

EFFECT OF COMPLEX ADDITIVE ON EXOTHERMIC KINETICS AND HYDRATION STAGES OF CEMENT SYSTEMS

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ABSTRACT: The influence of complex additives on the kinetics of cement exothermia and the stage of cement hydration at hardening of heavy concrete in industrial conditions has been studied. It is established that in heavy concrete B35 with the use of cement CEM I 42.5 N SS with complex admixture maximum temperature of hydration - 79.0 ° C reaches 26 hours 30 minutes after pouring and preservation of maximum temperature within 78.0-79.0 ° C continues for 9 hours. Complex additives significantly increase the degree of hydration of cement. At the same time, the initial hardening time is 4-4.5 % and 28 day hardening age is up to 15 %. It is established that the introduction of complex additive in the cement composition of CEM I 42.5 N SS promotes the formation of smaller and mostly oriented secondary crystals of Portlandite. The considered complex additives consisting of the active mineral additive (wastes of enrichment of polymetallic ores), nanofiller (microsilica) and chemical additives (master air 200 and master rheobuid 1000) changing speed of hydration and time of achievement of the maximum temperature index, define the periods of hydrate formation.

Keywords: Exothermia of cement, Hydration stages, Hydrate phases, Binder composition, Complex additive

1. INTRODUCTION

The work objective is studying the effect of complex agents on the kinetics of exotherm and hydration stages of cement systems.

The considered complex additives consisting of an active mineral additive (wastes of enrichment of polymetallic ores), nanofiller (microsilica) and chemical additives (Master air 200 and Master rheobuild 1000) changing speed of hydration and time of achievement of the maximum temperature index, define the periods of hydrate formation.

The use of new technologies and materials in the construction industry is of high social importance and is the basis for innovative processes in construction.

In the world construction practice, modified heavy concrete has firmly taken one of the leading positions as a universal material that allows to solve a wide range of engineering problems and provides modern quality and competitiveness of construction products.

Physico-technical properties of the modified heavy concrete allow its use for the construction of supporting and enclosing structures in various areas of construction. Due to its qualities, this type of concrete is most widespread in industrial and civil construction.

It is known that concrete hardening is

accompanied by heat generation which intensity depends on the mineralogical composition of cement.

There are scientific studies of domestic and foreign scientists in this field, however, their results do not contain any data on the effect of various mineral and chemical agents on the hydration heat of clinker minerals and cement.

2. LITERATURE REVIEW

As an active mineral admixture to cement, the authors [1] used the support of Aysar mine. Cement with mineral mixture was activated under the action of complex catalysts containing gypsum and ROH, where R - Na, K. According to the authors, the presence of amorphous silica in the active mineral mixture added to cement promotes the same increase in catalytic reaction rate. The role of active minerals in cement, which hardens very quickly, is important. $C_{11}A_7SOH$ (where X is fluorine or chlorine) and C_3S rapidly hydrogenated cement at hydrogenation for 6-10 hours are characterized by the influence of internal stress in the zone of fully crystallizable hydroaluminate, in most cases, a decrease in strength. To reduce the level of internal stress of very quickly solidified cement, it includes materials with pozzolanic properties (ash, tuff, road, soot, diatomite, fired

clay or slate, blast-furnace slags) [2].

The authors [3,4] have determined that the structure of strong cement stone is formed within 28 days with the addition of 5-10% of mineral additives. They determined that the hydration of cement stabilizes the process of formation of ethtringite mineral mixture and provide an even distribution of moisture in the cement rolled products.

The authors [5,6] have determined that the construction of strong cement stone is formed within 28 days with the addition of 5-10% of mineral additives. They have determined that the hydration of cement stabilizes the formation of ethtringite mineral mixture and provide an even distribution of moisture in the composition of cement rolled products. In M.M. Sychev's work the influence of bentonite mixture on solid cement system is investigated. The strength of cement in the initial period (1-7 days) increases to 50%, including activated bentonite at 2-5% of the binder mass, 460 °C.

Sychev M.M. has shown in his studies that the introduction of a mixture containing 15-20% carbonate does not affect the initial strength of cement stone (3 and 7 days), but its 28 and 180 daily strength limits exceed 15 and 25%, and in the period of 1080 daily hardening the strength limit is 68.5 MPa. Increase in strength of the cement stone containing carbonate, with growth of terms of hardening is explained by mutual growth of crystals hydrohydrates and their epitaxial combination.

Based on studies [7], the hydration mechanism of cement using a polymetallic mixture can be divided into two stages:

- intensive hydration stage (from the beginning of hydration to 28 days). In this case, the hydration of clinker minerals, alkaline sensitivity of the mixture glass phase, gypsum solder and limestone to complex hydrate;

- slow hydration stage from 30 days to 1080 days. During this period of hardening is the process of levelling destructive phenomena due to the pozzolanic reaction.

In this regard, it is important to study the effect of a complex agent consisting of micro-filler (silica fume and waste from the processing of polymetal ores) and chemical agents (Master Air 200 and Master Rheobuid 1000) on the kinetics of heat generation and concrete hardening processes.

In present day concrete technology increasing attention is paid to the development of energy-saving technologies of accelerated concrete and reinforced concrete products hardening, the most important element of which is the use of the binder hydration heat.

The relevance of this task consists in the fact that the efficiency of the cement own heat

dissipation can be considered as a hardening accelerator at the initial stages in monolithic construction and production of concrete and reinforced concrete.

In work the complex method of researches including the analysis of theory and practice of use of the modified heavy concrete, standard and physicochemical methods is used.

3. HEADINGS

The composition of the modified binder includes (in weight %): cement CEM 1 42.5 H CC - 70-80; silica fume - 5-10; tailings - 10-20. The binder is prepared by joint dry grinding of the components to the specific surface of 320-350 m²/kg.

It has been experimentally established that the optimal content of chemical agents Master Air 200 (air-entraining agent) and Glenium is 0.08 and 1.6 % of the cement mass, respectively. When adding the air-entraining agent Master Air 200 in the amount of 0.08 % by weight of cement, the amount of air entrained is 4.4 %. The introduction of Glenium superplasticizer in the amount of 1.6 % by weight of cement with the concrete mix mobility of 14 cm allows reducing the W/C ratio to 0.39. Table 1 shows optimal composition of heavy concrete B35 using complex modifying agents.

Table 1 The optimal composition of heavy concrete B35 using complex modifying agents

Type of chemical agent	Material consumption per 1 m ³ of concrete, kg						
	Chem. agent	Stones, fr., mm		Sand	Cement*	Water	W/C
		5-10	10-20				
Master Air 200-0.08 %*	0.336	500	700	690	400	157	0,39
Glenium-1.6 %	6.75						

*in the cement composition there has been introduced a modifying agent: 20 % of the weight.

The density of the concrete mixture is 2455 kg/m³; the amount of air entrained is 4.4 %. The mobility of the concrete mixture along the draft of the cone is 14 cm at the W/C ratio 0.39. Under normal conditions of hardening, compressive strength after 7 days is 38.5 MPa and after 28 days 49.0 MPa.

Experimental data show that the introduction of additives significantly affects the processes of

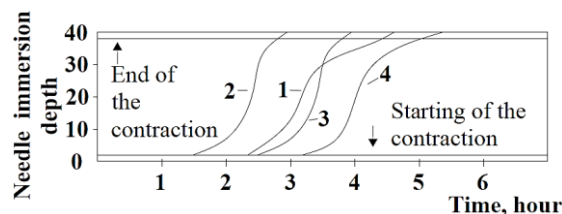
structure formation of the cement test. Introduction of "Glenium" superplasticizer accelerates the beginning of cement test setting and reduces the period of structure formation. In this case, the period from the beginning to the end of setting is reduced by 60 minutes, compared to cement dough without additives (Table 2).

Table 2 Influence of additive content on properties of low-clinker modified binders

Type of binding material	Normal density, %	The timing of the contraction, h-min	
		starting	end
CEM I 42.5H SS	26,0	2-20	4-30
CEM I 42.5H SS + 1.6% JV "Glenium 51".	24,0	1-30	2-40
CEMENT I 42.5N CC + 15% MD	25,5	2-35	3-40
CEMENT I 42.5N CC + 20% MD	26,5	3-15	5-10

The introduction of a 15% modified additive (CEM I 42.5H SS + 15% MD) has very little effect on the rate of structural formation in the initial period, which is confirmed by an almost identical slope angle of the curves to the time axis. By the end of the setting period, CEM I 42.5H SS + 15% MD significantly reduces the rate of structure formation. In this case the period from the beginning to the end of the setting is 1 h 05 min, which is 65 min less compared to the cement test without additives.

Cement dough with 20% modifying additive (CEM I 42.5H SS + 15% MD) at the end of the period reduces the rate of formation and increases the period from start to finish setting. The setting period is reduced by 15 min.



- 1 - CEM I 42,5H SS;
- 2 - CEM I 42,5H SS+1,6 % JV "Glenium 51";
- 3 and 4 - with 15 and 20 % modifying additives respectively

Fig.1 Effect of additives on cement dough setting kinetics

The introduction of complex additives into the cement increases the water consumption of the mixture. This is due not only to the diversion of water to wet additives, but also to the fact that they change the properties of the adjacent cement dough layers due to surface forces. By changing the normal density of the cement dough, additives also have a noticeable effect on the setting time, the rate of structural formation as shown at Fig.1.

The research methodology is as follows: at the construction site, reinforcing cage of type-02 foundations (the volume is 2.8 m³) there are mounted the TM-1288/1 type thermometers. The thermometer readings are recorded in the eight-channel microprocessor device TM 5103 (entered in the State Register of Measuring Instruments under No. 54183-13, TU 4210-024-13282997-03). Multichannel thermometers TM 5103 are designed to measure, control and archive the temperature values. The arrangement of the thermometers in the structure is shown in Fig.1.

The first foundation was made of concrete mix B 35 using cement CEM 1 42.5 H CC without agents, and for the second foundation cement was used with a complex modifying additive (Table 1).

In Fig.2 we can see that in the first and second foundations, there are mounted thermometers No. 1 and No. 6 in the center, respectively, thermometers No. 2, No. 3 and No. 7 and No. 5 are mounted along the edge of the formwork at different levels. Thermometers No. 4 and No. 8 are mounted to control the outside temperature or the air temperature.

The results of measuring the temperature of concrete over time are shown in Table 3.

To exclude the effect of the environment external temperature, after pouring concrete the foundations were completely covered with thermal insulation material.

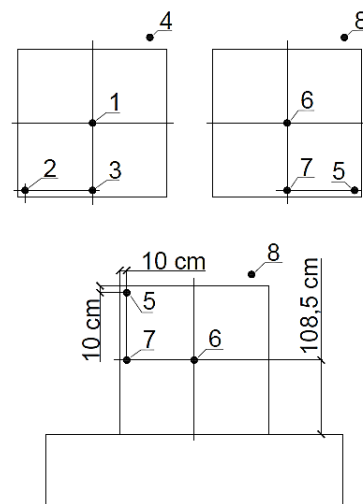


Fig.2 Diagram of the thermometers 1-8

arrangement in the structures

The temperature of concrete was recorded continuously by the microprocessor device TM 5103.

The analysis of the results of the concrete temperature changes shows that 5 hours after pouring, the temperature of concrete began to rise markedly and amounted to 37.1-38.3 °C (Table 3). Such relatively slow rise of the temperature is explained by the regular process of cement hydration that can conditionally be divided into three stages [8,9].

Table 3 Changing the concrete temperature over time

Time	Thermometer No.							
	1	2	3	4	5	6	7	8
1 st day								
10:00	29.9	30.5	30.3	25.3	31.1	30.0	30.3	29.0
10:30	30.3	31.0	30.7	26.0	31.2	29.7	30.9	28.1
11:00	30.5	31.0	30.9	26.7	31.2	30.6	31.0	28.8
11:30	30.5	31.2	31.1	26.9	31.2	31.2	31.3	28.9
12:00	31.1	31.3	31.4	28.3	31.3	31.6	31.6	29.9
12:30	31.6	31.8	31.9	29.3	31.6	31.8	32.1	30.8
13:00	32.7	32.8	33.2	29.2	32.6	32.1	33.1	31.1
13:30	33.2	33.2	33.7	29.5	33.0	32.6	33.6	31.3
14:00	34.3	34.3	34.8	30.8	34.0	33.6	34.7	31.5
14:30	35.7	35.5	36.2	31.5	35.2	35.3	36.0	32.4
15:00	37.7	37.3	38.3	31.6	37.1	37.9	37.9	31.8
15:30	39.9	39.3	40.8	32.9	39.0	40.2	40.1	31.4
16:00	42.9	41.9	43.8	34.3	42.5	43.2	43.1	31.9
16:30	45.5	44.1	46.6	34.4	43.6	46.1	45.7	32.3
17:00	48.7	46.5	49.9	34.6	46.0	49.2	48.6	32.6
17:30	51.8	48.7	52.7	34.4	47.9	51.9	51.3	32.3
2 nd day								
08:30	76.2	47.1	69.1	26.8	60.3	77.8	68.7	28.8
09:00	76.1	47.2	69.0	27.3	60.4	77.9	68.7	29.7
09:30	76.2	47.4	68.9	28.9	60.5	78.0	68.8	31.6
10:00	76.2	47.7	68.8	30.8	60.7	78.2	68.8	34.0
10:30	76.1	47.9	68.6	31.5	60.7	78.3	68.7	34.7
11:00	76.1	48.0	68.4	31.4	60.7	78.3	68.7	34.1
11:30	76.0	48.3	68.4	32.5	60.7	78.7	68.7	36.0
12:00	75.9	48.4	68.3	33.3	60.6	78.6	68.7	36.0
12:30	75.4	48.8	67.9	36.6	60.4	79.0	68.6	35.4
13:00	75.3	48.9	67.6	37.4	60.2	78.9	68.5	34.7
13:30	75.1	49.1	67.6	37.4	60.1	78.8	68.4	35.4
14:00	75.1	49.3	67.5	37.9	60.2	78.8	68.3	35.2
14:30	75.0	49.4	67.4	37.9	60.2	78.8	68.2	35.6
15:00	74.8	49.5	67.2	37.7	60.0	78.6	68.2	35.7
15:30	74.6	49.6	67.1	36.4	59.7	78.4	68.1	34.9
16:00	74.3	49.3	66.9	36.2	59.5	78.2	68.1	34.4
16:30	74.1	49.1	66.6	34.7	59.4	78.1	68.2	33.6
17:00	73.9	49.0	66.4	34.7	59.1	77.8	68.1	33.0
17:30	73.8	48.7	66.2	34.3	58.6	77.7	67.9	32.7
18:00	73.6	48.4	66.1	33.9	58.2	77.3	67.9	32.5
19:00	73.1	47.8	65.6	30.2	57.3	76.5	67.7	29.8

At the first stage, when cement is mixed with water during hydrolysis of tricalcium silicate, calcium hydroxide is released, forming a supersaturated solution [10,11]. In this solution

there are ions of sulfate, hydroxide and alkali, as well as a small amount of silica, alumina and iron.

A high concentration of calcium ions and sulfate ions is observed within a short time after mixing cement with water, since within a few minutes the first newgrowths begin to precipitate from the solution: calcium hydroxide and ettringite.

Complex agents change significantly the rate of hydrate nucleation. The studies show [12,13] that 3 minutes after mixing of the binder with water, the first needle-like newgrowths appear, indicating intensification of the hydration process. With 2000 time magnification, micropores with the size of 2-3 microns are observed.

About an hour later, the second stage of hydration begins that is characterized by the formation of very small calcium hydrosilicates [14]. Due to the fact that only surface layers of cement grains are involved in the reaction, the grain size of the cement decreases slightly. The newly formed hydrated phases, called the cement gel, are characterized by the nanostructure.

Newgrowths primarily appear on the surface of cement grains [15]. With increasing the number of newgrowths and their packing density, the boundary layer becomes poorly permeable to water within about 2 to 5 hours.

The second stage of delayed hydration is usually called the "latent, or induction period" of cement hydration.

Then there comes the third stage of the hydration process. In our experiments this period begins in 5 hours 30 minutes. At this, the temperature of concrete rises to 39.3-40.8 °C. Increasing the concrete temperature indicates the onset of crystallization of calcium hydroxide from the solution [16].

This process is very intense. Since at this stage the number of hydrated phases is relatively small, in the space between the cement particles there takes place free growth of thin plates of calcium hydroxide and calcium hydrosilicates and ettringite in the form of long fibers that are formed simultaneously [17].

After 3 days of hardening, hexagonal prismatic Portlandite crystals are found at the bottom of such pores, which indicates strong initial supersaturation of the liquid phase with Ca²⁺ ions. The further recrystallization and growth of hexagonal Portlandite crystals obey the laws of collective growth and proceed metasomatically [18].

In the process of intense hydration of clinker minerals C₃A C₄AF and C₃S, after 22 hours and 30 minutes, thermometers No. 1 and No. 3 (foundation No. 1) respectively reach the maximum temperature of 76.3 and 69.1 °C.

In foundation No. 1 (cement CEM 1 42.5 H CC without agents), the maximum temperature was recorded by the thermometers:

- No. 1 after 22 hours 30 minutes - 76.2 °C;
- No. 3 after 22 hours 30 minutes - 69.1 °C;
- No. 2 after 29 hours - 49.6 °C.

In foundation No. 2 (cement CEM 1 42.5 H CC with a complex modifying agent), the maximum temperature was recorded by the thermometers:

- No. 7 after 23 hours 30 minutes - 68.8 °C;
- No. 5 after 24 hours - 60.7 °C;
- No. 6 after 26 hours 30 minutes - 79.0 °C.

Preservation of the reached maximum temperature of concrete can be observed from 4 to 9 hours, depending on the location of the thermometers.

The above data confirm that the third stage of the hydration process occurring after 5 hours 30 minutes, lasts up to 27 hours.

After 27-30 hours, a uniform and slow decreasing of the temperature is observed at all studied points of concrete structures (Fig.2). This confirms the completion of intensive C₃A and C₄AF hydration, as well as completion of calcium hydroxide crystallization through the liquid phase [12].

The X-ray analysis (Fig.3) shows that the phase composition of cement CEM 1 42.5 H CC with a complex agent at the age of 1 and 7 days consists mainly of Ca(OH)₂ (d = 0.493; 0.310; 2.262; 0.192; 0.179; 0.148 nm), γ-hydrate C₂S (d = 0.304; 0.270; 0.247; 0.235; 0.189; 0.179; 0.165; 0.154 nm), tobermorite-like hydrosilicate CSH₂ (d = 0.281; 0.183; 0.167 nm) and non-hydrated C₃S (d = 0.277; 0.267; 0.244; 0.198; 0.194; 0.177; 0.163; 0.149 nm).

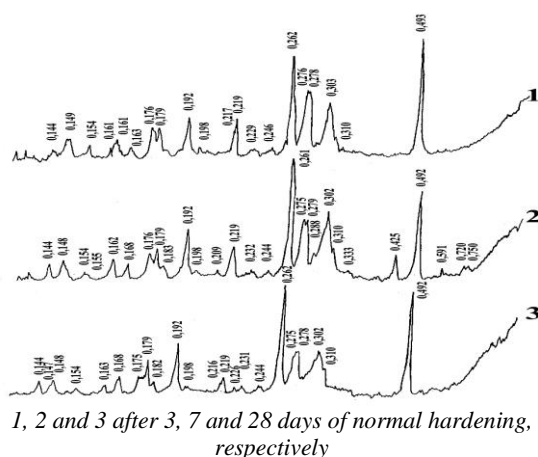


Fig.3 X-ray pattern of cement stone CEM 1 42.5 H CC with a complex agent

At the 28 days age, the amount of non-hydrated C₃S is significantly reduced, which is accompanied

by increasing the degree of hydration of CEM 1 42.5 H CC without agents after 3.7 and 28 days, respectively, by 50, 62 and 71 % (Table 3).

Table 3 Agents effect on the degree of hydration

Binder type	Hydration degree, % at the hardening age, days		
	3	7	28
CEM 1 42.5 H CC without agents	50.0	62.0	71.0
CEM 1 42.5 H CC with a complex agent	54.5	66.0	86.0

Complex agents increase significantly the degree of cement hydration, at this at the initial hardening periods by 4–4.5 % and at the 28-day age of hardening up to 15 %.

The studies have shown [19,20] that in X-ray diffraction patterns of pure calcium hydroxide, the intensity of the line of the (0001) plane with d = 0.490 nm is 70-80 % of the intensity of the line of the (0011) plane with d = 0.262 nm. This is explained by the predominant formation of large crystals of Ca(OH)₂. However, on the X-ray pattern of the cement stone CEM 1 42.5 H CC with a complex agent hydrated within 3 days, the intensity of the Ca(OH)₂ line with d = 0.490 nm is higher than that d = 0.262 nm (Fig.2).

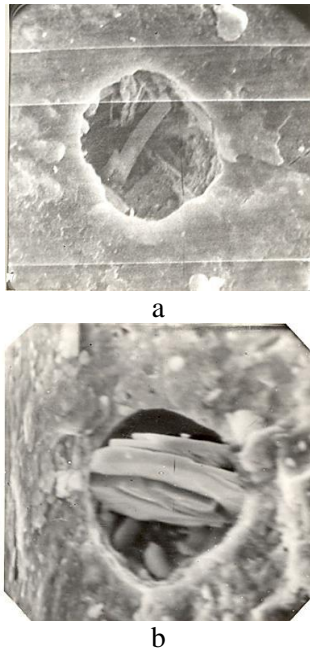
With further hydration of cement CEM 1 42.5 H CC with a complex additive, the line intensity d = 0.262 nm increases, and after 28 days of hardening, the intensity of these lines is equalized on X-ray patterns. This shows that in CEM 1 42.5 H CC cement with a complex agent, there are formed smaller and predominantly oriented secondary Portlandite crystals (Table 4, Fig.4).

Table 4 Binder type effect on the Portlandite amount in the cement stone

Binder type	Ca(OH) ₂ amount, % , at the age, days			
	3	7	28	90
CEM 1 42.5 H CC without agents	12.7	15	23.6	25.9
CEM 1 42.5 H CC with a complex agent	13.1	13.4	17.3	16.03

According to studies [21], the analysis of line intensities makes it possible to determine the structure of crystals of variable composition, where the matrix structure has different positions suitable for the placement of impurity elements. Hence, it can be assumed that changing the intensity of the Ca(OH)₂ lines is caused by the formation of C₂S₃H₂ as a result of isomorphism of individual (OH)⁺ tetrahedra on the discrete [SiO₂]⁺, [Fe₂O₃]⁰ inside the Portlandite base. The

studies have shown [22-29] that the genesis of hydrate formation is the result of physical-and-chemical processes accompanied by penetration of water into particles, internal, topochemical hydration. This process leads to the gradual filling of the free space between them, which contributes to the dense coalescence of hydrates and favorably acts on concrete strength.



Forming laminated packages of Portlandite,
X 2000

Fig.4 Microphotos of cement stone CEM 1 42.5 H CC with a complex agent at the age of hardening 7 days (a) and 28 days (b)

Thus, at the first stage, when the components of the concrete mixture are mixed with water, a supersaturated solution is formed. In this solution there are ions of sulfate, hydroxide and alkali, as well as a small amount of silica, alumina and iron. A high concentration of calcium ions and sulfate ions is observed within a short time after mixing the concrete mixture, since within a few minutes the first newgrowths begin to precipitate from the solution: calcium hydroxide and ettringite.

After about an hour, the second stage of hydration begins that is characterized by the formation of very small calcium hydrosilicates. In this case, newgrowths are primarily formed on the surface of cement grains. With increasing the number of newgrowths and their packing density, the boundary layer becomes impermeable to water within about 2 to 5 hours. This stage is called the “latent, or induction period”, as a result, the process of cement hydration is slowed down.

The studies have shown that the third stage of the hydration process occurs after 5 hours 30

minutes. At the same time, the temperature of concrete rises to 39.3-40.8 °C. Increasing the concrete temperature indicates the onset of crystallization of calcium hydroxide from the solution. This process is very intense. Since at this stage the number of hydrated phases is relatively small, there takes place free growth of ettringite in the form of long fibers, small Portlandite blocks and calcium hydrosilicates in the space between the cement particles.

4. CONCLUSION

It was found that the considered chemical additives, changing the rate of hydration and time to reach the maximum temperature, determine the periods of formation of hydrate phases. It was found that complex additives significantly increase the degree of hydration of cement. The results obtained allow considering the effects of complex additives as a factor of directional impact on the processes of structural formation and hardening of heavy concrete.

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