	4.8	10.5	0.58	1223

FS: flexural strength; CS.: compressive strength

4 Discussion of experiential results

The optimum firing time of clay gypsum is the interval from 2 to 4 hours. The softening coefficient at firing up to 4 hours increases from 0.3 to 0.58. The change in compressive strength most appreciably depends on the firing time at a temperature of 250oC, which provides the best yield of binding material. The optimum result is obtained by firing for 2-3 hours. Then there is a slight (up to 10%) decrease in compressive strength.

The properties of fired clay-gypsum binder depend on firing modes: firstly, nonlinearly, and secondly, on changes of several technological parameters simultaneously. This makes it necessary to conduct additional studies using the technique of mathematical planning of experiments.

The clay-gypsum binder mixed with water is a gypsum dough with evenly distributed (dispersed) clay particles in it (Fig. 1, 2). Conventionally, the particles are assumed to be elliptical in shape with the ratio of longer length to shorter length ($l_2/l_1 = 28$). Gypsum contained in this system, both according to a priori data and according to the results of research, corresponds to grades G5-G8, and the properties of clay particles are determined by the properties of clay minerals before and after heat treatment of clay gypsum.

Clay particles are evenly distributed in the material and gradually disperse, creating in the solution structures similar to colloidal systems. This effect, first of all, affects the reoplastic properties of clay gypsum, namely its thixotropy, workability, and elasticity of mortars. It can be said that the effect of “sliding lubrication” is manifest.

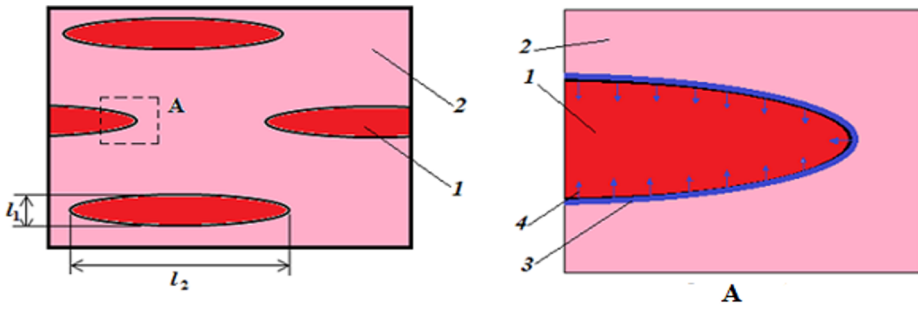


Fig. 1. Model structure of clay-gypsum suspension: 1: clay particle; 2: gypsum binder; 3: water film; 4: water movement inside the particle

The layered structure of silicates forming clay minerals is the reason for the high energy potential of clay particles. Removal of physically bound water and a part of interpacket water (physico-chemically bound water) during thermal treatment of clay gypsum creates a surface energy potential, which contributes to the increase of adsorption capacity of clay micelles, which determines the contribution of clay components to the formation of properties of clay gypsum dough in the process of its mixing, as well as the properties of hardened materials. The activity and water consumption of clays—components of clay gypsum—is determined by the type of clay minerals, clay dispersity, and the degree of dehydration of clay minerals.

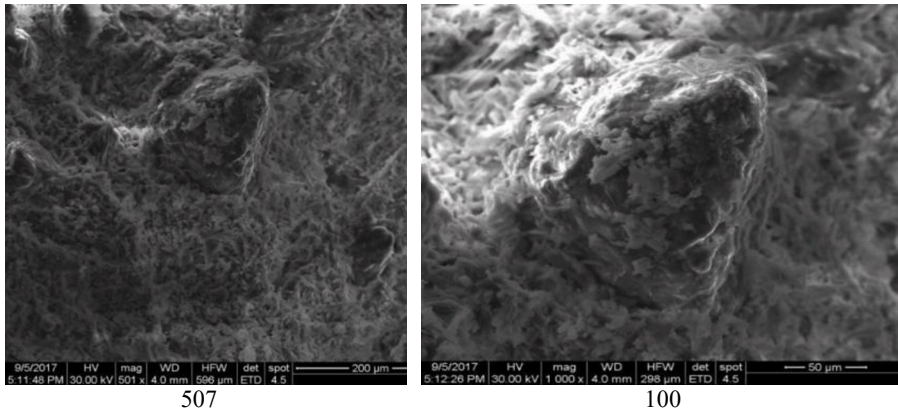


Fig. 2. Microphotographs of the contact zone "clay particle-gypsum," magnification 507; 1000 respectively.

It is the presence of clay minerals that determines the higher temperature of thermal preparation, the longer setting time of clay gypsum, and the increase in its strength as a result of heat treatment at temperatures close to 300 oC. At temperatures close to 300 oC, due to fluctuations in the properties of the clay-hypsum mixture, it may appear anhydrite, which also leads to an increase in setting time and strength.

After mixing with water, clay-gypsum dough is formed, during processing, setting, and subsequent hardening, of which three groups of processes can be distinguished: in clay minerals, in gypsum dough, and on the contact surfaces of clay particles and gypsum binder. Gypsum in solution behaves as a gypsum binder, but on the contact surfaces of clay particles and gypsum mixed with water, special physical and chemical conditions are created, and we

can talk about the creation of a multifactor system: “gypsum binder-water-gas-clay minerals.”

In solution, water films are formed on the surface of clay particles. Mono- or poly-molecular layers on the surface of clay minerals and thicker ones on the particles. Under the condition of preservation of the thermodynamic and mechanical equilibrium of the system, a force arises that balances the pressure in a thin layer of liquid. Consequently, the equilibrium pressure differs from the pressure that is conserved in the volume of the liquid phase. The excess pressure that arises in the interfacial layer of the liquid at a sufficiently significant thinning of the liquid is called wedging pressure. As a result of the action of wedging pressure, the surfaces of solid-phase particles do not stick together, which increases the aggregative stability of the heterogeneous system.

The regularities of wedging pressure for liquid interlayers between solid planar surfaces were studied by B.V. Deryagin and co-workers. At present, this phenomenon is widely used in wet grinding of inert materials, first applied by P.A. Rebinder [34-36]. As a result of the action of wedging pressure, there is an additional dispersion of clay particles, as well as penetration of water molecules containing dissolved and dissociated gypsum into the interpacket layers of clay minerals with the possibility of subsequent creation of chelate complexes. Grains of clay minerals are surrounded by concentric layers of water (with dissolved and dissociated modifications of gypsum), each of which is held by clay particles with different strengths.

Formation of hydration structures in the hardening of the gypsum component of clay gypsum binder is the basis for the hardening of binders of this group. For clay gypsum fired at 250-300 °C, the reaction agent is semi-aqueous gypsum, $\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$. As for pure gypsum, dehydrated to 180 °C, the hardening processes of clay-gypsum binder are determined by the reaction conditions:

The phenomenon of hardening is that the initial substance, dissolving in water, forms a solution, which is saturated with respect to it but supersaturated with respect to the products of hardening, released from it in the form of crystals.

The structure of solidified clay gypsum (at different degrees of magnification) is shown in Fig. 8. In the area of hardening of the gypsum component, characteristic crystals of bi-aqueous gypsum are presented.

The process of gypsum hardening has been studied by many specialists and from different directions. At present, based on the results of these works and studies performed by P.A. Rebinder and E.E. Segalova and co-workers, as well as V.B. Ratinoff, we can consider that hydration and solidification of semi-aqueous gypsum occur by crystallization mechanism, i.e., through the dissolution of semi-aqueous salt.

This statement refers to the principle provisions of hydration and hardening of clay gypsum, reflected in the concept of interaction of components and formation of properties of stabilized clay gypsum binder.

Clay minerals are natural modifiers of clay gypsum. As a result of experiments, it was established that clay minerals should not be considered as an “inert” filler: they have a direct influence on the conditions of dehydration of dihydrated calcium sulfate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and on the properties of the resulting binder.

In particular, in the firing process, dehydration of $\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$ in clay gypsum begins at 110-120 °C, and the highest content (yield) of semi-aqueous gypsum ($\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$) falls on the temperature range of 250-300 °C. Formation of anhydrite begins at temperatures above 300 °C. At temperatures above 300 °C presumably begins the thermal destruction of clay minerals with the possible formation of portlandite (in the presence of CaO in the fired clay gypsum), which explains the compatibility of clay-gypsum mixtures and bases based on Portland cement.

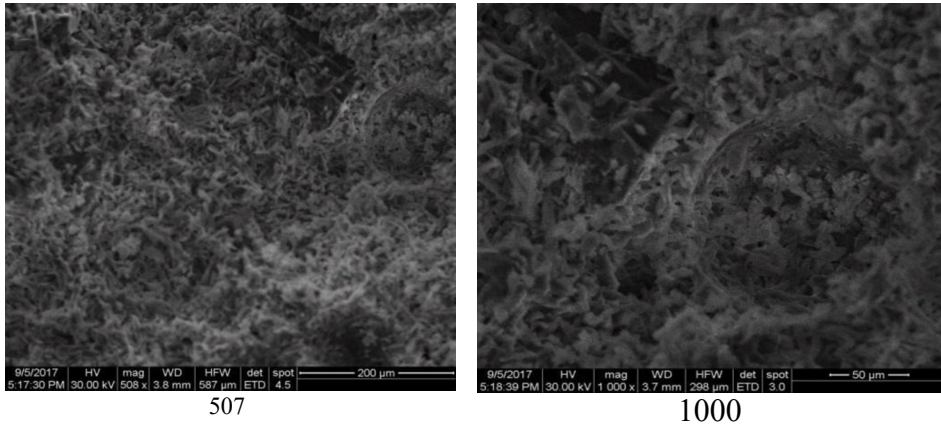


Fig. 3. Microphotographs of the pore structure, magnification, respectively 507; 1000

At mixing with water in the clay-gypsum suspension, dissociation of clay particles increases in their specific surface with increasing energy component, which increases their reactivity. Thus, areas of increased reactivity are formed on the surfaces of clay particles.

An important feature of hydration of clay-gypsum binder is the possibility of self-repair of the damaged structure. The pore formed at the early stages of curing is gradually filled by new formations at subsequent stages (Fig. 8).

The formation of hydration structures during the hardening of the gypsum component of clay-hypsum binder as a result of interaction with water is carried out according to classical schemes. For clay-gypsum fired at 240-260 °C, the main structure-forming element is semi-aqueous gypsum, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. For clay-gypsum binder fired at high temperatures (up to 300-320 °C), the role of soluble anhydrite CaSO_4 in the processes of structure formation increases.

5 Conclusion

One of the promising types of gypsum-containing binders is a binder based on clay-gypsum raw material. Clay gypsum is widespread both on the territory of Russia and in the countries of Central and West Asia, the Caucasus, and Transcaucasia, as well as in regions with developing economies where there are appropriate raw materials. By its properties, this material is close to gypsum group binders, including industrial gypsum-containing waste. However, the properties and possibilities of clay-hypsum binder are practically not studied, and there is no theoretical basis for the formation of the properties of this material. The use of clay-hypsum is also limited by the lack of effective technologies in its processing.

Obtaining clay-hypsum binder is possible as a result of the complex influence on clay-hypsum of various parameters of heat treatment, as well as types of modifying additives. The optimal firing parameters are firing temperature (250 oC) and time (3.5–4.0 hours). Softening coefficient at firing increases up to 0,58; compressive strength is 8,8-8,6 MPa; and bending strength is up to 4,5 MPa.

The most expedient fields of application of gypsum-containing binder are the production of modified clay-gypsum binder and dry building mixtures on its basis, as well as interior products. It is especially important for regions experiencing a deficit in natural gypsum and materials based on it.

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