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The features of the hydration and structure formation process of modified low-clinker binders

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Abstract. The effect of a complex additive consisting of silica fume and enrichment waste from the Karagailinsky mining and processing plant on the hydration and structure formation processes of cement systems is investigated. It was established that the introduction of a complex modifying additive consisting of silica fume and enrichment waste from the Karagailinsky mining and processing plant into the cement composition CEM I 42.5 N SR increases the compressive strength at the age of 28 days by 15.7–28 %. It was revealed that the studied complex additives contribute to an increase in the amount of chemically bound water. The processes of hydration and hardening of multicomponent modified binders were investigated. It is shown that the high strength of cement stone on a modified binder is due to the formation of stable low-base hydro silicates. The source of the formation of low-base calcium hydro silicates is the pozzolanic reaction, which proceeds with the binding of the clinker minerals released during hydration, portlandite silica complex additive. It is shown that purposefully changing the composition of the binder and hydration conditions, it is possible to improve the phase composition, morphology of hydrates and influence its final properties.

1. Introduction

Objective: to study the effect of a complex additive consisting of silica fume and enrichment waste from the Karagaila mining and processing plant on the hydration and structure formation of cement systems.

To achieve this goal, the following scientific problems are formulated and solved in this work:

- analysis of scientific research on the effects of various active mineral and finely dispersed additives on the properties of concrete;
- substantiation of scientific and practical aspects of the use of complex modifiers and fine industrial wastes in concrete technology;
- study of the effect of a complex additive consisting of silica fume and enrichment waste from the Karagailinsky mining and processing plant on the composition and morphology of hydrated neoplasms, as well as the hardening processes of cement systems.

The production of high-performance binders of the new generation today is accompanied by the use of complex compositions of components in order to obtain high-strength concrete of different functional purposes with high construction and operational properties [1–3]. It is known [4] that the addition of tripoli dolomite, limestone, slag, and calcined marl during grinding of Portland cement clinker accelerates cement hydration.

V.V. Timashev developed a quick-hardening binder containing 3–7 % alumina cement and 25–30 % active mineral additives (tripoli or diatomite). The reason for hardening acceleration is the formation of calcium hydrosulfoaluminate [5].



As an active mineral additive to cement, W.A. Ayapov used the flask of the Aisar deposit. Cement with an active mineral additive was activated by complex catalysts – gypsum and ROH, where R-Na, K. According to the authors, the presence of an active mineral additive containing amorphous silica in the cement contributes to an equal increase in the rate of catalytic reactions [7].

Vlasov V.K. investigated the role of active mineral additives in ultra-hardening cements. These cements, including the rapidly hydrating $C_{11}A_7COX$ (where X is fluorine or chlorine) and C3S, are characterized in some cases by strength drops caused by internal stresses, especially at the boundaries of hydro aluminates, which crystallize completely in 6–10 hours of hydration. To reduce the level of internal stresses of ultra-hardening cement, pozzolan type materials (ground volcanic ashes, tuffs, slopes, soot, diatomite, tripoli, calcined clay or slate, blast furnace slag) were introduced into its composition [8].

V.V. Timashev and V.M. Kolbasov established that the introduction of 5–10 % of active mineral additives favors the formation of a strong structure of cement stone by 28 days. The stabilizing role of active mineral additives for ettringite in cement stone and for moisture redistribution presses during cement hydration was confirmed.

The basis for the creation of modified low-linker binders is based on the principle of purposeful management of the technology at all its stages: the use of active components, the development of optimal compositions, the use of chemical modifiers and some other techniques [5, 6].

The introduction of active mineral additives into the cement composition reduces the likelihood of false setting, due to a reduction in the excess of $Ca(OH)_2$ in the liquid phase of the test and the adsorption of R_2O from it, and also hindering the formation of continuous aluminate aggregates with cement grains in the first minutes of hydration. In addition, active additives accelerate the movement of material through the mill, reduce the likelihood of grinding and “steaming” the mixture, disaggregate cement and prevent undesirable surface reactions.

The most important tasks of science in the field of construction: simplification of structures, acceleration and reduction in cost of technological processes, in particular, reducing the duration of heat and moisture treatment of products, increasing the corrosion resistance of concrete can be solved only by giving special properties to cements [8–10]. The way to solve this problem is through the expansion of the nomenclature of hardening modifiers [11–13]. The use of non-ferrous metallurgy waste as additives to cement will significantly expand the raw material base of the cement industry and meets modern requirements to ensure an increase in the demand for raw materials and materials due to their economy and more complete use of secondary raw materials, slags and other wastes [14, 15].

The basis for the creation of modified low-clinker binders is the principle of targeted technology management at all its stages: the use of active components, the development of optimal compositions, the use of chemical modifiers and some other techniques [16, 17].

According to this principle, a multicomponent binder containing up to 45 % of non-ferrous metallurgy waste and chemical additives was obtained [18]. At the same time, the improvement of the submicroscopic structure with a slight increase in the size of the effective radius of micropores from 10.5 to 10.8 nm was determined, which may be associated with the filling of large pores (more than 100 nm) with calcium hydro silicates [19].

As shown in [20], the applied complex additives based on enrichment waste do not increase the normal density of the dough, does not slow down as usual, but accelerate the rate of hydration of the binder. In combination with the use of plasticizing additives, these low-linker binders can be used for the manufacture of concrete and reinforced concrete products under optimal hardening conditions.

2. Research methods

An X-ray diffraction study of the materials was carried out on an URS-50I X-ray diffractometer. Radiographs were taken from flat powdered samples rotating in the plane of the axis of the goniometer at a speed of 25 rev/m, in the range of angles from 20° to 61° . X-ray mode: X-ray radiation ScK° – filter Ni; 14 microns thick, anode current of the X-ray tube 11.5 mA, counter slit 0.25×8 mm, slit limiting the primary beam 1×5 mm, recording constant $R^\circ - IV$, recording range of intensities D-1000 imp /c, speed movement of the chart tape 360 mm/hour.

Differential thermal studies were carried out on a MOM-1000 derivatograph system F. Poulik, I. Powlik and L. Erdey (Germany) in the temperature range 25–1000 °C in an air stream. The rate of temperature rise is 7.5 deg/min. Thermal transformations were determined from the curves of mass loss (TG), differential mass loss (DTA) and the change in differential temperature (DTA).

1. Spectral analysis of cement stone was carried out on a Spekord two-channel spectrophotometer. Using electron microscopes REM-200 and EVM-100 BR, the shapes, sizes, arrangement, and type of crystals

were investigated, volumetric images were obtained, and the composition of individual sections of cement stone was determined.

2. The studies were carried out by the powder method. Samples prepared for REM-200 and EVM-100 BR studies were dried to constant weight and crushed to a specific surface of 480.0 m²/kg [21, 22].

The optimal content of complex modifying additives was determined based on the greatest compressive strength of cement stone by testing 4x4x16 cm (mortar) after normal hardening according to Russian State Standard GOST 310.4-81 "Cements. Methods for determining the tensile strength in bending and compression" [23]. For a comparative analysis of the effect of complex additives on the compressive strength (Table 1), concrete samples based on cement CEM I 42.5 N SR without additives and with complex modifying additives were prepared with W/C = 0.4.

Heat and moisture treatment of prototypes is carried out according to the mode 2 + 4 + 1 h at a maximum temperature of 65 °C. In this case, the maximum processing temperature is reduced by 20 °C against the usual, and the duration of the isothermal exposure is reduced by 3 hours. Heat treatment was also carried out using solar energy at a maximum temperature of 65 °C during daylight hours in translucent chambers [24, 25].

3. Results and Discussion

The composition of the binder includes (in % by weight): cement CEM I 42.5 N SR – 70-80; silica fume – 5–10; enrichment waste – 10–20. The binder is prepared by joint dry grinding of the components to a specific surface of 320–350 m²/kg.

Sulfate-resistant Portland cement CEM I 42.5N SR GOST 22266-2013 Standard-Cement LLP (Republic of Kazakhstan, Shymkent) was used as a binder without additives [26].

The enrichment waste of the Karagailinsky mining and processing plant (Kazakhstan) with a specific surface area of 160–205 m²/kg consists mainly of quartz (76-85 %); there are minerals: montmorillonite (5–8 %), dolomite (5–10 %), repidomite (3–5 %), pyrite (1–6 %), albite (2–4 %), leuchtenbergite (3–10 %). Chemical composition of enrichment waste (% by weight): SiO₂ – 84.19; Fe₂O₃ 1.18; Al₂O₃ – 1.58; CaO – 2.58; MgO – 0.60; C – 1.52; S_{total} – 0.54.

Silica fume, a nanomaterial, is a waste product of the production of silicon-containing alloys: ferrosilicon, crystalline silicon, etc. During melting of the charge and reduction of quartz at temperatures above 1800 °C, gel-like silicon is formed, when cooled and in contact with air, it oxidizes to SiO₂ and condenses in the form of ultrafine silica particles. The particle size of silica fume is 0.1–0.5 micrometers.

Table 1. Properties of modified low-linker binder.

cement	Composition of the binder, mass %		Tensile strength at compression, MPa
	microsilica	enrichment waste	
100	-	-	44.5
84,5	5	10	51.5
74,2	7.5	15	57.0
64,0	10	20	54.0

The compositions and strength properties of the developed modified low-clinker binder are given in Table. 1. The test results show that the compressive strength of the modified small clinker binder at the age of 28 days hardening under normal conditions is 51.5–57.0 MPa. The introduction of a complex modifying additive to the cement composition CEM I 42.5 N SR increases the compressive strength at the age of 28 days by 15.7–28 %.

On roentgenograms of the modified low-clinker binder, there are also lines with $d = 0.422; 0.390; 0.180; 0.260; 0.241$ nm, indicating the presence of a C₂SH (A) type hydro silicate (Fig. 1), as well as the formation of two types of CSH (II) hydro silicates and gyrolite. They correspond to diffraction maxima with $d = 0.304; 0.280; 0.182; 0.167$ nm – CSH (II) and $d = 0.424; 0.336; 0.384; 0.285; 0.265; 0.225$ nm – gyrolite (Fig. 1). The formation in the stone of a modified low-clinker binder mineral gyrolite is explained by the high reactivity of silica fume and is associated with the presence in the latter of the predominant amount of amorphous finely divided silica. The results of X-ray phase analysis are confirmed by differential thermal analysis (DTA) (Fig. 2).

Analysis of thermograms showed that all thermograms in the low-temperature region have endo-effects associated with the primary dehydration of calcium hydro silicates. Moreover, as is known from the literature, the endo-effect at $T = 120–150$ °C corresponds to a gyrolite, and the exo-effect at $T = 200–250$ °C corresponds to CSH (II).

For C_2SH (A), dehydration is characteristic at a temperature of 420–480 °C. Such endo effects were found only in steamed samples. This corresponds to a change in the stoichiometric ratio of CaO and SiO_2 oxides in raw mixtures.

Differential thermal studies (DTG curves and the results of moisture loss during heating) of a cement stone indicate a close correlation between the heating temperature and the ability of the hydrated phases to retain water when heated (Table 2).

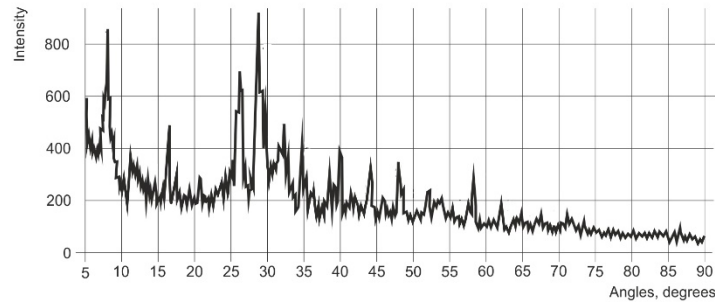
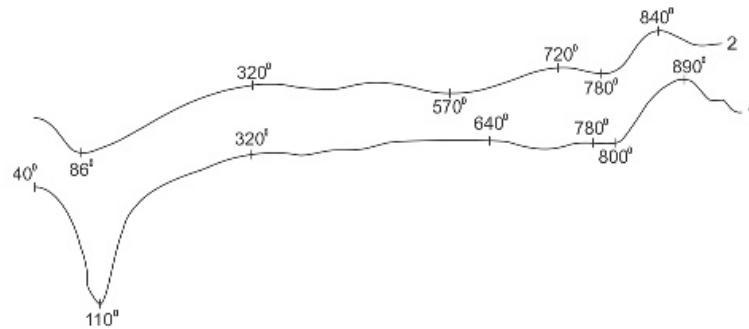


Figure 1. Radiograph of a modified low-clinker binder at 28 days of hardening.

Analysis of the mass loss during the heating of cement stone (Table 2) shows that complex additives contribute to an increase in the amount of chemically bound water.



**Figure 2. Thermogram of binders at the 28-day age of hardening:
1 – modified low-clinker binder; 2 – cement without additives.**

Differential thermal studies confirm the data of x-ray analyzes. DTG curves and the results of cement stone moisture loss (Table 2) upon heating indicates a close correlation between the heating temperature and the ability of the hydrated phases to retain water when heated. Complex additives contribute to the increase in the amount of chemically bound water.

Table 2. Mass Loss during heating of cement stone from modified low-linker binder (MMV) according to DTA data.

Type of binder	Mass loss in % in the temperature interval, °C			Relative mass loss, %
	20–200	20–600	20–1000	
CEM I 42.5 H SR	4.9	13.5	23	36
IIM	5.2	12.3	22	41

It can be assumed that these additives are enrichment waste, increasing the centers of crystallization and favor the growth of inter-crystalline cavities inside, which arise during the formation of a supramolecular layered structure. Such cavities are able to keep water molecules in a particularly oriented state, in which the rotational degrees of freedom of the molecule are inhibited and the translational ones are partially limited [27]. Within the monolayer, such water molecules have significant mobility and create conditions for easy sliding of the cement gel, which in turn facilitates the appearance of irreversible plastic deformations.

In the IR spectrum of cement stone, after 1 day in the wave number range of 700–1200 cm^{-1} , a wide band is distinguished, split into parts characteristic of calcium silicates. Absorption maxima at 930, 885, 840 cm^{-1} indicate the presence of dehydrated C3S (Fig. 3).

The infrared spectrum of the cement stone of modified low-clinker cement (Fig. 3) after 3 days shows absorption bands at 730, 780, 820, 860 and 880 cm^{-1} , which indicates the presence of a large degree of distortion in the structure of the mineral $[\text{AlO}_4]$.

In the cement stone of modified low-clinker cement, $[\text{AlO}_4]$ tetrahedra are severely deformed, which explains the high hydration activity in the initial stages of hardening. The absorption bands at 890, 860, 820, 780 cm^{-1} are due to the stretching vibrations of the bound $[\text{AlO}_4]$ groups, and the band at 730 cm^{-1} are due to the stretching vibrations of the isolated $[\text{AlO}_4]$ tetrahedra.

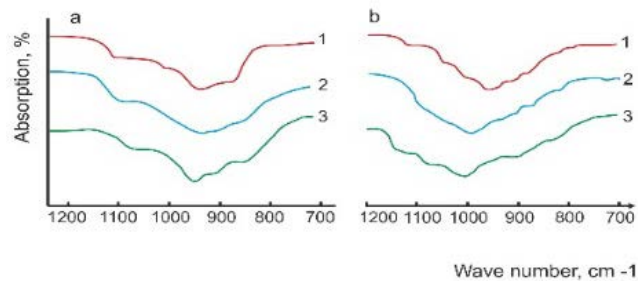


Figure 3. The infrared spectrum of cement stone
a – cement CEM I 42.5 N SR, b – modified low-clinker cement stone.
1, 2 and 3 after 1,3 and 28 days of normal hardening.

At the 28-day hardening age, the absorption band at 940 cm^{-1} observed in cement CEM I 42.5 N SR with the introduction of complex additives in it shifts toward large wavenumbers and is absorbed at 970 cm^{-1} . Such a shift of the bands shows the process of polycondensation of $[\text{SiO}_4]$ – tetrahedra; due to the modified binder, the basicity of hydro silicates decreases [28].

Using electron microscopes, the shapes, sizes, arrangement, and appearance of crystals were studied, volumetric images were obtained, and the compositions of individual sections of cement stone were determined. The processes of germination of the phases CH and C-S-H, as well as changes in the morphology of hydrates, were studied.

With an increase of 2500 times, micropores with a size of 2–3 microns are observed. Recrystallization and growth of hexagonal portlandite crystals obey the laws of collective growth and proceed metasomatically [29]. With an increase in the hardening time, crystallization of portlandite occurs by topochemical hydration processes inside anhydrous silicate crystals [30, 31]. The resulting calcium hydroxide can be almost amorphous due to the lack of space for diffusion, as well as due to disturbances created by inclusions of silicon-containing groups.



Figure 4. Microstructure of a modified low clinker binder, x2500
a – micropores of cement stone are compacted with thin sheets with CSH foil (1)
(at 180 days of hardening age); b – Intergrowth of portlandite blocks with gel-like CSH.

Amorphous portlandite is very difficult to distinguish from poorly crystallized gel phases of CSH during X-ray phase and electron microscopy studies. However, the authors of [5] proved the existence of amorphous portlandite in the hydration products of C_3S and $\beta\text{-C}_2\text{S}$.

During hardening, Portlandite binds to active silica complex additives. If portlandite is in an amorphous form, the pozzolanic reaction (the binding process) is faster. In this case, the most typical form of CSH (1) is formed, which is large, but very thin sheets or foil with a thickness equal to the thickness of the main layer (Fig. 4, a). These sheets or foil stick together easily, forming loose folded aggregates up to 2 micrometers in size. The leaves of CSH (1) foil are so thin and transparent that only folds are observed, giving the impression of a fine-fiber structure.

Unlike the control one, in a cement stone with complex additives in portlandite blocks they are tightly fused with the cement gel, forming a monolith of gel and CH or areas of their mutual germination (Fig. 4, b). The structure of the external rhythm consists of embryos and growing crystals, obeying the laws of collective growth.

With an increase in the hardening time, crystals grow in the structures of the external rhythm and crystals arise in the structures of the internal rhythm, which leads to the recrystallization of crystals and their nuclei. Complex additives, affecting the nature of crystallization, can change the composition of the crystal, forming solid solutions.

In the process of cement stone hardening, portlandite layers are the matrix for the incorporation of various elements and ions into it with the subsequent formation of hydrated compounds. This explains the presence of structural elements of calcium hydroxide in many hydrated compounds of cement stone.

4. Conclusion

1. It was established that the introduction of a complex modifying additive to the composition of cement CEM I 42.5 N SR consisting of silica fume and enrichment waste from the Karagailinsky mining and processing plant increases compressive strength at the age of 28 days by 15.7–28 %.

2. DTG curves and the results of cement stone moisture loss upon heating indicates a close correlation between the heating temperature and the ability of the hydrated phases to retain water when heated. Complex additives contribute to the increase in the amount of chemically bound water.

3. To ensure the formation of the structure of a cement stone with minimal porosity and increased strength, it is necessary to stabilize the composition of hydrated neoplasms, prevent their phase transitions, regulate the hydration process, the optimal ratio of crystalline gel-like masses in hydration products by selecting the composition of mineral additives and micro-fillers.

4. An additional source of the formation of stable calcium hydro silicates is the pozzolanic reaction, which takes place with the absorption of portlandite released by hydration of clinker minerals with amorphous silica of a complex modifying additive.

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