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High performance lightweight concretes for 3D printing

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Abstract. The paper is devoted to developing high-performance nanostructured concrete for 3D printing and studying its strength and operational characteristics. The research addresses issues related to the use of modern concrete chemistry, such as plasticisers and concrete hardening accelerators, and nanomodification by two types of nanocarbon: nanotores (Astralene) and sulphur graphene (Ugleron). The designed lightweight concrete for 3D printing has a compressive strength of about 70 MPa and 9 MPa after extrusion at a density of about 1.55–1.6 kg/m^3. Pozzolan additives from industrial waste in concrete are used, such as silica fume and oil shale ash. Microspheres, which are also industrial wastes, are used as lightweight aggregate and thixotropy regulators. As a result, lightweight, high-performance concrete for 3D printing was developed, which allows the disposal of industrial waste inside it.

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1. Introduction

The rapid development of a digital economy requires an equally quick transition of industrial production to robotic technologies with digital control [1, 2]. This also applies to such an inertial sector of the industry as construction is.

Such a tool can be the introduction of 3D printing technology in construction [3, 4].

The introduction of 3D printing in construction will reduce labour costs by automatization [5]. In this case, for the manufacturing of concrete walls, for example, there is no need to work with several masons teams, but the operator who will monitor the building process and prepare the concrete mixture is enough. Moreover, it is possible to reduce the number of materials used in construction due to optimizing the geometry of structures[6]. In addition, account must be taken of that automatization of construction is not only a matter of cost reduction. It is a hard and traumatic job, where there are not rarely fatalities. Automatization of even a part of the processes will reduce injuries and even save human lives.

These actions will significantly increase the environmental friendliness of construction and relieve the environmental burden from the construction region. It is worth pointing out that cement production is responsible for 5–9 % CO_2 emissions [7]. Moreover, massive waste is formed in construction, especially when utilizing old buildings [8, 9].

With the advent and development of 3D printers and their penetration into the technology of forming products made of polymers and metals [10, 11], 3D printing in construction technologies also began to develop [12, 13]. However, each such transition becomes possible only after the creation and development of the industry producing the related specialized materials such as unique thermoplastics for digital printing of plastic parts and special powders [14], tapes, and wires for the technology of printing metal parts [15]. The same new materials are also required for printing structures in construction. It would seem that the properties of the traditional compositions based on mineral binders (concretes) are very close to the tasks of 3D printing in construction technologies [16–18]. Industrial concretes based on Portland cement, aluminous (high alumina), sulphoaluminate and slag-portland cement binders according to the current regulatory and technical base, do not have (in their majority) the ability to set quickly (in less than 30 minutes) [19]. Also, they cannot promptly gain hardness, providing an intensive increase in crack resistance and stability of the shape of the cross-section of the concrete jet which is being laid.

In this regard, it is advisable to use concrete hardening accelerators that accelerate the concrete setting and contribute to an accelerated set of strength in the first hours of hardening. However, hardening accelerators should not lead to cracking and other problems when used [20]. The cause of cracks in such situations is typically the unequal start of hardening or overheating of concrete. In the case of concrete printing 3D, you should not fear overheating concrete. When using this technology, massive monolithic structures of tens of cubic meters of concrete are not formed. Multilayer structures in 3D printing are used that allow excess heat to leave the concrete.

As a result of the information given above, it becomes a highly urgent task to develop and organize materials (dry admixes of raw materials) for concrete mixtures intended for industrial 3D printing of reliable and safe buildings and structures.

The primary purposes of this work and tasks of its experimental part were as follows:

- select suitable additive materials to develop concrete formulations for 3D printing;
- to develop some variants of working formulations of concrete mixtures for 3D printing;
- to conduct studies on the workability and effectiveness of the selected base materials in the composition of experimental working formulations of concrete mixtures for 3D printing;
- to determine mechanical and operational characteristics, as well as strength gain dynamics.

2. Methods

2.1. Used materials

In this research, the authors are focused on Portland cement as a primary binder which is the most affordable and relatively cheap in mass production. Still, they took it without additives, with a minimum amount of free alkalis in Na_2O (not more than 0.8 % of the mass). Such cement is currently represented in Russia only by Portland cement CEM I 42.5 N, produced by PJSC Mordovcement and portland cement CEM I 42.5 N produced by LLC Asia-Cement (Penza). All other cement manufacturers cannot provide a high quality of the composition and characteristics of their products, especially in terms of exceeding the permissible amount of free alkalis, which adversely affects the freeze and thaw resistance of concrete and on their longevity.

However, in this research, the choice was favouring cement manufactured by PJSC Mordovement since it is more accessible in Russia's northern and central regions.

Active microcrystalline silica fume manufactured by pilot production of the Irkutsk National Research Technical University (IRNITU) and electro filter slate fly ash “Zoles Bet” obtained from State District Electric Station (SDES) in Narva were used as pozzolanic and fine-grained additives. Modified basalt microfiber by TC 5761-014-13800624-2004 was used for dispersal reinforcement. Carbon torus-like nanoparticles of the fulleroid type Astralenes® by TC 2166-001-13800624-2003 were used to control the properties of cement stone crystallization and its micro-dispersion self-reinforcement. Ash aluminosilicate cenospheres (from Kemerovskaya CHPP) with average diameters of 100-130 microns were used to control the thixotropy of the concrete mixture. High-efficiency antifreeze additive NitCal® was used as an accelerator for setting and obtaining high strength values. The water quantity was reduced with the help of the carboxylate plasticizer REOMAX PC 3901P manufactured by KUBAN-POLYMER LLC with admix of hydrophilic sulphur adduct carbon nanoclusters Ugleron® (TC 2166-049-91957749-2011), which has been used for these purposes in [21].

Aluminosilicate cenospheres were used to control the thixotropy of the concrete mixture. Aluminosilicate hollow microspheres are glass-crystalline aluminosilicate cenospheres that are formed during the high-temperature flaring of the coal. They are the most valuable components of ash waste from thermal power plants. They are hollow, almost ideal in shape silicate balls with a smooth surface, with a diameter of 10 to several hundred micrometers, on average about 100 micrometers. The microspheres walls are solid non-porous with a thickness of 2 to 10 microns, a melting point of 1400–1500 °C, a density of 580–690 kg/m^3. The inner cavity of the particles is filled mainly with nitrogen and carbon dioxide.

2.2. 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 No. 1

Components	recipe No. 1	recipe No. 1
	Kg per m^3	
Portland Cement CEM I 42.5 N manufactured by JSC Mordovcement	552	
Gabbro diabase manufactured by Lysaya Gora Ltd. (fraction of 0 – 2.5 mm)	405	397
Dry admix of:		
Silica fume manufactured by pilot production of the INRTU [22]		
Shale ash Zolest-bet manufactured by PCV LLC		
Modified basalt microfiber manufactured by NTC of Applied Nanotechnologies	396	
Aluminosilicate cenospheres (from Novo-Kemerovo CHPP)		
Plasticizing agent REOMAX PC 3901P manufactured by KUBAN-POLYMER LLC		
Carbon nanoparticles Astralene manufactured by NTC of Applied Nanotechnologies		
Antifreeze additive NitCal®		
Modified basalt microfiber manufactured by NTC of Applied Nanotechnologies	–	8
The water of mixing according to GOST 23732-2011	220	220

The concrete was mixed as follows. Water, binding materials and plasticizing agent were first incorporated in the concrete gravity batch mixer Eco CM-71 for one minute. After that, fine aggregates were introduced and mixing lasted another minute. Fiber and Astralene were introduced last into the concrete mixture. The final mixing also lasted for one minute.

Nanomodifiers (Astralene) were introduced into concrete by serial dilution method [23]. An aqueous suspension of Astralenes was made, which was added to the concrete mixture using the laboratory homogenizer I100-840 LLC Ultrasonic Technique.

The volume of each batch of concrete mixing was 35 litres.

Concrete density was determined according to Russian State Standard GOST 10181-2014.

Concrete cubes with dimensions of 100×100×100 mm in the quantity of 15 pieces and concrete prisms with dimensions of 100×100×400 mm in the amount of 3 samples were made according to Russian State 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 according to Russian State Standard GOST 10180-2012.

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

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

Figure 1. Extrusion Equipment.

The extrusion was carried out using the construction syringe Long Blast and the Injection screw pump KRIN-706 PRO.

Control samples of extrusion concrete were made as follows: fine-grained concrete was fed into the pump funnel. Concrete was pumped through a flexible pipe with a diameter of 3 cm and a length of 2 meters. Extrusion was performed through a rectangular nozzle of 2.5 cm × 0.5 cm. Extrusion was carried out by the "layer by layer" method [24]. Prisms were filled with concrete perpendicular to the application of the load during the mechanical testing.

Moreover, in samples with fiber, this was also done to create the effect of fiber orientation [25]. Next, the forms with concrete were compacted using vibration according to Russian State Standard GOST 10181-2014. During concrete compacting, additional concrete was supplied to fill the resulting space using the construction syringe Long Blast.

The test load was applied perpendicular to the direction of laying.

3. Results and Discussion

The main concrete properties are given in Table 2. And Table 2 shows how extrusion, fiber, concrete hardening accelerators influence the main concrete properties.

Table 2. Main properties of concrete.

Properties	Units	Without fiber			With fiber	
		Without Nitcal	Before extrusion	After extrusion	Before extrusion	After extrusion
Compressive strength (12 hours)	MPa	9.8	15.9	17.5	16.4	17.9
Compressive strength (1 day)	MPa	30.1	39.6	42.4	40.4	43.1
Compressive strength (2 days)	MPa	43.1	48.3	51.2	48.7	52
Compressive strength (3 days)	MPa	46.8	51.9	54.3	52.3	55.1
Compressive strength (7 days)	MPa	51.4	56.3	60.1	56.7	61
Compressive strength (21 days)	MPa	60.7	62.1	66.2	62.6	66.8
Compressive strength (28 days)	MPa	65.4	64.8	69.6	65.7	70.9
Flexural strength (28 days)	MPa	7.3	7.1	8.9	7.7	10.2
Fresh concrete density	kg/m³	1568	1573	1609	1587	1621
Concrete density (28 days)	kg/m³	1521	1529	1554	1541	1572
Waterproofing	Class	W18	W18	W20	W20	W20
Flowability	Class	Self-Compacting				

The use of basalt microfiber led to an increase in the strength of concrete before extrusion. The compression strength of concrete increased from 64.8 MPa to 65.7 MPa (+ 1.4 %), and the flexural strength from 7.1 MPa to 7.7 MPa (+ 8.4 %).

It can be seen from Table 2 the concrete strength enhancement after extrusion. The compression strength of concrete without fiber increased from 64.8 MPa to 69.6 MPa (+ 7.4 %), and the flexural strength from 7.1 MPa to 8.9 MPa (+ 25 %). The compressive strength of fiber reinforced concrete increased from 64.8 MPa to 70.9 MPa (+ 9.4 %), and the flexural strength from 7.1 MPa to 10.2 MPa (+ 42 %). It should be noted that the concrete was placed in the molds perpendicular to the direction of application of the load during the tests, which led to the orientation of the fibers in the direction of extrusion [25]. This explains such a significant increase in flexural strength with a relatively low fiber content in concrete.

The extrusion improves the structure of concrete rock, leading to an increase in its strength. The basalt microfiber serves as a dispersed micro-reinforcement, leading to the concrete strength enhancement, especially flexural strength enhancement. During concrete extrusion, the fiber is oriented in the extrusion direction. It led to a significant increase in the strength of the concrete in the plane perpendicular to the extrusion direction. The orientation of the fiber during extrusion has also been established by other researchers [26–28].

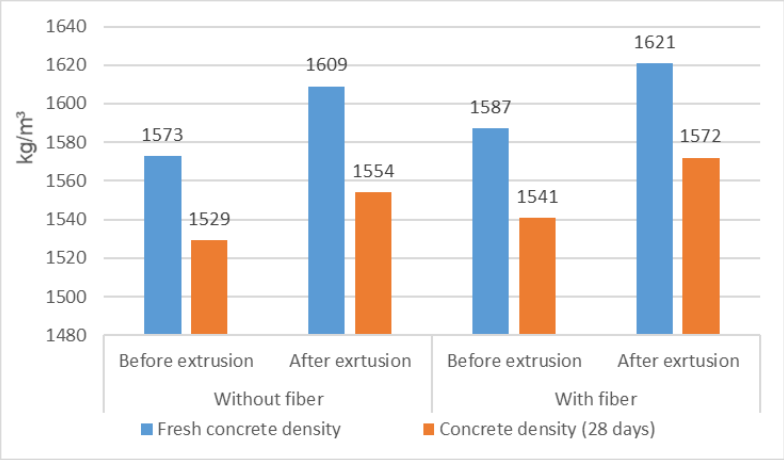

Figure 2. The concrete density.

In the Fig. 2, it can be seen that the density of concrete after extrusion increases by about 35 kg/m^3. During extrusion, air bubbles are removed, which are involved in the concrete mixture during the mixing process. This suggests that after extrusion, the string-round of concrete is improved and the interaction between the matrix and the inert aggregate is improved. Also worth noting is that after extrusion, the waterproofing of concrete increased by the next class (from W18 up to W20) and reached the highest class W20. This also indicates that the structure of the concrete after extrusion is improved as its permeability is reduced. In turn, it should lead to an increase in the durability and operational properties of concrete.

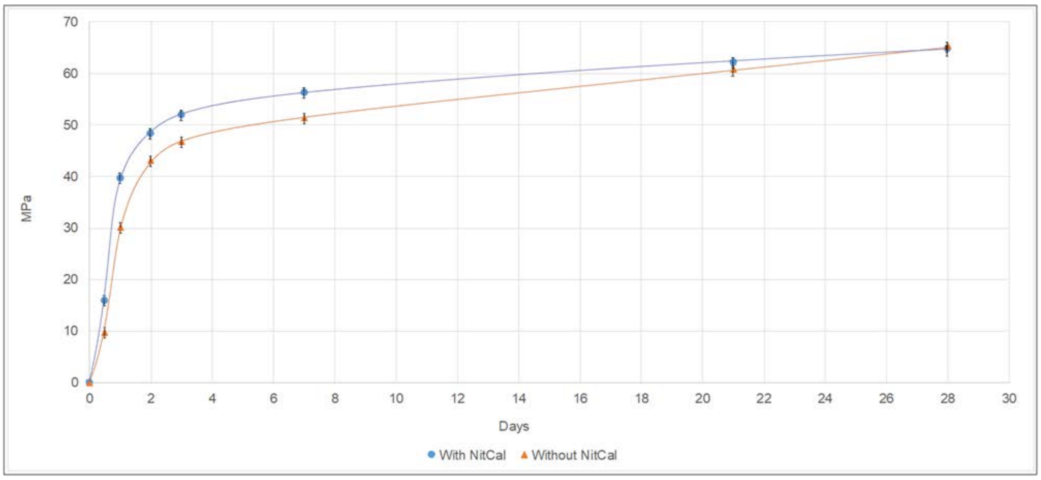

Figure 3. Concrete hardening dynamic.

Fig. 3 shows that the strength of concrete in the presence and absence of a hardening accelerator agent differs, especially in the first two days. The concrete gains 24 % of the class strength (28 days of hardening) in the first 12 hours when using a strength gain accelerator. And without Nitcal using the concrete gains only 15 % of class strength. After a day of hardening, concrete gains 61 % of class strength by using a hardening accelerator and only 46 % of class strength when no hardening accelerators were used. The gap between values in the process of time is reduced, and on the 28th day, the strength of concrete without Nitcal slightly exceeds the value of concrete strength with NItcal. The authors of this research [20] also concluded in favour of using a concrete hardener based on calcium nitrite, since alternative versions led to the appearance of cracks or other defects.

4. Conclusions

1. Nanostructured concrete formulations with high mechanical and operational characteristics were developed, and strength gain dynamics were investigated.

2. The use of a calcium nitrite hardening accelerator has resulted in the accelerated hardening of concrete in the early stages. In addition, this did not lead to any defects in concrete, such as cracking. This is consistent with the results of other researchers who have concluded that it is advantageous to use a concrete hardening accelerator based on calcium nitrite [20].

3. On day 28, the strength of concrete without fiber before extrusion was 64.8 MPa, 65.7 MPa and 65.4 MPa using a hardening accelerator (Nitcal).

4. It has been found that after extrusion, the strength of concrete increases significantly. This is because excess air is removed from the concrete mixture. There was an increase in concrete strength in the current research by 7.4 % (from 64.8 MPa to 69.6 MPa), and when using fiber by 7.9 % (from 65.7 MPa to 70.9 MPa).

5. The developed concrete can be safely called eco-friendly since it has many industrial waste products, such as silica fume, shale ash, aluminosilicate cenospheres. This reduces the impact of industrial waste on the region.

6. In terms of strength and operational properties, the developed concrete surpasses most founded analogues with a similar density. For example, this research [27] developed a concrete with similar strength values but with higher density values (1.7 – 1.8 t/m³).

7. The developed concrete is suitable for extrusion and further development of lightweight concrete structures for 3D printing.

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