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Process of hydration and structure formation of the modified self-compacting concrete

Процессы гидратации и структурообразования модифицированного самоуплотняющегося бетона

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Ключевые слова: модификация; добавки; суперпластификатор; гидрофобизатор; метакаолин; самоуплотняющийся бетон; гидратация

Abstract. The article reviews the research results of influence of a complex modifying agent on rheological properties of cement-water paste and cement stone strength. The article describes the processes of hydration and structure formation of cement stone, the special aspects of the phase constitution of Portland cement hydration products in the process of modifying by complex admixture. The behavior of cement hydration in composition with the complex modifying agent have been shown by means of measuring the hydrogen-ion concentration, by sedimentation, contraction and heat emission of cement suspension. There is the decreasing of the degree of cement stone hydration because of blocking action of the superplasticizing admix and hydrophobisator during the initial stage. Studying the cement stone spalls with the aid of electron microscopy has showed that there are the crystallized hydrated newgrowths with smaller dispersive capacity in composition with the complex modifying agent than the ones without introduction of admixtures. The increasing of concentration of hydrated calcium sulfoaluminate in pores and capillaries, the increasing of the specific surface area of hydrated phases both in the general structure of cement stone and in structure with regions of imperfections, the voids content decreasing lead to the material hardening. The way of cement stone structure formation in composition with the complex modifying agent is found by means of differential thermal and X-ray phase analyses. This way is shown in the composition with a complex modifying agent manifested in blocking effect SP and HP, resulting in a reduced amount of portlandite and high content of the starting phase the cement clinker, wherein the MTK is reacted with calcium hydroxide, which helps to seal material. Reduction of ettringite in the composition with a complex modifying agent is associated with precipitation of superplasticizing admix molecules on C3A particles, which limits interaction with water.

Аннотация. В статье рассмотрены вопросы повышения эксплуатационных характеристик самоуплотняющегося бетона, путем модифицирования структуры цементного камня разработанным комплексным модификатором. Изучено влияние комплексного модификатора и его компонентов: гиперпластификатора (ГП), гидрофобизатора (ГФ), метакаолина (МтК) на реологию, тепловыделение, контракцию цементного теста, а также на предел прочности при сжатии, микроструктуру и фазовый состав цементного камня. Введение комплексного модификатора позволяет повысить прочность цементного камня на 75 % по сравнению с контрольным. Определение кинетики изменения показателя pH, седиментации, контракции и тепловыделения цементных суспензий выявлен характер гидратации цемента в составе с комплексным модификатором, проявляющийся в начальном замедлении степени гидратации цементного камня из-за блокирующего действия гиперпластификатора и гидрофобизатора. Изучение сколов цементного камня с помощью электронной микроскопии показало, что в составе с комплексным модификатором кристаллические новообразования формируются значительно меньшей дисперсности, чем в составе без добавки. Увеличение концентрации гидросульфоалюмината кальция в порах и капиллярах, увеличение удельной поверхности гидратных фаз, как в общей структуре цементного камня, так и в дефектных областях пространственного скелета, уменьшение пористости приводит к упрочнению материала. Методами дифференциально термического и рентгенофазового анализа установлен механизм

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структурообразования цементного камня в составе с комплексным модификатором, проявляющийся в блокирующем эффекте ГП и ГФ, что выражается в пониженном количестве портландита и большем содержании исходных фаз цементного клинкера, при этом МТК взаимодействует с гидроксидом кальция, что способствует уплотнению материала. Уменьшение эттрингита в составе с комплексным модификатором связано с осаждением молекул гиперпластификатора на частицы С3А, ограничивающем взаимодействие с водой.

Introduction

The problem of high-functional durable concrete production is topical to the present day. Such kind of concrete is usually used as underlay of buildings. Moreover, structuring the hardened cement paste, that has high density, low capillary porosity and consists of predominantly low-basic hydrated phases, is top priority [1–4]. One of the simplest and most effective methods of improving the cement composition properties is introduction of complex admixtures, which contains effective superplasticizing admixes, hydrophobic waterproofing agents and active mineral admixtures. Literature review and data analysis of these components have shown the following.

The effective superplasticizing admix is an important part of self-compacting concrete (SCC) [5]. This modifying agent makes it possible not only to increase the concrete consistency under the low water to cement proportion, but also to get high-performance, durable concrete with high density. Furthermore, most advanced superplasticizing admixes based on polycarboxylic ethers are the most effective [6-8, 13].

The increasing of freeze-thaw resistance, waterproofing capacity and exterior resistance can be achieved by introduction of water-repellent admixtures [9-11]. The silicone waters based on sodium and potassium siliconates are of primary concern. Moreover, consideration must be given to retardancy of cement hydration under the high hydrophobisator' dosages because of hydrophobic film occurring on the surface of reactants and impeding the process of hydration during the initial period [12].

With the purpose of optimizing the SCC's grain size composition and exclusion of water gain and concrete disintegration it is necessary to add fine-dispersed components to concrete composition. Ground meal, microsilicasuspension, boiler fly ash, rice husk ash, floured glass sand and metakaolin are used for this component. Scientists highlight metakaolin among the above listed [14-16], which has stable content and properties. Besides, metakaolin has a pozzolanic effect, as it reacts with $Ca(OH)_2$ during the late curing time and therewith increases the resistance to aggressive media and cement stone strength[17, 18].

The authors have developed a complex modifying agent for self-compacting concrete, comprising: superplasticizer – 1.5 %, hydrophobisator – 0.15 %, metakaolin – 5 % by weight of cement. Optimization of complex modifying agent components is shown in [19, 20].

Therefore the aim of the research is studying the influence of the complex modifying agent and its components on the special aspects of micropatterning and structuring phase composition of cement stone.

The effective and simple way of achievement of the polyfunctional effect and the full realization of a capability of all the components is introduction of complex admixtures. There are famous scientists S.S. Kaprielov, V.I. Kalashnikov, L.Ya. Kramar, A.V. Sheinfeld, B.Ja. Trofimov, H.-S. Kim, S.-H. Lee celebrated for scientific research of the investigation the influence of the complex admixtures, consist of plasticizing agents and active mineral admixtures (AMA), on process of hydration and structure formation of cement stone [2, 17, 18, 21, 22]. V.G. Batrakov's studies about the complexes with plasticizing agent and hydrophobisator are also well known [12]. But the complexes which contain a plasticizing agent based on polycarboxylic ether, an organosilicone hydrophobisator and metakaolin as AMA are underinvestigated. Consequently, the investigation of the influence of superplasticizing admixture, hydrophobisator and metakaolin in composition of complex modifying agent on the strength, micropatterning and structuring phase composition of cement stone is the site of special scientific interest.

The object of the research is studying the influence of the complex modifying agent and its components on the special aspects of micropatterning and structuring phase composition of cement stone.

In order to learn these aspects it is necessary to get around the following problems:

– to learn the influence of complex modifying agent and its components on setting up time of cement-water paste and on the strength of cement stone;

– to learn the influence of the complex modifying agent and its components on the processes of hydration by means of calorimetric measurements, contraction of cement-water paste, determination cement suspension hydrogen-ion concentration (pH-value);

– to learn the influence of the complex modifying agent on the microstructure of cement stone with the aid of electron microscopy;

– to learn the influence of the complex modifying agent on the phase composition of cement stone hydrated newgrowths by means of differential thermal analysis and X-ray phase analysis.

Materials and Methods of Research

The Portland cement CEM II/A-S 32.5 corresponding to Russian State Standard GOST 31108-2003 "Cements. Technical conditions" produced by Holsim (Rus) ("Volskcement" Open Joint Stock Company), and Portland cement CEM II/A-S 32.5 corresponding to Russian State Standard GOST 31108-2003 "Cements. Technical conditions", produced by Ulyanovskcement LLC (hereafter brands of Portland cement will be denoted by C1 and C2 correspondingly) were used as a cementitious matter. The C1 consists of the following main minerals: C_3S - 68 %, C_2S - 10 %, C_3A - 3.7 %, C4AF - 15 % and admixtures: gaize - 6 %, SO_3 - 2.2 %. The C2 consists of the following main minerals: C_3S - 57 %, C_2S - 17 %, C_3A - 7.5 %, C_4AF - 12.8 % and admixtures: gaize - 9.1 %, SO_3 - 2.36 %.

In the research the following admixtures are used as modifying agents: superplasticizer (SP) Remicrete SP 10 produced by SCHOMBURG GmbH company (Germany) (admixture is compatible with PN-EN 934-2:T3.1 и 3.2), organosilicone hydrophobisator (HP) "Tiprom S" produced by "Proizvodstvennoe obedinenie "SAZI" LLC (TS 2229-069-32478306-2003). Metakaolin (MtK) was used as active mineral admixture - noncrystalline aluminium silicate occurrence in ZHuravlinyj Log (TS 5729-095-51460677-2009).

The research of heat emission kinetics of cement-water paste was made by means of calorimetric measurements with the use of a measuring system Termokhron DS1921.

The research of contraction of cement-water paste was made by means of the measurement procedures MI 2486-98 and MI 2487-98 on the contraction measuring tester of cement activity "Cement-prognoz".

The hydrogen-ion concentration of cement-water paste liquidus was measured with the use of pH-meter pH-metra testo 206-pH1.

The kinetics of sedimentation of cement suspension (1:100) was experimented in spirit on the torsion balance sort of BT 500.

X-ray patterns were measured on the automatic X-ray diffractometer D2 Phaser (manufacturing company Bruker AXS GmbH). Processing the received interference spectrums was made with the use of the DIFFRAC.SUITE software package. The diffractive database named ICDD PDF-2 Release 2013 helped to make the phase identification with the use of the DIFFRAC.EVA-v3.1 program module. The statement of amount of the phases was made by Rietveld method with the use of DIFFRAC.TOPAS-v4.2 program module.

Microstructure of cement stone was determined by means of a high-resolution autoemissive electronic scanning microscope Merlin produced by CARL ZEISS Company. The spalls of cement stone samples were mist by composite metal Au/Pd in the ratio 80/20 on the high-vacuum unit named Quorum T150 ES.

Differential scanning calorimetry was studied by means of the instrument of synchronal thermoanalysis STA 443 F3 Jupiter (Netzsch, Germany). During the assay performance the sample's weight was 30-50 mg, the rate of temperature elevation was 10 °C/min, the temperature interval was from 30 °C to 1000 °C.

Results and Discussion

The results of the research of the influence of the complex modifying agent and its components on the water-reducing effectiveness, on the setting up time of cement-water paste and on the cement stone strength are shown in Table 1.

Table 1. The influence of the complex modifying agent and its components on the normal consistency, setting up time of cement-water paste

No.	Name of admixture	Admixtures content, %	Normal consistency of cement-water paste, %		Setting up time, min		Cement stone compressive strength (MPa)	
			C1	C2	beginning	ending	C1	C2
1	-	–	0.29 100 %	0.27 100 %	195 190	290 280	45.38 100 %	44.97 100 %
2	Remicrete SP 10	1,5	0.21 72.4 %	0.203 75.2 %	410 390	650 610	74.1 163 %	72.6 161 %
3	HP Tiprom S	0.15	0.284 98 %	0.266 98.5 %	620 630	830 810	45.83 101 %	46.3 103 %
4	MtK	5	0.324 112 %	0.292 108 %	180 190	320 330	56.83 125 %	55.16 123 %
5	Complex modifying agent	6.65	0.235 81 %	0.215 79.6 %	530 510	740 720	79.41 175 %	77.68 173 %

Note: For the setting up time there are results for C1 above the line and amounts for C2 under the line. The numerator is strength of compression in MPa, the denominator is fractional strength of compression in %.

As you can see in the table (Table1), water to cement proportion (WC) decreases by 1.5-27.6 % during the introduction of admixtures, moreover, water to cement proportion (WC) increases by 8-12 % during the introduction of metakaolin. The increasing of cement-water paste setting up time for all studied admixtures (except metakaolin), especially for the cement with superplasticizing admix, and HP can be observed.

Adding the complex modifying agent to the cement-water paste increases the cement stone compressive strength by 73-75 %, depending on the type of cement. Furthermore, during the introduction of superplasticizing admix and MtK severally cement stone compressive strength increases by 61-63 % and 25-23 % correspondingly, and the introduction of HP doesn't have influence on the increasing of compressive strength. The combined effect of introduction of all complex modifying agent components helps to increase compressive strength by 73-75 %, which shows the synergism of chemical admixtures.

Degree of hydration of cement stone was analyzed by measuring the hydrogen-ion concentration of cement-water paste liquidus, the heat emission, by the sedimentation of cement suspension (the Portland cement made at the Ulyanovskiy factory), by the contraction on both types of cement.

Here are the research results of measuring the hydrogen-ion concentration of cement-water paste liquidus (1:10) under the influence of the complex modifying agent and its components.

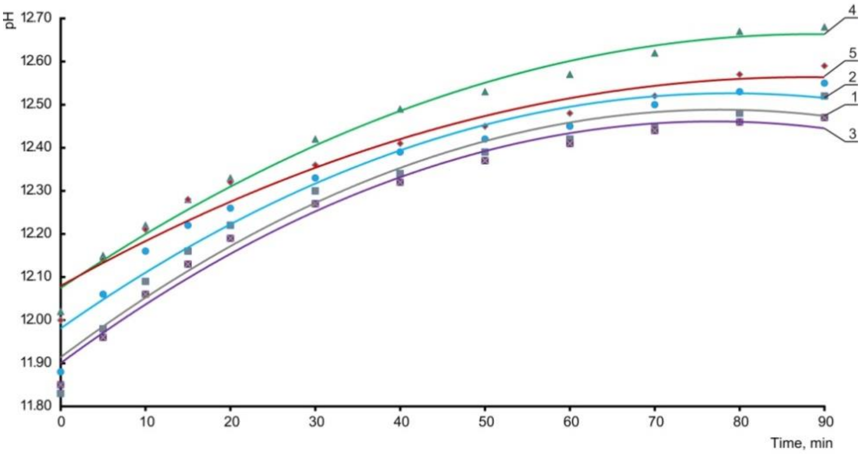

Figure 1. The influence of complex modifying agent and its components on changing the hydrogen-ion concentration of cement-water paste liquidus:
1 – with no admixtures; 2 – 1.5 % Remicrete SP 10; 3 – 0.15 % Tiprom S; 4 – 5 % MtK;
5 – 6.65 % complex modifying agent by weight of cement

In accordance with the findings in Figure 1, the hydrogen-ion concentration of separate components of complex modifying agent (superplasticizing admix and hydrophobisator) is less than the hydrogen-ion concentration of complex modifying agent. The decrease of hydrogen-ion concentration of superplasticizing admix and HP are connected with the blocking action of their molecules during the cement hydration. Furthermore, the hydrogen-ion concentration of cement with complex modifying agent is higher in comparison with compositions with mono-admixtures (superplasticizing admix and HP), which is probably connected with the superplasticizing admix and HP molecules adsorbtion by metakaolin and finally leads to the hydrogen-ion concentration increase in comparison with compositions with mono-admixtures (superplasticizing admix and HP). The hydrogen-ion concentration of complex modifying agent is more than 12.4, which is the condition of formation and accretion of the calcium hydroxide nucleating seeds and C-S-H phase.

The increasing of the hydrogen-ion concentration of cement suspensions is connected with intensive cement hydration. In view of this, there are the results of sedimentary test of cement suspensions in Figure 2. According to the data from Figure 2, the rate of cement sedimentation with admixtures is highly decreased, especially with Remicrete SP 10 modifying agent and the complex modifying agent, which is the result of the more intensive physical-chemical cement dispersion, which leads to strengthening of the degree of hydration. The superplasticizing admix mechanism of action is based on the quick molecules adsorption of the admix on the surface of cement. The molecules of admix are adsorbed on the surface of cement clinker, besides this, intensive cement dispersion in liquidus is due to the steric effect of the molecules action of superplasticizing admix. The intensity of dispersion in composition with complex modifying agent is less in comparison with composition with HP (by 17 %), which is also connected with local superplasticizing admix molecules adsorption on the particles of MtK.

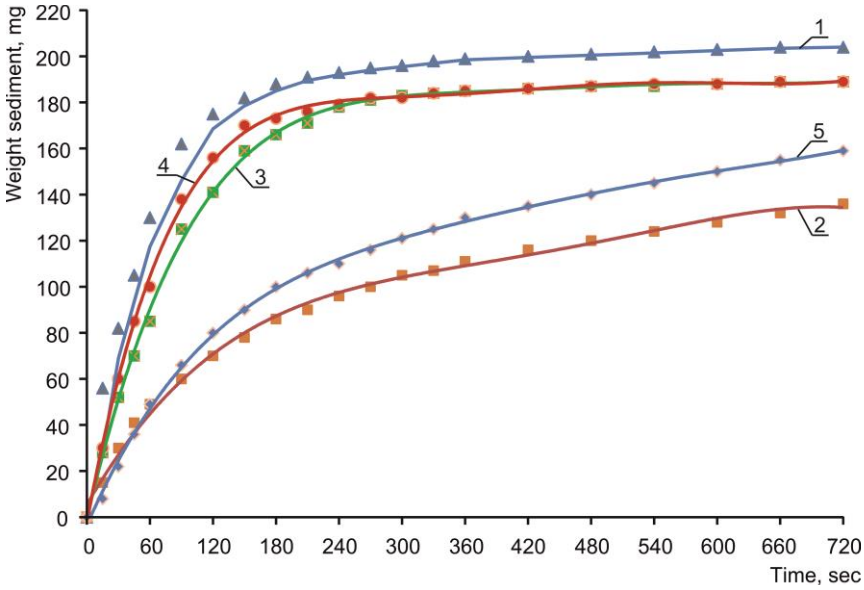

Figure 2. Kinetics of cement sedimentation with admixtures. Conventional signs are shown in the Figure 1

Intensive cement dispersion and increasing of the hydrogen-ion concentration of cement suspensions with complex modifying agent comes with changing the absolute volume of the hydrating cement-water paste. For this reason we have measured the contraction of cement-water paste (Figure 3).

As you can see in Figure 3, the highest rate of contraction of cement-water paste with MtK during the initial stage of hydration can be observed, which illustrates the increasing of degree of hydration with this admixture. There is the decreasing of the contraction rate in composition with complex modifying agent during the first hours of cement hardening due to the hydrophobisator's blocking action.

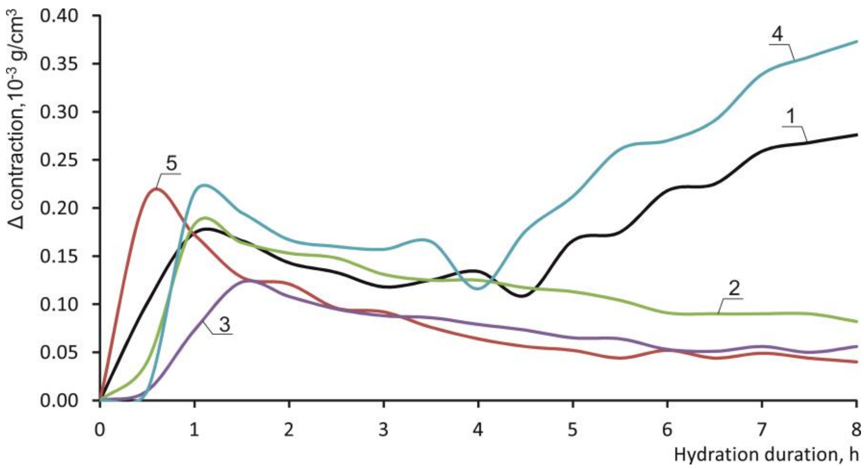

Figure 3. The rate of contraction of cement-water paste differential curve. Conventional signs are shown in the Figure 1

The contraction of cement-water paste closely connected with heat emission kinetics. In connection with this, the heat emission curves of cement-water paste with admixtures are shown in Figure 4.

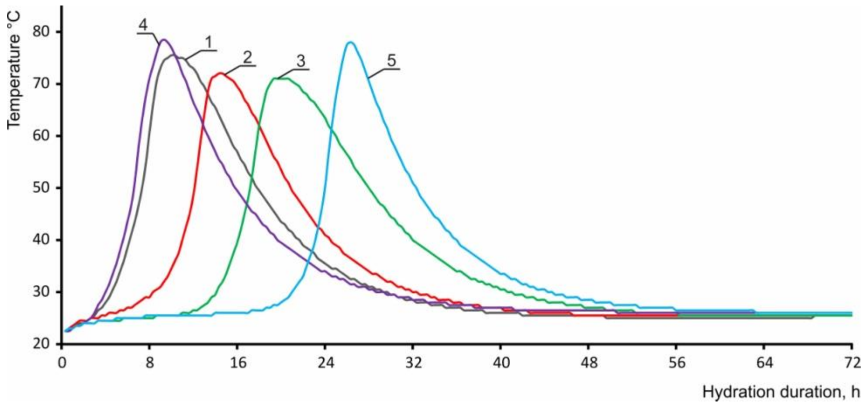

Figure 4. The heat emission during the hydration of Portland cement. Conventional signs are shown in the Figure 1

It can be seen from Figure 4 that the duration of induction process over superplasticizing admix, HP and the complex modifying agent is longer by 4, 8 and 6 hours in comparison with composition with no admixture correspondingly and shorter during the introduction of MtK. In accordance with the opinion of Batrakov V.G. [12] the slowing-down effect of hydration during the introduction of HP is explained, on the one hand, by the interaction of potassium alkylsiliconate and calcium hydroxide, accompanied by the release of hydrogen, which envelops cement particles and prevents the hydration, and on the other hand, by accumulation the admixtures interaction products and cement grains in the system.

The MtK admixture leads to an increase in the rate of heat emission of cement-water paste and the maximum temperature of hydration. HP and superplasticizing admix reduces the hydration, which becomes evident in the shifting the temperature peaks to the right by 8-12 hours. However, there is the reducing of hydration by 20 hours in composition with complex modifying agent, what is the result of hydrophobisator and superplasticizing admix synergistic effect. Nevertheless, the highest amount of waste heat in composition with complex modifying agent can be observed, that becomes evident in the increasing the degree of hydration of cement-water paste.

Studying the structure formation of cement stone with addition the complex modifying agent is a one of the top scientific problems.

There is an intricate structure of cement in Figure 5. There are needle crystals of ettringite in jellous mass of hydrated newgrowths in composition with complex admixture, which fill pore spaces in cement stone. According to the data from X-ray phase analysis, the hydrated newgrowths of ettringite are also produced in free volume in the sample without admix.

The increasing of hydrated calcium sulfoaluminate concentration in pore spaces, specific surface area of hydrated phases lead to the hardening of material both in the general structure of cement stone and in structure with regions of imperfections.

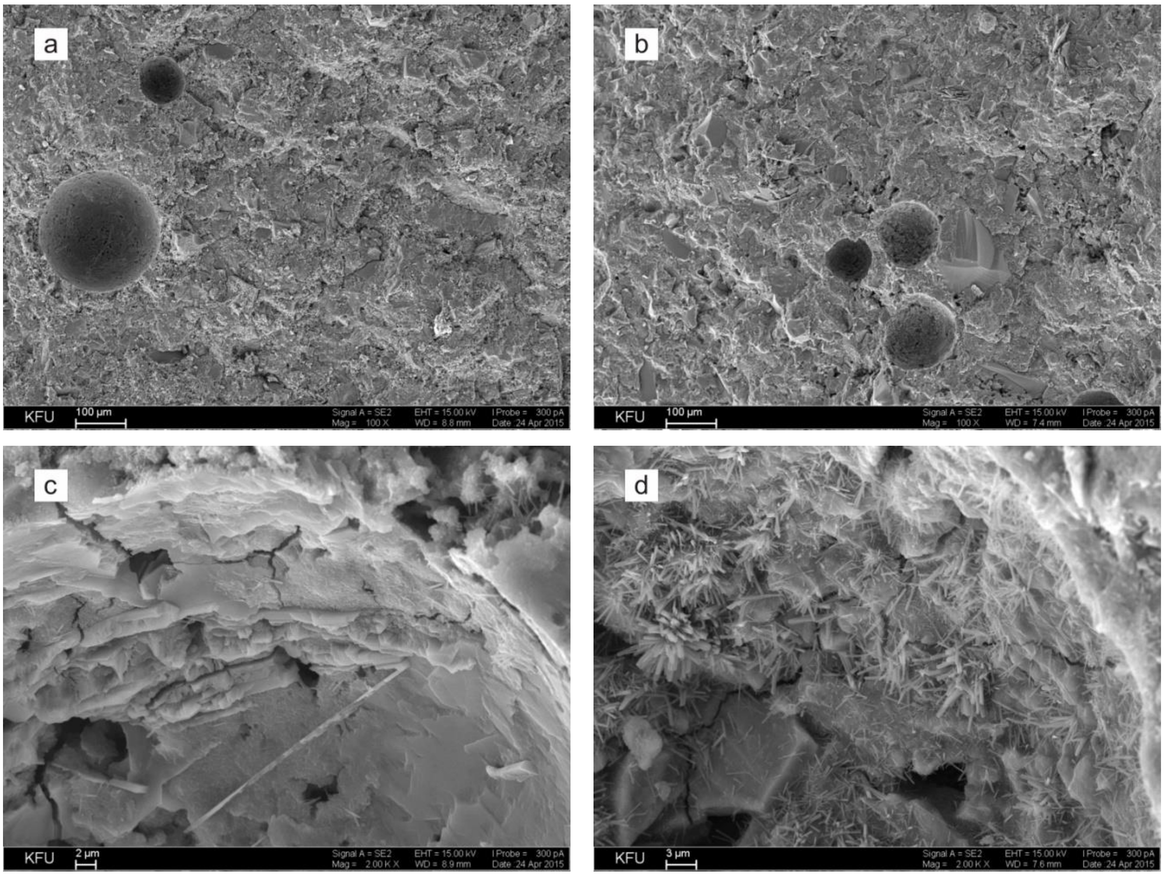

Figure 5. Electron microphotographs of samples of cement stone:
a, c – spall of the sample with no admixture;
b, d – spall of the sample with complex modifying agent.
Remarks: a, b – magnification 100^x
c, d – magnification 2000^x

The special aspect of influence of complex modifying agent is the fact that new crystallized formations have lower dispersion than formations in composition with no admixture. Moreover, the hydrated newgrowths crystallized in composition with complex modifying agent colmatage pores of cement stone, also hardening it.

Volume increasing and decreasing of the hydration products can be measured on the samples of cement stone hardening under normal conditions during 28 days by means of differential thermal (Figure 6) and X-ray phase analyses (Figure 7).

The first endoeffect at a temperature of 100–102 °C is connected with removal of gravitational moisture from pores and capillaries, and also with the fractional dehydration of ettringite. This effect is bigger in composition 1, what is confirmed by lower voids content in composition with complex modifying agent and by a high content of ettringite in composition 1, which is conformed to the data from X-ray phase analyses.

The second endoeffect at a temperature of 470–480 °C is connected with decompounding of $Ca(OH)_2$., and this effect is bigger in composition 1, which is also conformed to the data from X-ray phase analyses. Furthermore, there is the banding the free Portlandite with metakaolin and formation of calcium silicate hydrate (CSH) with lowered basicity in composition 2 [2, 17].

The third endoeffect is probably connected with decompounding of calcium carbonate. This effect is equal in both compositions, which is confirmed by the data from X-ray phase analyses, videlicet by the Rietveld method calculation (taking into account 20% of add phase); the effect is up by 8% in the sample with no admix in comparison with the sample with the complex modifying agent. Also endoeffect at a temperature 830–860 °C can be associated with dehydration of low-basic calcium hydrosilicates. At the same time, no endoeffect is observed in the DTA curves at a temperature of 830–860 °C.

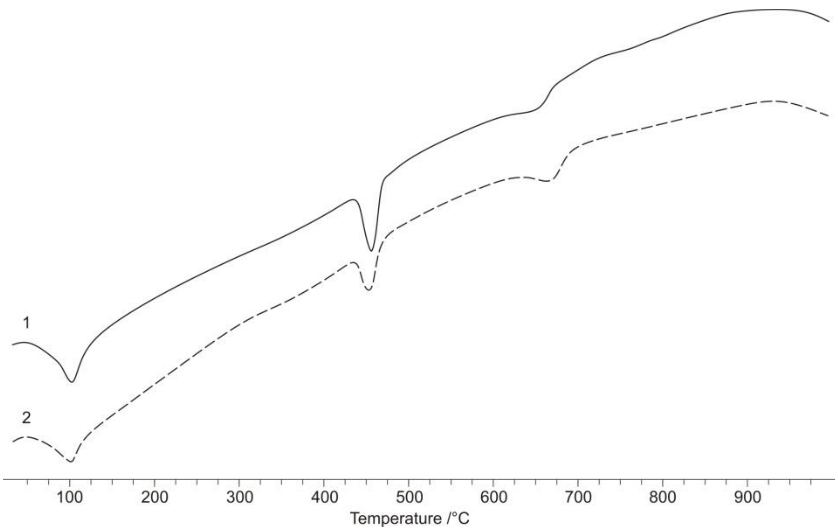

Figure 6. The curves of differential thermal analysis:
1 – the sample, 2 – the sample with complex modifying agent

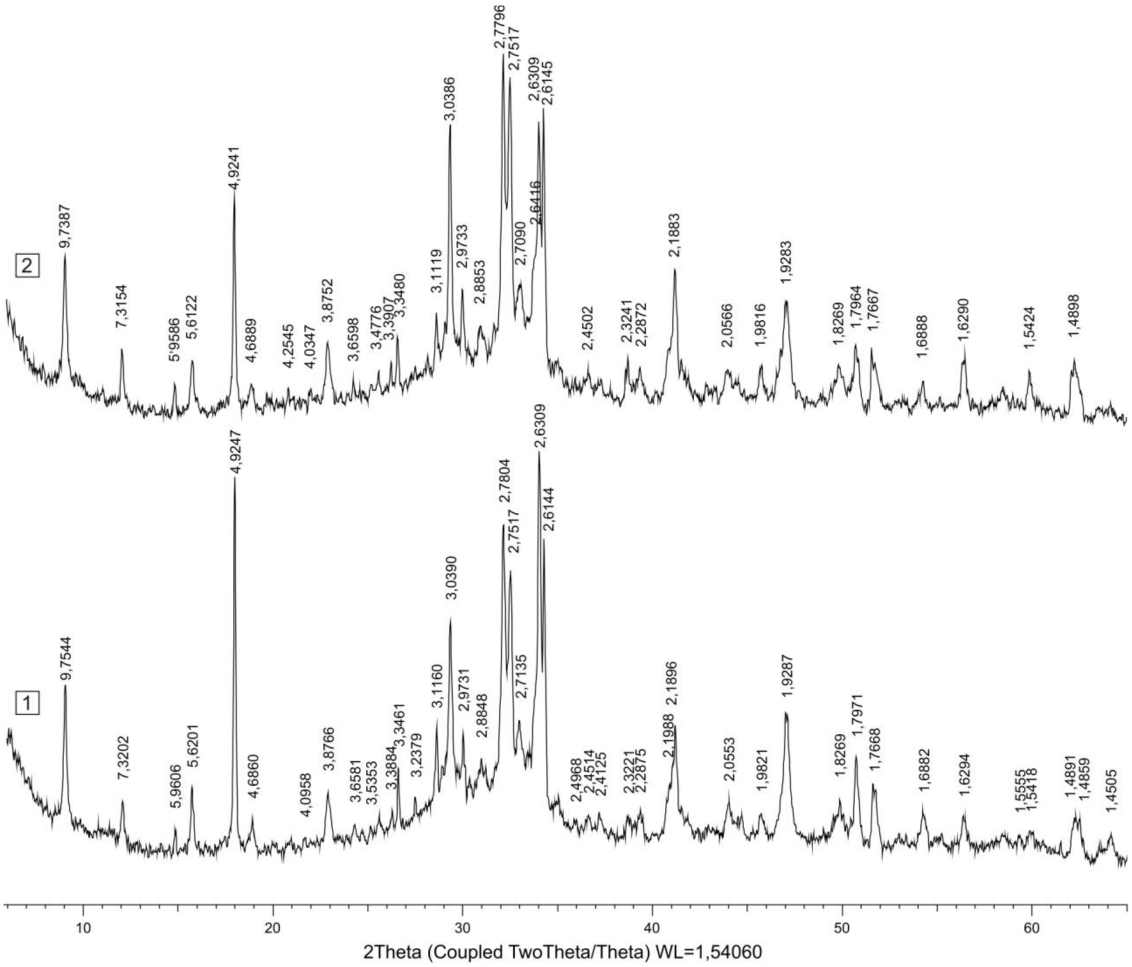

Figure 7. The curves of the X-ray phase analysis of cement stone: 1 – the sample, 2 – the sample with complex modifying agent

X-ray phase analysis of samples of cement stone without additive and with the complex modifying agent was performed (Figure 7). A quantitative calculation was also made using by the Rietveld method taking into account 20% of add phase. It has been established that the amount of the initial phase - alite (C_3S d = 3.0386; 2.7796; 2.7517; 2.6145; 2.3241; 2.1883; 1.7667; 1.6290 Å) is observed in the composition with a complex modifier and tricalcium aluminate (C3A d = 2.7090, 1.5555 Å) is 43 % higher than the control composition (C_3S d = 3.0390; 2.7804; 2.7517; 2.6144; 2.3221; 2.1988; 1.7668; 1.6294 Å;

C_3A d = 2.7135; 1.5555 Å), which indicates a delay in hydration of cement due to the blocking effect of SP and HP, while the amount of whitet decreases by 35% (C_2S d = 2.8853; 2.7796; 2.7517; 2.7090; 2.2872; 2.1883 Å). This effect is probably due to the fact that the adsorption of the superplasticizing admix occurs on hydrated neoplasms, and the greatest adsorption capacity is possessed by C_3A, the smallest C_2S [12]. The decrease in the amount of portlandite ($Ca(OH)_2$ d = 4.9241, 3.1119, 2.6309, 1.9283, 1.7964, 1.6888, 1.4859, 1.4505 Å) in the composition with a complex modifying agent (by 50 %) is due to the interaction of portlandite with metakaolin. This helps to compact the material, which is indirectly confirmed by a decrease in the amount of calcium carbonate ($CaCO_3$ d = 3.8752, 3.0386, 2.6309 Å) by 8 %, which increases the resistance of the material to carbonization. In the composition with the complex modifying agent, the amount of ettringite (d = 9.7387, 5.6122, 4.6889, 3.8752, 2.1988, 2.1504 Å) decreases by 32 %, which is possibly due to a large amount of unreacted C_3A in the composition with a complex modifying agent (43 %). And a decrease in the concentration of calcium hydroxide in solution, while according to electron microscopy, ettringite crystallizes in the pore spaces and cracks, consolidating and strengthening the structure of the material.

Taken all round according to the data from X-ray phase analysis, differential thermal analysis, electron microscopy, the mechanism of action of the complex modifying agent is manifested in the blocking effect of SP and HP, which is expressed in a reduced amount of portlandite and a greater content of the initial phases of cement clinker, while MTK interacts with calcium hydroxide, which contributes to the compaction of the material. Reduction of ettringite in the composition with a complex modifying agent is associated with precipitation of superplasticizing admix molecules on C_3A particles, which limits interaction with water.

Conclusions

1. The complex modifying agent for self-compacting concrete has been developed. We have researched the influence of the complex modifying agent on setting up time of cement-water paste and on the cement stone strength depending on Portland cement mineralogical composition.

2. The behavior of cement hydration in composition with the complex modifying agent have been shown by means of measuring the hydrogen-ion concentration, by sedimentation, contraction and heat emission of cement suspension. There is the decreasing of the degree of cement stone hydration because of blocking action of the superplasticizing admix and hydrophobisator during the initial stage.

3. Studying the cement stone spalls with the aid of electron microscopy has showed that there are the crystallized hydrated newgrowths with smaller dispersive capacity in composition with the complex modifying agent than the ones without introduction of admixtures. The increasing of concentration of hydrated calcium sulfoaluminate in pores and capillaries, the increasing of the specific surface area of hydrated phases both in the general structure of cement stone and in structure with regions of imperfections, the voids content decreasing lead to the material hardening.

4. By differential thermal and X-ray phase analysis established the mechanism of structure formation of cement stone in the composition with a complex modifying agent manifested in blocking effect SP and HP, resulting in a reduced amount of portlandite and high content of the starting phase the cement clinker, wherein the MTK is reacted with calcium hydroxide, which helps to seal material. Reduction of ettringite in the composition with a complex modifying agent is associated with precipitation of superplasticizing admix molecules on C_3A particles, which limits interaction with water.

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