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COMPOSITE PORTLAND CEMENTS MODIFIED WITH CERAMIC PRODUCTION WASTES

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ABSTRACT

The article presents research into the possibility of using ceramic waste as an active mineral additive for producing composite Portland cements. This work is devoted to the development of the composition and technology for obtaining Portland cement modified with a technogenic aluminosilicate material that has undergone a preliminary stage of thermal activation - ceramic waste. The element compositions, structural features of the initial and final components were studied using a modern instrumental method. Due to the intensive process of nucleation and genetic formation of new formations during hydration, a cement stone of a dense structure is formed and as a result, despite a decrease in the proportion of highly active clinker by 20%, by the 28th day its hydraulic activity is 41.5-42.8 MPa, which ensures the cement grade PC 400-D20 according to State Standard 10178-85. Physicomechanical and physicochemical research methods have scientifically substantiated and proven the practical possibility of obtaining PC400-D20 grade CPC, the production technology of which has been mastered at JSC «Akhangarancement».

Keywords: Ceramic Waste, Active Mineral Additive, Composite Portland Cement, Hydration, Construction Industry.

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INTRODUCTION

Construction engineers develop various building materials and additives in order to provide high-quality cement-containing materials, and at the same time, reduce the cost of their production. For this reason, it is necessary to conduct a search and experimental work to produce cement components from various natural raw materials and industrial waste. One of the priority areas of development of the cement industry, satisfying the principles of energy and resource conservation, is the creation of Portland cements containing active mineral additives of natural and technogenic origin.[1] The resources of currently used additives do not meet the ever-increasing needs of the cement industry.[2] In this regard, researchers are faced with the task of expanding the raw material base to obtain accessible and widespread mineral supplements with sufficient reserves. Their use is one of the most effective and simple ways to save clinker today, allowing for the simultaneous reduction of the costs of both material and energy resources, as well as reducing the negative impact on the environment of the load exerted by cement plants.[3,4,5] This waste material is stored in dumps. The presence of these dumps has an adverse effect on the surrounding environment. This article presents the first results of basic research dealing with the treatment process of waste cellular concrete rubble by means of a crushing process and its subsequent use as filler in the production of new porous concretes. The use of substitute additives from secondary raw materials will help solve the problem of Portland cement shortage, especially in those regions where reserves of natural active mineral additives are limited or completely absent. The most promising in this regard are considered to be metakaolin, thermally activated clays, or their compositions with other mineral ingredients and silica fillers.[6,7,8,9]

EXPERIMENTAL

Materials and Methods

This work is devoted to the development of the composition and technology for obtaining Portland cement modified with a technogenic aluminosilicate material that has undergone a preliminary stage of

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thermal activation - ceramic waste. In conducting the research work, the authors applied the following analysis techniques: synchronous thermal analysis using STA 449 F3 Jupiter (Munich, Germany); a scanning electron microscope JSM-6390LV (JEOL, Japan) integrated with an Oxford INCAEnergy energy-dispersive X-ray microanalyzer (Oxford Instrument, UK) to determine the elemental composition and microstructure of the sample under investigation; and an IR Fourier spectrometer (Zhimadzu IR Prestige-21 with an attenuated total internal reflection attachment Miracle Pike Technologies (Tokyo, Japan)) to study the structural features of the studied sample.

General Procedure

The objects of research used were clinker from JSC «Akhangarancement», gypsum stone from the «Karnab» deposit, and fired ceramic waste from the dump of JSC «Kulol». To establish the suitability of ceramic waste as an active mineral additive to cement, its chemical composition was determined according to State Standard 5382-91, and its hydraulic activity under compression was determined according to the method of State Standard 25094, followed by calculating the value of the Student criterion. The results obtained were assessed according to State Standard 31108-2020, the hydraulic activity of which was assessed according to State Standard 10178.

Detection Method

Physicochemical studies of the processes of hydration and structure formation during the hardening of additional cements with an optimal content of ceramic waste were carried out using chemical, X-ray phase, and electron microscopic analyses. The technological sample of ceramic waste in terms of chemical composition meets the requirements of Uz DSt 901-98 "Additives for cements. Active mineral additives and filler additives. The fired pieces of ceramic waste, separated by passing through a large mesh, are crushed into smaller pieces and accumulated in a separate area (warehouse) near the waste heap, from where they are shipped to the consumer. Samples of ceramic waste selected from the warehouse, represented by sintered pieces of various sizes of ceramic tiles and products made of clay (kaolin) raw materials.[10,11,12] The chemical composition of the starting components is given in Table-1.

Table-1: The Chemical Composition of the Starting Components

Name Components	SiO_2	$A1_2O_3$	Fe_2O_3	CaO	MgO	SO_3
PC clinker,%	20.54	5.19	3.56	62.04	3.60	0.62
Gypsum stone,%	1.52	0.13	0.14	33.04	0.20	43.46
Ceramic fight,%	64.85	23.89	2.79	2.24	1.00	1.28

To study the effect of adding fractionated broken ceramic tiles on the physical and mechanical properties of Portland cement, raw material mixtures were prepared, including 75-85% clinker, 10-20% broken ceramic tiles, and 5% gypsum stone (Table-1). As a control sample, we used a cement without additives, containing 95% clinker and 5% gypsum rock. The raw materials were ground in a laboratory ball mill for 50 min.

RESULTS AND DISCUSSION

The obtained value of the t criterion for ceramic waste was t=21.21, which is greater than its standard value of at least 15 according to State Standard 31108-2020. Consequently, the ceramic waste has passed the strength activity test and can be used as an active mineral additive in the production of Portland cements in accordance with the relevant regulatory documents. It was established that the grindability of the “clinker-gypsum” batch depends on the content of ceramic waste: the grinding fineness, determined by the residue on a sieve with mesh No.008 of cements containing 10-15% of the additive and the batch without the additive, is the same and amounts to 10%. With an increase in the additive content to 20%, the process of grinding the batch accelerates, and with an identical grinding time, the residue on sieve No. 008 was 8%. The addition of ceramic waste at the initial stage (up to 3 days) somewhat slows down the process of water binding by the mineral components of the binder composition (Fig.-1). This is apparently due to the fact that the smallest particles of ceramic waste envelop the surface of the clinker grains and prevent their intensive contact with water.

By the 7th day and in the subsequent periods, up to 90 days, water binding accelerates, and its quantity somewhat exceeds the indicator of matrix Portland cement. This indicates that in the forming cement

composite based on a binder with ceramic waste, the number of new formations also increases, which ensures its density and strength are not lower than that of Portland cement without additives. An electron microscope made it possible to observe the process of the formation and growth of new formations and the formation of the structure of the additional cement stone. After mixing the cement powder with water, a vigorous chemical interaction of its constituent components immediately begins, as a result of which, by the age of one day, a significant number of needle-shaped ettringite crystals are formed on the surface of the clinker particles, beginning to grow in air pores, in pores filled with water, as well as in intergranular spaces, oriented in different directions (Fig.-2).

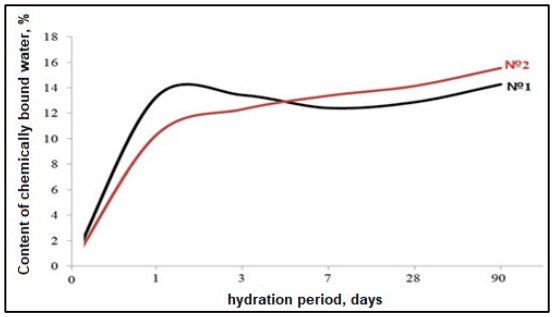

Fig.-1: Change in the Rate of Hydration of Cements

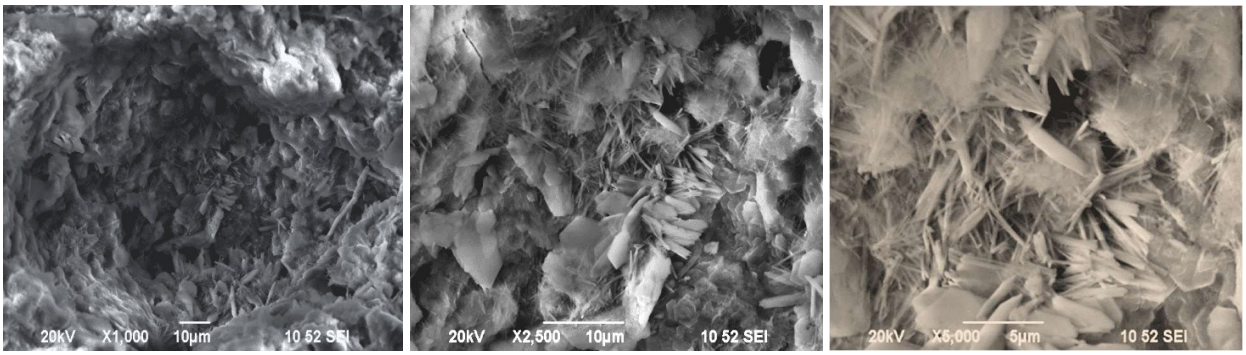

Fig.-2: Surface Relief of a Cement Chip Containing 20% Ceramic Waste after 1 Day of Hardening

In the work[14], during the crystallization of new formations in pores filled with water, they gradually increase in size and the pores become overgrown, and crystallization in air pores has a completely different character: new formations crystallize on the surface of the pore walls and form a comb-like structure and the overgrowth of such pores occurs over a long period of time. By the 3rd day of hardening, the number of crystals of needle-shaped, fibrous, and prismatic forms becomes greater, and the level of filling of pores is quite high. Fibrous crystals of calcium hydrosilicates grow together chaotically and, like a web, entangle the grains of cement, forming a framework of crystalline intergrowths, reducing the size of macropores in the forming artificial conglomerate (Fig.-3). In the surface layers and in the pores, crystals of calcium hydrosilicates also intensively appear in the form of short and thick prisms and their intergrowths in the form of druse and plates.

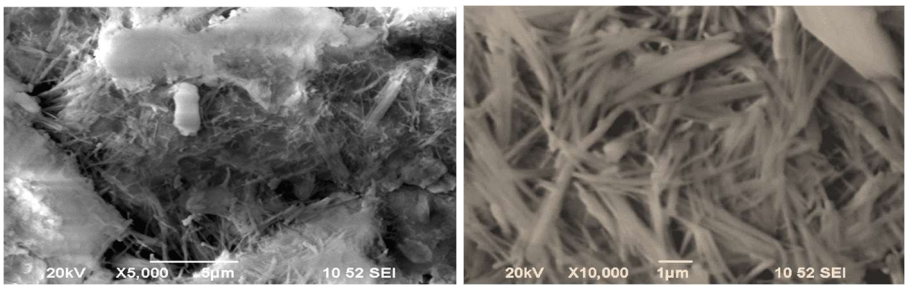

Fig.-3: Filling of Pores and Intergranular Voids of Additional Cement with Crystalline Products by 3 Days of Hardening

By the 7th day, prismatic crystals of hydrosilicates – tobermorite type – begin to tightly pack and grow together to form a block structure (Fig.-4).

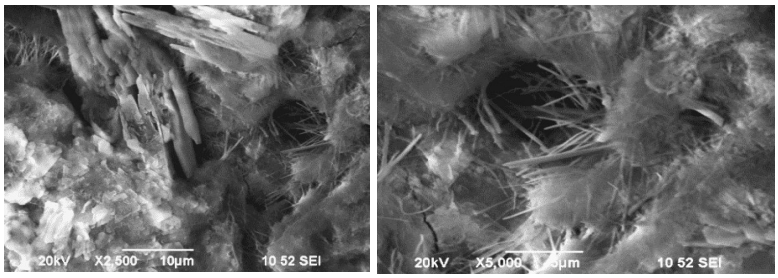

Fig.-4: Clogged Pores and Formation of a Layered-Block Structure of Cement Stone after 7 days of Hardening

By the 28th day, the microstructure of the cement stone chip surface is characterized by a denser packing of prismatic crystals of calcium hydrosilicates CSH (B) and lamellar crystals of tobermorite, which form fibrous aggregates and blocks. Fibrous (filamentous) crystals are located between the blocks of these crystals, which, dissolving on their surface, penetrate into the fine-grained mass and, as it were, "stitch" the grains of clinker minerals together (Fig.-5).

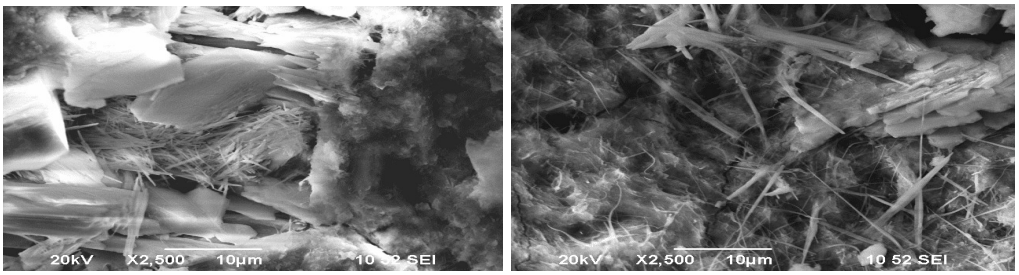

Fig.-5: Surface Relief of a Cement Stone Chip after 28 days (1) and 90 days (2) of Hardening Under Normal Conditions

The intensive growth of hydrosilicate crystals and their aggregation inside the pores of the cement stone leads to the formation of cracks and their deformation. During the recrystallization of hydrosilicates, the process of hydration, hydrolysis of silicate minerals and the release of Ca^{2+} ions into the liquid phase continues, due to which the process of "self-healing" of cracks occurs in parallel due to the formation of additional portions of calcium hydrosilicates and intensification of polymerization processes that contribute to the monolithization of the hydrate structure of cement stone and a decrease in its overall porosity, which is the main factor in achieving strength properties. indicators of cement with ceramic waste at the PC-D0 level. To determine the structural features of the composite material, IR-spectral studies were carried out. The results are shown in Fig.-6.

From Fig.-6 shows the IR spectrum of composite portlandcements, from which it follows that: absorption spectra with wavelengths of 2330.01 cm^{-1} characterize the presence of compound ($CaSO_4$); absorption spectra with wavelengths of 1950.03-1876.74-1782.23 cm^{-1} characterize the presence of functional groups (-SO-; -S-O-C); absorption spectra with wavelengths in the range of 1670.35-1620.21 cm^{-1} 1714.72-1622.13 cm^{-1} characterize the presence of compound ($CaSO_4*2H_2O$); absorption spectra with wavelengths in the range of 1116.78-1056.99-1026.13 cm^{-1} characterize the presence of compounds ($MeSiO_3$,); absorption spectra with wavelengths in the range of 962.48-902.69 cm^{-1} characterize the presence of characterize of metals (Al_2O_3); absorption spectra with wavelengths in the range of 786.96 cm^{-1} characterize the presence of functional groups (CaO); absorption spectra with wavelengths in the range of 675.09-644.22 cm^{-1} characterize the presence of characterize the presence of compounds (MgO); absorption spectra with wavelengths in the range of 596.00-501.49 cm^{-1} characterize the presence of functional groups (FeO, Fe_2O_3). The physical and mechanical properties of Portland cements with the addition of ceramic waste differ from those of the control cement PC-D0. In the presence of (5-20) % of the additive, the water requirement of the cement paste increases by (5-9) % compared to the control cement, and the setting time is extended from 200 min (3 h 25 min) to 345 min (5 h 45 min). The additive

introduced in the amount of (5-20) % has an accelerating effect on the hardening process of cements, especially in the initial stages. Thus, after 7 days, the compressive strength limit of the additional cements is (6-12) % higher than the strength of the control cement PC-D0. The same patterns of strength gain of the additional cements are noted by the 28th day of hardening. Cements containing 5 to 20% ceramic waste have a compressive strength of (41.9-42.8) MPa at 28 days of normal hardening, which is higher than the strength of PC-D0 and meets the requirements of State Standard 10178 for PC400-D20 grade Portland cements.[15,16] In this regard, studies were conducted to determine the effect of ceramic waste on the rate of hydration, the formation of the phase composition, and the microstructure of stone based on Portland cement containing 20% of the additive. In the works of the authors, waste was used as a raw material mixture, and the physical and chemical processes of clinker formation were studied for the production of Portland cement in the construction industry.[17] In the work of the authors[18,19,20], the complex of positive properties of cement stone is determined by a favorable combination of phases of different structure and morphology: crystalline hydrosulfo-aluminates, hydroaluminates, portlandite and X-ray semi-amorphous hydrosilicate-calcium gel C–S–H, etc. Tobermorite gel is the main structural component of cement stone, the crystals of which occupy approximately 70% of its volume. It consists of tiny crystals with a layered structure, and, as researchers note, the thicker and shorter the size of the calcium hydrosilicate crystals, the stronger the cement stone.[21,22,23]

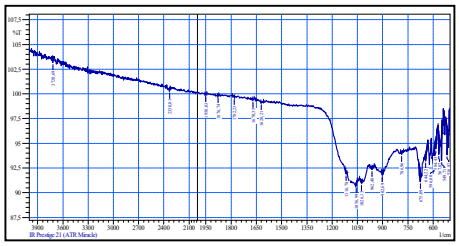

Fig.-6: IR-Spectrum of Composite Portlandcements

CONCLUSION

Thus, by studying the route of evolution of new formations in the system “finely ground clinker - ceramic waste - gypsum stone - water”, it was established that the introduction of up to 20% of the additive during clinker grinding has a positive effect on the process of formation of the stone structure during hardening of Portland cement. Due to the intensive process of nucleation and genetic formation of new formations during hydration, a cement stone of a dense structure is formed and as a result, despite a decrease in the proportion of highly active clinker by 20%, by the 28th day its hydraulic activity is 41.5-42.8 MPa, which ensures the cement grade PC 400-D20 according to State Standard 10178-85. Based on the research results, the technology of separating ceramic waste into fired and clay fractions was recommended for practical application, and the technology of producing general construction Portland cement grade PC400-D20 using fired ceramic waste was implemented at JSC «Akhangarancement».

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

This work is related to *SDG-12: Responsible Consumption and Production*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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