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Structure and properties of mortar printed on a 3D printer

R.Kh. Mukhametrakhimov*, L.V. Lukmanova
Kazan State University of Architecture and Engineering, Kazan, Russia
**E-mail: muhametrahimov@mail.ru*

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Abstract. The influence of the molding process of sand-cement mortar printed on a 3D printer on its structure and properties is investigated. The mortar mix was characterized by a mobility P_k = 2, which corresponds to an immersion depth of the etalon cone of 5 cm. Determination of compressive strength was carried out on standard samples of beams with dimensions of 40×40×160 mm by loading them on a press in pure compression mode. Water absorption was defined as the ratio of the difference between the mass of a water-saturated sample and the mass of a dried sample to the mass of a dried sample. The porosity of the hardened mortar samples was determined by the results of determining their density, water absorption and sorption moisture. Defects of the sand-cement mortar mix and hardened composites, formed by the extrusion (3D printing), were determined by the visual and instrumental methods using a measuring metal rule. It is shown that the raw mixes currently used (similar to accepted in experimental studies) are not adapted for their extrusion (3D printing), as reflected in the appearance of various defects – different layer thicknesses, crushing of the underlying layers, cracks, skew of the mixture, inhomogeneous structure of the hardened composite, mix spreadability, high porosity. It was found that the molding of the studied sand-cement mortar by the extrusion (3D printing) leads to increased total pore volume by 10 %, open capillary pore volume by 22 %, conditionally closed pore volume by 9 %, microporosity by 8 %, reduction in open non-capillary volume by 65 % compared to the traditional injection molding samples of a similar composition with further compaction. This leads to a decrease in compressive strength by half compared with the injection molding method with further compaction, and an increase in water absorption by 22 %. Based on the results, the directions of improving the raw mixes for 3D printing are determined.

1. Introduction

There is a quick growth of additive manufacturing market in different areas of human activities at the present time. In 2018 market volume exceeded $5 billion, in 2025 projected market volume will be more $21 billion (Fig. 1). It should be noted that more than 50 % of the global additive manufacturing market is controlled by such countries as the USA, Germany, Great Britain, Japan, etc. In the long-term perspective it defines them as leaders in the development of this technology. While addressing manufacturers of equipment for additive manufacturing, the following leading companies should be highlighted: 3D Systems (USA), EOS Gmbh (Germany), SLM Solutions (Germany), Stratasys (USA), ObjectGeometries (USA-Israel), Envisiontec (USA-Germany (DLP), ExOne (USA), Voxeljet (Germany), Arcam AB (Sweden).

Considering the market of 3D construction printing, it should be noted that the largest market share in terms of cost and volume is 3D printing by the extrusion, the volume of which can grow to $56.4 million by 2021 [2].

This technology allows to create (grow) objects by layer-by-layer deposition (extrusion) of the raw material mix in accordance with a three-dimensional digital model [3], [4]. The essence of this method is to extrude a construction mix (sand-cement mortar, fine-grained concrete, gypsum, gypsum-cement-pozzolanic mixtures, etc.) with various mineral, chemical additives and reinforcing fibers through a ‘nozzle’ or an extruder of a 3D printer [5–8]. Each next layer is laid on top of the previous one, which results in a certain structure.

Concrete extrusion can be carried out by various types of 3D printers: portal, with a delta drive working in angular coordinates, based on industrial manipulators [5, 9, 10].

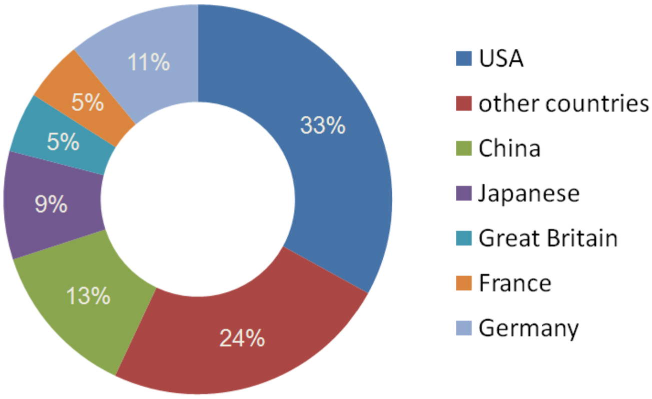


Figure 1. Forecast of the additive manufacturing market structure by 2025 by region. The 'other countries' segment includes India, Latin America, Russia, Australia, Sweden, Italy, Belgium, Spain and the Netherlands [1].

An analysis of the world experience of existing technological solutions for 3D printing in construction performed in [11] made it possible to identify organizations implementing 3D printing technology in construction – 'WinSun' (China), OOO 'SPECAVIA' (Russia), 'ApisCor' (Russia), 'StroyBot' (Rudenko 3D Printer), 'BetAbram' (Slovenia), 'Contour Crafting Corp.' (USA), 'MIT Media Lab' (USA), Loughborough University (Great Britain), 'CyBe Construction' (Holland), 'DUS Architects' (Holland), 'Batiprint 3D' (France), as well as recommendations for concrete mixes used and characteristics of composites based on them. For example, the width of the layer of the extrudable raw mix of different types of 3D printers varies in the range of 20–60 mm, the thickness is 10-40 mm, the average density is 2000–2350 kg/m^3, the bending strength is 6–13 MPa, and the compressive strength is 27.4–110 MPa. It should be noted that the indicators of the properties of construction mixes and hardened compositions based on them, claimed by the listed companies, are often promotional references and they are not always the result of scientific research or certified tests, which does not allow to use these data in the planning of scientific research. The tests performed on some of the proposed compositions in laboratory conditions using a 3D printer confirm this assumption.

An analysis of the quality of construction products printed on a 3D printer shows that the concrete mixes currently used (mainly fine grains) are not adapted for use in extrusion technology (3D printing), which is reflected in the disruption of the geometrical dimensions of the products due to spreading of the underlying layers ruptures, voids and fractures, low crack resistance, high shrinkage deformations, low adhesion of layers caused by not optimal compositions and rheological and technological properties of the mixes, which leads to reduced physical and mechanical characteristics and durability of the products on their basis (Fig. 2-4).

a) b)

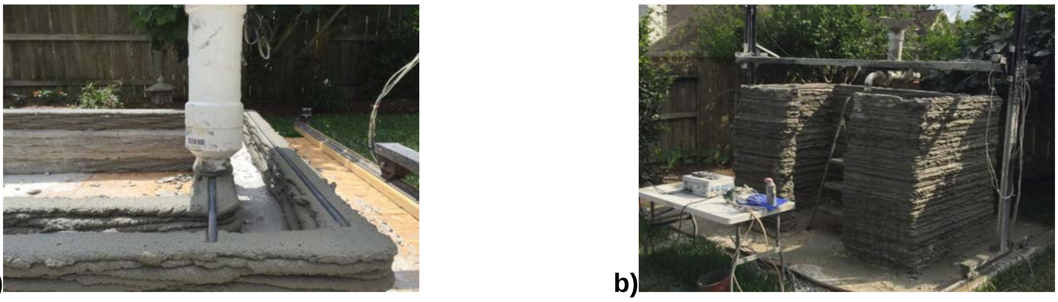


Figure 2. Typical 3D printing defects on the example of a residential building by Alex Le Roux (USA) [12]: a), b) disruption of the geometrical dimensions due to spreading of the underlying layers, poor adhesion of the layers.

a) b)

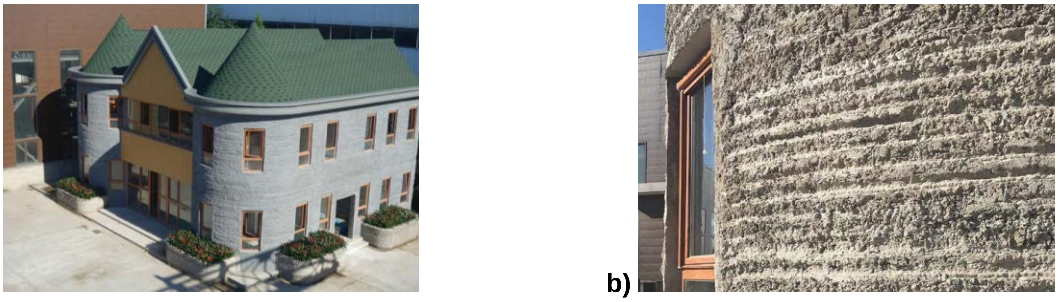


Figure 3. Typical 3D printing defects on the example of residential building by 'Tengda' (China) [12]: a), b) disruption of the geometrical dimensions due to spreading of the underlying layers, surface heterogeneity.

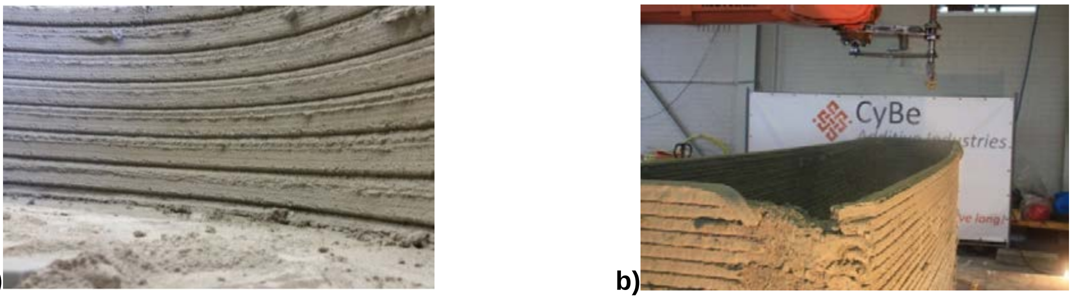
a) b)


Figure 4. Typical 3D printing defects on the example of concrete wall by ‘CyBe Additive Industries’ (Holland) [13]: a) disruption of the geometrical dimensions, concrete flows b) destruction of the concrete product due to the low structural strength of the concrete mix.

Molding process furthermore can directly influence on the quality of the molded products along with a composition of the concrete mix. Distinctive features of concrete molding by extrusion from the widely used injection molding method is the absence of formwork (form) resulting in significant open surface module of product and difficulties in curing of concrete; the absence of a technological stage aimed at compaction of the molded concrete mix after its extrusion, difficulties in realizing a full-fledged traditional core reinforcement using flat grids, volume frames, etc. (although there are some positive results in this area [14–17]).

As a result of our experimental studies and literature data, the main requirements for concrete mixtures in additive manufacturing based on operational, prescription and technological factors were identified. It should be noted that this is consistent with the results. At the stage of molding and hardening the mix, the main requirements should include: rheological and technological properties [18, 19] (formability or workability, the ability to transport through pipes, plastic strength [20], thixotropy), dispersion, adhesive properties (tight adhesion of the layers), the absence of fractures of the mixture, the absence of cracking, low shrinkage, uniformity hardening (setting), high setting rate after extrusion. The finished products are required to provide the necessary strength, high uniformity and stability of properties, low density and thermal conductivity, high adhesion strength, frost resistance [21]. M.Y. Elisrtatkin at al. [22] established links between the process factors and properties of molding components; principles of their practical implementation are proposed.

There are a number of works aimed at developing new compositions of raw mixes adapted for the 3D printing by extrusion, as well as methods for controlling their properties [23]. G.S. Slavcheva at al. [24, 25] established that the W/C-ratio defining the concentration of solid phase particles in the system is the main factor of structuring and strengthening of cement paste for 3D construction printing. R.A. Buswell at al. [26] explored the relationship between raw material mix and geometry of the created hardened composite (mortar and concrete). Based on the obtained data, a matrix of issues has been created that identifies the spectrum of future research exploration in this emerging field. Y. Zhang at al. [27] propose to use recycled pieces of glass as a fine aggregate, which makes it possible to control the rheological parameters of the mixture, thus introducing them into the composition increases the mobility of the mix, however, the strength of products made from such concrete is lower compared to conventional concrete.

An alternative way to improve the rheological properties of concrete mixtures for 3D printing is to use active mineral and chemical additives [28, 29]. To reduce the rate of setting concrete mix, authors [30] proposed ultrasonic activation of the binder during the hydration process in the pre-induction period, during which a larger number of ettringite crystals is created, which can significantly increase the speed of 3D printing, while the concrete strength reaches approximately 1 MPa within 20 minutes. In another work [21], to reduce the setting time, it is proposed to introduce a solution of sodium silicate in the amount of 5 and 8 % into the formulation of the raw material mix, which allows reducing the setting time from 90 minutes to 20 and 12 minutes, respectively. To reduce the shrinkage deformation of concrete, the authors [31] propose using silica fume together with a plasticizing additive.

To solve the problems of low strength and fracture toughness of products obtained on a 3D printer by the extrusion, it is proposed to use dispersed reinforcement with various types of fibers: polyvinyl [32, 33], polyethylene [34], polypropylene [35, 36], glass [37], basalt [38]. The introduction of polypropylene fibers in an amount of 1 % [36] has a positive effect on a decrease in the spreadability of printed samples (of seven layers) upon application of a vertical load, which is associated with an increase in the compressive strength of composites to 31 %.

P. Lubin at al. [39] proposed replacing cement paste with clay soil up to 25 %, which leads to a reduction in cost and an increase in printability on a 3D printer with a slight decrease in the strength of the material to 7 %.

One of the ways to control the structure formation of building composites is the use of nanomodifying additives [40–42], including creating construction products using 3D printing [43, 44].

There is no study comparing the properties of building mixtures from the molding method, the influence of 3D printing technology on the appearance of defects and damages, the causes of their occurrence in many works. Solution of these problems allows to determine the main directions of development and optimization of concrete raw mixes adapted for 3D printing, which ultimately will lead to high-quality construction products.

The purpose of this study is to examine the impact of the molding process of a raw mix not adapted for 3D printing on the example of a sand-cement mortar on its structure and properties during the injection method with compaction and extrusion without compaction (3D printing) and, based on the results, to determine directions for improving compositions raw mixes for 3D printing.

2. Materials and Methods

The studies were carried out in the laboratory of additive manufacturing in construction (Kazan State University of Architecture and Engineering, Russia).

The process of creating a sample by extrusion (3D printing) included the following sequence of technological operations:

1. Development of object digital three-dimensional model in ‘AutoCad’ (Autodesk, Inc.);
2. Division of the model into layers in cross section in ‘Sheet Cam’ (Stable Design);
3. Translation the model into G-code, which allows to simulate, generate codes and control a 3D printer by Mach3 (Artsoft founder Art Fenerty);
4. Preparation of a raw mix with specified properties;
5. Transfer of the developed code to the print head-extruder;
6. Extrusion of the raw mix in accordance with a specified digital three-dimensional model;
7. Curing of the raw mix material;
8. Secondary processing: removal of the supporting structure (substrate).

The sample was formed from a sand-cement mix by extrusion in workshop 3D printer ‘AMT S-6044’ OOO ‘SPETSAVIA’ (Yaroslavl, Russia), organized by a portal system (Fig. 5), in accordance with a specified digital three-dimensional model (G-code). Technical specifications of 3D printer ‘AMT-S-6044’ are shown in Table 1.

Table 1. Technical specifications of 3D printer ‘AMT-S-6044’.

Length, mm	4000
Width, mm	4000
Height, mm	2800
Weight, kg	870
Producer	OOO ‘SPETSAVIA’
Manufacturing country	Russia. The equipment is certified in the territory of the Customs Union
Type of drive	Stepper motors with parallel-shaft
Type	S Series Small-format Portal Construction 3D Printer
Assignment	Printing of building elements up to 12.6 m^2, hardscape elements, street furniture, architectural decor, reinforced concrete products. The printer is intended for installation in the workshop
Productivity, m^3/h	0.6
Operational zone, mm	3500×3600×1000
Working power, кВт	1.6
Print layer size, mm (height, width)	10×30

The following materials were used in the research process:

a) sand-cement mix (manufacturer Samara gypsum plant) complying with GOST 28013-98 (Russian standard);

b) modifying additives:

- comprehensive additive ‘Cemfix’ superplasticizer-hardening accelerator based on polymethylene naphthalene sulfonates (C-3) and inorganic sodium salts (‘Cemmix’), conforming to TU 2499-007-90557835-2014;

c) polypropylene fibers with a length of 12 mm (VSM-II), conforming to TU 5458-001-82255741-2008. Technical specifications are shown in Table 2;

d) tap drinking water complying with GOST 23732-2011 (Russian standard).

Table 2. Technical specifications of polypropylene fibers 'VSM-II'.

Name	Index
Fiber diameter, μm	20
Fiber lenght, mm	12
Tensible strength, MPa	550
Lengthening	20 %
Elastic modulus, MPa	no less than 10000
Number of single fiber, million pieces/kg	510-550
Surface fiber area, m^2/kg	150
Temperature of fusion, °C	160

Mixing of the components of the raw materials was carried out in a cyclic concrete mixer 'CMI 46' for 5 minutes 30 seconds until homogeneous mass was obtained.

The mortar mix was characterized by the mobility P_k = 2 conforming to GOST 28013-98 (Russian standard), which corresponds to immersion depth of the etalon cone of 5 cm. The mobility of the mortar mixture was determined to GOST 5802-86 (Russian standard).

Defects of the sand-cement mortar mix and hardened composites based on it, formed by extrusion (3D printing), were determined by the visual and instrumental methods using a measuring metal rule.

Samples were formed in two ways: extrusion without mold (formwork) and compaction of the mix (3D printing) and injection molding with further compaction in the mold 40×40×160 mm.

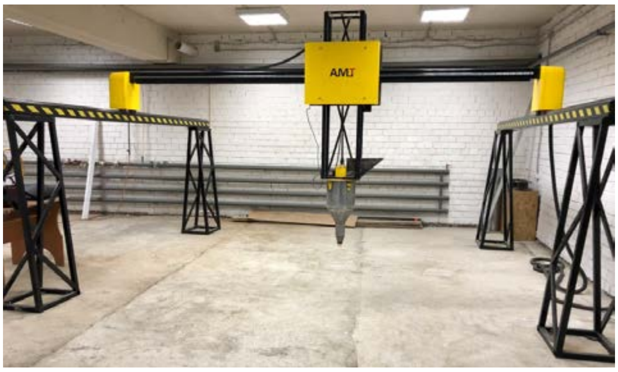


Figure 5. 3D printer 'AMT-S-6044' at the laboratory of additive manufacturing in construction (Kazan State University of Architecture and Engineering, Russia).

The samples were tested after hardening for 28 days in an air-moist environment. Determination of compressive strength was carried out on standard samples of beams with dimensions of 40×40×160 mm by loading them on a press in pure compression mode complying with GOST 310.4-81 (Russian standard).

Water absorption and density of the samples were evaluated according to GOST 5802-86 (Russian standard). Water absorption was defined as the ratio of the difference between the mass of a water-saturated sample and the mass of a dried sample to the mass of a dried sample.

The porosity of the samples of the hardened composite was determined by the results of determining their density, water absorption and sorption moisture according to GOST 12730.4-78 (Russian standard).

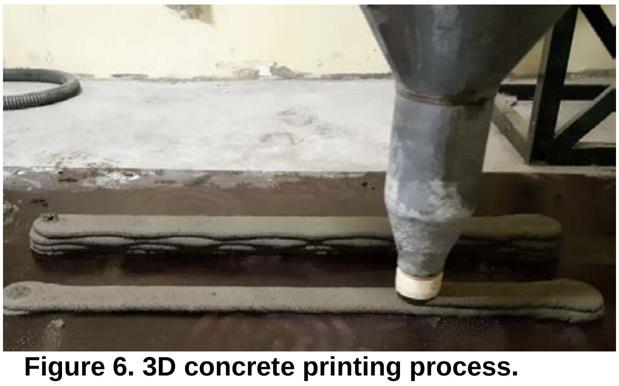


Figure 6. 3D concrete printing process.

3. Results and Discussion

According to the results of the first stage of experimental studies, it was found that the molding process of raw mix affects the pore structure parameters and the character of the pore distribution.

Table 3. Effect of molding process on pore structure sand-cement composite.

Molding process	Total pore volume	Volume of open capillary pores	Volume of open non-capillary pores	Volume of conditionally closed pores	Microporosity
Injection molding with further compaction in the mold	30.36	8.38	1.50	20.48	0.85
Extrusion without mold and compaction (3D printing)	33.53	10.24	0.97	22.39	0.92

As can be seen from Table 3, molding process of raw mix affects the pore structure parameters and the character of the pore distribution. Thus, the extrusion molding process (3D printing) leads to an increase in total pore volume by 10 %, volume of open capillary pores – 22 %, volume of conditionally closed pores – 9 %, microporosity – 8 % and it leads to decrease in volume of conditionally closed pores by 65 %.

At the second stage effect of molding process on compressive strength of specimens was investigated (Fig. 7).

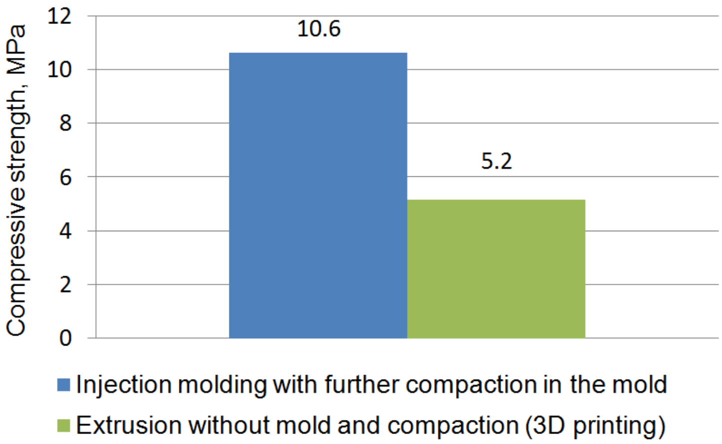


Figure 7. Effect of molding process on compressive strength of sand-cement composite.

As follows from Fig. 7, molding process of raw mix significantly affects the strength of sand-cement composite. Thus, compressive strength of sample formed by extrusion (3D printing) is 5 MPa, which is two times lower than sample formed by injection molding with further compaction. The low compressive strength of a sample formed by extrusion (3D printing) is primarily a consequence of the resulting pore structure of hardened composites, poor adhesion of the printed layers.

Water absorption of sand-cement mortar formed by extrusion (3D printing) and injection molding with further compaction is shown in Fig. 8.

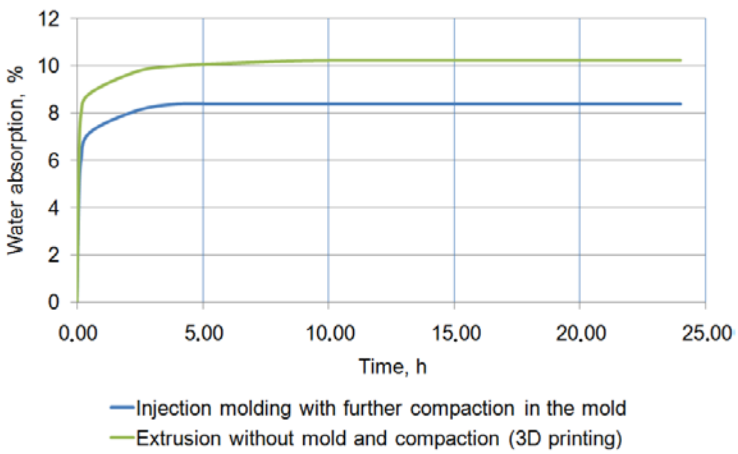


Figure 8. Effect of molding process on water absorption of sand-cement composite.

As follows from Fig. 8, specimen formed by extrusion (3D printing) has the highest water absorption – 10.2 % owing to high pore structure parameters of hardened composite.

In general, the obtained data on the average density of water absorption of cement mortars are consistent with the data presented in the research [45, 46].

The following defects and damages of specimens formed by extrusion (3D printing) were observed: different layer thickness, crushing of the underlying layers, cracks, skew of the mixture, inhomogeneous structure of the hardened composite, mix spreadability, high porosity. Some of the listed defects were also noted by the authors in the research [26].

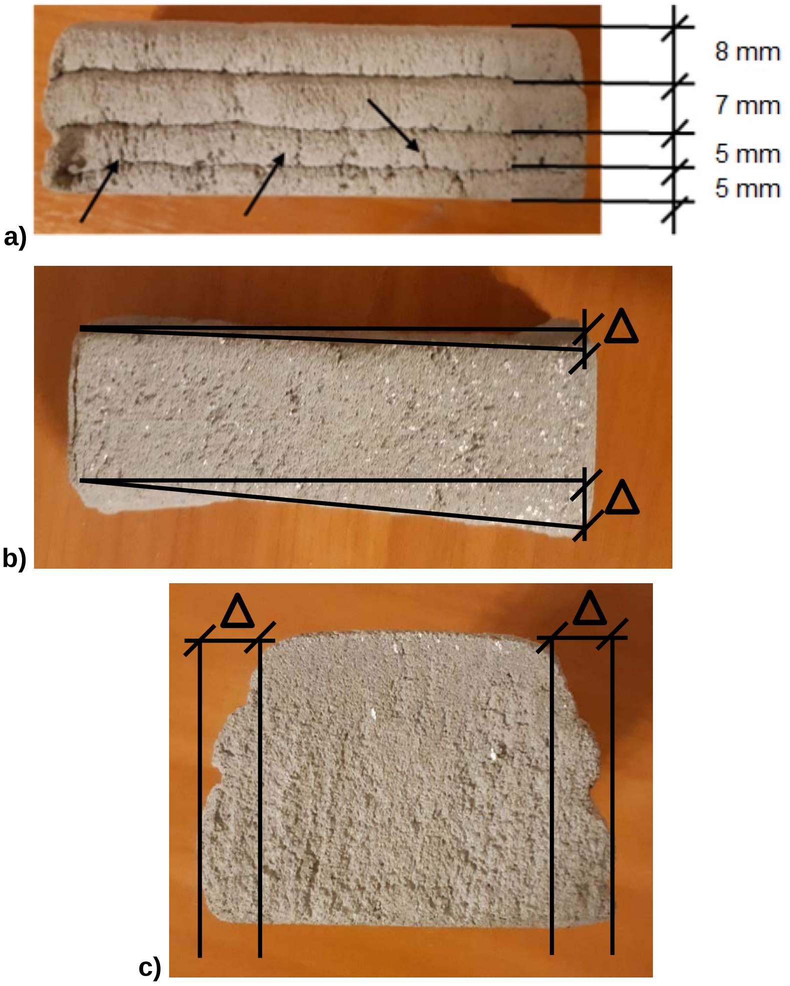


Figure 9. Defects of specimen formed by extrusion (3D printing):
a – different layer thickness (5-8 mm), crushing of the underlying layers, cracks;
b – skew of the mixture, inhomogeneous structure of the hardened composite;
c – mix spreadability, high porosity.

In accordance with the results, the main directions for improving the formulation of fine-grained concrete for extrusion (3D printing) are determined:

- imparting a denser and more uniform structure to fine-grained concrete mix, for example, by introducing the optimal type and amount of active and inert mineral additives;
- imparting required rheological properties to fine-grained concrete mix, accelerating the kinetics of the initial structure formation and hardening of the raw material mix and the strength properties of the hardened composite, for example, by introducing the optimal type and amount of plasticizing additive and hardening accelerator additive;
- imparting increased crack resistance and strength to fine-grained concrete, for example, by reinforcing with fibers of an optimal form and content;
- imparting increased water resistance, frost resistance and other indicators of durability to fine-grained concrete, for example, by introducing a water-repellent additive.

4. Conclusions

1. Concrete mixes currently used are not adapted for extrusion (3D printing), as reflected in the appearance of various defects – disruption of the geometrical dimensions due to spreading of the underlying layers ruptures, voids and fractures, low crack resistance, high shrinkage deformations, low adhesion of layers caused by not optimal compositions and rheological and technological properties of the mixes which leads to reduced physical and mechanical characteristics and durability of the products on their basis.

2. It has been established that the extrusion molding process (3D printing) of sand-cement mortar leads to an increase in total pore volume by 10 %, volume of open capillary pores – 22 %, volume of conditionally closed pores – 9 %, and microporosity – 8 %, and it leads to decrease in volume of conditionally closed pores by 65 % compared with the traditional injection molding with further compaction of samples of a similar formulation. This leads to a deterioration in the physical and mechanical characteristics and durability of 3D printing products.

3. It was revealed that the molding of a cement-sand mortar by extrusion (3D printing) leads to a decrease in compressive strength by half compared to the injection molding with further compaction, and an increase in water absorption by 22 %.

4. The results of research confirm that conventional (not adapted) compositions of raw mixes are not effective for use in 3D printing as "ink", indicating the need for further theoretical and experimental studies on development of raw mixes, primarily based on fine-grained concrete, as the most effective and used in existing 3D printers for extrusion (3D printing).

5. The main directions of improving the composition of fine-grained concrete for extrusion (3D printing) are following: imparting a denser and more uniform structure, the required rheological and technological properties to fine-grained concrete mix, acceleration of the kinetics of the initial structure formation and hardening of the raw material mix and the strength properties of the hardened composite, increased crack resistance , increased water resistance, frost resistance and other indicators of durability, for example, by introducing optimum type and amount of active and inert mineral additives, chemical additives (plasticizing additives, hardening accelerator additives, water-repellent additives) into formulation, fiber reinforcement.

5. Acknowledgements

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Contacts:

Rustem Mukhametrakhimov, muhametrahimov@mail.ru

Liliya Lukmanova, lilya0503199@gmail.com