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Physicochemical Basis for the Production of Heat-Resistant Non-Shrinking and Expanding Backfill Cements

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ABSTRACT: We searched for the composition of the cement in the direction of selection, the properties of the composite mixture, consisting of an expanding component (as a factor providing density and strength in the early stages of hardening) and silica (to ensure the durability of the cement stone). The processes of hydration of expanding components at elevated temperature and pressure have been investigated.

The hydrothermal crystallization of compounds during the hydration of cements of various compositions has been studied. We also studied the change in the plugging properties of cements (spreadability, thickening, setting time, strength at normal and hydrothermal hardening), depending on the fineness of their grinding, mineralogical composition of Portland cement clinker and sulfoaluminosilicate components.

KEY WORDS:rock, clay, drilling, well, fluid, flow, dependence, filtration, experiment, opening, crust, pressure

I. INTRODUCTION

The problem of casing high-temperature and steam injection wells remains unresolved, but it is very important due to the increase in the volume of drilling of deep, superdeep wells, as well as the prospects for steam injection into productive formations in order to increase the oil recovery of productive horizons. In this case, the cement should have a high density and relatively increased strength in the early stages of hardening at ordinary temperatures, and then, under the influence of elevated temperature and pressure, the cement stone should acquire stability and durability

II. LITERATURE STUDY

To solve the problem, we searched for the composition of the cement in the direction of selection, the properties of the composite mixture, consisting of an expanding component (as a factor providing density and strength in the early stages of hardening) and silica (to ensure the durability of the cement stone). However, at first it was necessary to study the nature of the change in the properties of these components during hardening under conditions of normal and elevated temperatures. The following expanding components are commonly used: alumina slag, sulfoaluminate clinker, fired alunites. Their chemical and mineralogical differences can determine the formation of cement stone of various structures [1-2]. Experiments were carried out to clarify the composition of the hydrated phases during the hydration of a mixture of ettringite with various compounds. Ettringite was synthesized by the authors of [1-2] according to the generally accepted method. On its basis, mixtures with silica, silica and lime, tricalcium silicate were obtained. After being placed in molds, they were placed in an autoclave at 523 K and 40 MPa. Under these conditions, ettiringite actively interacted with SiO_2.

III. EXPERIMENTAL RESULTS

The specified composition of the products is apparently due to the insufficient saturation of the system with lime, which led to the decomposition of ettringite in the presence of SiO2 and the presence of the latter in free form, therefore, further studies of the mixture were carried out with the addition of lime to it [3-6].

When calcium hydroxide was added to a mixture of ettringite with silica, low-resistance calcium hydrosilicates CSH (I), calcium hydrogen garnets C3AS1.5H, and anhydrite were formed. The study of a mixture of ettringite with C3S revealed the formation of anhydrite, calcium hydroaluminate C3AH6, calcium hydrogarnet C3ASH4, and calcium hydrosilicate C2SH.

Thus, in all cases at elevated temperatures and pressures, the interaction of ettringite with silica, a mixture (SiO2 + Ca (OH) 2), or C3A led to the formation of calcium hydrogens and calcium hydrosilicate, the basicity of which depends on the (C + A) S ratio in the system ...

The processes of hydration of expanding components at elevated temperature and pressure have been investigated. Calcium sulfoaluminate, alumina slag, and fired rock were used as sealing components at ordinary temperatures.

It should be emphasized that if during the firing of mixtures containing CaO, Al2O3 and SiO2, the formation of gehlenite is prevented by the introduction of gypsum into the raw mixture, then under conditions of hydrothermal crystallization of compounds, the introduction of gypsum into a system containing helenite leads not to the formation of helenite hydrate, but to crystallization calcium hydrant of composition C3ASH4.

The hydrothermal crystallization of compounds during the hydration of cements of various compositions has been studied. The following mixtures were taken for the study:
- Portland cement + sulfoaluminate clinker (SAK), gypsum and silica (composition 1);
- Portland cement + alumina slag, gypsum and silica (composition 2);
- Portland cement + alunite + gypsum and silica (composition 3).

When hydration of composition 1, containing sulfoaluminate clinker, without silica and in its presence at ordinary temperature in the structure of the cement stone, the hydration products are presented in the form of well-formed acicular crystals of calcium hydrosulfoaluminate of the three-sulfate form, lamellar crystals of calcium hydrosulfoaluminate, hexagonal calcium plates and tonic aluminosilicate mass.

The hydration products of composition 2, hardened under normal conditions, include ettringite, hydroaluminates, and highly basic calcium hydrosilicates. Under the conditions of hydrothermal action, calcium hydrogarnates xonotlite, anhydrite and boehmite were formed in the cement stone.

The hydration of composition 3 was studied in two versions. The first involved the use of fired alunite rock. The results were similar to those described. The second option included the use of natural alunite rock without prior firing. It was found that when using "raw" alunite under hydrothermal conditions, xonotlite, tobermorite, calcium hydrogarnates and anhydrite were formed.

Comprehensive analysis of data on the setting time, strength characteristics and volumes of expansion of cement stone for the studied compositions (1-Portland cement + alumina slag + sand; 2-Portland cement + sulfoaluminate clinker + sand and 3-Portland cement + alunite + granular slag) showed that optimal characteristics that meet the requirements for oil well cements are implemented using the following cement compositions containing (wt.,%):
- 47-52% Portland cement clinker, 8-12% alumina slag, 35% sand (composition 1);
- 45-50% Portland cement clinker, 10-15% sulfoaluminate clinker, 30-35% sand (composition 2);
- 15-20% Portland cement clinker, 35-39% crude alunite, 38-43% granular slag (composition 3).

We also studied the change in the plugging properties of cements (spreadability, thickening, setting time, strength at normal and hydrothermal hardening), depending on the fineness of their grinding, mineralogical composition of Portland cement clinker and sulfoaluminosilicate components.

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Taking into account the results of determining the optimal properties of the compositions of cements and production parameters, a technological procedure for the production of cements based on sulfoaluminate clinker and alumina slag has been developed.

The fineness of cement grinding, characterized by the specific surface, should be at least 3000 cm2 / g. The content of C3S in clinker should be in the range of 50-60%, C3A - 5.8 wt. %. The amount of sulfates in cement in terms of SO3 is no more than 2-4 wt.%.

The ratio of components in cement is determined by the value (CaO + Al2O3): SiO2, which should be at least 0.9 and not more than 1.3.

Thus, using expanding components (sulfoaluminate clinker, alumina slag, alunite), non-shrinkage cements were obtained that are heat-resistant at 523 K and a pressure of 40 MPa.

Valuable results were obtained [6] in the study of expanding oil well cements, a technology for their preparation was developed.

Attempts have been made to develop compositions of expandable sulphate-based grouting binders from industrial waste with a temperature range from 0 to 358 K.

The chemical and mineralogical compositions of industrial waste, their yield, efficiency, stability of the chemical composition, availability, efficiency, as well as the distance from waste storage sites to cement plants have been analyzed, and a conclusion has been drawn that it is advisable to use phosphogypsum as a gypsum component, and calcium alumina as an alumina component. slag, which is a waste of electric steel-making production.

Phosphogypsum is produced during the production of phosphoric acid, its main component is gypsum dihydrate. It includes (% by mass) 70-95 CaSO42H2O, 1.77 not decomposed apatite Ca5F (PO4) 3, 1.8 monocalcium phosphate CaH (PO4) 2, 0.22 fluorosilicic acid H2SiF6 and silica.

There are more than 100 million tons of Phosphogypsum in the dumps of enterprises of Uzbekistan, and these dumps are constantly increasing.

In terms of chemical and mineralogical composition, the alumina-calcium slag of electric steel-making production is close to alumina cement, as evidenced by the following data (% by weight): 44.95 - CaO; 38.11 - Al2O3; 9.97 - SiO; 5.4 - MgO; 0.8 - Fe2O3; 0.6 - MnO; 0.17 - S. Its mineralogical composition is represented by the following minerals (% by weight): 31.73 - C12A7; 20.07 - CA; 14.62 C2S; 5.36 - C2AS; 2.72 - periclase MgO; 9.51 - shrinel MgOAl2O3; 15.99 - glass.

Studied the processes of hydration and hardening, crushed alumina-calcium slag (ASH) (table. 1) and found that the main products of gyration of ASH. at an early stage of hardening at a temperature of 295 K, these are neoplasms of CAH10, C2AH8 and a small amount of C3AH6 and AH3. With further hydration of CABG, the content of C2AH8 and AH3 increased, and by days 28 and 180, partial recrystallization of hexagonal calcium hydroaluminates CAH10 and C2AH8 into cubic C3AH6 was observed.

During the hydration and hardening of calcium alumina slag at an air temperature of 288-293 K, mainly lamellar crystals of hexagonal calcium hydroaluminates CAH10, C2AH8 and cubic calcium hydroaluminates C3AH6 are formed, giving a relatively strong crystalline intergrowth, as indicated by the determination of the strength of cement stone (Table 1). Hydration and hardening of CABG at elevated temperatures leads to the intensity of all processes. The rate of recrystallization of hexagonal calcium hydroaluminates CAH10 and C2AH8 into cubic C3AH6 increases, which leads to a slight decrease in the strength of the cement stone in comparison with natural hardening (see Table 1).

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Table 1. Changes in important physical and mechanical properties of cement stone based on alumino-calcium slag within 1-180 days.

Indicator	First experience						Second experience	Third experience
	Hardening time, days							
	1	2	3	7	28	180	2	2
Compressive strength, MPa	4,8	9,1	12,9	19,6	20,1	21,1	12,0	11,2
Flexural strength, MPa	2,3	3,0	3,4	3,7	4,1	4,4	3,7	3,0
Shrinkage,%	0,27	0,30	0,32	0,38	0,45	0,51	0,47	0,90
Note:	1. Research was carried out on equally mobile solutions (18-19 cm along the cone of AzNII 2. Curing conditions: from the first to 180 days t = 295 K, p = 0.1 MPa; two days at t = 327 K, p = 10 MPa; two days at t = 348 K, p = 10 MPa							

This can also be explained by the appearance in a short period of time in the hardening cement stone of a large amount of low-basic calcium hydroaluminates, which had the form of extremely small crystals that did not form a strong crystalline intergrowth. In addition, the formation of a certain amount of AH3 inhibited the recrystallization of hexagonal calcium hydroaluminates into cubic hydroaluminates. It has been shown that the calcium alumina slag has good binding properties, at the same time it has a short setting time, and the cement stone has a large shrinkage value (0.45-0.90), which does not allow its use in its pure form.

Expanding binder can be obtained by combining calcium-alumina slag and phosphogypsum. The spreadability of the grouting slurry along the cone of AzNII was 18-19 cm.Only with an optimal ratio between alumocalcium slag and phosphogypsum, the maximum content of hydrosulfoaluminates is ensured, therefore, the maximum strength of the cement stone, while the content of sand must be assigned from the condition of obtaining the required thickening time.
To slow down the setting time, thickening time and heat transfer rate, the addition of ground basalt sand is introduced into the expanding binder. It had an effective effect on some other important properties of the grouting slurry and cement stone (sedimentation stability, gas permeability, etc.). Conditionally, this binder is called aluminophosphogypsum sand (AFP).

According to the experimental study, it was found that the strength of cement stone based on binder AFP decreased in proportion to the increase in the content of quartz sand. The thickening of the solution lengthened with an increase in the sand content. For example, when adding up to 30% to the mixture, the thickening time of the cement slurry increased from 57 to 116 minutes. After the introduction of restrictions on the time of thickening of the grouting slurry and the strength of the cement stone, the dosage limits of the binder AFP components, by weight, were proposed: calcium alumina slag - 40-55; phosphogypsum - 20-30; ground basalt sand - 10-30.

The analysis of X-ray structural and differential thermal allowed us to establish that only with the optimal ratio of gypsum and alumina components, the reactions most fully proceed with the formation of the largest amount of calcium hydrosulfoaluminates.

It has been established that the timing of thickening of the cement slurry largely depends on the composition, binder AFP and the hardening temperature (Table 2).

Table 2. Influence of the ratios of the binder AFP components and the amount of setting retarders and temperature on the time of thickening of the cement slurry

Binder composition,% by weight			Additive amount,% by weight			Thickening time of the solution, h-min	
Alumina-calcium slag	Phosphogypsum	Ground basalt sand	Bura	Dextrin	KVTs-1	At 295K	At 323K
60	30	10	-	-	-	1-35	0-29
50	30	20	-	-	-	1-46	-
50	20	30	-	-	-	1-56	-

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42	23	25	-	-	-	2-15	-
60	30	10	-	0,2	-	1-48	-
60	30	10	-	0,3	-	2-05	-
60	30	10	0,125	-	-	2-34	1-55
60	30	10	0,150	-	-	2-50	2-06
60	30	10	-	-	0,100	3-00	2-10
60	30	10	-	-	0,125	-	2-54
Gypsum-alumina cement (GHC)			-	-	-	1-10	-
Backfill Portland cement (TPTs)			-	-	-	5-40	-

IV. CONCLUSION

At the stage of searching for the composition, binder AFP, the required thickening time of the cement slurry was provided at a normal temperature of 295 K.

The thickening time is largely overestimated by the fineness of the grinding of the binder. The higher the specific surface area of the binder, the shorter the thickening time, and vice versa.

The cement stone based on binder AFP acquired the main strength in 7 days of natural hardening (90% of the strength after a month of hardening) and further hardened without decreasing strength. It should be noted that prolonged exposure to temperature on the AFP cement stone did not lead to a decrease in strength.

The proposed grouting compositions based on alumina-calcium slag - phosphorogypsum - ground basalt sand have been successfully tested at well 3 in the Uchsai area, at well 3 in the SevernyBerdak area, at well 2 in the Shege area.

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