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Composite binders for concrete of protective structures

Композиционные вяжущие для бетонов защитных сооружений

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Ключевые слова: самоуплотняющийся бетон; композиционное вяжущее; пуццолановые материалы; техногенное сырье; свойства бетонной смеси; механические свойства

Abstract. The composite binders, obtained as a result of joint mechanochemical activation of Portland cement, rice husk ash, quartz sand, screening of limestone crushing and plasticizing additive were considered. The type of binder influence to the rheological and mechanical characteristics of self-compacting concrete mixtures and fiber-reinforced concrete is established. By scanning electron microscopy, it was revealed that the application of the developed binder leads to the compacting of the microstructure, with lamellar and acicular neoplasms filling the isometric and anisometric pores. This leades to the formation of a rigid matrix with a lower porosity, which in turn leads to an increase in the compressive strength of the formed cement stone above 70 MPa. The use of a composite binder increases the physical and mechanical characteristics of fine-grained concrete accordingly (Rcompr over 80 MPa, elastic modulus greater than 40 GPa). The studied rheological characteristics showed that all mixtures with the use of composite binder meet the requirements of fluidity and segregation resistance for self-compacting concrete mixtures. The revealed indexes of shock endurance of fiber-reinforced concrete (30 % higher than for standard reinforced concrete) allow using this material for protective structures.

Аннотация. Рассмотрены композиционные вяжущие, полученные в результате совместной механохимической активации портландцемента, золы рисовой шелухи, кварцевого песка, отсева дробления известняка и пластифицирующей добавки. Установлен характер влияния вяжущего на реологические и физико-механические характеристики самоуплотняющихся бетонных смесей и фибробетонов. С помощью растровой электронной микроскопии выявлено, что применение разработанного вяжущего ведет к уплотнению микроструктуры, при этом наблюдаются пластинчатые и игольчатые новообразования, которые заполняют изометричные и анизометричные

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поры. Это способствует образованию жесткой матрицы с меньшей пористостью, что и приводит к увеличению прочности на сжатие формируемого цементного камня выше 70 МПа Использование композиционного вяжущего соответственно повышает физико-механические характеристики мелкозернистого бетона (Rсж более 80 МПа, модуль упругости выше 40 ГПа). Изученные реологические характеристики показали, что все смеси с применением композиционных вяжущих отвечают требованиям текучести и сопротивляемости расслоению для самоуплотняющихся бетонных смесей. Выявленные показатели ударной выносливости фибробетонов (на 30 % выше, чем для стандартных железобетонов) позволяют использовать данный материал для несущих конструкций защитных сооружений.

1. Introduction

Concretes for protective structures in connection with the increased natural (including global climate change) and technogenic (including the increase of international tension and terrorist acts) catastrophes acquire special significance. For these concrete, a special set of characteristics is required - compressive strength, impact endurance, crack resistance, impermeability, and workability. Thus, it seems expedient to develop promising composite binders using crop waste to enhance the efficiency of self-compacting fiber-reinforced concrete on their basis.

In earlier studies [1–5], theoretical bases for the creation of composite binders with the use of various pozzolanic additives – ashes of thermal power plants, blast-furnace slags, diatomite shales, volcanic ash, etc., were used as active silica components. However, the issue of the application of new species nanodispersed mineral additives, as well as the principles of their compatibility to ensure the required performance characteristics of composite binders has not been studied sufficiently. To expand the use of composite binder in construction, it is necessary to study the compositions of Portland cement and multicomponent finely dispersed mineral and organic additives to obtain the required properties of binders and composites based on them for protective structures [6–11].

In the previous article [12] we proved that using of rice husk ash can effectively serve as an alternative material in the production of concretes.

Accordingly with the foregoing, the paper formulates a working hypothesis that the use of rice husk ash (RHA) in combination with Portland cement, quartz sand, limestone and effective hyperplasticizer for base Portland cement will make it possible to obtain a composite binder that will ensure the cohesion of a highly mobile concrete mixture, to obtain high-strength concretes due to the provision of increased cohesion of cement stone with aggregate due to the application of RHA, regulation of pore structure, and forced deformations due to the use of composite binder.

Thus, the purpose of the research is development of composite binders and high-strength self-compacting concretes on its basis.

To achieve this purpose, accomplished tasks include the following:

- research of the physical properties and chemical composition, and the structure of the raw materials;
- determination of the optimal technology for the preparation of composite binder;
- study of microstructure and chemical composition of cement stone neoplasms;
- research of rheological characteristics of self-compacting concrete mixes;
- study of mechanical and exploitation characteristics of fine-grained fibrous concrete.

2. Research methods

The mineral composition of the raw components was obtained by X-ray diffraction analysis using the method of full-scale quantitative analysis. The granulometric composition of powdery materials was determined by laser granulometry, which allows one to directly determine the particle sizes and the percentage of their content in the material being analyzed.

A study of the morphological features of the microstructure was carried out using a scanning electron microscope Carl Zeiss CrossBeam 1540XB. Derivatograms of the samples were obtained on the thermogravimetric analyzer Shimadzu DTG-60H.

Specific surface of binder as well as used mineral additives was measured, according to Russian State Standard GOST 310.2-76, with the help of the device PSH-11, which operates on the principle of air permeability through a layer of pre-compacted material.

The standard viscosity of the cement paste was determined with the aid of a Vic device. To study the viscosity of concrete mixtures, a rotary viscometer "HAAKE RheoStress 600" with measuring system FL22 (propeller type) was used.

Tests of self-compacting concrete mixtures (SUBM) were carried out as per Russian State Standard GOST 10181-2000 "Concrete mixtures. Methods of testing", and per international regulatory documents. First, the spreading cone (mm) was determined and the time taken to achieve a spread of the standard cone of the concrete mix to a diameter of 500 mm (s), that is, a measure of speed.

The mixes were then subjected to the following tests:

1. Testing of a self-compacting concrete mix by V-funnel test.
2. Testing of self-compacting concrete mix by L-box.
3. Test of self-compacting concrete mix by U-box.
4. Research of the segregation of the mix using the Static Segregation Column Mold-HC-3666.

To determine the average density of concrete on samples of regular shape, three samples of a cubic shape were taken, cured under the same conditions and having the same age of curing. Compressive strength at the static load action (cubic strength) was determined in accordance with Russian State Standard GOST 310.4-81. Prism strength and modulus of elasticity were determined in accordance with Russian State Standard GOST 24452-80. Elastic modulus was calculated for each sample at a loading level of 30% of the destructive one, according to formula

$$E_\sigma = \frac{\sigma_1}{\varepsilon_{1y}}$$

where σ_1 is the increment of the voltage from the conditional zero to the level of the external load equal to 30% of the destructive one;

ε_{1y} is the increment in elasticity of the relative longitudinal deformation of the sample, corresponding to the load level $P_1 = 0.3P_p$ and measured at the beginning of each stage of its application;

P_p is the destructive load, measured on the scale of the press force meter;

P_1 is the corresponding increment of the external load.

The porosity of the cement stone was determined on samples measuring 1x1x30 cm and 3x3x3 cm at the age of 28 days by mutually complementary methods, namely: proton magnetic resonance with a pore measurement range of $1x10^{-3}$ ... $1x10^{-1}$ μm in diameter; small-angle X-ray diffraction with a measurement range of $2x10^{-3}$ ... $3x10^{-1}$ μm; mercury porosimetry with a measurable range of $1x10^{-1}$ ... 4x10 μm; optical microscopy of thin sections with a measuring range of 4x10 ... $1x10^3$ μm.

For the purpose of comparative analysis of the behavior of brittle materials during impact, two methods of shock interaction of solids with targets from concrete were realized. In the first case, when there is a blind introduction of the shock striker into the obstacle, the latter was in contact with the measuring rod. Measured and recorded values in this experiment were the impact velocity, residual depth and shape of the crater, as well as the deformation impulse in the measuring rod. At higher speeds to study the penetration and piercing processes, the plate target was located in a special chamber, and the penetration process was recorded by a high-speed movie camera (for a detailed description of the experimental setup used, see [13]). The main fixed values in this case were the character and type of scattering of the fragments of the rear surface of the target, as well as the movement of the rear end of the shock striker.

3. Results and Discussions

At the Institute of Chemistry, Far-Eastern Branch of the Russian Academy of Sciences under the leadership of L.A. Zemnukhova [14–15] obtained amorphous silica by thermal method and precipitation from rice husks. The sample of the raw material was treated with 5 % potassium hydroxide solution at 90 °C for 1 hour. The remainder of the raw material was separated from the solution, from which silica was then precipitated with concentrated hydrochloric acid HCl. The precipitate of the material was washed with water until the sodium chloride or potassium chloride was completely removed and dried in air at 60°C.

A silica sample obtained by precipitation from an alkaline solution consists of irregularly shaped, acute-angled particles, the size of which is 200 μm to 1 μm or less (Figure 1). The investigated raw material visually represents a powder of light gray color with bulk density of 570 kg / m^3.

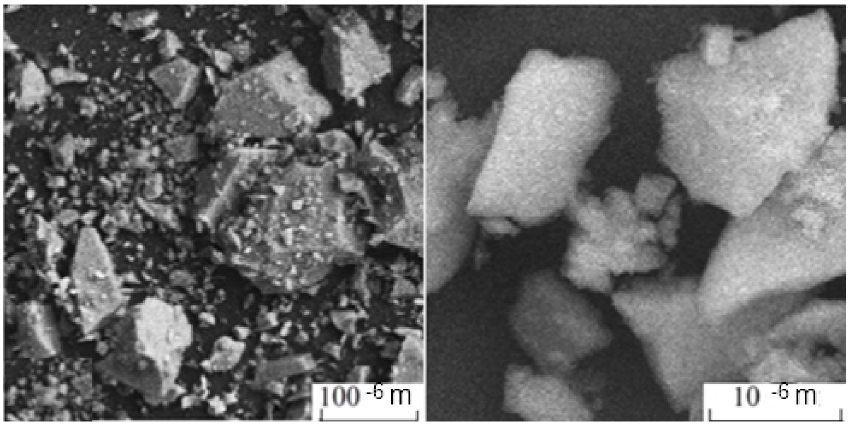

Figure 1. Microstructure of amorphous silica samples

An analysis of the mineral composition of the raw material studied, obtained by treatment of XRD by the method of full-profile quantitative analysis, showed the following chemical composition (Table 1).

Table 1. Chemical composition of rice husk and its ash

Raw material	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Na_2O	K_2O	LOI
Husk	0.61	15.64	0.24	0.12	0.45	0.18	0.48	0.28	82
Ash	3.36	85.48	1.33	0.64	1.93	0.45	2.09	1.57	1.68

To achieve the purpose of the work, a composite binder was prepared, which was prepared by joint grinding of the following components: 60 % of cement, 25 % of rice husk ash, 5 % of quartz sand, 5 % of crushed limestone, 5 % of hyperplasticizer. The composite binder was reduced to 500–900 m^2/kg. Water was added in the amount necessary to ensure the same mobility, but from the calculation of the water-binding ratio not higher than 0.25. Cement stone was studied at the age of 1, 3, 7 and 28 days.

To determine the "best" hyperplasticizer, the six most common super- and hyperplasticizers in the Russian Far East were evaluated [16–17]. Primary studies were conducted only for a reference sample, pure CEM I 42.5 H (Spassk Cement). The results of the fluidity test are shown in Figure 2. Although the results for Pantarhit PC160, Melflux 5581F and Melflux 1641 F are almost the same, all further experiments were conducted using Pantarhit PC 160, due to the lower cost of this product.

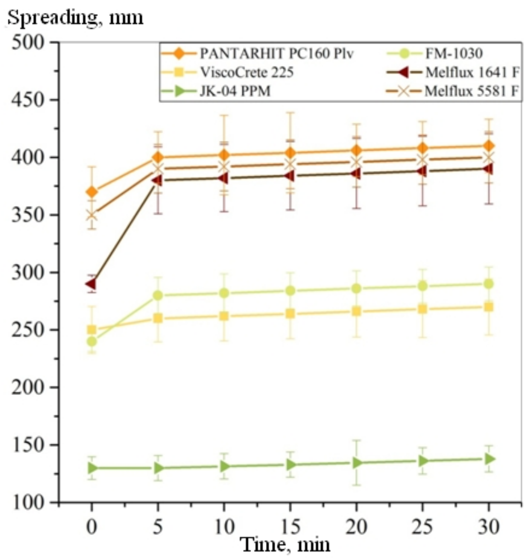

Figure 2. Spreading of cement paste with various hyperplasticizers (each point represents the average of six dimensions)

In order to determine the maximum effectiveness of the action of RHA to cement stone, a two-factor variation was made: the first factor is the optimal dosage (0-30 %), the second factor is the fineness of grinding (500–900 m^2/kg). As a control sample, a pure cement stone from fine-grained cement was used.

The analysis of the obtained results showed that the maximum increase in strength of about 30 % in comparison with pure Portland cement is achieved with the introduction of RHA in the amount of 25 %. Further increase in the content of the additive in the system leads to a drop in strength (Figure 3), which is due to dilution of the system, as well as an increase in the water demand of the mixture.

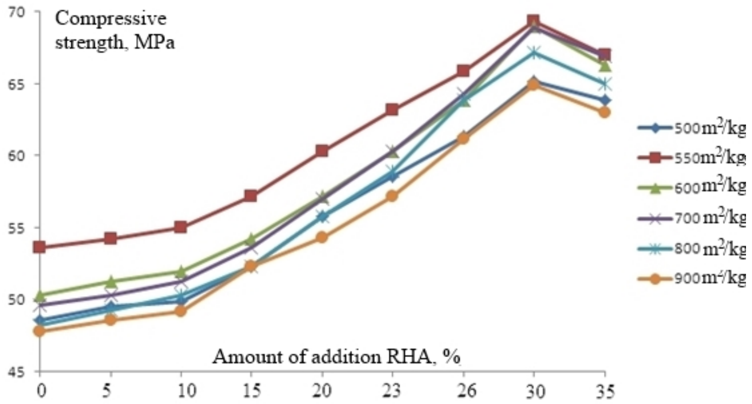

Figure 3. Dependence of compressive strength of cement stone on the amount of addition RHA

Next, an almost linear dependence of the required time of grinding of composite binder was revealed to achieve a different specific surface in the range from 280 to 900 m^2/kg (Figure 4a).

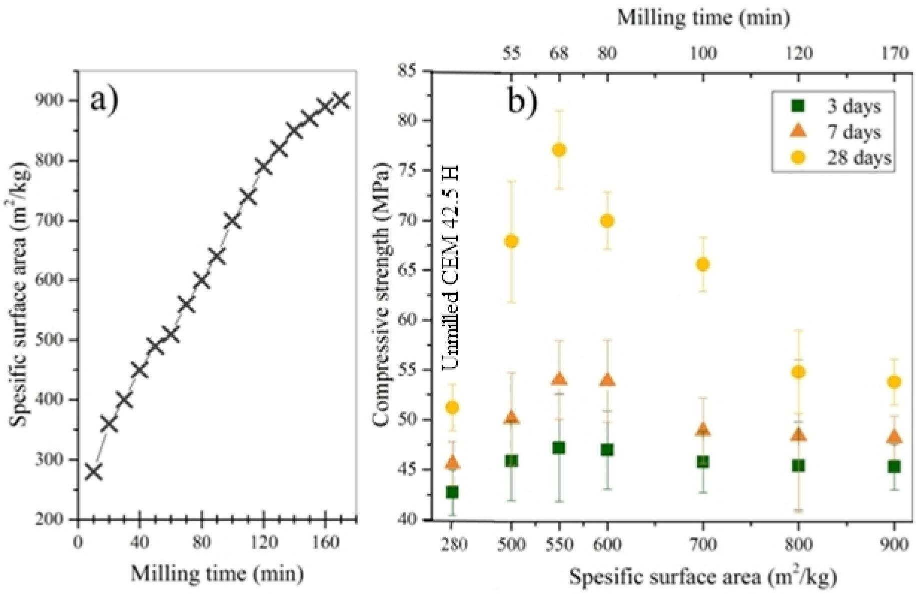

Figure 4. Bond between compressive strength of the cementitious stone samples and the composite binder surface area. Each point represents the average of six measurements

Obviously, with these data, the required grinding time can be predicted to reach a certain surface area. After grinding the composite binder components and measuring the surface area, water was added and the compressive strength was measured at the age of 28 days. The results are shown in Figure 4b. It can be seen that the maximum compressive strength was obtained at a surface area of 550 to 600 m^2/kg. The increase in the surface area does not lead to a further increase in strength, and even leads to a decrease. This is due to the excess of fine particles, because the limit of the action of the hyperplasticizer has been reached, which we investigated earlier [18, 23, 25]. This behavior was also observed in the change in the viscosity of the mixture when the surface area of the particles was above 600 m^2 / kg. It is expected that an increase in the amount of hyperplasticizer will lead to the creation of concrete with an even greater compressive strength [13–17, 19–22, 24, 26–27].

Analysis of the microstructure of the samples made it possible to establish that for pure Portland cement, a matrix with a large number of pores and microcracks is characteristic, the main mass is represented by crystallized X-ray amorphous neoplasms against which hexagonal plates of portlandite are viewed (Figure 5a).

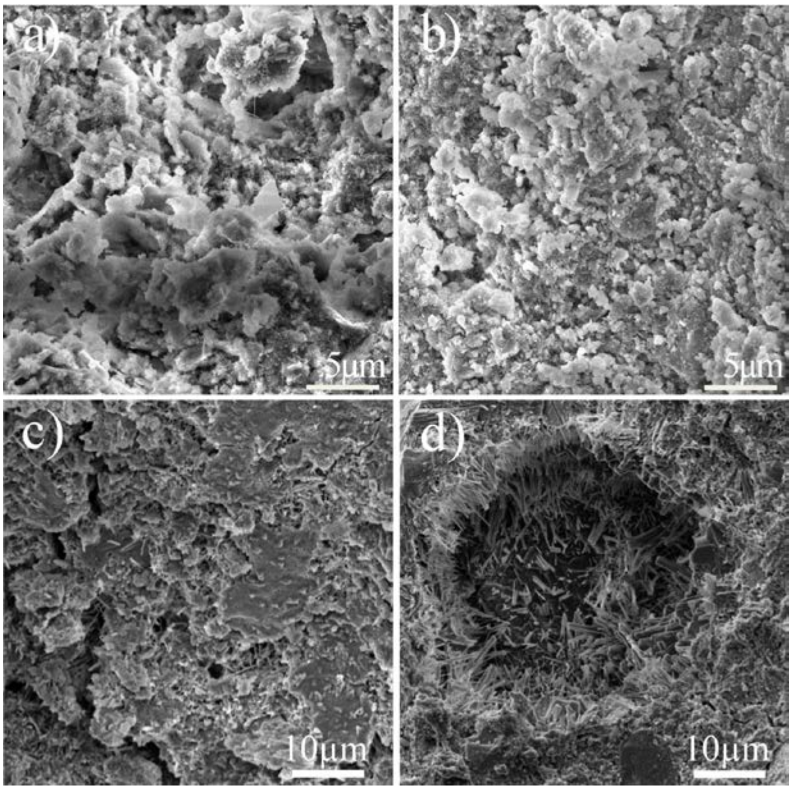

Figure 5. Microstructure of neoplasms (age 28 days): pure cement stone (a, c) and cement stone on the developed composite binder (b, d)

Using of the developed composite binder promotes the formation of a denser microstructure, with clearly distinct systems of needle and lamellar neoplasms filling anisometric and isometric pores (Figure 6, *b, d*). This contributes to the formation of a rigid matrix with a smaller number of pores, which predetermines an increase in the compressive strength of cement stone.

According to the results of X-ray diffraction analysis (Figure 6) for the diffractogram of cement stone, a lower intensity of peaks corresponding to clinker minerals of C_3S with d / n = 3.04; 2.97; 2.78; 2.74; 2.75; 2.61; 2.18; 1.77 Å is characteristic for the developed composite binder; C_2S with d / n = 2.89; 2.67; 2.72; 2.76; 2.75; 2.78; 1.77 Å, which indicates the intensification of hydration processes with using of the composite binder. Also, composite binder contributes to a decrease in the intensity of the portlandite peaks.

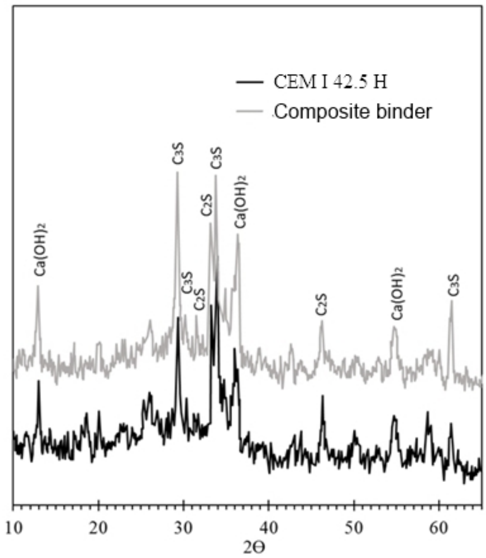

Figure 6. XRD of neoplasms

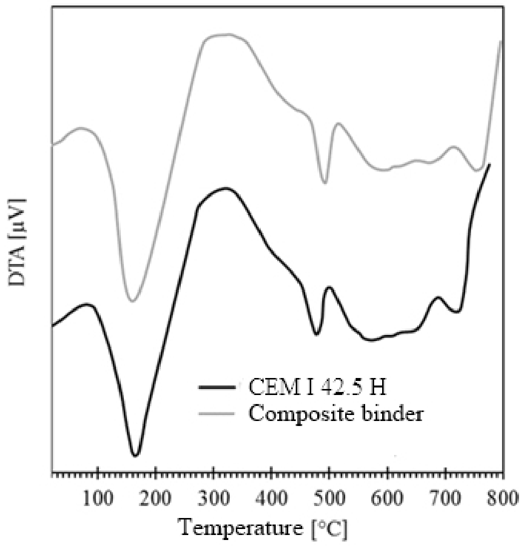

Figure 7. Results of DTA for pure cement stone and cement stone of composite binder

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Analysis of the thermograms of pure cement stone and cement stone of composite binder made it possible to identify the presence of three main endothermic effects (Figure 7). The first (at a temperature of about 160 °C) is caused by the loss of adsorption water present in the gel-like hydration products. The decrease in the area of this effect on the thermogram of cement stone of composite binder indicates a decrease in the content of gel-like neoplasms due to their transition to a crystalline state. The second endothermic effect (at a temperature of about 475 °C corresponds to the dehydration of $Ca(OH)_2$. The increase in the area of this peak on the DTA of pure cement stone indicates a greater content of portlandite in it. Third endothermic effect (at a temperature of 525–650 °C) can be associated with the decomposition of $CaCO_3$.

Further studies were aimed at developing the composition of binder on the basis of Portland cement, RHA, quartz sand, screening of limestone crushing and hyperplasticizer. For this purpose, a matrix was planned, where as factors of variation were taken: the amount of inert filler including quartz sand and limestone crushing (7–14 % of the mass of binder) and the content of the hyperplasticizer (3–9 % of the mass of the composite binder mixture). The output parameters were the compressive strength. The control stones were cement stones obtained from the following binders: pure Portland cement CEM 42.5 H, Portland cement CEM 42.5 H + RHA, Portland cement CEM 42.5 H + Pantarhit PC-160 (PC + HP) and Composite binder (CB) (Table 2).

Table 2. Control compositions

Binders	Standard viscosity, %	Average density, kg/m^3	Compressive strength (28 days), MPa	Percentage increase in compressive strength in relation to pure Portland cement
CEM I 42,5 H	25.9	2269	43.89	-
CEM 42.5 H +31 % RHA*	24.4	2359	69.02	57.26
CEM 42.5 H + 6 % HP	21.6	2431	60.63	38.14
CB	21.5	2340	71.21	62.25

* - additives to cement are ground to a specific surface of 550 m^2/kg

The analysis of the obtained results showed (Table 3) that the maximum compressive strength indexes are characteristic for cement stone with a cement content of 52.5 % cement CEM 42.5 H, 31 % RHA, 10.5 % complex of inert fillers (quartz sand and limestone crushing) and 6 % hyperplasticizer. As optimal we take the composition 2-2.

Table 3. The results of selecting the optimal composition of binder

Sample number	Binder composition				Standard viscosity, %	Density, kg/m^3	Compressive strength, MPa
	CEM 42.5 H	Inert fillers	RHA	Hyper-plasticizer			
1-1	58	7	32	3	22.3	2369	62.36
1-2	56	7	31	6	21.6	2372	63.96
1-3	54	7	30	9	20.3	2370	64.21
2-1	54.5	10.5	32	3	22.7	2336	67.36
2-2	52.5	10.5	31	6	21.5	2340	71.21
2-3	50.5	10.5	30	9	20.8	2346	66.39
3-1	51	14	32	3	23.0	2320	63.36
3-2	49	14	31	6	22.1	2323	64.65
3-3	47	14	30	9	22.6	2325	62.11

Investigation of the mechanical properties of fine-grained concrete (Table 4) showed that using of the composite binder allows to increase the technical characteristics of concrete, in comparison with similar compositions made with the use of traditional binder materials. This fact is explained by the denser structure of the cement stone on the developed composite binder, with a lower porosity, due to less water in the concrete. The best physical and mechanical characteristics showed composition 2-2. It should be noted that increasing the amount of ash and reducing the amount of cement to ensure equi-mobility of the compositions (slump flow = 10-12), it is necessary to increase the amount of mixing water introduced into the concrete mix.

Table 4. Mechanical characteristics of fine-grained concrete depending on the composition of the binder

Composition	Consumption of materials per 1 m^3				Cubic strength, MPa	Prism strength, MPa	Elastic modulus, GPa
	Cement, kg	Fillers of binder, kg	Sand, kg	Water, l			
1-2	646	508	1020	223	73.6	54.0	41.0
2-2	606	548	1020	231	82.6	65.2	55.3
3-2	565	589	1020	236	75.3	50.3	41.3
CEM I 42.5H	545	-	1634	218	62.9	41.8	35.2
CEM I 42.5H + 31% RHA*	376	169	1634	241	71.2	52.3	44.0
CEM I 42.5H + 6% HP	512	33	1634	182	65.3	49.2	41.2

*- RHA is ground to a specific surface of 550 m^2/kg

To establish the optimal percentage of reinforcement of fine-grained steel-fiber-concrete, concrete samples of the same composition (2-2) with different contents of steel and basalt fiber were molded. The results are shown in Figure 8.

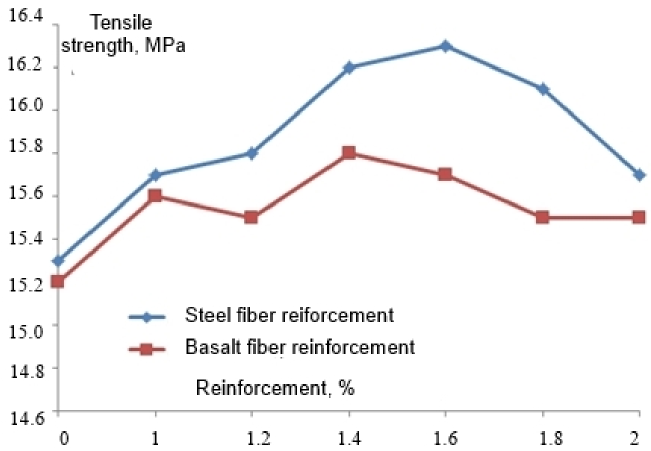

Figure 8. Dependence of tensile strength of fiber-reinforced concrete on percentage of reinforcement by different types of fiber

To determine the workability, all concrete mixtures were designed in such a way as to have a spreading cone of average diameter of 680 ± 30 mm, which was achieved due to variation of the water-binding ratio. It should be noted here that an increase in the content of RHA in mixtures led to a decrease in workability due to a higher specific surface area of ash particles, which led to greater water consumption to facilitate the movement and sliding of particles over each other. As mentioned earlier, RHA particles were ground to make a more favorable material. However, in the RHA still have some unground or insufficiently ground particles (Figure 1), which are extremely porous and sintered, while the Portland cement particles are denser than even shredded RHA. As the water content increases, the porosity increases, which can lead to an unfavorable effect on the properties of the concrete mix. The results of investigations of rheological characteristics are shown in Figure 9. In addition, the data obtained show that all binder mixtures meet the requirements for fluidity and resistance to delamination in accordance with EFNARC requirements.

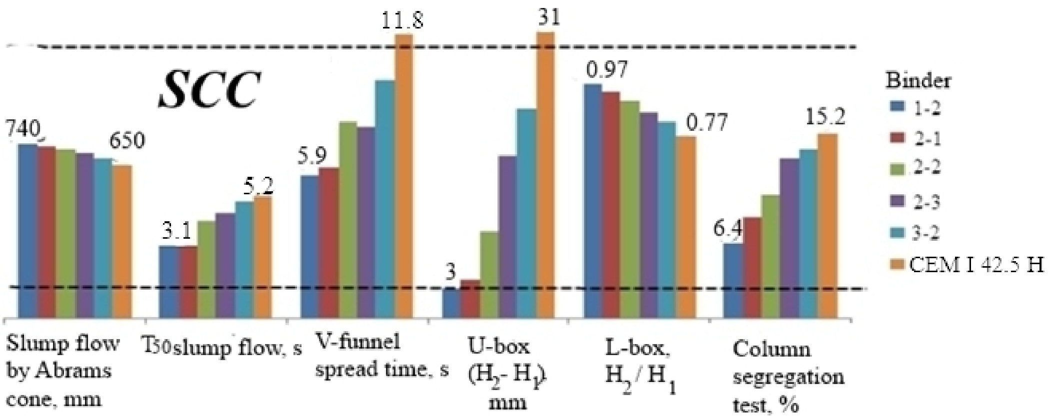

Figure 9. Results of the study of the rheological characteristics of self-compacting concrete mixes

To evaluate the resistance of fiber-reinforced concrete to impact, experiments were carried out to introduce a shock impactor into the target in the form of disks with a diameter of 100 mm and a thickness of 20 mm. As a shock impactor, a steel cylinder was used, which was accelerated by 20-mm gas cannon with a diaphragm-type shutter. Air and helium were used as the working gas. In the study of shock endurance of fiber-reinforced concrete reinforced with various types of fiber, results were obtained that produced 1600–1700 shocks before failure at 1.4–1.6 % reinforcement.

4. Conclusion

1. There was offered the new composite binders and high-strength self-compacting concretes on its basis.
2. The optimal technology for the preparation of composite binder is joint grinding of raw components to specific surface area of 550–600 m^2/kg.
3. The composite binder contributes to the formation of a rigid matrix with a smaller number of pores, which predetermines an increase in the compressive strength of cement stone.
4. Application of these composite binders allows to increase the efficiency of self-compacting fiber-reinforced concretes for various mechanical and operational characteristics.
5. The obtained characteristics of impact strength of the developed fiber-reinforced concrete (up to 30 % higher than those for standard reinforced concrete) allow this material to be used for protective structures.

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