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Plastic shrinkage of concrete modified by metakaolin

K. Usanova*, Yu.G. Barabanshchikov, A.V. Krasova, S.V. Akimov, S.V. Belyaeva Peter the Great St. Petersburg Polytechnic University, St. Petersburg, Russia *E-mail: plml@mail.ru

Keywords: cement, concrete, heat release, heat of hydration, plastic shrinkage, early age shrinkage, autogenous shrinkage, superplasticizer, metakaolin, concrete mixtures, concrete additives, cracks

Abstract. The subject of research is concrete containing a polycarboxylate superplasticizer admixture and metakaolin. The effect of water-cement ratio, superplasticizer, and metakaolin on plastic shrinkage of cement paste was investigated. Two types of cement are also compared. One of them was alite-aluminate cement (cement A). Another one was belite-celite cement (cement B). If the water-cement ratio is increased, then the plastic shrinkage of samples with both cements develops more intensively and achieves higher values. However, the w/c ratio affects the shrinkage of the cement A more. For the first 8 hours, the cement A shrinkage is 1.71-5.21 mm/m at W/C = 0.2-0.35. For cement B, the corresponding values are 0.953-2.63 mm/m. The cement A's plastic shrinkage is 1.51-1.98 times higher than cement B's plastic shrinkage. If polycarboxylate superplasticizer content is increased, then the shrinkage of both cements increases significantly due to the release of part of physically bound water and its evaporation. The addition of the superplasticizer increases the shrinkage of the cement A by an average of 40 %, and the shrinkage of cement B by approximately twice. Simultaneously, the shrinkage of two cements becomes approximately equal due to the same rate of interaction of water with the cement, limited by the diffusion of water through the polycarboxylate adsorption film. Selective capacity of polycarboxylate concerning two types of cement is shown. The alite-aluminate cement gives the same shrinkage as the belite-celite cement, with a 1.89 times higher superplasticizer content. Replacing part of the cement by metakaolin reduces the initial shrinkage, however, not so significantly as the other two factors. If the metakaolin content in the binder is increased, then the shrinkage of both cements decreases. More noticeable is the effect of metakaolin on the cement A. The shrinkage of the cement B has not changed much.

1. Introduction

One of the main problems of concrete pouring is ensuring the crack resistance of concrete during its hardening. Uneven and restrained deformations cause cracks in hardening concrete. They develop due to shrinkage and heat release of concrete [1]. A specific area of concern is the early-age cracking of selfcompacting concrete, which is an extremely fluid mix with a high content of powdered additives and superplasticizers [2]. The plastic shrinkage reviewed in [3–5].

It is known that the self-compacting concrete requires a careful combination of the various components of the mixture. For this reason, much attention needs to be paid to the influence of additives and powdered fillers on the early shrinkage of concrete [6-10]. Results [11] show that shrinkage depends on the mineral admixture type and more particularly on the developed microstructure. Replacing 15% of Portland cement by metakaolin increases the plastic state's shrinkage and decreases it significantly at the hardened state in free drying conditions. A study [12] showed that concrete mixtures blended with mineral admixtures (silica fume, microsilica or fly ash) also had more plastic cracks but below average crack compared to plain Portland cement.

An important role in early shrinkage was the water-cement ratio. According to [13], a low watercement ratio could cause large shrinkage. The experimental results showed that an increasing the watercement ratio in the range from 0.13 to 0.24 reduced the chemical and autogenous shrinkage after 8-10

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hours of hardening. With further hardening in air, starting from 42 hours from mixing and up to 28 days, shrinkage, on the contrary, increased at an increase in W/C. Moreover, air shrinkage was less than plastic shrinkage. Minimizing the autogenous shrinkage of concrete was an important task. The way to solve this was the use of low-heat belite types of cement and shrink-resistant additives [14–16].

The use of mineral fillers to reduce creep and shrinkage was well known [17–23]. According to [24], fly ash could significantly reduce early age autogenic shrinkage, while silica fume caused an increase in the autogenic shrinkage. The effect of mineral fillers was related to their quantity and fineness of grinding. If the specific surface area of fly ash was above 4000 cm²/g, then the autogenous shrinkage increased at an increasing amount of additive. A mixture of fly ash and slag significantly reduced the autogenous shrinkage and concrete creep [25]. If more than 20% of the cement was replaced by fly ash, then the shrinkage decrease was not so significant. The influence of the slag on early age shrinkage, on the contrary, was more noticeable when replacing cement above 20%. A mixture of fly ash and slag had lower shrinkage than only ash had [26].

The study [27] showed that limestone filler with a large specific surface area increased autogenous shrinkage, reduced the rate of water evaporation, and increased plastic cracking of self-compacting concrete.

On the other hand, fillers reduced concrete bleeding, which [27, 28] significantly reduced cracking, as shown in [27]. The addition of 10 % of water reduced the cracks' length, and a decrease in the amount of superplasticizer reduced the cracking width of the concrete mixture.

The study of various fillers used as partially replacing cement showed [29] that the shrinkage depends on the type of mineral additive and, in particular, on the fineness of grinding. Replacing 15% of Portland cement by metakaolin increased the plastic shrinkage and significantly reduced it in the hardened concrete if air drying was used [30]. The fillers from waste marble and ceramic tiles increased the resistance to cracking of early age concrete, tested at temperatures of 25 and 40 °C, and the presence and absence of wind (ventilation) [31].

In addition to mineral additives, superplasticizers have a great effect on the concrete mixture. They can significantly improve the quality and characteristics of the mixture; regulate the concrete setting and its hardening [32–35].

In [36], two kinds of naphthalene compounded superplasticizers were tested. One was a retarder for concrete, and the other one was an early strength-air entraining-retarding superplasticizer. The use of this mixture had a positive effect not only on strength but also on the shrinkage of cement paste, mortar, and concrete. In [37], the effect of naphthalene based plasticizers, polycarboxylate superplasticizer, a retarding component, and shrinkage-reducing admixtures on concrete creep was studied. The results showed that polycarboxylates can significantly reduce the shrinkage and the creep of concrete compared to the naphthalene based plasticizers. The shrinkage-reducing admixtures also reduced the concrete creep. However, in [38], a mixture modified with a superplasticizer suddenly cracked, and the air-entraining admixture significantly reduced shrinkage.

As the literature review shows, the results of various studies can be contradictory. Therefore, a separate study is required to evaluate specific cases.

The subject of research is concrete containing a polycarboxylate superplasticizer admixture and metakaolin.

The objective of the work is a comparison of concretes from two cements with different capacity of reaction. The plastic shrinkage is analyzed. The water-cement ratio, the polycarboxylate superplasticizer content, and the metakaolin content are varied.

2. Materials and Methods

2.1. Testing laboratory

Concrete mixtures were tested in the laboratory of Peter the Great St. Petersburg Polytechnic University (Russian Federation).

2.2. Materials

The mixtures were tested using two types of cement produced by Holsim (Rus) LLC.

- 1. Cement A: Portland cement without mineral additives based on clinker of normalized composition.
- 2. Cement *B*: class G oil well Portland cement without additives. It is high-sulfate-resistant cement. API Specification 10A 24 Edition: API Well Cement Class G at Grade HSR.

The physical and mechanical properties of two types of cement are presented in Table 1.

Table 1. Physical and mechanical properties of two types of cement.

Properties	Cement A	Cement B		
Blaine fineness [cm²/g]	3488	3027		
Normal consistency [%]	27.0	26.5		
Initial setting time [min]	177	212		
Final setting time [min]	245	466		
Soundness Test on Cement	sound cement	sound cement		
Compressive strength [MPa]				
at the age:				
3 days	44.9	32.2		
28 days	61.2	50.8		
Flexural strength [MPa]				
at the age:				
3 days	5.7	3.5		
28 days	7.4	5.9		

Chemical composition and mineralogical composition of the cements are presented in Table 2.

Table 2. Chemical composition and mineralogical composition of the cements.

Tune of coment	Chemical composition [%]			Mineralogical composition [%]									
Type of cement	SiO ₂	Al_2O_3	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na₂O	C ₃ S	C_2S	C ₃ A	C ₄ AF	CaO _{free}
Cement A	20.7	4.6	3.5	65.0	1.2	2.9	8.0	0.2	73.0	8.1	5.2	12.0	0.5
Cement B	20.9	4.1	4.9	64.4	1.1	2.5	0.5	0.2	65.0	12.5	2.6	15.7	0.3

The concrete mixes contained additives produced by MC Bauhemi LLC: MC-PowerFlow PF 7951 polycarboxylate superplasticizer and powdered metakaolin produced under the trade name Centrilit NC.

These cements were tested for heat release in concrete.

The concrete composition is shown in Table 3.

Table 3. Concrete composition.

Ingredient	Content [kg/m³]			
Cement	365			
Sand with fineness modulus of 1.6	810			
Crushed stone with a fraction of 5-20 mm	1040			
Water	181			
Additive Centrilit NC	10.95			
superplasticizer MC-PowerFlow PF 7951	1.64			
Total	2409			

2.3. Plastic shrinkage measurement

Cement paste samples in rectangular plastic containers with dimensions $90\times50\times25$ mm were made to determine plastic shrinkage. The upper edge of the samples was in contact with the room air. The relative air humidity was 30–32 % and a temperature of 22–24 °C. Water evaporated through this upper edge of the samples during the experiment. The other faces were in contact with the walls of the containers.

The MIR-12 microscope measured the plastic shrinkage by marks applied to the sample's exposed surface (see Fig. 1). The gauge length was 50 mm. Measurements started 10 minutes after mixing of the cement and ended after 24 hours.



Figure 1. Measuring microscope.

2.4. Heat release of concrete measurement

The heat release of concrete was determined by the semi-adiabatic method by EN 196-9:2010 Methods of testing cement. Part 9: Heat of hydration. Semi adiabatic method. The initial temperature was 20 $^{\circ}$ C. The sample was placed in a glass Dewar flask (thermos). It had a high thermal resistance due to the silvering flask surfaces and a vacuum between two glass layers. However, heat exchange between the sample and the surroundings was not completely excluded. Part of the heat generated by cement Q_{I} was consumed to increase the sample's temperature, and the other one Q_{2} was scattered into the environment with a constant temperature.

Total heat release Q generated by cement during the hydration is composed of two parts:

$$Q = Q_{1+} Q_{2.}$$

The heat Q_1 is proportional to the increase in the temperature of the sample t_x relative to the initial temperature of the sample t_0 with a proportionality coefficient equal to the specific heat capacity C_t [J/°C] of all heated parts of the calorimeter:

$$Q_{1=} C_t(t_x-t_0).$$

The specific heat capacity C_t of all heated parts of the calorimeter was determined experimentally for every calorimeter.

The heat Q_2 is the heat loss to the surroundings, and this heat loss determined by the heat conductivity coefficient α . The heat conductivity coefficient α is the amount of heat loss from the body in 1 c of time from 1 m² surface area with a temperature difference between the body's surface and the surroundings of 1 °C. Since the thermos' contact area with the surrounding S is constant value for this thermos, the heat conductivity constant of the calorimeter $Q_c = \alpha S$ (W/°C) was used. The Qc value was determined experimentally.

The heat Q_2 was determined by the formula:

$$Q_2 = Q_c \int_0^{\tau} (t_x - t_n) d\tau,$$

where τ is time and $t_n = const$ is environment temperature.

The integration over time was replaced by summing over the area F_x . These areas were enclosed between the curve $t_x = f(\tau)$ and the line $t_n = const$. Hence

$$Q_2 = Q_k F_{x}$$
.

The heat Q was the amount of heat generated by unit volume of concrete during its hardening. The value of the heat Q depended on the cement content C in concrete. Therefore, the results of the experimental measurement of heat release are presented further in the article in the form of heat release per unit mass of cement:

$$q=Q/C$$
.

The heat release of the same concrete mixture tested in different thermoses was not the same. The reason for this is the different Q_k values for different thermoses and, accordingly, the samples' measured temperatures.

The experimental results were recalculated for the isothermal hardening mode at a temperature of 20 °C

$$\frac{v_1}{v_2} = \frac{\tau_2}{\tau_1} = f_t = const$$

for the correct comparison of different cements.

The characteristical temperature difference ε was used further in the article to present the experimental data. The characteristical temperature difference ε is related to the temperature function by the following formula:

$$f_t = 2\frac{t_1 - t_2}{\varepsilon},$$

where f_t is temperature function. If t_1 - t_2 = ε , then f_t =2; this means if the temperature rises by ε degrees, the rate of heat release will double.

The hypothesis on the ratio of the heat release rate was used to determine the temperature function f_t . According to the hypothesis, the heat release rates $v=dQ/d\tau$ and corresponding terms τ_I and τ_2 remains constant at moments of equal heat release at $O_I=O_2$:

$$v_1v_2=\tau_2\tau_1=f_t=const.$$

The value of ε is obtained from experimental data on the heat release of concrete at three (not less) temperatures. It is established that the characteristic temperature difference ε is not constant, but depends on temperature.

This dependence is approximated by the linear function $\varepsilon = k_t + l$, where $k \approx 0.13$; $l \approx 8$ are empirical characteristics of the process.

2.5. Flexural strength and compressive strength measurement

The compressive strength of concrete was determined on cubes with a size of 70.7×70.7×70.7 mm according to Russian State Standard GOST 10180-2012 "Concretes. Methods for strength determination using reference specimens". The test results of the concrete specimens for the compressive strength at the age of 3 and 28 days are presented in Table 4.

The flexural strength was determined on beams of a square section with a size of $40\times40\times160$ mm according to Russian State Standard GOST 310.4-81 "Cements. Methods of bending and compression strength determination". The test results of the concrete specimens for the flexural strength at the age of 3 and 28 days are presented in Table 4.

3. Results and Discussion

The effects of the water-cement ratio, polycarboxylate superplasticizer MC-PowerFlow PF 7951, and metakaolin additive Centrilit NC on plastic shrinkage of cement paste were investigated. Two types of cements were compared. One of them was an alite-aluminate cement (cement *A*), and the other one was a belite-celite cement (cement *B*).

The manufacturing environment makes it possible to protect concrete from water evaporation only a few hours after concrete pouring when the concrete acquires some initial strength necessary for workers to walk on it. For this reason, the air-hardening of cement is used in the study.

The reaction capacity of cements is evaluated by the value of heat release per unit mass of cement.

Figure 2 show the results of determining the heat release per unit mass of cement q.

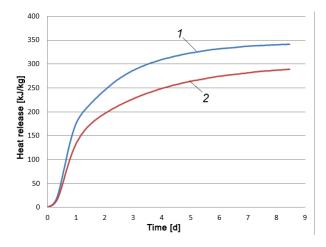


Figure 2. The specific heat release per unit mass of cement q: 1 is cement A, 2 is cement B.

The Figure 2 shows that the cement *A* generates a greater amount of heat by any period and has a higher heat release rate in the initial period of hardening than the cement *B*. This ratio of heat releases is consistent with cements' strength characteristics and with differences in their mineralogical composition. In contrast to cement *B*, the cement *A* has an increased amount of tricalcium aluminate C₃A and tricalcium silicate C₃S. Therefore, cement *A* has accelerated hydration and hardening. However, after about 5 days, the heat release rates of cements begin to change oppositely. The angle of arrival to curve 1 becomes slightly less than the angle of arrival to curve 2. The ratio of angles suggests that the difference in the strength of two cements in the later stages of hardening will not be so significant. This suggestion is partially confirmed by the ratio in cements' strength for the ages of 3 and 28 days (see Table 4).

Table 4. Ratio $R_{\it cA}/R_{\it cB}$ ultimate strengths of cement A and cement B.

Age of cement samples —	R_c	A/R_{cB}
Age of cement samples	Flexural strength	Compressive strength
3	1.63	1.39
28	1.25	1.20

As can be seen from Table 4, the ratio R_{cA}/R_{cB} decreases with age.

The results of determining the plastic shrinkage of cement *A* and cement *B* depending on the watercement ratio are presented in Figure 3.

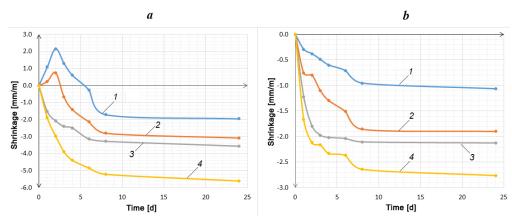


Figure 3. The plastic shrinkage of cement pastes A (a) and B (b) at water-cement ratio: 1 is 0.20; 2 is 0.25; 3 is 0.30; 4 is 0.35.

As shown in Figure 3, if the W/C ratio is increased, then the samples' shrinkage develops more intensively and reaches higher values. The plastic shrinkage ends after about 8 hours, which almost corresponds to the final set of cement. After that, shrinkage occurs in the set cement paste. For the first 8 hours, the cement A shrinkage is 1.71–5.21 mm/m, and for the next 16 hours, it is only 0.247–0.392 mm/m, depending on the W/C ratio. For the cement B, the corresponding values are 0.953–2.63 and 0.022–0.123 mm/m. Hence, cement A's plastic shrinkage is 1.51–1.98 times higher than cement B's plastic shrinkage. This plastic shrinkages rates are logical due to the difference in the

mineralogical composition of cements. Unlike cement B, the cement A is characterized by a 2 times higher content of C_3A and 12% higher content of C_3S .

In addition to high initial shrinkage, the difference between the cement A and the cement B is a significant expansion during the first three hours at W/C = 0.20-0.25. This difference in the shrinkages is also due to its chemical and mineralogical composition, which leads to the formation of an increased amount of hydrated calcium sulfoaluminate. The cement samples with a W/C = 0.30-0.35 have no expansion due to the high shrinkage that compensates for the expansion.

The results of determining the plastic shrinkage of cement A and cement B depending on the polycarboxylate superplasticizer content at a constant W/C = 0.25 are presented in Figure 4.

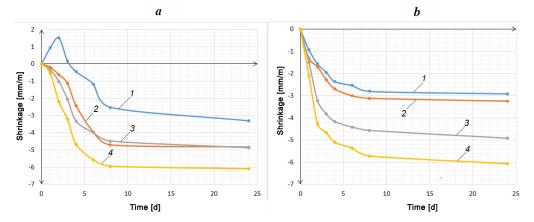


Figure 4. The plastic shrinkage of cement pastes A (a) and B (b) with the superplasticizer content as a percentage by mass of cement:

1 is 0.2; 2 is 0.4; 3 is 0.6; 4 is 0.8.

The addition of the superplasticizer MC-PowerFlow PF 7951 increases the shrinkage of the cement *A* by an average of 40%, and the shrinkage of the cement *B* by approximately twice, when compared to the cement paste without admixture. In this case, the order of the shrinkage values of the two cements is approximately equal. There is an assumption that equalizing the shrinkage of two cements is associated with two things. The first of these is the peptization of particles. The second one is the water-repellent effect of the screening adsorption layers of polycarboxylate on cement grains. Thus, the rate of interaction of water with cement is equally limited by water diffusion through the film. If the admixture content increases, then the shrinkage of cements also increases. This increasing due to the release of part of physically bound water. Water goes into a weakly bound or free state. This state allows it to evaporate more easily and allows solid particles to come close.

A normal consistency test was conducted to determine the effect of metakaolin additive Centrilit NC on the plastic shrinkage. Metakaolin was introduced instead of cement. The total amount of the solid component remained constant.

The results of determining the plastic shrinkage of cements depending on the metakaolin content are presented in Figure 5.

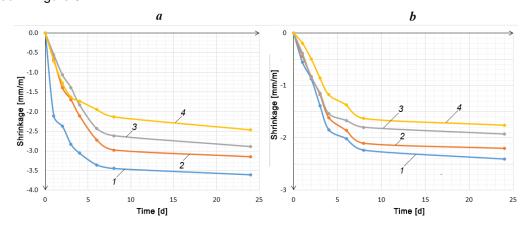


Figure 5. The plastic shrinkage of cement pastes A (a) and B (b) with the metakaolin content as a percentage by mass of binder (cement and metakaolin):

1 is 5; 2 is 10; 3 is 15; 4 is 20.

There is no expansion of cement A test in this case, as in previous experiments, possibly because of metakaolin's pozzolanic effect. Replacing part of the cement by metakaolin reduces the initial shrinkage, however, not so significantly. More noticeable is the effect of metakaolin on the cement A. The shrinkage of the cement B has not changed much. If the metakaolin content in the binder is increased, then the shrinkage of both cements decreases.

Figure 6 shows that metakaolin, replacing part of the cement, reduces plastic shrinkage the more, the higher its content. The shrinkage trend continues for both cements, but faster for cement *A*. Plastic shrinkage is reduced, starting from 10 % metakaolin content, compared to normal consistency mix without additives.

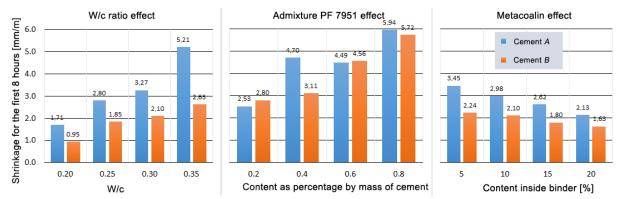


Figure 6 shows a histogram of shrinkage values for the first 8 hours for all testes.

Figure 6. Histograms of shrinkage values for the first 8 hours.

The histogram shows that two factors are essential for plastic shrinkage in air, which are the watercement ratio and the polycarboxylate superplasticizer content. Both factors affect the amount of free water in the cement paste. A significant part of the shrinkage is due to the water evaporation.

An analysis showing the superplasticizer's contribution to plastic shrinkage compared to the equivalent amount of evaporation water is performed below. Two dependences of plastic shrinkage ε are plotted on one graph. The first dependence is ε on the W/C ratio of cement without additive. The second one is the dependence of ε on the additive D content at W/C = 0.25. Since the values of W/C and D have the same numbers, it is convenient to use a single scale along the abscissa axis, using it both in one and in the other case.

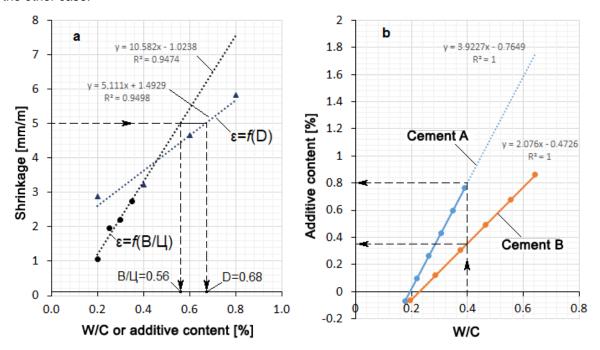


Figure 7. a. Linear approximation of shrinkage dependencies: ε in the function of W/C of mix without additive and ε in the function of superplasticizer content D at W/C=0.25 (cement B); b. correlation between W/C of normal consistency mix without additives and the superplasticizer content at W/C=0.25, causing the same plastic shrinkage.

Both dependences give a linear correlation with the coefficient of determination R^2 = 0.95 (Figure 7a). According to the graphs in Figure 7a, we find the values of W/C and D, giving the same plastic shrinkage. For example, for shrinkage, 5 mm/m, we find W/C=0.56 and D=0.68. That is, the initial shrinkage of the cement paste equal to 5 mm/m can be obtained in two ways: 1) preparing of the mix with W/C =0.56; 2) preparing the mix with W/C =0.25 and adding a superplasticizer in the amount of 0.68% of the cement mass. For any shrinkage value, a corresponding pair of W/C and D values can be found. Figure 7b shows the correlation between W/C and D for the two types of cement. This diagram gives an infinite series of W/C and D values associated with the same plastic shrinkage. So the shrinkage of the mix with W/C=0.4 can be obtained at W/C=0.25 using a superplasticizer. In the case of cement A, 0.8% of the superplasticizer by cement weight must be added, and in the case of cement B, 0.36% of the superplasticizer by cement weight must be added. At a linear correlation, the slopes or dD/d (W/C) ratio for cements A and B remains constant, equal to 3.923/2.076 = 1.89. This constant ratio means that cement A requires the addition of a superplasticizer (to compensate for the decrease in W/C) 1.89 times more than cement B, regardless of the value of the shrinkage deformation.

The selectivity of polycarboxylates for plasticizing cementitious compositions is well known. In this case, it manifested itself in relation to the initial shrinkage.

4. Conclusions

- 1. Based on the experimental research, the influence of three factors (water-cement ratio, superplasticizer MC-PowerFlow PF 7951 content, and content of metakaolin as a partial replacement of cement) on the initial shrinkage of Portland cement and high-sulfate-resistant cement is established.
- 2. The tested cements vary significantly in their activity, both in terms of strength (structure formation) and heat release (hydration of cement), which is determined by their chemical and mineralogical composition. According to the mineralogical composition, the first cement is the alite-aluminate cement, and the second one is the belite-celite cement.
- 3. If W/C ratio is increased, then the shrinkage of the samples develops more intensively and reaches higher values. The W/C ratio has a greater effect on shrinkage of cement A. For the first 8 hours, the cement A shrinkage is 1.71–5.21 mm/m at W/C = 0.2–0.35. For cement B, the corresponding values are 0.953–2.63 mm/m. The plastic shrinkage of cement A is 1.51–1.98 times higher than cement B's plastic shrinkage. In addition to high initial shrinkage, the cement A has a significant expansion of up to 2.2 mm/m during the first three hours at W/C = 0.20–0.25.
- 4. If superplasticizer PF 7951 content is increased, then the shrinkage of both cements increases significantly. This is due to the release of part of physically bound water and its evaporation. The addition of the superplasticizer MC-PowerFlow PF 7951 increases the shrinkage of the cement *A* by an average of 40%, and the shrinkage of the cement *B* by approximately twice. At the same time, the values of shrinkage of two cements become approximately equal. This is due to the same rate of interaction of water with the cement, limited by the diffusion of water through the polycarboxylate adsorption film.

Selective capacity of polycarboxylate in relation to two types of cement is shown. The alite-aluminate cement gives the same shrinkage as the belite-celite cement, with a 1.89 times higher superplasticizer content.

5. Replacing part of the cement by metakaolin reduces the initial shrinkage, however, not so significantly as the other two factors. If the metakaolin content in the binder is increased, then the shrinkage of both cements decreases. More noticeable is the effect of metakaolin on the cement *A*. The shrinkage of the cement *B* has not changed much.

Further research on this topic may be experimental studies of the influence of these factors on the initial shrinkage of cold-bonded fly ash aggregate concrete. Fly ash can be used as an additive in the mix [39] or as a large aggregate [40]. If the presoaked aggregate is added to the concrete mix, this will create "internal curing" for the concrete and reduce cracks caused by the heat release [41].

5. Acknowledgement

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Contacts:

Kseniia Usanova, plml@mail.ru Yuriy Barabanshchikov, ugb@mail.ru Anastasiya Krasova, krasova-nastya14@mail.ru Stanislav Akimov, akimov_sv@spbstu.ru Svetlana Belyaeva, belyaeva_sv@spbstu.ru

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