Heavyweight Concrete Based on Hydromechanochemically Activated Binder

Sergey Pimenov

1Kazan State University of Architecture and Engineering, Kazan, Russia
E-mail: SergeyPimenov12@yandex.ru

Abstract. The results of the influence hydromechanochemical activation of the cement with superplasticizing admixtures, both individually and jointly, on the structure formation of cement stone, the physical and technical properties of heavyweight concrete. An analysis of the microstructure and chemical elements in the spectrum of the surface of a cement stone prepared with preliminary hydromechanochemical activation of the binder with superplasticizing admixtures is presented. The method of hydromechanochemical activation of cement with a superplasticizing admixtures can be recommended for the production of quick-hardening concrete and mortars. The production of quick-hardening concrete is especially important: monolithic concreting in winter, when it is necessary to gain critical strength in concrete in a short time; precast concrete production at precast concrete plants, where it is possible to reduce energy costs for steaming products; modern additive printing technologies using a 3D building printer, requiring the development of new effective quick-hardening mortars and concrete.

Key words: heavyweight concrete, hydromechanochemical activation of the cement, superplasticizing admixture, chemical base, modifiers, hardening accelerator, structure formation.

1 Introduction
The structure formation kinetics of cement stone and, as a result, the kinetics of hardening of cement compositions is determined by many factors, including characteristics of binders, conditions of hardened stage, influence of modifying supplements. The phase-mineralogical composition, material composition, fineness of cement milling and its surface activity are refers to the main characteristics of cement, which affect the kinetics of curing [1-4].

All listed characteristics are determine the degree of hydration of the clinker phases and microstructure of hardened paste in various ways, which also define its physical properties, including strength [5-8].

In addition to increasing the activity of the binder, which affects the strength and physical and technical properties of mortar of cement and concretes, the degree of homogeneity of the micro- and macrostructure of the composite also greatly affects this. Distribution of water and surfactant molecules in a cement-water system within the flocculus and between them is not uniform, that this doesn’t ensure uniform flow of hydration processes and structure formation of cement stone in the volume of the system. One of the solutions to this problem is the use of a method of mechanically activating a binder, including cement, with surfactants in an aqueous medium [9, 10]. Such a method also can be called hydromechanochemical activation (HMCA) of cement with surfactants. The method allows to obtain the required activity of cement with reduced energy consumption compared with
mechanical activation in a dry environment, as well as a more uniform cement suspended load before preparing the concrete mix [10].

Effective surfactants include the superplasticizer [11, 12]. Superplasticizer that are currently on the market can be classified according to two characteristics: the composition of materials and the main effect in the mechanism of action on cement systems. The classification according to the second criterion seems more convincing, because as a result of the appearance of various new materials with the properties of superplasticizer, it becomes difficult to group them depending on the composition [13].

Numerous studies have been carrying out to study the mechanism of action of superplasticizer in cement systems [13-17].

Influencing the processes of structure formation, especially at the initial (coagulation) stage, superplasticizer change the rheological properties of the cement system, this helps to reduce its water demand, which subsequently affects the parameters of the crystallization structure.

According to academic circle [13-19], the mechanism of action of superplasticizer in cement systems is the physical adsorption of macromolecules on the active centers of the binder, leading to a decrease in the internal friction of the particles of the solid phase, as well as its dispersion. Subsequently, chemical interaction of the functional groups of the superplasticizer occurs with calcium hydroxide, as a result of the appearance and accumulation of calcium hydroxide in the system, which leads to the neutralization of molecules and their removal from the surface of cement grains.

It has been established that in the mechanism of action of superplasticizer of the types of naphthalene formaldehyde, melamine formaldehyde, lignosulfonates, the effect of electrostatic repulsion of cement particles and stabilization prevails, this is because the adsorption layers of the molecules of superplasticizer increase the value of the zeta-potential on the surface of cement particles. The value of the zeta potential depends on the adsorption capacity of superplasticizer (the higher the adsorption value, the greater the absolute value of this potential, which has a negative mark) [14].

In the mechanism of action of superplasticizer such as polycarboxylates, the role of the zeta potential is less, and the mutual repulsion of cement particles and stabilization of the suspension is ensured by the predominant steric effect. Many experts attribute this difference to the structure of various types of superplasticizer molecules: naphthalene formaldehydes, melamine formaldehydes, lignosulfonates are characterized by a linear polymer chain; superplasticizer based on polycarboxylate esters are characterized by cross-links and a two- or three-dimensional form [14].

The promising way to accelerate hardening and improve the quality of concrete is a method based on the integrated effect of hydromechanochemical activation of cement together with a superplasticizer [20].

Thus, the use of superplasticizer to reduce the total and mainly capillary porosity of concrete, to more fully use the energy of Portland cement and the use of hydromechanochemical activation of cement are prerequisites for the synthesis of early strength and durability of heavyweight concrete.

This academic work presents the results of the influence of superplasticizing additives, superplasticizing additives with an additive-accelerator hardening and hydromechanochemical activation of cement on the structural formation of cement stone and the physical and technical properties of heavyweight concrete.

2 Materials and Methods
Portland cement was used as a binder with slag CEM II/A-Sh 32,5N grade, which manufactured by JSC «Ulyanovskcement» Ulyanovsk plant and was in compliance with the GOST 31108-2016. The choice of medium-grade Portland cement is due to its high efficiency in hydromechanochemical activation [14], so the use of granulated blast furnace slag as a mineral additive provides clinker savings and environmental safety, as well as its positive effect on the structure formation of cement stone after activation by interaction with calcium hydroxide with the formation of predominantly strong low-basic calcium hydrosilicates [21-23].
The chemical and mineralogical composition of Portland cement is show in table 1 and table 2.

**Table 1.** The chemical composition of Portland cement.

| Type of Portland cement | The chemical composition, % |
|-------------------------|-----------------------------|
|                         | SiO<sub>2</sub> | Al<sub>2</sub>O<sub>3</sub> | Fe<sub>2</sub>O<sub>3</sub> | CaO   | MgO   | SO<sub>3</sub> | Alkalis | CaO<sub>eq</sub> |
| Ulyanovsk               | 22,1            | 5,0                | 9,0              | 64,0  | 0,92  | 0,94        | 1,01    | 0,27            |

**Table 2.** The mineralogical composition of Portland cement.

| Type of Portland cement | The content of main minerals: in % | The content of additives: in % |
|-------------------------|-----------------------------------|--------------------------------|
|                         | C<sub>3</sub>S | C<sub>2</sub>S | C<sub>3</sub>A | C<sub>4</sub>AF | Granulated blast furnace slag | SO<sub>3</sub> |
| Ulyanovsk               | 54             | 20            | 11             | 12           | 9,2           | 2,8          |

Sand of the Kamsko-Ustiynskiy deposit with the fineness modulus of 2.7 was used as a fine filler and was in compliance with the GOST 8736-2014 with a true density of 2.6 g/cm<sup>3</sup>, bulk density of 1.550 g/cm<sup>3</sup>, porosity V=(1- 1,653/2,6)·100 = 36,42 %.

Granite rubble of the Ural deposit with the fraction sized 5-20 mm was used as a fine filler and was in compliance with the GOST 8267-93 with a true density of 2.75 g/cm<sup>3</sup>, bulk density of 1.40 g/cm<sup>3</sup>, porosity – 40.8 %.

Naphthalene formaldehyde superplasticizer was used as a Relamix T-2 admixture manufactured by the Russian company LLC «Polyplast» according to TU 5870-002-14153664-04 in the amount of 1 % by the mass of cement [24, 25].

Figure 1 shows the structure of the molecules of the C-3 admixture - an analogue of the Relamix T-2 admixture.

![Figure 1. Molecule structure of sulfonaphthalene formaldehyde (C-3 admixture) [14].](image)

Remicrete SP60 admixture based on polycarboxylate ester was used as the second studied superplasticizer which manufactured by the German company «Schomburg», it has certification number: 0764-CPD-0012 (according to EN 934-2:2001) in the amount of 1% by the mass of cement. Figure 2 shows the molecule structure of superplasticizer based on polycarboxylate ester.
Figure 2. Molecule structure of polycarboxylate ester [14].

As an additional additive - hardening accelerator was used sodium sulfate (SS) in the form of an anhydrous salt of white crystals with a yellow tint according to GOST 6318-77. The optimal dosage is 1-2% by the mass of cement in the composition of the concrete mixture. In this work, SS was used in an amount of 1% by the mass of cement.

Industrial water was used for the preparation of cement paste, concrete mixes, as well as for washing aggregates, that meets the requirements of GOST 23732-2011 «Water for concrete and mortar. Technical conditions».

Heavyweight concrete of industrial composition class with the strength V25 was manufactured and tested with a ratio of components in the following proportions: cement:sand:granite rubble = 490:555:1315. The water-cement ratio (W/C) of the studied compositions was selected from the condition of achieving the same mobility of mark P2 (O.K.=6-8 cm according to GOST 10181-2014). The choice of this mobility of the concrete mixture was made in accordance with the requirements for the production of various concrete and reinforced concrete products (floor slabs, beams, columns, lintels, etc.) according to the CR (the code of rules) 130.13330.2018 «Production of prefabricated reinforced concrete structures and products».

Cement was subjected to hydromechanochemical activation in a rotary pulsation equipment (RPE) manufactured by LLC «Promservice» according to TU 5132-001-70447062. The advantage of conducting studies with hydromechanochemical activation of cement in this equipment is the availability of industrial samples of this apparatus.

The samples were made of the prepared concrete mixtures - cubes in size 10×10×10 cm, which at the age of 1, 3, and 28 days of normal hardening, the specimens were subjected to mechanical tests.

The average density of concrete was determined in accordance with GOST 12730.1-78 «Concretes. Density determination methods».

The strength of the samples was determined in accordance with GOST 18105-2018 «Concretes. Rules of control and strength assessment».

Optical research of the cement stone samples were carried out using an electron microscope which equipped with an AZtec X-MAX energy dispersion spectrometer. The resolution of the spectrometer is 127 eV.

The cement stone was preliminarily sprayed with Au/Pd alloy in a ratio of 80/20 on a high-vacuum Quorum T150 ES installation and then we was shot of its surface at an accelerating voltage of 5 kV. Elemental analysis was carried out at an accelerating voltage of 20 kV and a working length of 9 mm, the probe depth was less than 1 micron.
### Results and discussion

An earlier analysis of the results of the research work of the effect of hydromechanochemical activation of cement on the technological properties of concrete mix and the hardening kinetics of heavyweight concrete showed distinctive features when using two superplasticizers admixture of different chemical bases:

- the use of the Relamix T-2 admixture by naphthalene formaldehyde base in an amount of 1% by the mass of cement during HMCA of cement doesn’t contribute to the increased mobility of the concrete and allows you to get heavyweight concretes of industrial composition class with the strength V25 with a tensile compressive strength of 17.4-23.5 MPa in 1 day hardening on Portland cement with slag CEM II/A-Sh 32,5N, and, I guess, it can be recommended for precast and reinforced concrete production technology [24].

Analysis of the structure and elemental composition of the cement stone obtained by HMCA of cement with the Relamix T-2 admixture (figure 3, table 3) showed the presence in the spectrum of an increased concentration of calcium, silicon, and oxygen atoms in an amount whose mass content can be used to suggest the formation of calcium hydrosilicate minerals (CSH), forming a dense crystalline structure of cement stone.

![Figure 3. The fragment of the structure of a sample of cement stone obtained by HMCA with the Relamix T-2 admixture, magnification 20000x.](image)

| Element | Weight.% | Atom. % | Reference name |
|---------|-----------|---------|----------------|
| C       | 11.35     | 17.45   | C Vit          |
| O       | 56.81     | 65.58   | SiO₂           |
| Na      | 0.62      | 0.50    | Albite         |
| Mg      | 1.07      | 0.82    | MgO            |
| Al      | 1.45      | 0.99    | Al₂O₃          |
| Si      | 7.60      | 5.00    | SiO₂           |
| P       | 0.15      | 0.09    | GaP            |
| S       | 0.96      | 0.55    | FeS₂           |
| K       | 0.66      | 0.31    | KBr            |
| Ca      | 17.75     | 8.18    | Wollastonite   |
| Ti      | 0.17      | 0.07    | Ti             |
| Fe      | 1.40      | 0.46    | Fe             |
| Amount: | 100.00    |         |                |

- the application of the Remicrete SP60 admixture based on polycarboxylate esters in an amount of 1% by the mass of cement with HMCA cement slurry allows for a longer time to maintain the mobility of the concrete mix and helps to increase the compressive strength of heavyweight concrete with the
strength V25 production composition at a more smooth pace, with reaching at the age of 28 days the maximum compressive strength of 57.9-65.7 MPa on Portland cement with slag CEM II/A-Sh 32.5N, and, I guess, it can be recommended for monolithic construction technology [24].

Analysis of the microstructure and elemental composition (figure 4, table 4) showed the formation of the surfaces of the CSH in mainly rounded shapes, which during hydration forming a dense crystalline structure of cement stone.

![Figure 4. The fragment of the structure of a sample of cement stone obtained by HMCA with the Remicrete SP60 admixture, increase 5000X.](image)

**Table 4.** Elemental composition in spectrum No. 3 of a sample of cement stone obtained by HMCA of cement with the Remicrete SP60 admixture.

| Element | Weight.% | Atom. % | Reference name |
|---------|----------|---------|----------------|
| C       | 0.00     | 0.00    | C Vit          |
| O       | 46.60    | 66.47   | SiO₂           |
| Na      | 0.11     | 0.11    | Albite         |
| Mg      | 0.24     | 0.23    | MgO            |
| Al      | 1.16     | 0.98    | Al₂O₃          |
| Si      | 16.57    | 11.59   | SiO₂           |
| S       | 1.31     | 0.93    | FeS₂           |
| K       | 1.27     | 0.74    | KBr            |
| Ca      | 32.11    | 18.70   | Wollastonite   |
| Fe      | 0.63     | 0.26    | Fe             |
| **Amount:** | 100.00  |         |                |

According to the opinion [17], the complex use of two or more admixture of a plasticizing group of a different chemical base can give a positive effect in the form of a decrease in their consumption or an increase in the plasticizing effect.

The author [17] conducted research on the influence of the combined effect of two superplasticizers admixture of different chemical bases, also identified the economic feasibility of using several superplasticizers, determined the effectiveness of the admixture, however, research work and determining the effectiveness of additives in joint HMCA cement were not carried out.

In this context, it is of interest to study the joint effect of two superplasticizers of different chemical bases during hydromechanochemical activation of cement. The effectiveness of the additives was evaluated by the physical and technical properties of heavyweight concrete.

In this regard, the research was conducted on the effect of hydromechanochemical activation of cement in the presence of two superplasticizers – Relamix T-2 and Remicrete SP60 admixtures in an amount of 0.5% of each additive by the mass of cement.
In order to increase the rate of concrete strength gain more, the research was also conducted on the effect of hydromechanochemical activation of cement with the admixture of superplasticizers and hardening accelerator SS.

For this experiment, concrete mixtures were prepared with the following methods:
1 – mixing of the components of the concrete mixture in the concrete mixer for 5 minutes without the use of additives (reference);

2 – previously, Relamix T-2 and Remicrete SP60 admixtures were introduced and mixed into the mixing water in an amount of 0.5% of each additive by the mass of cement, and then mixed with aggregates and Portland cement in a concrete mixer for 5 minutes;

3 – previously, estimated amount of mixing water containing Relamix T-2 and Remicrete SP60 admixtures in an amount of 0.5% of each additive of the calculated cement mass, it was mixed with Portland cement in an amount of 50% of its calculated mass, then the resulting suspension was loaded into the PRE bunker and it was subjected to activation during 2 min, then the suspension was discharged into a concrete mixer and mixed with aggregates and the remainder of Portland cement for 5 minutes;

4 – previously, estimated amount of mixing water containing Relamix T-2 admixture in an amount of 0.5% and SS in an amount of 1% of the mass of Portland cement, it was mixed with Portland cement in an amount of 50% of its calculated mass, then the resulting suspension was loaded into the PRE bunker and it was subjected to activation during 2 min; then the suspension was discharged from PRE, Remicrete SP60 admixture was added to it in an amount of 0.5% of the mass of Portland cement; after that, the suspension was mixed in a concrete mixer with aggregates and the remainder of Portland cement for 5 minutes;

5 – previously, estimated amount of mixing water containing Relamix T-2 admixture in an amount of 1% and SS in an amount of 1% of the total mass of Portland cement, it was mixed with Portland cement in an amount of 50% of its calculated mass before receiving the more homogeneous suspension; then the resulting suspension was loaded into the PRE bunker and subjected to activation during 2 min; then the suspension was discharged into a concrete mixer and mixed with aggregates and the remainder of Portland cement for 5 minutes.

The results of the research are show in the table 5.

| No. composition | Admixture (content in% by weight of cement) | Duration of activation in PRE, min | W/C | Average density of concrete mix, кг/м³ | The compressive strength, MPa: at age: |
|-----------------|-------------------------------------------|----------------------------------|-----|----------------------------------------|--------------------------------------|
|                 |                                           |                                  |     |                                        | 1 day | 3 days | 28 days |
| 1               | -                                         | -                                | -   | 0,42                                   | 2435  | 9,02*  | 24,60*  | 45,80*  |
|                 |                                           |                                  |     |                                        | 100% | 100% | 100% |
| 2               | +                                         | +                                | -   | 0,30                                   | 2478  | 17,54  | 47,80  | 77,80  |
|                 |                                           |                                  |     |                                        | 194% | 194% | 170% |
| 3               | +                                         | +                                | -   | 0,30                                   | 2465  | 30,02  | 52,60  | 77,90  |
|                 |                                           |                                  |     |                                        | 333% | 214% | 170% |
| 4               | +                                         | +                                | +   | 0,30                                   | 2470  | 32,07  | 56,40  | 74,20  |
|                 |                                           |                                  |     |                                        | 356% | 229% | 162% |
| 5               | +                                         | +                                | +   | 0,30                                   | 2474  | 32,74  | 48,46  | 76,03  |
|                 |                                           |                                  |     |                                        | 363% | 197% | 166% |

Note: above the line is the average value of the indicator; below the line is the relative value of the indicator in% of the reference.
As can be seen from Table 5, the joint introduction of superplasticizing Relamix T-2 and Remicrete SP60 admixtures in a total amount of 1% of the mass of cement in composition of the concrete mix (composition 2) can reduce its water demand by 29%, increase the density. The compressive strength of hardening concrete rises during all hardening periods: at the age of one day - by 94%, at the age of three days – by 94%, at the age of 28 days – by 70% compared with the control composition.

Concretes have high compressive strengths at the age of one day and it is 30.02-32.74 MPa, which obtained by 3, 4 and 5 methods, including hydromechanochemical activation of cement together with a complex of additives. That is higher by 3.33-3.63 times of the compressive strength of the control composition sample. At the age of three days of hardening, the compressive strength of the compositions is 48.46-56.40 MPa, which is higher by 1.97-2.29 times than the compressive strength of the control composition. At the age of 28 days, the compositions of heavyweight concrete save a high rate of hardening and compressive strength is 74.20-77.90 MPa, which is higher than the compressive strength of the control composition by 62-70%.

The use of Relamix T-2 and Remicrete SP60 admixtures both individually and together, in the process of HMCA of the cement can significantly increase the compressive strength of heavyweight concrete, especially in the early stages of hardening. Taking into account the results of previous tests[24], in order to increase the compressive strength to a greater extent, it is more efficient to use one Relamix T-2 admixture at a young age than in combination with the Remicrete SP60 admixture. Taking into account the results of previous tests, in order to increase the compressive strength at a young age to greater extent, it is more efficient to use one superplasticizer - Relamix T-2 admixture, than in combination with the Remicrete SP60 admixture.

The research of the kinetics of heavyweight concrete hardening was of practical interest in the first 24 hours of hardening. For the research, we selected the compositions which obtained by HMCA of cement with Relamix T-2 admixture and HMCA of cement with Relamix T-2 admixture and hardening accelerator - SS, which had the greatest increase in strength on the first day of hardening according to the results of previous experiments. For the reference was selected composition without additives and activation.

Samples were made from concrete mixtures - cubes in size 10×10×10 cm, which were aged under normal humidity conditions and subjected to mechanical tests at the age of 16, 20 and 24 hours. The results of the research are show on the figure 5.

![Figure 5](image)

**Figure 5.** The hardening kinetics of heavyweight concrete on the first day. Compositions:
1 – reference; 2 – with HMCA of cement and RelamixT-2 admixture;
3 – with HMCA of cement and Relamix T-2 and SS admixtures.
The equations of approximation of the hardening kinetics of compositions 1-3 of heavyweight concrete of figure 5 are shown in Table 6.

Table 6. The equations and reliability of approximation. The hardening kinetics of heavyweight concrete on the first day.

| № composition in the figure 5 | The equation of approximation | The reliability of approximation |
|-------------------------------|-------------------------------|---------------------------------|
| 1                             | \( y = -0.0096x^2 + 0.5514x + 0.0083 \) | 0.9984                          |
| 2                             | \( y = -0.0276x^2 + 1.8194x - 0.0153 \) | 0.9996                          |
| 3                             | \( y = -0.0303x^2 + 1.9896x - 0.036 \) | 0.998                           |

As we can see from the data of figure 5, the curves of hardening kinetics of concrete which obtained by HMCA of cement (compositions № 2-3), they are differ from the curve of hardening kinetics of the control composition at a higher rate of curing.

There is a slight difference in compressive strengths in compositions № 2-3 during the first days of hardening: after 16 hours – the difference is 5%, after 24 hours – 7%, which may be an experimental error.

Taking into account these results and also the reduced compressive strength at the age of 28 days of a composition with SS (compositions No. 4 or No. 5, table 5), the increased corrosion of metal reinforcement in reinforced concrete products and the structures with the additive of SS, the use of SS in these compositions can be considered unjustified.

4 Conclusions

1. Analysis of the microstructure and elemental composition of cement stone obtained with using hydromechanochemical activation of cement and modification with a superplasticizer showed the formation of calcium hydrasilicate (CSH) minerals, which are mostly crystalline, which form a dense cement stone microstructure during hydration;

2. With the aim of increasing the early strength of heavyweight concrete by hydromechanochemical activation of cement it is more expedient to use the naphthalene formaldehyde base Relamix T-2 admixture, than the Remicrete SP60 admixture based on polycarboxylate esters. This conclusion made by analysis of the physical and technical properties of heavyweight concrete, and is possibly related to the structural features of the molecules of the studied additives. In the structure of the admixture based on polycarboxylate esters, there are longer side chains, which as a result of intensive mechanical activation, undergo destruction and deformation, which is reflected in a decrease in the efficient use of admixture together with intensive dynamic effects;

3. Due to the small difference in the tensile strengths for saturation of the compositions of heavyweight concrete obtained by hydromechanochemical activation of cement with a superplasticizer admixture and a superplasticizer + hardening accelerator, on the first day of hardening (up to 7%), it was decided for further studies don’t use a complex additive with a hardening accelerator, but to use one superplasticizer – Relamix T-2 admixture on a naphthalene formaldehyde basis;

4. The method of hydromechanochemical activation of cement with a superplasticizer admixture can be recommended for production of quick-hardening concrete, mortars. Production of quick-hardening concrete is especially relevant: monolithic concreting in winter, when it is necessary to gain critical strength in concrete in a short time; production of precast reinforced concrete in factories of reinforced concrete products, where there is an opportunity to reduce energy costs for steaming products; modern additive printing technologies using a 3D building printer, requiring the development of new effective quick-hardening mortars and concrete [26-31].
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