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High-performance fine-grained nanostructured concrete based on low strength aggregates

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Abstract. This paper continues the authors' previous research on developing high-performance concretes based on low-strength aggregates. Unlike the previous work, where a low-strength crushed stone was used, the current research investigates a low-strength fine aggregate-based concrete. With modern construction chemistry in combination with pozzolan and high dispersed ground additives and nanomodification, it is possible to obtain high strength and operational characteristics concretes. The work carried out studies of three cement manufacturers for suitability in high-strength hydrotechnical concrete and phase analysis of the aggregates used in this study. Strength (7, 28, 180 days), density, waterproofness, freeze-thaw resistance of fine-grained concrete based on low-strength and high-strength aggregates were compared in the research. The final result is a concrete recipe with freeze-thaw resistance class F400, Waterproofing class W20 and compressive strength of 60.5 MPa at the age of 28 days (71.9 MPa at 180 days).

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1. Introduction

This paper is a path forward from the author's previous work on this topic [1], devoted to developing heavy hydrotechnical structural concrete based on low-strength aggregate. In this work, a matrix was developed to obtain high-strength hydrotechnical concrete based on low-strength aggregate. Concretes with a strength of about 100 MPA has been developed using a crushed stone fraction of 10–20 mm and about 80 MPA when using a crushed stone fraction of 3–10 mm. The freeze-thaw resistance of these concretes was F400 and the waterproofing class was W20. Using the proposed matrix, it became possible to achieve high strength and operational characteristics of concrete based on low-strength aggregate from gneissic granite.

The developed matrix is of huge interest for use in concrete and other low-strength aggregates, which can be crushed construction waste [2], as well as industrial waste, such as slag [3–5]. Therefore, it was decided to develop formulations of high-performance concrete based on this matrix but using fine aggregate.

The study on the suitability of using a matrix with small aggregate opens up even greater possibilities for reuse, for example, construction waste when recycling old buildings [6, 7]. Moreover, fine-grained concrete has advantages over coarse-grained concrete, [8, 9], especially in conditions of lower temperatures, since it has higher freeze-thaw resistance [10]. However, fine-grained concrete has extensive air entrainment [11] and, accordingly, a lower density, leading to a decrease in strength. For this reason, more cement is used to obtain a similar mechanical strength and performance in fine-grained concrete than coarse aggregate-based concrete. However, to get high strength fine-grained concrete, it is impossible to infinitely increase the cement content.

Moreover, the maximum amount of cement in concrete is regulated by regulatory documents and recommendations. In addition, 21st-century construction science allows using less cement to produce high-strength concretes. Modern high-efficiency plasticisers significantly reduce the water-cement ratio while maintaining the same flowability of the concrete mixture, which leads to a significant increase in strength and performance. Also, strength and performance characteristics can be improved by using industrial waste such as silica fume [12, 13] and shale ash. They have pozzolan activity and can be used as additional binders for civil concretes and even unique, heat-resistant concretes and alternative binders for composite binders [14, 15]. Silica fume and shale ash combined with water reduction minimize the permeability of concrete, leading to an increase in frost resistance and chemical resistance of concrete. They are also waste from industries, so their using in concrete helps to remove the environmental load from the region since these materials can be disposed of in concrete. In addition, the use of complex additives consisting of pozzolan and finely ground additives, concrete chemistry in combination with fibers allows you to obtain high-performance concretes with increased operational characteristics. This issue has been investigated in these publications [16–19].

Based on the above, the following research tasks can be distinguished:

- study of the characteristics of gneissic granite crushed stone extracted from the Kem river bed, as well as an alternative crushed stone;
- investigation of the properties of cement available in the northern regions of Russia in large volumes that are suitable for hydrotechnical construction;
- development of concrete recipes of high-performance fine-grained hydrotechnical nanostructured concrete based on scientific literary data, as well as author's research;
- production of high-performance fine-grained concrete using cement amount not exceeding 550 kg/m³;
- preparation of the concrete samples for determination of principal characteristics, including mechanical parameters, water absorption, waterproofing, freeze-thaw resistance;
- testing of principal characteristics of concrete, defining quality of high-performance fine-grained nanostructured concrete: mechanical characteristics, water absorption, waterproofing, freeze-thaw resistance.

2. Methods

2.1. Concrete aggregates

Fine inert aggregate from the boulders of gneissic granite has been used for the development of empirical research. Boulders have been mined in the Kem river bed (Karelia, northern Russia) and have been crushed down in several steps on a laboratory jar mill 80JM-1a.

At the first stage, the boulders were crushed into small fragments, into fractions of 150–200 mm, and further on, into fractions of crushed stone of 3–10, of 10–20 mm. Fractions were obtained by sieving on laboratory sieves. Gneissic granite sand of a fraction of 0–2.5 mm was pulverized from the fraction of 0–3 mm.

For comparison, gabbro-diabase sand of Goloday-gora Ltd. mine was also investigated. Also, concrete was made on the basis of this sand for comparison with gneissic granite sand-based concrete. Crushability was determined by the fraction of sand of 5–10 mm, which was taken during crushing large stones into sand. This is since regulatory documents do not provide for the determination of crushability by smaller fractions.

2.2. Determination of gneissic granite sand characteristics

The crushability (ΔC) of gneissic granite sand was determined by the grain destructiveness during compression (crushing) in the cylinder. A steel cylinder with a diameter and height of 75 mm according to Russian State Standard GOST 8269.0-97 was used for the purpose. Sand samples are filled into the cylinder, and 50 kN load was made using the hydraulic laboratory testing machine WK-18 ZARZAD SPRZETU (Poland). The loading speed was 1 kN/s. Laboratory scales VTB-12 were used to determine the mass.

$$\Delta C = \frac{m - m_1}{m} 100, \quad (1)$$

where m is the mass of crushed stone test sample, g; m_1 is the mass of residue on the control sieve after sieving of crushed stone sample crushed in the cylinder, g.

Radiographic studies were conducted for the determination of the mineral composition of the crushed stone. Radiographic studies were conducted by using the automatic powder diffractometer D2Phaser (Bruker) (radiation of an x-ray tube is CoKα1+2, wavelengths CoKα1 = 1.78900 Å и CoKα2 = 1.79283 Å, tube operating mode 30 kW/10 mA, position-sensitive detector, geometry on reflection, scheme of focusing Bregg-Brentano, speed of rotation of a sample of 20 revolutions per minute, the interval of angles of diffraction 2theta = 5–80°, scanning step 0.02°, exposition in a point is 1.0 seconds, T = 25 °C, the atmosphere is air).

The sample was made by dry pressing of the studied substance in low-background to a ditch of single-crystal silicon (depth is 0.5 mm, the diameter of the studied area is 20 mm). The identification of the phases was contacted by using the base of powder diffraction data of the Powder Diffraction File. The results of the quantitative X-ray phase analysis are given in Table 3.

Grain size distribution, the content of dust and clay particles, specific gravity, packed density were determined according to the method described in Russian State Standard GOST 8735-88.

2.3. Preparation of concrete samples and determination of their characteristics

Laboratory mixing of concrete according to Russian State Standard GOST 10180-2012 was carried out according to the following recipes:

Table 1. Concrete recipe №2

Components	recipe №1	recipe №2
	kg per m³	
Portland Cement CEM I 42.5 N manufactured by JSC Mordovcement	550	550
Gneissic granite sand (fraction of 0 – 2.5)	1442	–
Gabbro-diabase sand (fraction of 0 – 2.5)	–	1442
Dry mix of: 1. Silica fume manufactured by pilot production of the INRTU 2. Shale ash Zolest-bet manufactured by PCV LLC 3. Modified basalt microfiber manufactured by NTC of Applied Nanotechnologies 4. Plasticizing agent REOMAX PC 3901P manufactured by KUBAN-POLYMER LLC 5. Carbon nanoparticles Astralene manufactured by NTC of Applied Nanotechnologies 6. Defoaming agent Geos P150 manufactured by KUBAN-POLYMER LLC	101	101
Water of mixing according to Russian national standard GOST 23732-2011	210	210

The concrete was mixed as follows. Initially, water and cement were first mixed in the concrete gravity batch mixer Eco CM-71 for one minute. After that, the dry mix of admixes was introduced and mixing lasted another minute. The final mixing also lasted for one minute. Finally, sand was introduced last into the concrete mixture.

Nanomodifiers (Astralene) were introduced into concrete by serial dilution method [20]. Astralene were deposited on the basalt microfiber (TC 5761-014-13800624-2004), and microfiber was introduced into the concrete as a component of dry mix. This resulted in a more uniform distribution of nanomaterials throughout the concrete volume.

The volume of concrete mixing was 40 litres.

Concrete density was determined according to Russian national standard GOST 10181-2014.

Concrete cubes with dimensions of 100x100x100 mm in the quantity of 30 pieces were made according to Russian national standard GOST 10180-2012.

The concrete samples were prepared in moulds and removed from the moulds after 1-day curing at room temperature. All the samples (and the control samples) have been hardened in thermo-humidity conditions for 28 days according to Russian national standard GOST 10180-2012.

The compressive strength test of concrete cubes with dimensions of 100x100x100 mm was carried out on the hydraulic laboratory testing machine MP-1000 «Nutcracker» according to Russian national standard GOST 10180-2012.

The freeze-thaw resistance was determined on the climatic chamber SM 55/50-120 SB according to Russian national standard GOST 10060-2012. The freeze-thaw resistance was determined in a water-saturated state $\left(F_1\right)$. The dispersion of the density values of individual samples in the series before their saturation did not exceed 30 kg/m³.

Waterproofing of concrete control samples by air permeability was determined by the device AGAMA-2 according to Russian national standard GOST 12730.5-2018.

3. Results and discussion

3.1. Cement analysis

The paper carried out a comparative analysis of two regional suppliers of Portland cement (Pikalevsky Cement JSC and Petersburg Cement LLC). These are some of the northernmost cement producers with large production volumes MordovCement PJSC. The results of this comparison give all reasons for the choice in favour of non-additive Portland cement 42.5, produced according to GOST 10178-85, GOST 30515 - 2013 MordovCement PJSC, as the best object for modelling concrete mixtures for the development of high-performance concrete formulations.

Table 2. Comparative table of characteristics of three types of cement of different manufacturers in the Russian Federation

Composition	Safe amount, %	Pikalevsky Cement JSC	Petersburg Cement LLC	Mordov Cement PJSC
Cl^- content	0.1	0,009	0,016	0,008
SO_3	3.5	2.9	3.41	3
C_3S	–	64.4	61,07	63,3
C_3A	–	5,3	8,03	6,6
C_2S	–	15,6	12,58	13,6
C_4AF	–	10,8	10,6	13,5
MgO	5	1.24	2,52	1,23
Free alkalis as per Na_2O	0,8	1,16	1,12	0,78

The cement of MordovCement PJSC has the best values for the minimum concentration of chlorine ions (0.008 %), which is essential for the operating conditions of reinforced structures [21, 22]. But most importantly, at present only MordovCement PJSC produces Portland cement with a level of free alkalis in 0.78 %, which meets the requirements of the existing regulatory and technical documentation (Russian national standard GOST R 55224-2012), while in the regulatory document "Bridges and Pipes" 46.13330-2010-2010 this level was adopted even tougher – no more than 0.6 %. The amount of free alkalis in terms of Na2O determines the degree of alkaline corrosion of concrete, especially if the aggregate contains compounds capable of reacting with free alkalis of cement to form products that change their volume and density [23–25]. As a result, cracks are formed and start to develop in concrete, up to the destruction of structures (especially with temperature differences towards lower temperatures) [22]. Thus, it can be argued that free alkalis in the cement composition and the presence/absence of reactive acidic components in the filler significantly determine the frost resistance and durability of concrete and concrete structures.

Unfortunately, in the northern regions of Russia, there is a problem with access to cement, which in quality would be suitable for use in special hydrotechnical concrete without complaints. The delivery of cement from other regions leads to an increase in the cost of construction and the need for more detailed incoming control of each batch of cement, since with long transportation, the probability of violation of cement storage conditions during operation increases.

3.2. Concrete fine aggregates

The Table 3 shows the quantitative phase analysis of the components that make up the gneissic granite crushed stone.The purpose of this analysis was to search for harmful components that make up the crushed stone.

Table 3. Quantitative phase analysis of gneissic granite sample (weight. %) according to the full-height analysis by Rietveld method

Minerals	Chemical formula	% of masses	
		10 – 20 mm	0 – 5 mm
Quartz	SiO_2	41.2	29.4
Microcline	$K(AlSi_3O_8)$	17.6	17.9
Albite	$Na(AlSi_3O_8)$	24.3	25.1
Biotite	$K(Mg,Fe)_3[AlSi_3O_{10}](OH,F)$	5.3	5.2
Amphibole	$(Na,Ca)_2(Mg,Fe^{3+},Fe^{2+},Al,Ti)_5Si_8O_{22}(OH,F)_2$	4.1	4.5
Magnetite	$Fe^{2+}Fe^{3+}{}_2O_4$	2.0	2.3
Chlorite	$(Mg,Fe)_6Si_4O_{10}(OH)_8$	3.9	4.2
Talc	$Mg_3Si_4O_{10}(OH)_2$	1.2	1.4
Calcite	$CaCO_3$	trace levels	trace levels
Zirconium silicate	$ZrSiO_4$	trace levels	trace levels

One of the first concerns was the fear that all the least stable waste in the production of other fractions fell into the 0–2.5 mm fraction. Because of this, a completely different chemical composition would arise compared to other fractions, which would lead to a significant deterioration in the properties of the 0–5 mm fraction and the impossibility of its use in concrete. However, after the phase analysis (Table 3), it was found that the fears were not justified. The mineral composition of the sand of a fraction of 0–2.5 mm had only slight differences from the composition of the starting material.

From these experiments it was concluded that the majority of the minerals, which are a part of crushed stone, received from sedimentary rocks of a bed of the Kem river (88.8 % of masses) is not specified on the "blacklist" of harmful components and impurity under the table A of the Russian national state standard GOST 8267-93 „Crushed stone and gravel of solid rocks for construction works. Specifications”. Chlorite (4.2 % of masses), magnetite (2.3 % of masses) and biotite (rock-forming mica, 5.2 %) make the exception.

Hovever, according to Table A of the Russian national standard GOST 8267-93 is allowed when the total amount of layered silicates (micas, hydromicas, chlorites) will not exceed 15 % of the mass. In this case this condition is met.

Based on Table 3, it can be concluded that there are no mineralogical and physicochemical restrictions for the use of gneissic granite crushed stone in concrete. This allows to study the mechanical characteristics of gneissic granite crushed stone, as well as the characteristics of concrete based on gneissic granite crushed stone and their compliance with actual Russian national construction standards GOST 8267-93 and GOST 26633-2015.

Table 4. Determined properties of gneissic granite sand

Parameter	Freeze-thaw resistance	Crushability	Specific gravity	Packed density	Water absorption
Value	300 cycles	28–35 %	2606 g/sm³	1.45 g.sm³	2.2 %

The value of crushability corresponds to the grade of 200 according to Russian national standard GOST 8267-93, the lowest rate declared in Russian national standard GOST.

Table 5. Main properties of gabbro-diabase sand declared by the manufacturer

Parameter	Freeze-thaw resistance	Crushability	Specific gravity	Packed density	Water absorption
Value	300 cycles	3.6 %	2986 g/sm³	1.62 g/sm³	0.1 %

The value of crushability corresponds to the grade of 1400 according to Russian national standard GOST 8267-93, the lowest rate declared in Russian national standard GOST.

Table 6. Main properties of concretes

Properties	Recipe №1	Recipe №2
Compressive strength (7 days), MPa	48.4	52.1
Compressive strength (28 days), MPa	60.5	68.3
Compressive strength (180 days), MPa	71.9	80.8
Fresch concrete density, kg/m³	2303	2327
Concrete density (28 days), kg/m³	2255	2261
Concrete density (180 days), kg/m³	2239	2248
Freeze-thaw resistance, cycles	400	400
Water absorption, %	3.9	3.6
Waterproofing, Class	W20	W20
Flowability, Class	F4	F4

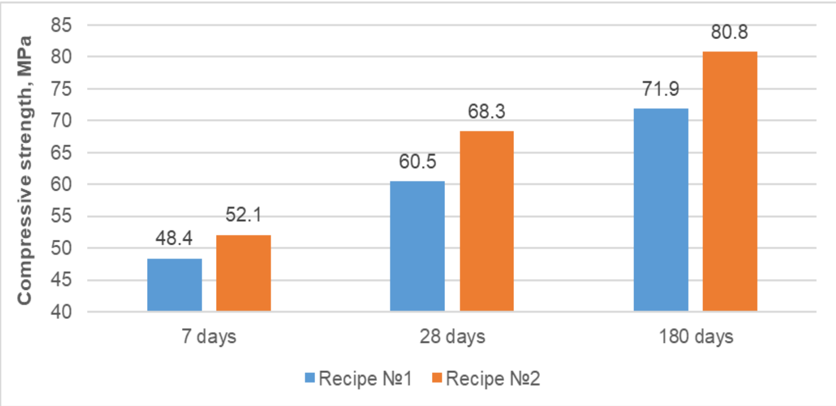

Figure 1. Compressive strength of concrete, MPa.

Table 6 and Fig. 1 show that the compressive strength of concrete with gneissic granite sand of fraction of 0–2.5 mm differs from the compressive strength with gabbro-diabase sand of fraction of 0–2.5 mm only by 12.9 % at the age of 28 days and 12.4 % at the age of 180 days. This is a rather nonsignificant difference considering the strength (crushability) of the sand. The grade of gneissic granite in strength (crushability) was only 200 according to Russian national standard GOST 8269.0-97. On the other hand, the grade of gabbro-diabase was 1400, which is the maximum strength (crushability) grade of crushed stone according to GOST 8269.0-97. By comparing absolute values, compressive strength of concrete with gneissic granite sand of fraction of 0–2.5 mm differs from the strength of concrete with gabbro-diabase sand of fraction 0–2.5 mm by 7.8 MPa at the age of 28 days, and 8.9 MPa at the age of 180 days.

Compressive strength values at the age of 7 days were determined as additional to characterize the concrete hardening process. At the age of 7 days, concrete with gneissic granite sand of fraction of 0–2.5 mm had 80 % of compressive strength compared to concrete compressive strength at the age of 28 days, on the concrete of gabbro-diabase sand of fraction of 0–2.5 mm had 76 % of compressive strength compared to concrete compressive strength at the age of 28 days.

However, following the regulatory documents for hydrotechnical concrete, it is possible to determine the grade strength at 180 days. Due to the operation of hydrotechnical concrete in a water-saturated state, the strength gain does not stop at the age of 28 days but continues to gain strength further.

The difference in compressive strength in 28 and 180 days corresponds to regulatory documents, which provide for increasing factors for analysing the strength gain of concrete after 28 days of maturing. This is also supported by numerous experimental data from other researchers [26–28].

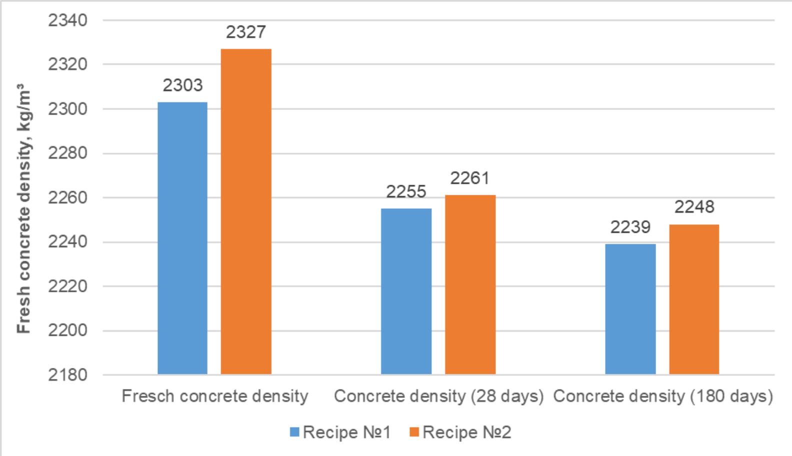

Figure 2. Fresh concrete density, kg/m³.

Table 6 and Fig. 2 show that the concrete density of all recipes is practically equal at the same age. The concrete density value is quite typical for sand concrete with coarse aggregate. This demonstrates that the structure of concrete is not disturbed when replacing one sand with another.

The high water absorption values of gneissic granite compared to gabbro diabase raised the author's concerns. This could affect the concrete mixture's flowability, rheology, and adhesion between the aggregate and the binder. However, as with coarse aggregate gneissic granite concrete [1], this does not significantly affect the flowability and rheology of the concrete. Furthermore, the introduction of toroidal nanoparticles (Astralene) led to a better penetration of suspension from water and binding materials deep into the crushed stone and directed the crystallization of cement stone. This mechanism lies in the fact that due to the shape of the torus, the Astralenes have. This allows the formation of volumetric structures several micrometers long in concrete, which is actually self-reinforcement of concrete. The mechanism is described in more details in the previous paper of the authors [29].

This moment also has a substantial positive effect. Gneissic granite crushed stone has relatively large pores, which leads to saturation with a suspension of water and binder materials. It also strengthens the crushed stone itself and stronger adhesion between the crushed stone and the binder.

The obtained freeze-thaw resistance value F400 according to Russian national standard GOST 10060-2012 is a very high class of freeze-thaw resistance even for concrete with high-quality aggregates. On the other hand, the freeze-thaw resistance class of concrete the most frequently used in civil engineering is class F50-150.

Concrete with freeze-thaw resistance class F400 belongs to the group of high freeze-thaw resistance. Concretes with this class of frost resistance belong to the group of special concretes. They are intended for use in exceptional cases. For example, such concretes are used when there is a variable level of water contacting the concrete structure in addition to low temperatures.

The high waterproofing class (maximum according to GOST 12730.5-2018) indicates that this concrete does not need additional water isolation. And even more, the concrete itself becomes a water isolation material. This is a significant factor, as it simplifies water isolation tasks during construction when temporary and financial resources are spent on additional water isolation.

A high class of freeze-thaw resistance, in sum with a high level of waterproofing, makes this one also more durable. When concrete is used in the north of Russia, the durability of concrete directly depends on its freeze-thaw resistance and waterproofing.

The pozzolan and finely ground additives used in this study have proven to be highly effective in improving the mechanical and operational characteristics of concrete. Their use as additional binders undoubtedly has a positive effect on the structure of concrete, as well as on its permeability and frost resistance. The effect of silica fume on concrete permeability has been considered in these papers [5, 30, 31]. Moreover, the used basalt microfiber has a complex positive effect on concrete properties, particularly on concrete's freeze-thaw and permeability.

These materials are also actively used by other researchers to obtain concretes with unique characteristics [32–34], as well as to reduce the amount of cement in the composition of concrete [35].

It should be noted that this study has initially been aimed at demonstrating the real possibility for developing high-strength concretes using low-quality aggregate. However, the matrix used provides extensive further concrete development opportunities for practical applications using solid industrial and construction waste, which meets the global challenges facing building in the 21st century [36]. Moreover, for the production of civil and even special-purpose concretes of low classes, it is possible to significantly decrease the amount of cement (up to 150–200 kg/m^3) using analogues of the developed matrix.

It also stands to mention that this paper was not aimed at studying the effect of nanomodifiers on the properties of concrete. The influence of nanomodifiers has already been studied in previous works by the authors of this paper and other researchers. Nanomodifiers were used as additives to concrete, such as silica fume, shale ash etc., the effectiveness of which has already been proven in concrete.

4. Conclusions

1. The properties of three types of cement available in large quantities in the northern regions of Russia were investigated. The most suitable for use in special concretes, in particular hydrotechnical, is the cement produced by MordrovCement PJSC since it has the least amount of free alkalis in terms of Na2O (0.78 %). And he also has the best indicators for the minimum concentration of chlorine ions (0.008 %), which is important for the operating conditions of reinforced structures [23, 37, 38].
2. The suitability of the proposed combination of pozzolan and fine-ground additives with modern construction chemistry for producing high-performance fine-grained concrete based on low-strength aggregates was tested.
3. High-strength fine-grained hydrotechnical concretes with a strength of 60.5 MPa at the age of 28 days on the basis of gneissic granite crushed sand were developed. Concrete strength increased up to 71.9 MPa after 180 days of maturation.
4. Obtained values of compressive strength of concrete based on low-quality gneissic granite crushed sand differed not too significantly from compressive strength of concrete made according to the same recipe using high-strength gabbro-diabase crushed sand of the same fraction. Compressive strength value was 68.3 MPa (28 days) and 80.8 MPa (180 days) for gneissic granite and gabbro-diabase crushed stone, respectively.
5. This nonsignificant difference in compressive strength of concrete compared to the huge difference in strength (crushability) of the crushed stone is due to the fact that in the case of using a low-strength aggregate, the main load was taken by the concrete matrix, not the aggregate. Due to the high value of water absorption of the crushed stone, crushed stone was saturated with a suspension of water and binder materials, which led to the strengthening of the crushed stone, as well as stronger adhesion to the concrete matrix, which made it possible to distribute the load more evenly.
6. The developed matrix makes it possible to produce high-strength concrete in hard-to-reach and remote regions with difficult access to high-strength concrete aggregates. It also makes it possible to obtain high-strength concretes when using aggregates from industrial and construction waste, which meets the global challenges facing construction in the 21st century [36].
7. The grade F400 of freeze-thaw resistance concrete is a severe achievement since a low-strength aggregate of gneissic granite was used. Moreover, the frost resistance of crushed stone was only 200 cycles. Frost resistance is a crucial characteristic of concrete for the construction of buildings and structures in the northern regions, as this determines the durability and reliability of the design.
8. The combination of high compressive strength, low water absorption value, high water waterproofing class and high freeze-thaw resistance makes the developed concrete unique since a low-strength aggregate was used.

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