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Influence of carbon black additives and finely ground waste from stone wool production on characteristics of cement systems

Klyuev S.V. * [1], Ayubov N.A. [4], Fomina E.V. [1], Ageeva M.S. [1], Klyuev A.V. [1], Nedoseko I.V. [2]

[1] Belgorod State Technological University named after V.G. Shukhov, Russia,
[2] Ufa State Petroleum Technical University, Russia,
[3] Kh. Ibragimov Complex Research Institute of the Russian Academy of Sciences, Russia

Abstract: The object of research is cement composites with additives of carbon black and finely ground waste stone wool production. The work aims to design a mix of a cement composite with the additives of carbon black and finely ground waste from stone wool production, which achieves the best strength characteristics. The results show that carbon black is represented on average by particles of 155 microns with inclusions of large agglomerates up to 1-2 mm in size, consisting of almost homogeneous nanoparticles 10-20 nm in size. Carbon black is distinguished by high hydrophobic properties with a true powder density of 900 kg/m3 and a bulk density of 300 kg/m3. The chemical composition of black carbon is 70-80% carbon and 10-15% oxygen, and it also contains impurity compounds of zinc, iron, sulfur, silicon, and other elements. Carbon additives acquire hydrophilic properties in the presence of a plasticizer, and the degree of their influence on hydration becomes less pronounced. The contraction of the binder during the first three hours of hardening is reduced when carbon black is introduced into the cement system in an amount of 8%. A composition with the best strength characteristics was obtained: the content of finely ground waste from stone wool production is 6% by weight of the binder; carbon black content is 4-5%; W/C = 0.2. However, there is difficulty in mixing the mixture at such a low W/C. With a water-cement ratio of 0.3, this problem is solved, and the strength characteristics remain quite high.
Keywords: carbon black, cement, stone wool, recycling, concrete, sem, differential thermal analysis

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*Corresponding author E-mail: Klyuyev@yandex.ru

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1. INTRODUCTION

The most important task of the global environmental safety is the development of technological methods and technical means, resource conservation and complex processing of man-made materials, and the production of products with high-performance characteristics based on them.

The most materials used in the construction industries are cement concretes [1]. In the production of mortars and concrete, the use of additives from recycled materials (recycled crushed stone [2], thermal insulation waste, multi-component packaging, old tires) and by-products of industrial processes (fly ash [3, 4], slag [5], rice husk [6], silica fume [7, 8], rock crushing screenings [9], marble and granite dust) makes it possible to obtain an intelligent nanoengineered composite.

An analysis of recent research in the field of building materials science has shown that in the development of "intelligent" building materials, combined carbon-based additives are used with a combination of different carbon forms (polymorphic modifications, linear dimensions, dispersion).

Following the concept of a closed cycle, as well as from an economic point of view, carbon black is of considerable interest [10]. Carbon black is the fundamental unstructured material that is obtained from rubber tires and utilized as filler material in rubber and plastic products [11]. Compactly located elementary crystallites of carbon black form its primary particles, which are firmly bound by covalent bonds into aggregates (Fig. 1) [12]. Moreover, the more particles the aggregate contains, the higher the carbon black structure. Individual aggregates consolidate the interaction of weak chemical and physical bonds (van der Waals, electrostatic forces, etc.), forming agglomerates.

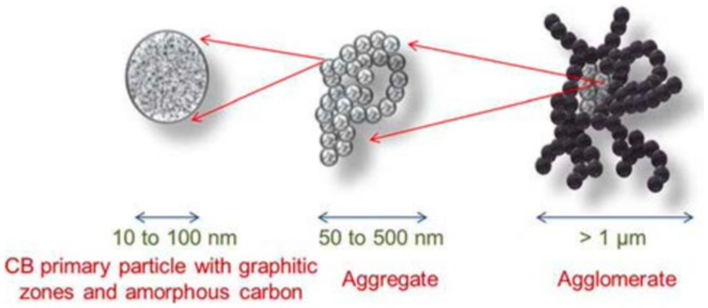


Fig. 1. Carbon black particles [12].

It is known that carbon black particles do not participate in cement hydration [13], and their high specific surface area (S.S.A.) makes it possible to effectively fill pores at the micro- and nanolevel of concrete. In this case, it becomes possible to control the propagation of microcracks in cementitious composite materials. Acting as an inert filler, carbon black proves to be an effective way to control the properties of concrete. For example, carbon black helps to minimize the crack appearance in concrete prisms when they are subjected to bending, and it also enhances their conductivity and mechanical assets [14, 15].

Another technogenic waste is mineral wool waste [16], the amount of which, in some cases, can reach 15-30% of the mass of the finished product [17]. According to experts, a significant part of them is disposed of in landfills. A solution to the problem may be the use of waste as fillers, primarily in the production of various concretes [18]. The inclusion of technogenic fibrous materials in binder systems is aimed at improving bending strength and thermal conductivity, which is confirmed by the study of lime [19], cement [20], and geopolymer binders [21, 22]. The use of technogenic fibrous materials in building mortars makes it possible to reduce the shrinkage of the mixture, increases the elastic modulus, and also increases the fire resistance of building composites [23, 24]. As a filler in wood-fiber composites to improve moisture-resistant properties [25]. Scientists estimate that geopolymer concrete with mineral wool produces approximately 80% less CO2 emissions than conventional concrete, and the final product is twice as strong as traditional cement concrete.

During the production of thermal insulation materials, fibers of various lengths and widths are formed, as well as a certain proportion of molten spherical particles in the form of loose, coarse material [26]. These particles are called beads in this work. In finely ground form, beads have astringent properties under the condition of alkaline activation for the production of slag-alkaline binder and cement concrete [27], [28]. The presence of amorphous silicate phases in this material allows it to be used as an effective active mineral additive in natural and autoclaved cement concrete [26]. The beads can also be effectively used as a replacement for fine aggregates in concrete, saving on the cost of natural aggregates. Another approach is to use beads as active mineral additives in concrete [29].

Most of the previous studies focused on the study of the physical and mechanical characteristics of cement systems using carbon black or waste mineral wool; however, it remains unclear whether it is possible to use these two additives together in cement systems to obtain a material with sufficient strength. This study aims to design a mix of a cement composite with the additives of carbon black and finely ground waste from stone wool production, which achieves the best strength characteristics of the material.

2. Materials and Methods

2.1. Materials

The following materials were used in the production of specimens:

1. Portland cement 42.5 N produced by AO Sebryakovcement (Volgograd region, Russia). The main characteristics of cement are given in Tables 1, 2.

Table 1. Chemical composition of cement, %.

CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	R_2O in terms of Na_2O
66.03±0.80	21.45±0.45	5.83±0.20	4.40±0.20	0.58±0,06	0.30±0.10	0.69±0.10

Table 2. Mineralogical composition of cement, %.

C_3S	C_2S	C_3A	C_4AF
65.10±2.50	13.86±1.50	8.96±1.00	11.95±1.50

1. Waste, called beads, from the production of mineral wool insulation "Izovol" (Belgorod, Russia). The bulk density of the beads is 1129-1366 kg/m^3, the true density is 2804–2884 kg/m^3, and the specific surface is 160-180 m^2/kg. The chemical composition of the beads is presented in Table 3.

Table 3. Chemical composition of beads, %.

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2
44.1	12.26	9.44	16.2	13.19	2.2	0.78	1.42

2. Carbon black – a product of low-temperature (T≤500°C) thermolysis technology for processing car tires.
3. Superplasticizer Reoplast PCE series produced by OOO Reoplast (Leningrad region, Russia).

1.1. Research on physical and chemical properties of carbon black

Contact angle measurements were carried out using the Kruss DSA 30 system (Hamburg, Germany) and ADVANCED software. The method was based on measuring changes in the contact angle of wetting on the surface of a specimen of cement stone with a size of at least (100±2) cm^2 with various additives under the influence of ultraviolet radiation at a temperature of (23±5) °C and a relative humidity of 40–70%.

Morpho-structural characterization was studied using a high-resolution scanning electron microscope TESCAN MIRA 3 LMU (Brno, Czech Republic).

The assessment of the mineral phase composition was carried out according to quantitative X-ray phase analysis using an ARLX'TRA X-ray diffractometer manufactured by Thermo Fisher Scientific (Waltham, USA).

The particle size distribution and the calculated area based on the particle size distribution were determined using a High-End laser particle analyzer Analisette 22 NanoTec plus manufactured by Fritsch GmbH (Idar-Oberstein, Germany), the measurement range of which is 0.01 – 2000 µm.

The specific surface area and average particle size were determined using the air permeability method using a PSH-12 SP device manufactured by OOO PSKH (Moscow, Russia), which averages particle sizes (measurement range 200 – 50000 cm^2/g).

1.2. Research on adsorption properties of carbon black and waste from stone wool production

The study of the physical and chemical properties of carbon black and waste from stone wool production included studies of sorption capacity in relation to aqueous solutions containing heavy metals. Model solutions containing Ni^{2+} ions were prepared by dissolving $NiSO_4 \cdot 7H_2O$ salt (reagent grade) in distilled water with pH=7. The concentration of $Ni^{2}+$ ions was determined using a KFK-3 spectrophotometer produced by OAO Zagorsk Optical-Mechanical Plant (Sergiev Posad, Russia). When establishing the adsorption value A (mg/g), model solutions had an adsorbent concentration from 10 to 4000 mg/dm^3. The solutions were purified statically by adding weighed portions of sorption material to a model solution of known concentration.

Carbon black was studied as an adsorbent for model solutions containing the dye methylene blue. When cleaning solutions, the amount of carbon black required for cleaning was determined. The work used model solutions containing dye in concentrations of 25 and 50 mg/l.

Carbon black was studied as a sorption material for petroleum products. The work used model solutions containing petroleum products in concentrations of 5 and 10 mg/dm^3. Carbon black was added to a solution with a volume of 1 dm^3; the content was varied in the range from 0.5 to 5 g. The mixing time of the suspension was 15 minutes.

1.3. Research into processes of structure formation and aggregative stability of cement system with carbon black

One of the main factors in the aggregative stability of dispersed systems is the electrokinetic potential (ς-potential). The work measured the ς-potential of carbon black powder in water, together with the superplasticizer, and in a solution of 0.001 M NaCl.

The work assessed the influence of carbon black on the processes of binder hydration. Measurements of volumetric deformations (contraction) of cement and the forecast of its contraction activity were determined using a cement contraction (volume deformations) meter "Cement-Prognoz" produced by OOO NPP Interpribor (Chelyabinsk, Russia). The principle of operation of the device was to measure the decrease in the volume of water in a hermetically sealed and water-filled measuring chamber, inside of which a glass with a specimen of the cement paste being tested is previously placed. The decrease in water volume was equal to the contraction ΔV (ml, cm^3) of the material and occurred over time. The change in volume was judged by the change in the level of the liquid column in the vertical capacitive sensor. The activity R (MPa) of cement was determined by the contraction ΔV_0 of a material specimen weighing 1000 g during the first 3 hours of hardening after mixing with water.

The heat release of cement during hydration was determined using an isothermal calorimeter ToniCAL 7338 at a temperature of 20 °C (Toni Technik Baustoffprüfsysteme, Berlin, Germany).

1.4. Regression analysis of influence of carbon black and finely ground waste additive from stone wool production on strength characteristics of cement systems

Mathematical modeling of the strength of cement stone depending on additives was carried out in order to optimize its composition. To describe the strength of the composite at any point in the experimental area, second-order polynomial models with three variable factors were used [30]:

$$Y(x)=a_0+a_1x_1+a_2x_2+a_3x_3+a_{11}x^2_1+a_{22}x^2_2+a_{33}x^2_3+a_{12}x_1x_2+a_{13}x_1x_3+a_{23}x_2x_3+a_{123}x_1x_2x_3 \quad (1)$$

where Y(x) is optimization parameter; x_1, x_2, x_3 are variable parameters; $a_0, a_1, a_2, a_3 \ldots a_{23}$, a_{123} are regression coefficients.

The experiment organization scheme provided for m=3 duplicate measurements of the output parameter at 3 points of the factor space.

Calculations of regression coefficients and analysis of the resulting model were performed using Excel.

The compressive strength of cement stone at the age of 28 days (R_{28}) was taken as the value to be optimized. The mass fraction of carbon black (x_1), the mass fraction of mineral additive (x_2), and the water-cement ratio (x_3) were considered variable factors.

2. Result and Discussion

2.1. Test results of physical and chemical properties of carbon black

The initial properties of various technical carbons directly depend on the source material and the production method; therefore, in each specific case, it is necessary to study the physicochemical properties. The carbon black considered in this work is a product of the thermolysis of automobile tires.

The study of the surface of graphite particles of carbon black showed that they are hydrophobic and atomically smooth, and therefore, a high degree of agglomeration is noted. The dispersion of particles is largely determined by their free surface energy, as well as the polar and dispersive parts of their components [31]. Consequently, non-polar carbon-based materials are not easily dispersed in highly polar media such as water. It is known that in aqueous solutions, carbon-containing components have hydrophobic properties [32].

To study the degree of hydrophobicity of soot, a drop of water was applied to its surface, and the contact angle was determined (Fig. 2).

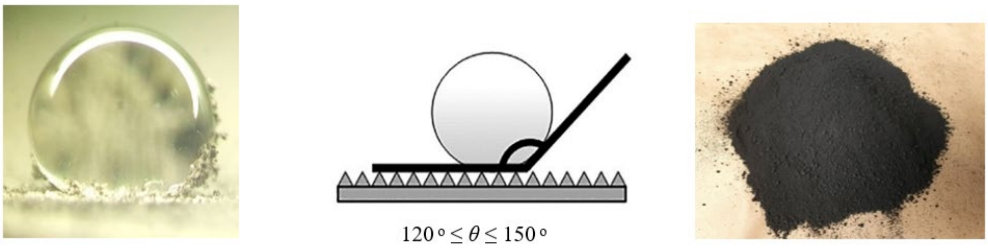


Fig. 2. Degree of hydrophobicity of soot based on droplet shape.

According to the classification of types of hydrophobic surfaces [33], the hydrophobicity of carbon black with a contact angle θ<120° is confirmed.

Using electron microscopy, it was found that the aggregated particles of carbon black have a size of 100-150 μm (Fig. 3). The aggregates are grouped by nanosized particles of 10-20 nm, close to a spherical shape. The particle size on a nanometer scale determines its high dispersity.

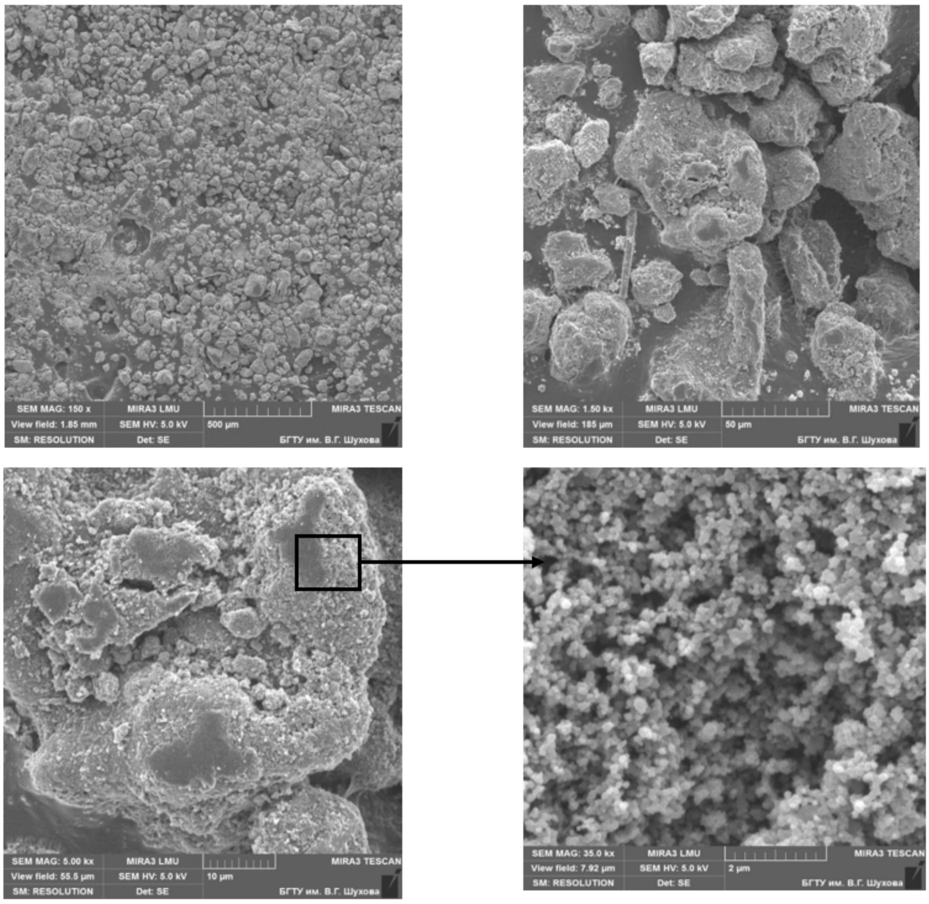


Fig. 3. Microstructure of carbon black.

Energy dispersive analysis of the chemical composition of the surface of carbon black particles is shown in Fig. 4.

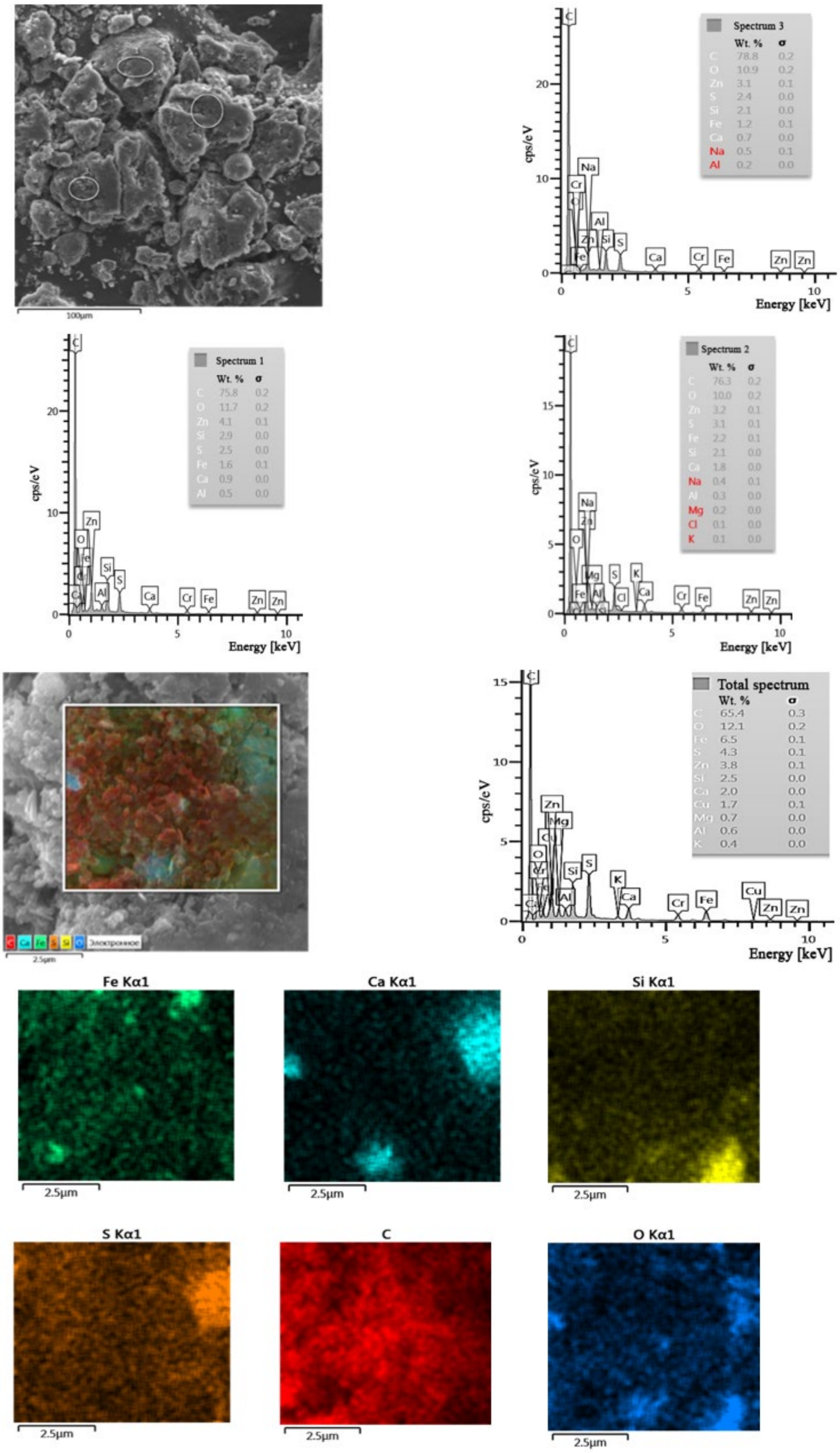


Fig. 4. Energy dispersive microanalysis of surface of carbon black particles.

Fig. 4 shows that on average carbon black contains over 70% carbon, 12% oxygen, and also contains zinc, iron, sulfur, silicon, and other elements.

To analyze the size distribution of carbon black particles, powder specimens were sifted through sieve 063. The results of the particle size distribution are shown in graph form in Fig. 5.

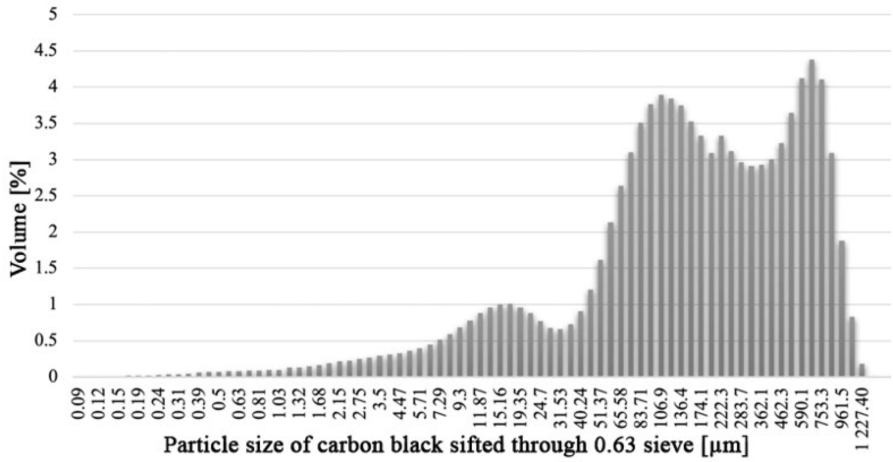


Fig. 5. Carbon black particle size distribution.

The quantitative content of particles of various fractions is determined in % by weight and is reflected in Table 4.

Table 4. Granulometric composition of carbon black.

Diameter [µm]	6.72	14.71	67.08	155.02	416.94	668.54	781.59	962.88
Percentile [%]	5	10	25	50	75	90	95	99

According to the data shown in the diagram (Fig. 5) and Table 4, the average particle size of carbon black is 155 microns, and the particle range is approximately from 1000 to 0.15 microns. Moreover, the graph has three pronounced peaks: in the range of 3–31 µm is peak 15 µm (up to 1% of the substance), 31–285 µm is 95 µm (3.8% of the substance), and 400–900 µm is 600 µm (4.3 %).

The main physical and chemical parameters of carbon black were determined experimentally (Table 5).

Table 5. Physical and chemical parameters of carbon black.

Parameter	Value
Specific surface area, m^2/g	12 – 18
Particle diameter, µm	155
pH of aqueous suspension	8
Bulk density, kg/m^3	300
True density, kg/m^3	990

From the results obtained, it is clear that carbon black is lighter than water, with an alkaline reaction in the aqueous suspension.

Thus, carbon black is represented on average by particles of 155 microns with inclusions of large agglomerates up to 1-2 mm in size, consisting of almost homogeneous nanoparticles 10-20 nm in size.

Carbon black is distinguished by high hydrophobic properties with a true powder density of 900 kg/m^3 and a bulk density of 300 kg/m^3. The chemical composition of black carbon is 70-80% carbon and 10-15% oxygen. It also contains impurity compounds of zinc, iron, sulfur, silicon, and other elements.

2.2. Test results of adsorption properties of carbon black and waste from stone wool production

The study of the physical and chemical properties of carbon black and waste from stone wool production included studies of sorption capacity in relation to aqueous solutions containing heavy metals. When establishing the adsorption value (*A*, mg/g), model solutions had an adsorbent concentration from 10 to 4000 mg/dm^3. The solutions were purified statically by adding weighed portions of sorption material to a model solution of known concentration. Ni^{2+} ion adsorption isotherms are shown in Fig. 6.

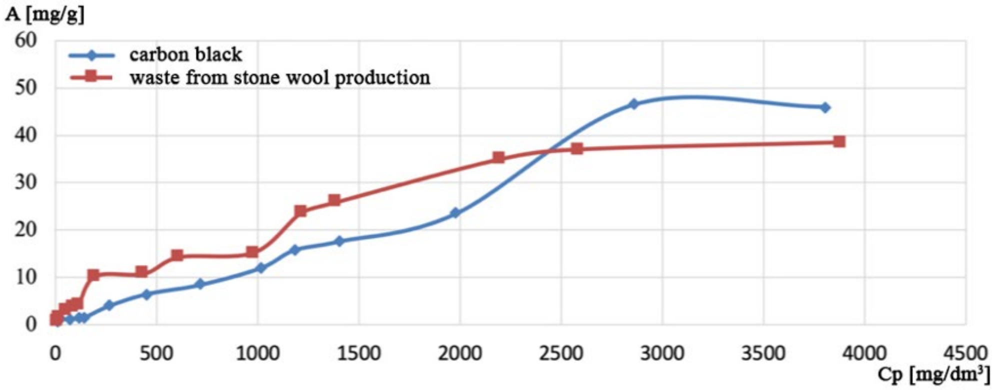


Fig. 6. Ion adsorption isotherms Ni^{2+}.

The highest value of sorption capacity *A* = 46 mg/g is achieved when using waste from stone wool production at Cp = 3000 mg/dm^3. Their particles have a highly developed surface due to depressions, irregularities, and notches, the presence of which is important in adsorption processes because it is to such zones that molecules of adsorbed substances attach. In the case of using carbon black, the maximum sorption capacity was *A* = 38.5 mg/g at *Cp* = 3000 mg/dm^3. The rough, highly developed surface of carbon black provides it with sorption activity. However, carbon black does not show high performance in relation to metal ions.

Carbon black was studied as an adsorbent for model solutions containing the dye methylene blue at concentrations of 25 and 50 mg/l. When cleaning solutions, the amount of technical specifications required for cleaning was determined (Fig. 7).

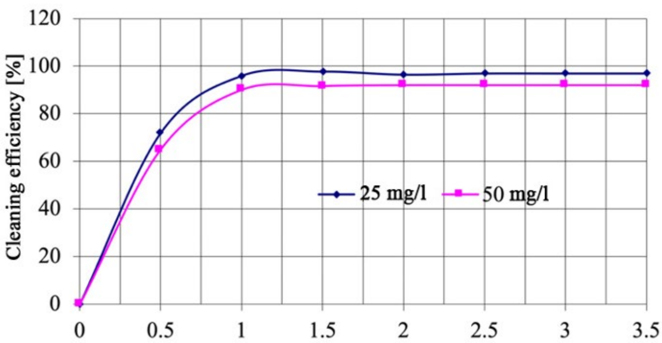


Fig. 7. Dependence of efficiency of purification of methylene blue dye on mass of additive for solutions with concentrations of 25 and 50 mg/l (T = 20 °C; τ = 10 min).

Fig. 6 shows that the maximum purification efficiency of model solutions with a concentration of 25 and 50 mg/l is achieved by adding 1 g of carbon black, which is 92-98%. Analysis of the obtained dependences showed that, depending on the mass of the adsorbent, the efficiency of purification of model solutions from the methylene blue dye also increases to a certain value. With the further addition of a larger mass of waste, the cleaning efficiency does not change significantly.

Carbon black was studied as a sorption material for petroleum products. The work used model solutions containing petroleum products in concentrations of 5 and 10 mg/dm^3. Carbon black was added to a solution with a volume of 1 dm^3; the content was varied in the range from 0.5 to 5 g. The mixing time of the suspension was 15 minutes.

Fig. 8 shows that the cleaning efficiency increases when adding material from 0.5 to 4 g and it is 85 and 74%, then the efficiency increases slightly, and, therefore, increasing the amount of added material is impractical.

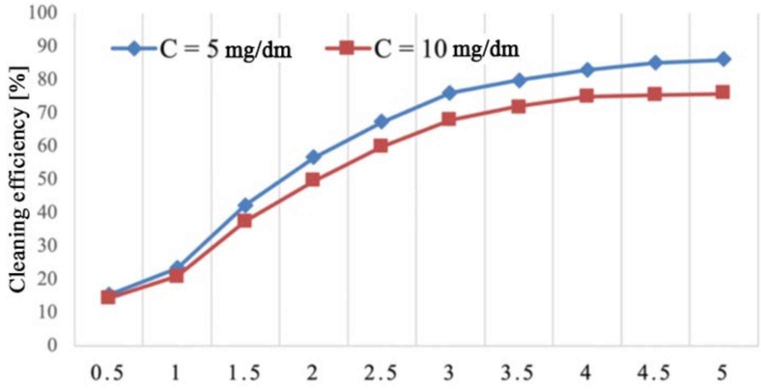


Fig. 8. Dependence of efficiency of solution purification on mass of carbon black.

For oil-containing emulsions, 4 g per 1 dm^3 of model solution should be considered a sufficient amount of sorption material.

The interaction of petroleum products with carbon black can be explained by the following processes. Petroleum products, like carbon-containing particles, are hydrophobic substances. Since the affinity of hydrophobic particles of petroleum products and technical carbon for water is less than for each other, under appropriate conditions (stirring, pH of the environment), they combine into globules. This process continues until the globules become larger and the gravitational forces begin to prevail over the repulsive forces. Increasing density allows particles to settle to form sediment. As the coagulated particles settle, suspended particles also settle with them, which is confirmed by experimental data.

Thus, there is a promising prospect for using waste from stone wool production as a sorption material that can provide high efficiency in wastewater treatment from heavy metals. Carbon black can be an effective alternative to existing sorption materials for the purification of wastewater from dyes and petroleum products.

2.3. Test results of studying the processes of structure formation and aggregative stability of a cement system with carbon black

Test results show when the modulus values of electrokinetic potential increase, the particle size decreases (Fig. 9).

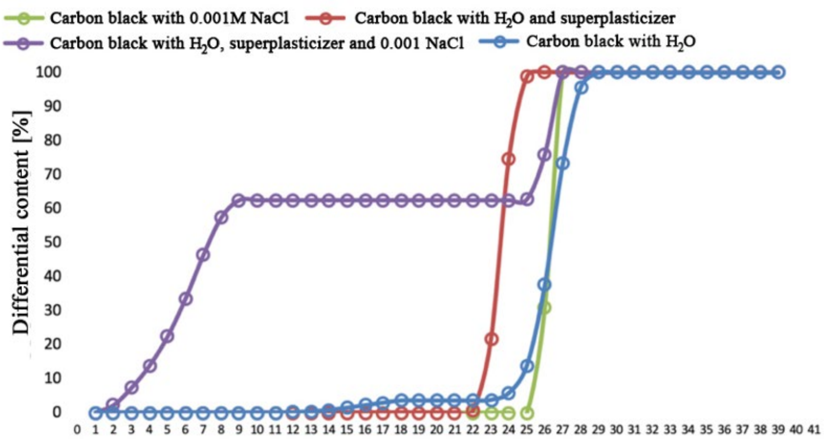


Fig. 9. Dependence of electrokinetic potential on size of carbon black particles under influence of aqueous solution of NaCl in presence of surfactant.

Fig. 9 shows that the ς-potential in distilled water is about +2.1 mV. The particles have a high degree of aggregation. When a surface-active agent is added, dispersion occurs, and the potential increases to +10.3 mV. A similar picture is observed when using a solution of 0.001 M NaCl. The ς-potential value increases with the introduction of a surface-active agent; it is 1.7 without the use of a surfactant, and it is 11.2 with the use one. Electrostatic repulsive forces increase the aggregative stability of the system, preventing the agglomeration of particles.

Literature data show that the addition of carbon-containing dispersed particles negatively affects hydration processes, which plays an important role in the microstructural development of processes and the final properties of cementitious composites.

The work assessed the influence of carbon black on the processes of binder hydration. An assessment of the contraction of cement paste during hardening of the binder with and without the addition of carbon black for 3 hours was carried out. The results of the accelerated determination of the characteristics of cement paste are presented in Table 6. The graph of changes in the volume of cement paste is shown in Fig. 10.

Table 6. Results of accelerated determination of cement paste characteristics.

Test result	Cement	Cement+8% carbon black
Chemical shrinkage ΔV(Co3), ml	0.47	0.29
Chemical shrinkage ΔV1k3, ml	0.65	0.38
Average temperature, °C	19.20	20.2
Temperature correction, ml	0.00	0.00
Final chemical shrinkage, ml/g	0.65	1.68
Cement activity R, MPa	<28.9	<28.9
Specific chemical shrinkage, ml	<0.025	<0.025

It was experimentally established that the contraction of the binder during the first three hours of hardening decreases when carbon black is introduced into the cement system in an amount of 8%, which indicates a slowdown in hydration during the initial stages of hardening (Fig. 10).

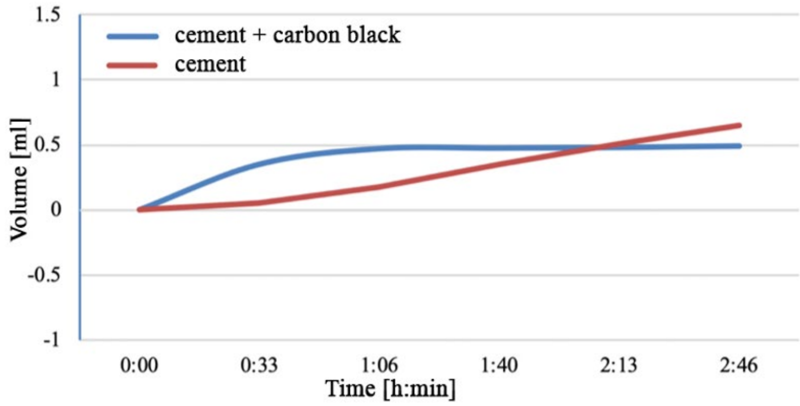


Fig. 10. Accelerated determination of hydration kinetics of cement paste.

However, the strength properties remain unchanged. The binder activity was about 30 MPa.

To clarify these results, an experiment was carried out with the introduction of carbon black at a concentration of 20 % by weight. Differences in the introduction of carbon black have slightly different effects on hydration. Two methods were investigated. In the first case, carbon black was mixed in dry form in a laboratory mixer for 2 minutes, and then water was added. In the second case, carbon black was introduced in the form of a suspension mixed with water and a superplasticizer. The effect of carbon black on hydration processes was observed using isothermal calorimetry measurements (Fig. 11).

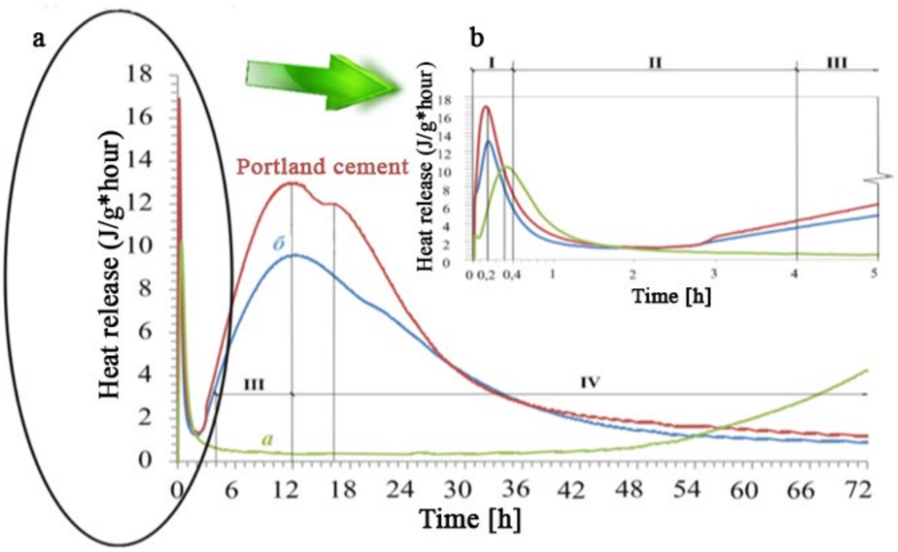


Fig. 11. Influence of carbon black on hydration of cement paste: a is raw mixture without plasticizer; b is raw mixture using plasticizer.

The maximum heat release of the studied systems is observed at the first stage of hydration when mixed with water, as evidenced by the first peak. The second stage characterizes the induction period of hydration. The third stage is characterized by the intensity of the processes of water interaction with clinker minerals. In the fourth stage, the activation of the processes of hydration of clinker minerals and binding of portlandite into hydrosilicate compounds occurs.

Analysis of the literature [34] shows that carbon additives do not affect hydration processes, and they are an inert material. When carbon black is used without the addition of a plasticizer, the water/solids ratio increases with an increase in the viscosity of the system. While mixing this mixture, the paste quickly thickened when water was added. The lack of water supply to cement grains leads to a slowdown in hydration processes, a shift in the first peak, and a subsequent decrease in the rate of hydration (Fig. 11 a).

As the carbon black acquired hydrophilic properties in the presence of a plasticizer, the degree of its influence on hydration became less pronounced, and the heat of hydration curves were closer to the control composition (Fig. 11 b). The intensity of the peaks decreases slightly, which can be explained by the action of the superplasticizer, which envelops the surface of clinker mineral particles [35], reducing the intensity of their hydration.

The surface morphology of the specimen using carbon black is characterized by a rough topography, in contrast to a purely cement specimen. The texture of the created surface allows the degree of hydrophobicity of the building composite to increase. According to studies of the contact wetting angle, a drop of water on the surface of the developed concrete forms $\theta > 90$, which is typical for hydrophobic surfaces (Fig. 12 a).

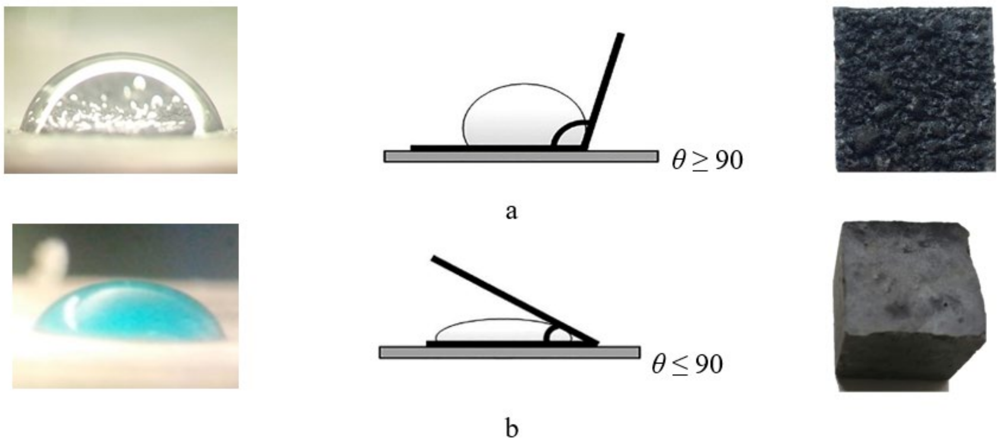


Fig. 12. Influence of carbon black on hydration of cement paste: a is raw mixture without plasticizer; b is raw mixture using plasticizer.

Thus, in aqueous solutions, carbon-containing components differ in their hydrophobic properties. Carbon additives acquire hydrophilic properties in the presence of a plasticizer, and the degree of their influence on hydration becomes less pronounced. The contraction of the binder during the first three hours of hardening is reduced when carbon black is introduced into the cement system in an amount of 8 %.

2.4. Regression analysis results

Mathematical modeling of the strength of cement stone depending on additives was carried out in order to optimize its composition.

In the study, a full factorial experiment design 2^3 was implemented. The center of the experiment (X_0) and the variation levels of variable factors (X_i) are given in Table 7.

Table 7. Initial data for planning experiments.

Variables	Variation levels			Variation interval
	-1	0	1	
x1 is carbon black content	4	8	12	4
x2 is beads content (S.S.A.=800 m^2/kg)	1.5	5	8.5	3.5
x3 - water-cement ratio	0.2	0.3	0.4	0.1

As a result of implementing the full factorial experiment plan, the following results were obtained (Table 8).

Table 8. Experiment planning matrix and test results.

№ series	*X1*	*X2*	*X3*	*x12*	*x22*	*x32*	*x1x2*	*x1x3*	*x2x3*	*x1x2x3*	*Yexp*
1	-1	-1	1	1	1	1	1	-1	-1	1	50
2	1	-1	-1	1	1	1	-1	-1	1	1	55
3	-1	1	-1	1	1	1	-1	1	-1	1	45
4	-1	1	1	1	1	1	-1	-1	1	-1	51
5	-1	-1	-1	1	1	1	1	1	1	-1	61
6	1	-1	1	1	1	1	-1	1	-1	-1	68
7	-1	1	1	1	1	1	-1	-1	1	-1	57
8	1	1	-1	1	1	1	1	-1	-1	-1	60
9	-1	0	0	1	0	0	0	0	0	0	53
10	1	0	0	1	0	0	0	0	0	0	55
11	0	-1	0	0	1	0	0	0	0	0	55
12	0	1	0	0	1	0	0	0	0	0	50
13	0	0	-1	0	0	1	0	0	0	0	49
14	0	0	1	0	0	1	0	0	0	0	65
15	0	0	0	0	0	0	0	0	0	0	57

According to visual analysis, at a water-cement ratio of 0.4 and 0.3, a rough surface of the specimen is noted, which is shown in the photographs in Fig. 13. With excess water, the raw mixture separated, and since the density of carbon black particles was lower than the density of water, they floated to the surface, imparting textural roughness.

a b c

Fig. 13. Appearance of specimens at different water-cement ratio (W/C): a is W/C=0.4; b is W/C=0.3; c is W/C=0.2.

At W/C = 0.2, the viscosity of the system increases, and carbon black is retained in the cement matrix, as evidenced by the homogeneous structure and texture of the specimen along all faces of the cube (Fig. 13 c).

Using the method of mathematical processing of the obtained experimental results, the coefficients of the equation describing the compressive strength of cement stone at the age of 28 days of normal hardening were calculated. The resulting equation has the following form:

$$R_C = 52.6\text{-}2.02X_1 + 3.52X_2 - 6.9X_3 + 2.5X_1^2 - 1.1X_2^2 - 5.5X_3^2 + 6.43X_1X_2 + 0.03X_1X_3 - 1.03X_2X_3 + 1.19X_1X_2X_3$$

Analysis of the mathematical model allowed us to draw the following conclusions:

- the most significant factor in the mathematical model is the water-cement ratio (X_3) since the coefficients at X_3, X_3^2 have maximum values compared to other coefficients in absolute value. The minus sign means that as the water-cement ratio increases, the strength will decrease. At W/C equal to 0.2, the strengths are the highest;

- the coefficients for X_2 (+3.52) and X_2^2 (-1.1) have different signs, which indicates that the zero level of the experiment is in the optimum region. As the amount of filler increases, the strength will increase. The optimal value of the additive content in the cement stone is found by studying the regression equation for the extremum of the variable – X_2, and it equals 2.1%;

- the next factor in the mathematical model is factor X_1 – carbon black content. The strength is inversely dependent on this factor: with an increase in technical carbon content, strength will decrease.

Models of the dependence of the compressive strength of cement stone on the amount of added additives and the water-cement ratio are shown in Fig. 14.

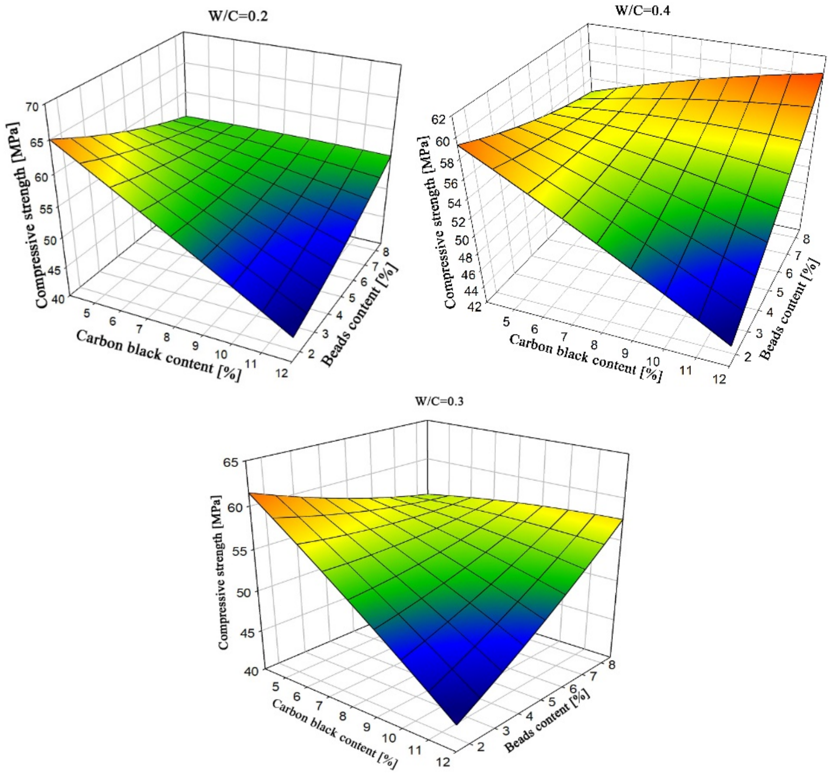


Fig. 14. Models of dependence of compressive strength of cement stone on additives content and water-cement ratio.

Thus, by varying the content of carbon black, beads, and water-cement ratio, it is possible to obtain a mix that is optimal in terms of strength with the following component values: beads content is 6 % by weight of the binder; carbon black content of 4–5 %; W/C=0.2.

Specimens with the obtained ratio of components (carbon black consumption of 4 %, beads (S.S.A.=800 m^2/kg) of 5.5 %, but at different water-cement ratios) were studied for water absorption according to the following equation:

$$W_m = (m_s - m_d) \cdot 100\% \,/\, m_d \qquad (2)$$

where m_w is saturate weight of the specimen after submersion, m_d is dry weight of the specimen.

1. W_m=(13.49-12.54)/12.54=7.6 % (W/C 0.4)
2. W_m=(16.48-15.69)/15.69=5.0% (W/C 0.3)
3. W_m=(16.36-15.96)/15.96=2.5% (W/C 0.2)

The composition with W/C=0.2 has a minimum water absorption of 3 times less compared to the composition with W/C=0.4, which is due to the presence of fewer open pores and voids in the specimens. At the same time, when analyzing the microstructure of composition No. 3, Fig. 15 shows the outlines of bubbles of entrained air forming closed porosity.

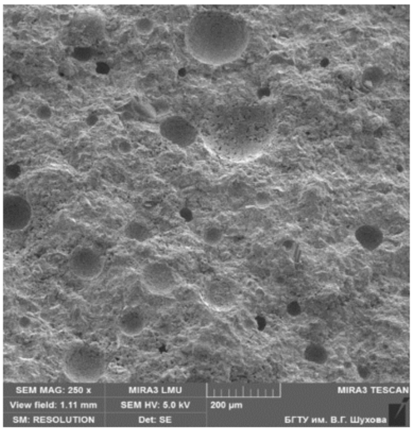


Fig. 15. Structure of specimen composition No. 3 (W/C=0.2).

Superplasticizers have high surface tension, and when mixing a mixture with low W/C, pinching and air entrainment occurs. The formation of closed porosity has a positive effect on the frost resistance of concrete. Macrostructural analysis also established a high degree of adhesion of carbon particles to the cement stone due to the fouling of their surface with an additional amount of hydrosilicate new formations (Fig. 16).

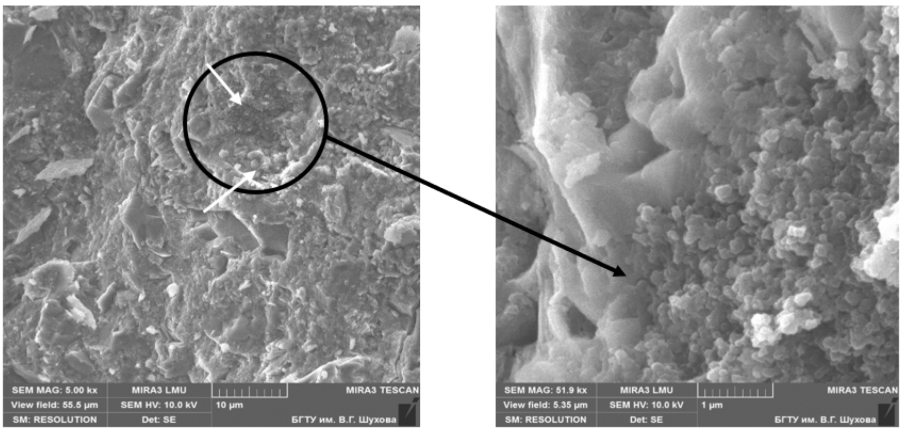
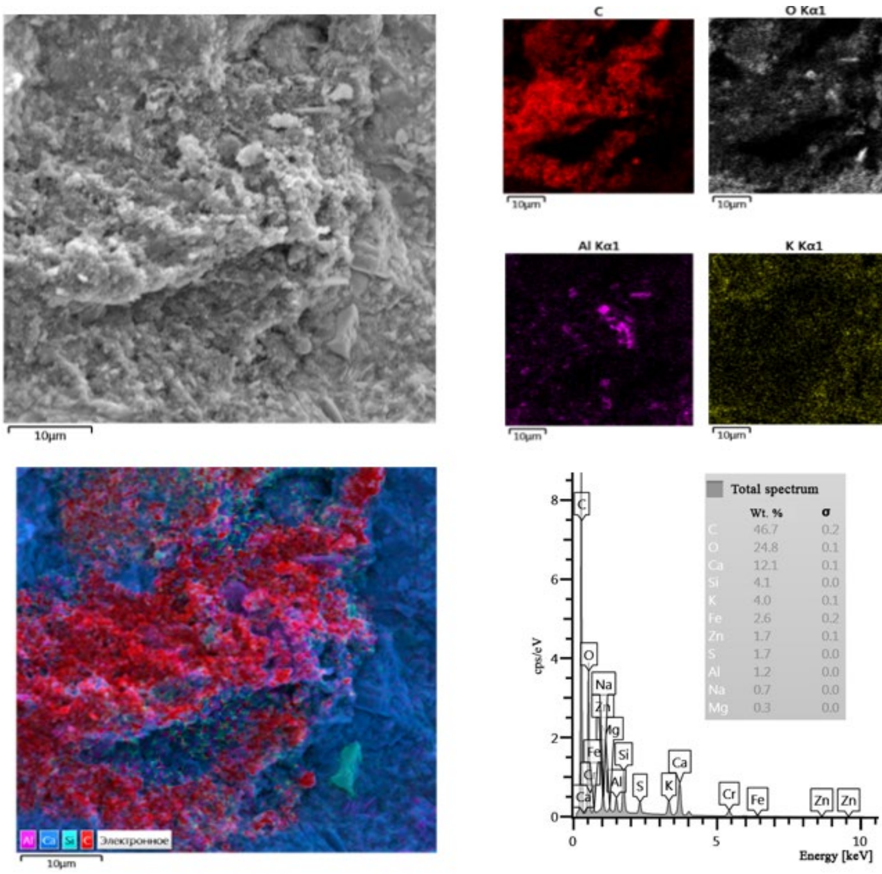
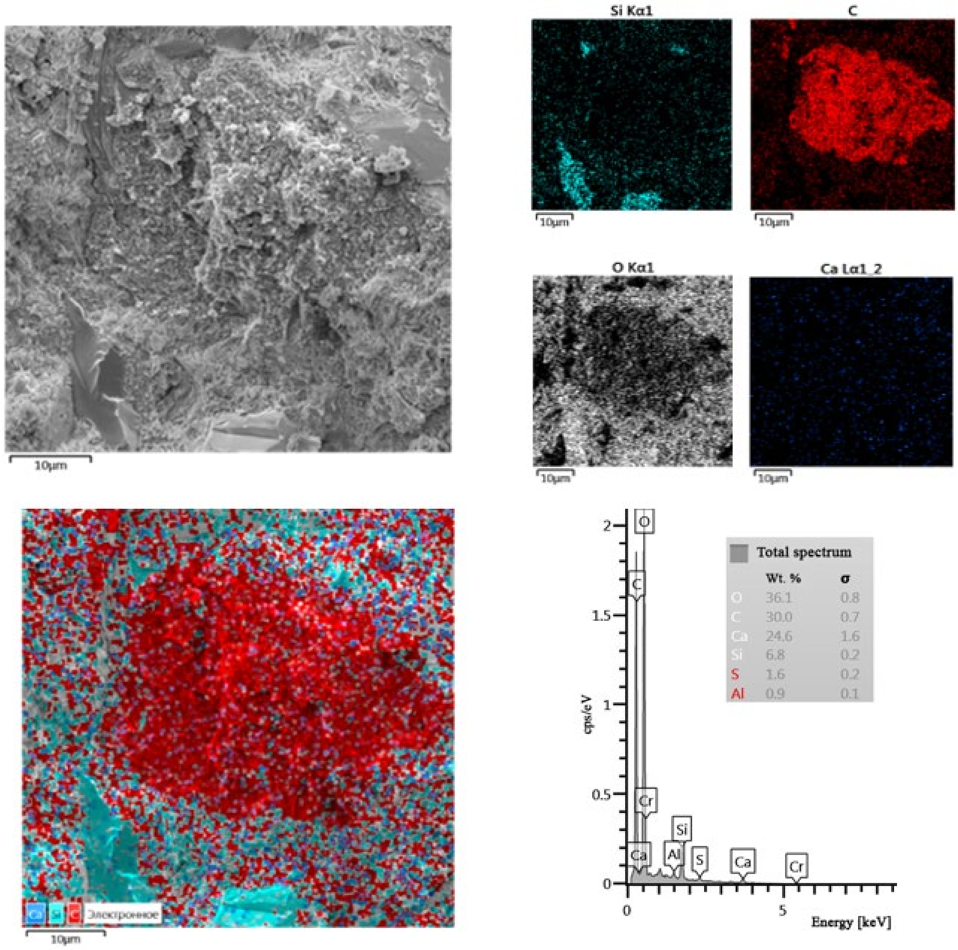


Fig. 16. Microstructure of specimen No. 3, where circle marks carbon particle in the cement matrix.

Analysis of the microstructure of the specimens at 3 different values of the water-cement ratio, carbon black content of 4 %, and the beads content of 5.5 % reflected a picture common to all compositions, where carbon particles are maximally completely covered with hydrate new formations. It is noted that the particle is embedded in the cement matrix, being the center of crystallization, which is clearly visible in the microphotographs of Fig. 17.

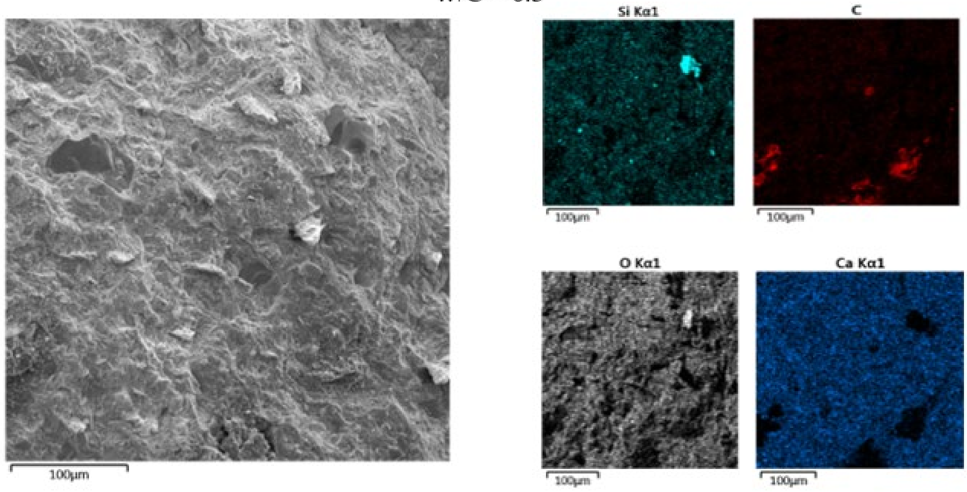


W/C = 0.4

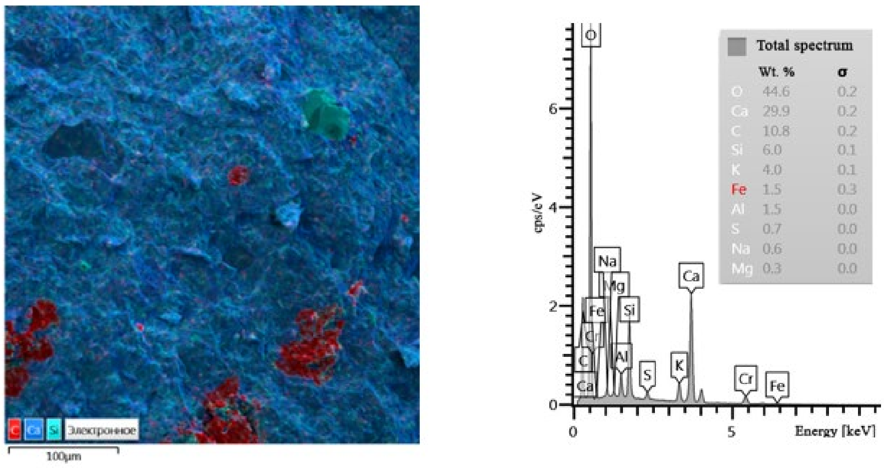


Si Kα1
C
O Kα1
Ca Lα1_2
10μm
Ca Si C Электронное
Total spectrum
Wt. % σ
O 36.1 0.8
C 30.0 0.7
Ca 24.6 1.6
Si 6.8 0.2
S 1.6 0.2
Al 0.9 0.1
cps/eV
Energy [keV]
O
C
Cr
Si
Ca
Al
S

W/C = 0.3



Si Kα1
C
O Kα1
Ca Kα1
100μm



W/C = 0.2

Fig. 17. Energy dispersive analysis of the developed compositions.

Thus, using the method of mathematical planning, a ratio of components was obtained at which the best strength characteristics of the material are achieved: beads content of 6% by weight of the binder; carbon black content of 4–5%; W/C = 0.2. However, there is difficulty in mixing the mixture at such a low W/C. With a water-cement ratio of 0.3, this problem is solved, and the strength characteristics remain quite high.

4. CONCLUSIONS

Based on the results obtained, the following conclusions can be underlined:

1. Carbon black is represented on average by particles of 155 microns with inclusions of large agglomerates up to 1-2 mm in size, consisting of almost homogeneous nanoparticles 10-20 nm in size. Carbon black is distinguished by high hydrophobic properties with a true powder density of 900 kg/m^3 and a bulk density of 300 kg/m^3. The chemical composition of black carbon is 70-80% carbon and 10-15% oxygen, and it also contains impurity compounds of zinc, iron, sulfur, silicon and other elements.
2. The prospect of using waste from stone wool production and carbon black as sorption materials has been established. The waste from stone wool production can provide high efficiency in wastewater treatment from heavy metals (sorption capacity A = 46 mg/g when using waste from stone wool production with Cp = 3000 mg/dm^3). Carbon black can be an effective alternative material, along with existing ones, for treating wastewater from dyes and petroleum products. The efficiency is achieved at a concentration of sorption material of 25-50 mg/l and 4 g/dm^3, respectively.
3. In aqueous solutions, carbon-containing components have hydrophobic properties. Carbon additives acquire hydrophilic properties in the presence of a plasticizer, and the degree of their influence on hydration becomes less pronounced. The contraction of the binder during the first three hours of hardening is reduced when carbon black is introduced into the cement system in an amount of 8%.
4. Using the method of mathematical planning, a ratio of components was obtained at which the best strength characteristics of the material are achieved: beads content of 6% by weight of the binder; carbon black content of 4–5%; W/C = 0.2. However, there is difficulty in mixing the mixture at such a low W/C. With a water-cement ratio of 0.3, this problem is solved, and the strength characteristics remain quite high.

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INFORMATION ABOUT THE AUTHORS

Klyuev S.V., e-mail: Klyuyev@yandex.ru, ORCID ID: https://orcid.org/0000-0002-1995-6139, SCOPUS: https://www.scopus.com/authid/detail.uri?authorId=57212454175, Belgorod State Technological University Named after V.G. Shukhov, Doctor of Engineering Sciences (Ph.D.), Professor

Ayubov N.A., e-mail: yrekly@mail.ru, ORCID ID: https://orcid.org/0009-0001-8129-9598, SCOPUS: https://www.scopus.com/authid/detail.uri?authorId=57221326693, Kh. Ibragimov Complex Research Institute of the Russian Academy of Sciences, Candidate of Economic Sciences (Ph.D.), Professor

Fomina E.V., e-mail: fomina.katerina@mail.ru, ORCID ID: https://orcid.org/0000-0003-0542-0963, SCOPUS: https://www.scopus.com/authid/detail.uri?authorId=55857656600, Belgorod State Technological University Named after V.G. Shukhov Department of Building Materials Science of Products and Structures, Candidate of Engineering Sciences (Ph.D.), Associate Professor

Ageeva M.S., e-mail: ageevams@yandex.ru, ORCID ID: https://orcid.org/0000-0002-3114-7078, SCOPUS: https://www.scopus.com/authid/detail.uri?authorId=56333430100, Belgorod State Technological University Named after V.G. Shukhov, Department of Building Materials Science of products and Structures, Candidate of Engineering Sciences (Ph.D.), Associate Professor

Klyuev A.V., e-mail: Klyuyevav@yandex.ru, ORCID ID: https://orcid.org/0000-0003-0845-8414, SCOPUS: https://www.scopus.com/authid/detail.uri?authorId=56567996100, Belgorod State Technological University Named after V.G. Shukhov, Department of Theoretical Mechanics and Resistance of Materials, Candidate of Engineering Sciences (Ph.D.), Associate Professor

Nedoseko I.V., e-mail: nedoseko1964@mail.ru, ORCID ID: https://orcid.org/0000-0001-6360-6112, SCOPUS: https://www.scopus.com/authid/detail.uri?authorId=6508383453, Ufa State Petroleum Technological University, Doctor of Engineering Sciences (Advanced Doctor), Professor of Building Constructions Departmen