Influence of the technological properties of cement-sand mortar on the quality of 3D printed products

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Abstract. The results of the influence of cement-sand mortar mobility on quality of hardened composites, made by 3D concrete printing (3DCP), are presented. Four formulations of cement-sand mortar mixes with mobility classes $P_k^1$, $P_k^2$, $P_k^3$, $P_k^4$ which corresponds to immersion depth of the reference cone of 3.3 cm, 6.5 cm, 8.7 cm, 12.0 cm, respectively, were prepared for testing. It has been established that regulatory framework regulating 3DCP test methods not sufficiently developed and requires further improvement. It was revealed 3D printing of cement-sand mortar mixes with mobility classes $P_k^2$-$P_k^4$ on researched 3D printer is possible, however, products have defects and damages. Mortar mobility has a significant impact on structure kinetics and geometric parameters deviation. The lower mobility class is, the higher structure kinetics of raw material mix is, which is due to the early formation of the crystallization structure of hardened composite. Cement-sand mortar with class mobility $P_k^2$ accepted for further research in the development of adapted raw mixes for 3DCP.

Keywords: concretes, cements, mortar, 3D printing, extrusion, additive manufacturing, mobility, rheology.

1 Introduction

In the technology of manufacturing concrete and reinforced concrete products, the choice of the molding method depends on a number of factors, among which one of the determining ones is the technological properties of the concrete mixes used – indicators of mobility or stiffness [1]. The technological properties of the concrete mix, in turn, are determined by its composition and properties of the materials used [2–4].

At the beginning of the twenty-first century, a new trend in the technology of manufacturing building products – 3D concrete (mortar) printing (3DCP) according to a three-dimensional digital model – has a wide degree of automation and mechanization of processes [5–9]. However, the products obtained by this molding method have various defects and damages – violation of geometric parameters, mix spreading, tears, voids, structural discontinuities, etc. [10–12]. These defects are clearly visible on a test print samples of a curved shape made by the authors (figure 1).

Figure 1. 3D printed cement-sand mortar samples of a curved shape with defects and damages.

The reason for such defects and damages is the non-optimal rheological and technological characteristics of the mixes [13–17], absence of universal test methods and quality control during 3D printing [18].
To solve the problems relating to increased crack formation of products printed on a 3D printer, cold-bonded fly ash lightweight aggregate concretes can be very effective. Pre-soaking of cold-bonded fly ash lightweight aggregate provides an “internal curing” of the mix, giving off its water. This will reduce the likelihood of shrinkage cracks during concrete drying [19].

The authors of study [20] suggested to use in 3DCP concrete with fine aggregate made of quartz sand with a particle size module of 1.12.

Analyzing regulatory documents in the field of 3D construction printing, it should be noted that ten national standards are currently approved and have been enacted in the Russian Federation, in addition, three draft national standards for additive manufacturing in construction using concrete and mortar mixes have been developed by the Technical Committee for Standardization “Additive Technologies” (TC 182):

1) “Construction material for additive manufacturing in construction. Terms and definitions” [21]. This standard applies to materials for additive manufacturing in the construction, reconstruction and repair of buildings and structures, and establishes the terms and definitions used in the technologies of additive manufacturing (AM) in construction, which are based on the additive principle of manufacturing building structures, i.e. creating physical features by sequentially adding material.

2) “Construction material for additive manufacturing in construction. Technical requirements” [22]. This standard applies to materials for additive manufacturing in construction, reconstruction and repair of buildings and structures, and establishes general technical requirements and acceptance rules.

3) “Construction material for additive manufacturing in construction. Test methods” [23]. This standard applies to materials for additive manufacturing in construction, reconstruction and repair of buildings and structures and establishes methods for determining the main quality indicators of materials for additive manufacturing in construction (determination of resistance to spalling, pumpability and adhesion of layers (continuity)) in the form of dry mixes, mortar (concrete) mixes, as well as hardened concrete (mortar).

It should be noted that test method for raw mixes (concrete and mortar) for 3DCP, proposed in this draft standard, is not universal, which is reflected in the complexity of printing test samples of shape and size (prism 100 mm wide, 50 mm high), which are regulated this standard. This is due, first of all, to the various technical capabilities of the printheads of 3D printers – width/thickness of the printed layers vary in the range of (5 ÷ 100)/(2 ÷ 40) mm [24].

An analysis of foreign and domestic studies in the field of 3DCP indicates that methods such as hand held rotational rheometer measurements [25–27], techniques such as ultrasonic pulse velocity (UPV) [28] and penetrometer tests [29] are used at present to control the quality of concrete mixes 3D printed and products based on them. Taking into account the fact that rheological and technological characteristics of raw mixes (concrete and mortar) are formed during the coagulation period and change during the hydration of the binder, determination the characteristics of mixes during this period, i.e. when the mixture is characterized by a plastic state (before its transition to a crystallization structure), is more appropriate. Penetrometer test fully meets these requirements, in addition, testing with a pocket penetrometer is easily feasible in a site.

The aim of this research is to study the influence of cement-sand mortar mobility on quality of hardened composites, made by 3DCP, and determine the optimal mobility of the mortar for further research.

At the first stage of research, the influence of the mobility of the mortar mix on the formation of the geometric parameters of the hardened composites (length, width, height of the printed layers) was studied. At the second stage, the influence of the mobility of the mortar mix on the type and nature of the defects and damages of the hardened composites (violation of the straightness of the layers, the presence of “waves” on the surface of the layers, the spread of the layers, the formation of gaps between the layers and along the length of the layer) was studied. In order to predict the ability of the raw material mixture to hold moldable layers without deforming the previous layers at the optimum printing speed, the influence of the mobility of the mortar mix on the structure kinetics within 12 hours was studied at the third stage.
2 Materials and methods

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

The following materials were used in the research process:
   a) Portland cement CEM II/A-S 32.5 N (manufacturer “SLK Cement” complying with GOST 31108-2016 (Russian standard);
   b) medium-sized river quartz sand;
   c) tap drinking water complying with GOST 23732-2011 (Russian standard).

Four formulations of cement-sand mortar mixes of different mobility classes were prepared (table 1) to carry out the studies. The mobility of the compositions was regulated by changing the water content at a constant flow of Portland cement and sand.

| Mobility class | Immersion depth of the reference cone, cm | cement/sand ratio |
|----------------|------------------------------------------|-------------------|
| Pk 1           | 3.3                                      | 1/2               |
| Pk 2           | 6.5                                      | 1/2               |
| Pk 3           | 8.7                                      | 1/2               |
| Pk 4           | 12.0                                     | 1/2               |

Mixing of the components of the raw materials was carried out in a forced action concrete mixer for 10 minutes until homogeneous mass was obtained.

The mobility of the mortar mixture was determined to GOST 5802-86 (Russian standard) (figure 2) by the depth of immersion in it of the reference cone.

The sample was formed from a sand-cement mix by extrusion in workshop 3D printer “AMT S-6044” “SPETSAVIA LLC” (Yaroslavl, Russia), organized by a portal system, in accordance with a specified digital three-dimensional model (G-code), which was a strip 40 cm long of four layers.
Defects of the cement-sand mortar mix and hardened composites based on it, formed by 3DCP, were determined by visual and instrumental methods using a measuring metal ruler according to GOST 427-75 (Russian standard).

The spread of the mortar mix of 3D printed samples was determined as the ratio of the maximum value of the actual width (b) to the minimum value of the actual layer height (h).

Structure kinetics of the mortar mix was determined in accordance with requirements of ASTM C403 using a pocket penetrometer C194 (figure 3).

Figure 3. Determination of structure kinetics of the mortar mix using a pocket penetrometer.

3 Results and discussion
Possibility of implementing the process of forming the raw mix on the used 3D printer was studied, depending on its mobility on the first stage of the study.

It was found that 3D printing of a mortar mix with a mobility class Pk 1 on researched 3D printer is impossible, which is manifested in blocking the rotation of the screw of 3D printer and, accordingly, the impossibility of extruding the raw mix through the 3D printer nozzle. 3D printing of a mortar mix with a mobility classes Pk 2, Pk 3, Pk 4 on researched 3D printer is feasible, however, the quality of the formed samples differs significantly from each other.

At the next stage, influence of the mortar mix mobility on formation of geometric parameters of the hardened composites was determined (table 2), as one of the most important conditions for ensuring product quality.

Table 2. Actual geometric parameters of hardened composites.

| Mobility class | length (L), mm | width, mm | height, mm |
|---------------|----------------|-----------|------------|
| Pk 1          | 3DCP impossible |           |            |
| Pk 2          | 485            | 53        | 80         |
| Pk 3          | 535            | 90        | 100        |
| Pk 4          | 580            | 120       | 65         |
As can be seen from Table 3, all the studied samples are characterized by significant deviations from the design length value \( L_d \), equal to 400 mm. Thus, the actual length \( L_a \) of a sample with mobility class Pk 2 exceeds the design length by 21.3\%, Pk 3 – by 33.8\%, the sample with mobility class Pk 4 has the greatest deviations of the geometric parameters, whose actual length increased by 45 \% in comparison with the design length value. In this case, this is due to some features of G-code generation of a three-dimensional digital model of the sample and its 3D printing (figure 5), namely: 1) design length value \( L_d \) is the distance between the points of the center of gravity of the extruded raw mix in the initial and final positions of 3D printer nozzle during printing; 2) raw material mix are spreading when 3DCP, accompanied by an increase in its length \( L_s \); the higher mobility of raw mix, the higher spreading value.

![Figure 4. Hardened composites printed on a 3D printer (top view): from left to right – Pk2, Pk3, Pk4.](image)

**Figure 4.** Hardened composites printed on a 3D printer (top view): from left to right – Pk2, Pk3, Pk4.

![Figure 5. Scheme for determining the actual length of samples printed on a 3D printer.](image)

**Figure 5.** Scheme for determining the actual length of samples printed on a 3D printer.
Thus, it is necessary to take into account the above features of G-code formation of a three-dimensional digital model to obtain products with the required geometric parameters.

The influence of the mortar mix mobility on the appearance of defects and damages on hardened composites is shown in table 3. The revealed defects and damages of hardened composites were characterized by three categories: “less significant”, “significant” and “more significant”.

Table 3. Influence of the mortar mix mobility on the appearance of defects and damages on 3D printed hardened composites.

| Mobility class | Violation of straightness | Presence of “waves” | Spread (b/h) | Gaps between layers | Gaps along the length (mm) |
|---------------|--------------------------|---------------------|-------------|--------------------|--------------------------|
| Pk 1          | 3DCP is impossible       |                     |             |                    |                          |
| Pk 2          | less significant         | no                  | 3.53        | significant        | up to 20 mm              |
| Pk 3          | more significant         | less significant    | 8.18        | less significant   | no                       |
| Pk 4          | more significant         | significant         | 12.0        | no                 | no                       |

As can be seen from Table 3, the sample with mobility class Pk 4 has the greatest degree of violation of straightness, “waves” (figure 6a), the highest mix spread, the appearance of which is caused by increased fluidity of the mix, however, for this reason, the composite has uniform and monolithic structure, as a result this sample is free from defects such as gaps between layers and gaps along the length of the layers. On the contrary, a sample with mobility class Pk 2 is characterized by the presence of gaps between the layers (figure 6b), gaps along the length of the layer up to 20 mm (figure 6c), however, defects such as a violation of straightness, presence of “waves” and spread are manifested less.

The results of studies of structure kinetics of a mortar mixes with mobility classes Pk 1 – Pk 4 using a pocket penetrometer are shown in figure 7.

As can be seen from figure 7, mortar mix mobility has a significant influence on the growth of its strength over time. Thus, with an increase of mortar mobility class from Pk 1 to Pk 4, the growth of strength is significantly reduced.

These differences are due to the peculiarities of the microstructure formation of cement-sand mortars, namely, that the crystallization period of hardening of specimen with mobility class Pk 1 occurs much earlier than other samples, which leads to an increase in the strength of the hardened composite. Obviously, this phenomenon is associated with a decrease in water content in the system of the sample under consideration. On the contrary, the higher mortat mobility class is, the longer the formation of its coagulation structure is, which causes thixotropy and plasticity of the hardening system. The above-described mechanisms of structure formation of cement systems are very important in 3DCP due to the need to predict the ability of the raw material mix to hold the formed layers without deforming the previous layers at the optimum printing speed.

Taking into account the fact that one of the main criteria for the quality of products is the provision of specified geometric parameters, minimizing the degree of appearance and development of defects and damages, high physical and mechanical properties, formulations with mobility class Pk 2 is more preferable to accept for further research on development of adapted formulations for 3DCP, which is characterized by the smallest deviations of geometric parameters and a relatively high speed of curing in time, with their subsequent modification. It is worth noting that the presence of defects such as gaps between layers, gaps along the length of a layer can affect the adhesion strength of layers between layers, however, this problem can be solved by modifying the compositions with active mineral and chemical admixtures reinforcing fibers and etc. which causes interest in further research in the field of development of adapted raw mixes for 3DCP.
a) Sample with mobility class Pk 4: presence of "waves" on layer surface

b) Sample with mobility class Pk 2: gaps between layers

c) Sample with mobility class Pk 2: gaps up to 20 mm along the length

**Figure 6.** Defects of hardened composites.

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**Figure 7.** Penetration resistance values of sand-cement mortar mix (mobility class Pk 1 - Pk 4) versus time.
4 Conclusions
1. Analysis of regulatory sources indicates the initial stage of formation and development of regulatory framework in the field of additive manufacturing construction using concrete and mortar mixes. Currently, the Russian Federation has developed three draft national standards regulated basic principles, terms and definitions, technical requirements for the materials used, and test methods. However, with regard to the proposed test methods, it is worth noting the limited use of them on various 3D printers, which requires further development of regulatory framework in this area.

2. It was found that the cement-sand mortar mobility affects the possibility of its molding on a 3D printer. It is possible to form mortar mixes with mobility classes Pk 2-Pk 4 on the researched 3D printer, while the quality of the molded products is significantly different from each other. The use of a mix with mobility class Pk 1 leads to blocking the rotation of the screw of the print head and to the impossibility of its extrusion through the 3D printer nozzle.

3. An increase mortar mobility class from Pk 2 to Pk 4 at the same printing speed (screw rotation speed and nozzle movement speed) leads to an increase in the deviation of the geometric dimensions of the molded products (length, width, layer height).

4. It was experimentally determined that, regardless of mortar mobility class (from Pk 2 to Pk 4), hardened composites have defects and damage, but their appearance and character are very different. So, with an increase mortar mobility class from Pk 2 to Pk 4, the degree of development of layer straightness and the spread of the layers increase; starting with Pk 3, “waves” are formed on the surface of the layers, which can be caused by the peculiarity of the rheological and technological properties of the compositions used or by uneven extrusion, which is well expressed when using more mobile raw material mixes. Moreover, the development of defects such as the formation of gaps between layers and gaps along the length of the layer decreases until they disappear with an increase in the mortar mobility. The opposite picture is observed when the mortar mobility class decreases from Pk 4 to Pk 2: the lower mortar mobility class, the higher likelihood of gaps between the layers and gaps along the length of the layer is, which adversely affects the adhesion strength of the formed layers and represents interest for further research.

5. The mobility of the 3D printed mortar mix has a significant influence on the growth of its strength over time. The lower mortar mobility is, the higher the rate of curing of the raw material mix is, which is due to the early formation of the crystallization structure of the hardened composite. This phenomenon in the formation of the stone microstructure is a very important factor for predicting the ability of the raw material mix to hold the formed layers without deforming the previous layers at the optimum printing speed to obtain high-quality construction products on a 3D printer.

6. It has been established that the formulation of the cement-sand mortar mix with mobility class Pk 2 meets the requirements of providing the specified geometric parameters of the product, a relatively high speed of curing and lower water demand of the raw material mixture; that is why raw mix mobility class Pk 2 is accepted for further research in the development of adapted raw mixes for 3DCP.

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