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Fine-grained cement-ash concrete for 3D-printing

L.I. Dvorkin ✉***, V.V. Marchuk*** ***, V.V. Zhitkovsky***

National University of Water and Environmental Engineering, Rivne city, Ukraine

✉ *dvorkin.leonid@gmail.com*

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Abstract. An analysis of the main types of basic building mixtures for 3D printing and the requirements for their basic properties is presented. Using the mathematical planning of experiments, a complex of polynomial models of the properties of cement-ash fine-grained concrete mixtures for 3D printing was obtained. These properties included their "open time" extrusion suitability, the structural strength required for layering the mixture after a certain time after mixing, tensile strength during splitting characterizing the adhesion of layers and the compressive strength of extruded concrete. As mineral admixtures, the concrete included fly ash and the hardening accelerator: building gypsum. A laboratory printer was designed and used for the research. On the basis of the obtained models, an analysis of the factors of the composition of cement-ash concrete mixtures for the investigated properties is carried out. The article shows a solution to the optimization problem according to the criterion of the minimum cost of the composition of a fine-grained concrete mixture for 3D printing using computer software.

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1. Introduction

A promising technology in construction production is 3D printing, which allows three-dimensional objects to be created by sequential application of layers of building mixture. 3D-printing allows to minimize the duration of the technological process of erection of structures, the cost of materials and labor, to ensure the creation of various volumetric shapes [1–12].

One of the difficult tasks arising in the implementation of 3D printing is the development of concretes and mortars that take into account the peculiarities of the 3D printer used, the technological requirements for the building mixture and the necessary physical and mechanical properties of the material at various stages of its hardening.

The most common materials used in 3D printing in the construction of buildings and structures are fine-grained fast-hardening mixtures that include various admixtures to improve certain characteristics, various types of fibers can also be introduced [3, 5, 9–18]. There is experience of using several types of concrete mixes [14]. The first one includes mixtures with water-binder ratio (W/B) 0.4–0.5 and superplasticizers to adjust the consistency. Their compressive strength after 28 days reaches 50–60 MPa. The second type of mixtures with W/B ≈ 0.23–0.3 contains silica fume, the required consistency is achieved by adding a superplasticizer. The compressive strength of these concretes reaches 125 MPa at 28 days of age. The use of such mixtures is restrained by the scarcity and high cost of silica fume.

An urgent problem in the design of concrete mixtures is to provide a set of necessary material properties when using available technogenic raw materials.

Table 1. The main indicators of the physical and mechanical properties of building mixtures and concretes based on them for 3D printers

№	Properties	Functional purpose	Index
1	Properties of mixtures		
1.1	Water holding capacity,%	Ensuring the homogeneity of the concrete mix during production, transportation through pipes and extrusion	No less 97
1.2	Delamination,%		No more 10
1.3	Viscosity, Pa·s	Providing the possibility of transportation through pipes and stable dosing of the mixture	10^9–10^{12}
1.4	Structural strength, Pa	Ensuring the necessary strength of the mixture, sufficient for laying the next layer	No less 800
1.5	Initial setting time, min	Providing the possibility of laying the layer	No less 20 after kneading
1.6	Mixture fluidity, by immersion of the cone, cm	Ensuring the mix's extrusion ability and stable geometry	8…12
	by Suttard viscometer, mm		150…170
2	Concretes properties		
2.1	Compressive strength, MPa	Ensuring the strength properties of structures	No less 20
2.2	Average density, kg/m^3	Providing thermal insulation and other physical and mechanical properties	600–2000
2.3	Adhesion, MPa	Provides adhesion between layers of the mixture for strength and uniformity of construction	No less 2
2.4	Thermal conductivity, W/(m·K)	Thermal insulation of building elements	No more 0.7
2.5	Frost resistance, cycles	Ensuring the durability of structures and resistance to alternating temperature changes	No less 100

Analysis of the results of the performed studies allows to establish the recommended [2] range of technical requirements for building mixtures for 3D printing (Table 1).

The properties of concrete mixture and concrete that determine the possibility of 3D printing include the necessary workability, structural strength after a certain time of hardening of the extruded layer, tensile strength at splitting, which characterizes adhesive strength of layers, as well as compressive strength.

Currently no standardized methods for determining the required properties of concrete mixtures for 3D printing. A number of studies have proposed requirements for the individual properties of the mixtures used [14, 18–24].

Workability (formability) is the rheological parameter of the consistency of the mixture over time. To determine the manufacturability of the extruded mixtures, can use some standard techniques, for example, Le [19] used the British test BS1337-9: 1990 and Kazemian [20] American standard ASTM C1437-15. For this purpose the Hagerman mini-cone is used [14].

Open time, or "window of opportunity for printing" is the period during which the mixture can be extruded. The mixture must also have certain adhesive properties for layering. In [19, 21], the open time was determined by measuring the shear stress in accordance with British standard BS1377-9: 1990 and German DIN 398. In another work [22] it was proposed to test open time using the V-funnel method, Vic's apparatus, shaking table and mini-cone immersion test.

The ability to build up, that is, to lay it in layers during the formation of mixtures, is determined by its structural strength after a certain period of time. To determine the ability of the mixture to build up, a method was proposed for determining the deformation when a load is applied to the sample [14]. This parameter correlates with the workability and extrudability of the mixture.

To ensure the necessary properties of mixtures for 3D-printing it is necessary to develop a methodology for the design of their compositions.

The purpose of the research was to develop a method for designing compositions of fine-grained concrete mixtures containing the mineral admixtures, providing a set of necessary properties for 3D printing.

2. Methods

In our studies, cement CEM I 42.5R of Cement Plant Dyckerhoff Ukraine, fly ash from Burshtynskaya thermal power plant, which belongs to type II ash of category B with a residue on a sieve with a mesh size of 45 µm no more than 25 % (class 2) (EN 450-1:2012) and quartz sand with a modulus fineness of

M_{fn} = 2.1 meeting the requirements of Russian standard GOST 8736-2014 were used. A polycarboxylate type superplasticizer was used (0.3 % by weight of the binder). As an accelerator for setting and hardening of the mixture, we used gypsum G-5 A II GOST 125-2018.

For determining the properties of cement-ash concrete mixtures was developed and used a laboratory 3D printer (Fig. 1).

The required water amount of was selected to obtain mixtures suitable for extrusion molding; the required workability of the mixture by immersion of a standard cone was within – 10 cm. The structural strength of the concrete mixture was determined using a device (Fig. 2), allowing allows measuring the specific loads in Pa (Fig. 3), which the mixture withstands after a certain period of time required to form one layer.

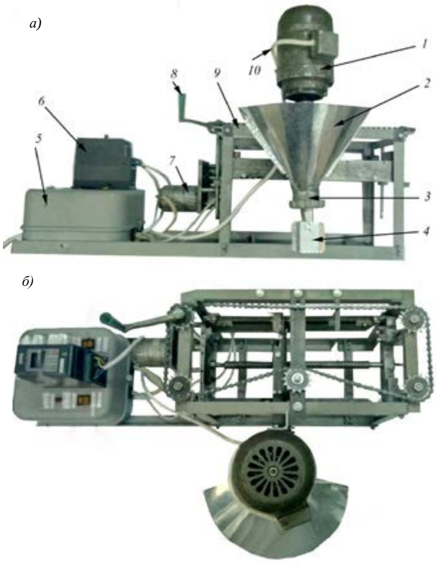

Figure 1. Laboratory printer a) front view, b) top view
1 – electric motor of the extruder; 2 – hopper of building mixture; 3 – auger; 4 – mouthpiece; 5 – control panel; 6 – frequency converter of electricity; 7 – reverse motor moving the extruder in the horizontal direction; 8 – manual drive moving the extruder in the vertical direction; 9 – frame; 10 – power cable of electric motors.

Figure 2. Device for determining the structural strength of an extruded layer of concrete mix.

a) *b)*

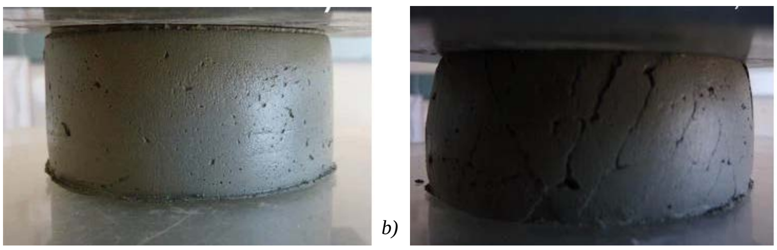

Figure 3. An example of determining the structural strength of extruded concrete,
a) the sample withstands the load (structural strength is provided), b) the sample is destroyed.

3. Results and Discussion

To study the effect of the composition of building mixtures on the their main properties, algorithmic experiments were performed in accordance with the three-level three-factor plan B_3 [25]. The conditions for planning experiments are given in Table 2.

Table 2. Conditions for planning experiments

Factors: Natural	Factors: Coded	Levels of variation: –1	0	+ 1	Interval
Content of cement-ash binder, kg/m³, (B)	x_1	400	600	800	200
Content of fly ash in the binder mixture (A),% by weight	x_2	40	30	20	10
Gypsum content (G),% by binder weight	x_3	0	5	10	5

As a result of statistical processing of experimental data, adequate polynomial models of the investigated properties were obtained with a 95 % confidence level.

General view of the models:

$$y = b_0 + \sum_{i=1}^{k} b_i x_i + \sum_{i=1}^{k} b_{ij} x_i x_j + \sum_{i=1}^{k} b_{ii} x_i^2 \quad (1)$$

where b_i, b_{ii}, b_{ij} is statistical estimates of the regression coefficients; x_i, x_j is factors taken into account; n is the number of factors.

The coefficients of the obtained mathematical models are given in Table 3.

Table 3. Cement-ash concrete properties polynomial mathematical models coefficients

Coefficients	Initial setting time, min	Structural strength after mixing and hardening, Pa: 10 min	25 min	40 min	Tensile splitting strength, MPa: 1 day	7 days	28 days	Compressive strength, MPa: 1 day	7 days	28 days
b_0	71.5	3083	4703	7845	3.2	7.6	11.4	20.8	41.7	56
b_1	–10.0	288	342	548	1.16	1.6	2.4	10.2	11.8	14.9
b_2	–6.0	553	888	1333	0.34	0.4	0.78	4.12	4.66	4.25
b_3	–20.0	831	885	1452	0.8	0.7	0.7	6.47	3.10	4.1
b_{11}	0.2	126	184	296	0.07	–0.5	–0.4	2.44	–1.69	–1.2
b_{22}	5.2	176	261	423	–0.01	0.1	–0.03	–0.36	-0.5	0.31
b_{33}	–10.2	149	163	260	–0.03	–0.4	–0.3	2.09	–0.25	1.41
b_{12}	–2.5	133	188	131	0.14	0.2	0.43	1.08	2.59	2.20
b_{13}	–2.5	230	117	167	–0.06	–0.6	–0.37	1.00	–2.10	–0.9
b_{23}	2.3	192	235	208	0.04	–0.1	–0.04	–0.78	–1.53	0.15

Based on the data on water demand in l/m³ of a mixture with the required workability and tested in accordance with the accepted planning conditions, a model was obtained:

$$W = 197 + 28.4x_1 + 3.3x_2 + 5.2x_3 + 13.9x_1^2 + \\ + 2.4x_2^2 - 5.1x_3^2 + 0.9x_1x_2 + 2.9x_1x_3 + 0.9x_2x_3. \quad (2)$$

Analysis of the obtained coefficients of mathematical models (Table 3) and graphical dependencies (Fig. 4–9) allows to obtain important technological conclusions.

The determining factor for the beginning of setting and the structural strength of the studied mixtures is the content of gypsum and fly-ash in the binder mass (Fig. 4, 5). For the splitting tensile strength of concrete, the content of the binder and part of the ash in its have a decisive influence (Fig. 6, 7). In a joint analysis of the models of compressive strength and water demand of a concrete mixture, as expected, the most influencing factors are the binder content and part of the ash in it. For the early strength of concrete, the content of gypsum is essential (Fig. 8, 9).

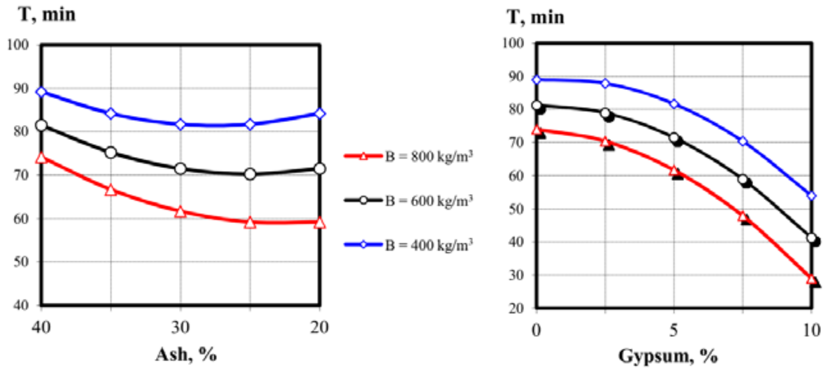

Figure 4. Graphical dependences of the mixtures initial setting for a 3D printer.

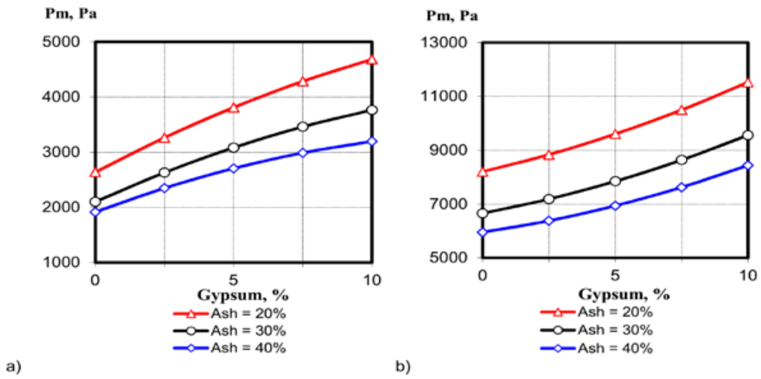

Figure 5. Graphical dependences of structural strength after 10 (a) and 40 (b) minutes of hardening of mixtures.

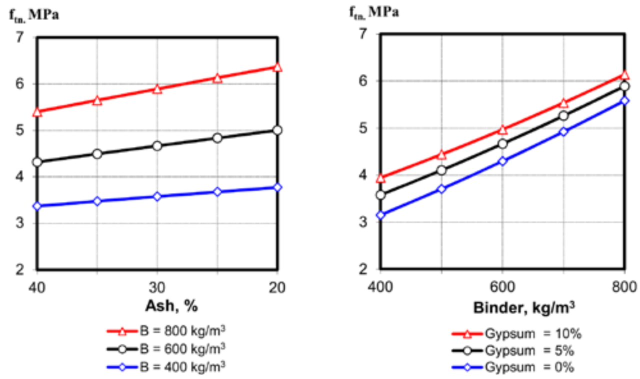

Figure 6. Graphical dependences of tensile strength at splitting at the age of 1 day.

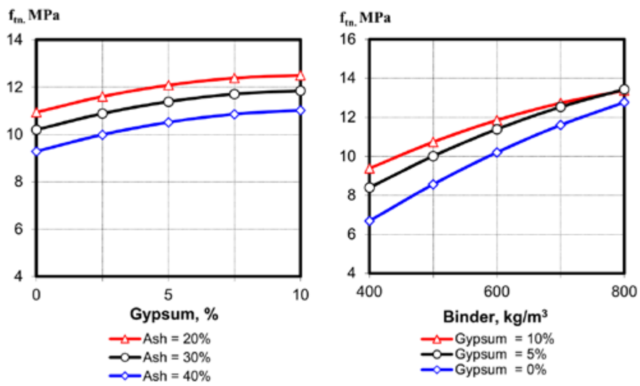

Figure 7. Graphic dependences of tensile strength at splitting at the age of 28 days.

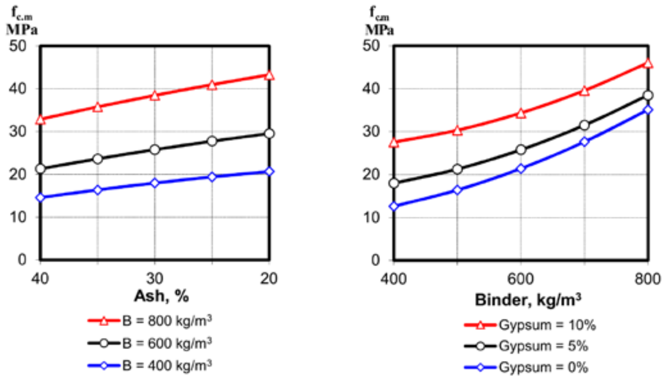

Figure 8. Graphical dependences of the compressive strength of mixtures at the age of 1 day.

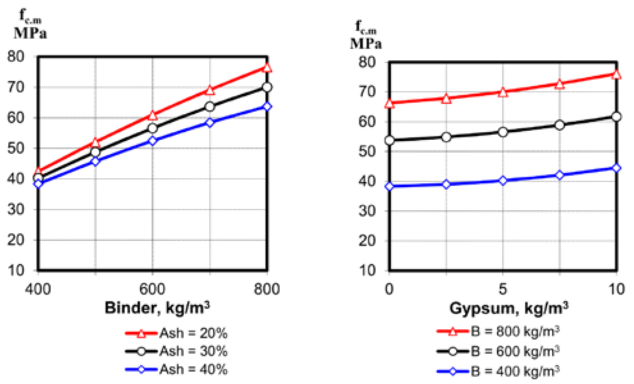

Figure 9. Graphical dependences of the compressive strength of mixtures at the age of 28 days.

The design of cement-ash concrete compositions for 3D concreting can be performed using the minimum cement consumption or minimum cost as the criteria for optimality [26].

At high cost of the admixtures, the optimal compositions of concrete mixtures by these two criteria may not coincide. In general, for a cement-ash grained concrete mixture, while minimizing its cost, the condition must be met:

$$C_c = C_{Cem} \cdot Cem + C_A \cdot A + C_G \cdot G + C_{Add} \cdot Add + C_S \cdot S \to \min \tag{3}$$

given that

$$\begin{gathered} P_1 \geq f\left(x_1, x_2, ..., x_n\right) \\ P_2 \geq f\left(x_1, x_2, ..., x_n\right) \\ P_n \geq f\left(x_1, x_2, ..., x_n\right) \\ x_1 \ldots x_n \in \left[a \ldots b\right], \end{gathered} \tag{4}$$

where C_c, C_{Cem}, C_A, C_G, C_{Add}, C_S is respectively the cost of concrete mixture, cement, fly ash, gypsum, admixtures and sand, for example in RUB/kg; Cem, A, G, Add, S is consumption of cement, fly-ash, gypsum, admixtures and sand, kg/m³; $P_1 \ldots P_m$ is set quality indicators of the mixture and concrete based on it; $x_1 \ldots\ x_n$ are the factors of the mixture composition taken into account in the models; a, b is restrictions on possible values of factors.

The conversion of the values of the parameters of the composition of the mixture, normalized in this study (Table 2) into a coded form for calculations by models was carried out according to the following dependencies:

$$x_1 = \frac{B-600}{200};\ x_1 = \frac{A-30}{10};\ x_1 = \frac{G-5}{5}. \tag{5}$$

The consumption values of fine aggregate (sand) are found by the formula:

$$S = \left(1000 - \left(\frac{Cem}{\rho_{Cem}} + \frac{A}{\rho_A} + \frac{G}{\rho_G} + \frac{W}{\rho_W}\right)\right) \cdot \rho_S. \quad (6)$$

where ρ_{Cem}, ρ_A, ρ_G, ρ_W and ρ_S are, respectively, the densities of cement, fly ash, gypsum, water and sand.

For materials used in the study ρ_{Cem} = 3.1 g/cm³, ρ_A = 2.9 g/cm³, ρ_G = 2.4 g/cm³, ρ_W = 1.0 g/cm³, ρ_S = 2.65 g/cm³.

The most rational way to solve this problem is to use the Microsoft Excel software, in particular, its "Search for a solution" application. At the given values of the indicators of the properties and costs of its components under certain restrictions on the values of the factors (in coded values from –1 to +1), the computer program enumerates the possible combinations of factors and parallel, determines, in order to achieve the minimum cost of the mixture, the water consumption necessary to ensure the required fluidity of the mixture according to the corresponding regression equation.

Example

Determine the composition of the cement-ash fine-grained concrete mixture for a 3D printer with compressive strength at the age of 1 and 28 days 15 MPa and 40 MPa, respectively, the tensile strength at splitting at the age of 28 days is 8 MPa, the initial setting time 55 min and structural strength after 10 min hardening 3300 Pa.

The characteristics of the used materials are given above.

We accept the cost of the main components of the mixture for a 3D printer as follows, RUB/kg: C_{Cem} = 5.2; C_A =1.5; C_G = 4.5; C_{Add} = 700; C_S = 1.

1. Using the experimental statistical models (Table 3) and substituting the values of the normalized parameters into them, we obtain the constraint functions (4):

The initial setting time, min

$$71.5 - 10.0x_1 - 5.0x_2 - 20x_3 + 0.2x_1^2 + 5.2x_2^2 - 10.2x_3^2 - 2.5x_1x_2 - 2.5x_1x_3 + 2.5x_2x_3 = 55$$

Structural strength after 10 minutes of mixture hardening, Pa

$$3083 + 288x_1 + 553x_2 + 831x_3 + 126x_1^2 + 176x_2^2 + 149x_3^2 + 133x_1x_2 + 230x_1x_3 + 192x_2x_3 \geq 3300$$

Tensile strength at splitting at the age of 28 days, MPa

$$11.4 + 2.4x_1 + 0.78x_2 + 0.7x_3 - 0.4x_1^2 - 0.3x_3^2 + 0.43x_1x_2 - 0.37x_1x_3 \geq 8$$

Compressive strength at the age of 1 day, MPa

$$20.8 + 10.2x_1 + 4.12x_2 + 6.47x_3 + 2.44x_1^2 - 0.36x_2^2 + 2.1x_3^2 + 1.08x_1x_2 + 1.0x_1x_3 + 0.78x_1x_3 \geq 15$$

Compressive strength at the age of 28 days, MPa

$$56.0 + 14.9x_1 + 4.25x_2 + 4.0x_3 - 1.2x_1^2 - 0.31x_2^2 + 1.41x_3^2 + 2.2x_1x_2 - 0.9x_1x_3 + 0.15x_1x_3 \geq 40$$

2. In equation (3) we substitute the value of the cost of the components of the mixture, and also set the constraints on the values of the factors: from –1 to 1 (in coded form).

3. Using the software application "Search for a solution", find the values of the factors that satisfy the accepted constraints, and minimize the total cost of the mixture: x_1 = –0.8; x_2 = –0.78; x_3 = 1. With such values of the factors according to coefficients regression equation (Table 3) T = 55 min and Pm_{10} = 3340 Pa, which corresponds to the required parameters. It should be noted that ensuring the required time for the initial setting time of the mixture setting required a certain increase in other normalized strength indicators, f_{cm}^{1} = 19.8 MPa, f_{cm}^{28} = 47.7 MPa and f_{tn}^{28} = 9.6 MPa.

4. The values of factors in natural form are determined by the equations (5):

$$Binder = -0.8x_1 + 600 = -0.8 \cdot 200 + 600 = 440 \text{ kg/m}^3$$

$$Fly\ ash = -0.78x_2 + 30 = -0.78 \cdot (-10) + 30 = 37.8\,\%$$

$$Gypsum = x_3 + 5 = 1 \cdot (5) + 5 = 10\,\%$$

5. Calculated nominal composition of the cement-ash mixture without taking into account the water consumption:

$$Fly\ ash = 440 \cdot 0.378 = 166.3 \text{ kg/m}^3$$
$$Cem = 440 - 166.3 = 273.7 \text{ kg/m}^3$$
$$Gypsum = 0.1 \cdot 440 = 44 \text{ kg/m}^3$$
$$Additive\ SP = 440 \cdot 0.003 = 1.32 \text{ kg/m}^3$$

6. Find the water consumption by the equation (2):

$$W = 197 + 28.4 \cdot (-0.) + 3.3 \cdot (-0.78) + 5.2 \cdot 1 + 13.9 \cdot (-0.8)^2 + 2.4 \cdot (-0.78)^2 - 5.1 \cdot 1^2 +$$
$$+ 0.9 \cdot (-0.8) \cdot (-0.78) + 2.9 \cdot (-0.8) \cdot 1 + 0.9 \cdot (-0.78) \cdot 1 = 170.6\, l / m^3$$

7. Sand consumption from equation (6):

$$S = \left(1000 - \left(\frac{273.7}{3.1} + \frac{166.3}{2.9} + \frac{44}{2.4} + \frac{170.6}{1}\right)\right) \cdot 2.65 = 1763 \text{ kg/m}^3$$

8. The value of the minimum possible cost of 1 m3 of the mixture, excluding the cost of water (found during iterations using the software application MS Excel "Solver"):

$$C_c = 5.2 \cdot 273.7 + 2 \cdot 166.3 + 4.9 \cdot 44 + 700 \cdot 1.32 + 1 \cdot 1763 = 4658.4\, RUB$$

4. Conclusions

1. Taking into account the peculiarities of the manufacture in the construction of structures using a 3D printer, the necessary properties of extruded mixtures and concretes based on them have been determined. Methods for determining the normalized properties of the concrete mixture, the design of the laboratory printer and the device for determining the required structural strength during its layer-by-layer laying are proposed.

2. A set of mathematical models describing the influence of composition factors on the most important properties of concrete mix on cement-ash binder in the presence of hardening accelerator was obtained using mathematical planning of experiments.

3. Using mathematical programming implemented by the Microsoft Excel software environment and its application "Solver", the possibility of using a set of experimental and statistical models to solve the problem of designing optimal compositions of construction mixtures used by a 3D printer is shown.

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Information about authors:

Leonid Dvorkin
Doctor in Technical Science
ORCID: https://orcid.org/0000-0001-8759-6318
E-mail: dvorkin.leonid@gmail.com

Vitalii Marchuk
PhD in Technical Science
ORCID: https://orcid.org/0000-0003-0999-0402
E-mail: v.v.marchuk@nuwm.edu.ua

Vadim Zhitkovsky
PhD in Technical Science
E-mail: zhitk@ukr.net

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