Mechanical and physical properties of fine-grained concrete for concrete additive manufacturing

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Abstract. Additive manufacturing (3D printing) is becoming more and more common in the field of modern construction. However, for wider implementation of this technology, it is necessary to solve a number of material-oriented scientific problems related to development of concrete composition with targeted rheological, stress-strain, physical and mechanical properties. It has been established that time periods between successful applications of layers play the crucial role in ensuring monolithic features of the “printed” structures. Application of mathematics planning of the experiment allowed establishing the main principles of formation of basic physical and mechanical properties of fine-grained concrete depending on material composition.

1 Introduction

The introduction of digital technologies for solving problems in construction practice has important practical significance. Digitalization technologies in the field of materials science, including construction, is particularly successful in additive manufacturing for creating materials, products and structures. One of such technologies is 3D printing construction, which allows creating objects by layer-by-layer extrusion of a viscoplastic mixture. The development of the method led to industrial production of purpose-targeted equipment by a number of Russian and foreign companies [1].

3D printing manufacturing involves the extrusion of a concrete mixture (Fig. 1). Ensuring of trouble-free extrusion, dimensional stability, as well as the necessary adhesion to the bottom layer and low deformability are the main requirements for concrete mixtures when adapting them for the latest 3D printing technology [2-3].

Construction material for erection of structural units (walls, floors) is a fast-hardening fine-grained or reaction-powder concrete reinforced with steel or polymer microfiber [4-6] and obligatory introducing of multifunctional additives [7-8]. Each next layer is extruded by 3D printer over the previous one. As a result, a certain structure is formed (see Fig. 2).

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Fig. 1. Additive construction of wall by extrusion [2].

Fig. 2. Formation of horizontal (1) and vertical (2) joints by using 3D printer.

The technology ensures significant speeding up of the process of erection of construction elements. Besides, under such conditions, fresh concrete is subjected to early loading because of the upper concrete layers, while having low plastic strength. In order to ensure estimated resistance the concrete mixture should provide the necessary adhesion of surfaces of the layers. For spatial stability and solidity of the concrete mass, it is crucial to take into account the adhesion strength in horizontal (marked by 1 in Fig. 2) and vertical joints (so-called abutting joints – marked by 2 in Fig. 2).

Early loading of the lower layers with the weight of the overlaid ones can improve adhesion of fresh concrete layers, as it is described in the work of A. V. Satalkin [9] and mentioned in later studies by S. G. Golovnev and colleagues [10].

Adhesion of Portland cement and aluminate cement layers after early loading was studied by the authors in [11-12] by bending tensile test on prisms, as well as by tensile splitting test on cubes as per GOST Standard 10180. Increased tensile strength has been observed when splitting samples subjected to early load. Besides, the lower was the age of hardening by the time of loading, the higher was the strength characteristics.

Concrete molded during 3D construction as described above are subjected to early load in a plastic state. It is appropriate to presume that reduction of time of next layer application will improve its adhesion with the underlying one. Composition and processing factors, such as presence and ratio between the modifying additives, that affect this parameter, are of particular interest. Because of the increased interest in mixed-media binder grouts for development of 3D printing, they were taken as an object of study [13].

2 Methods and materials

Construction structure erected by using additive manufacturing must have structural integrity after completion of hardening process of the cement. In case of layer-by-layer
application of fine-grained concrete, there is a high probability of cold joint formation in horizontal layers (Fig. 2-1) or vertical abutments of the structure (Fig. 2-2), which can reduce the adhesion strength at certain places and, as a result, reduce degree of solidity of the structure.

In order to identify relationship between the adhesion strength in horizontal layers and duration of breaks between laying the cubic samples were manufactured in two phases. For simulation of the first layer the test mold was filled to half of the height; after the break, which spanned for 30 to 120 minutes, the second layer of fresh fine-grained concrete mixture (FGC) was placed, filling the test mold to the top. Vibro-compaction was not carried out. The adhesion strength between the layers at the age of 7 days was estimated by the strength of the sample cubes in tension when splitting. The splitting plane went along the contact area of the layers.

One promising way for reduction of risk of shattering in the process of hardening is application of controlled-shrinkage binder grouts, such as expanding gypsum-alumina cement (hereinafter EGAC) [14]. Applied EGAC consists of high alumina cement, Portland cement and grinded gypsum stone (calcium sulfate dehydrate). The ratio of mineral components of the selected composition of the EGAC ensures a slight expansion of fine-grained concrete during the first 3-5 days of air-dry hardening (1-2 mm/m), and then a slight shrinkage of 0.8 mm/m or less from the nominal size by 28th day of hardening.

The developed fine-grained concrete mixture is proposed for use as a dry building mortar (hereinafter DBM). In addition to mineral components, this mortar contains water-soluble polymers (water-holding additive Mecellose 24502 and adhesive additive VINNAPAS 7220Е), as well as a dry superplasticizing admixture Melflux 5581F based on polycarboxylic ethers. These additives, despite the relatively small dosages, have a significant impact on the properties of the prepared composite. At the same time, taking into account the possible occurrence of synergistic or antagonistic effects due to the combined effect of the three components in the composition, it is reasonable to clarify the optimal dosages of the additives experimentally by using mathematical planning - a second-order three-factor orthogonal plan. The experiment plan is shown in Table 1.

**Table 1.** Orthogonal matrix for three-factor experiment.

| Test No | X1  | X2  | X3  |
|---------|-----|-----|-----|
| 1       | +1  | -1  | -1  |
| 2       | -1  | -1  | -1  |
| 3       | +1  | +1  | -1  |
| 4       | -1  | +1  | -1  |
| 5       | +1  | -1  | +1  |
| 6       | -1  | -1  | +1  |
| 7       | +1  | +1  | +1  |
| 8       | -1  | +1  | +1  |
| 9       | 0   | 0   | 0   |
| 10      | 0   | 0   | 0   |
| 11      | 0   | 0   | 0   |
| 12      | +1.35 | 0  | 0   |
| 13      | -1.35 | 0  | 0   |
| 14      | 0   | +1.35 | 0  |
| 15      | 0   | -1.35 | 0  |
| 16      | 0   | 0   | +1.35 |
| 17      | 0   | 0   | -1.35 |

The initial data for the development of orthogonal matrix for three-factor experiment are shown in Table 2.

https://doi.org/10.1051/e3sconf/20199102041
Table 2. Initial data for the development of orthogonal matrix for three-factor experiment.

| Parameter                                | Experiment factor, $X_i$ |
|------------------------------------------|--------------------------|
| Coordinates of plan center, $x^{0_i}$    | $X_1$ | $X_2$ | $X_3$    |
| Variability interval, $\Delta x_i$      | $0.074$ | $0.741$ | $0.07$   |
| Investigation levels                     | +1    | 2     | 0.2     |
|                                          | -1    | 0.076 | 1.259   |
|                                          | +1.35 | 0.25  | 3       |
|                                          | -1.35 | 0.05  | 1       |

Table 2 introduces the following notation:

$X_1$ – content of water-holding additive, % of dry solid matter weight;
$X_2$ – content of adhesion additive, % of dry solid matter weight;
$X_3$ – content of superplasticizing admixture, % of EGAC weight.

The following factors have been accepted as resulting parameters:

$Y_1$ – flexural strength at the age of 7 days, MPa;
$Y_2$ – compression strength at the age of 7 days, MPa;
$Y_3$ – adhesion strength at horizontal joint at the age of 7 days, MPa

Compression strength and flexural strength were determined on reference samples – beams $40 \times 40 \times 160$ mm stored in air-dry conditions after formwork removal, at the age of 7 days. In order to simulate the laying of two layers of the mixture with a further determination of the adhesion strength between them cubic samples with an edge of 70.7 mm were fabricated. The samples were manufactured by placing two layers of equal thickness with a break between laying for 1 hour and 40 minutes. At the age of 7 days, the samples were tested for tensile strength by splitting along the contact plane of two layers.

### 3 Results and discussion

#### 3.1 Adhesion strength in horizontal and vertical joints

Figure 3 presents the results of test for strength by splitting two types of fine-grained concrete mixture – one based on Portland cement and the other based on the modified expanding gypsum - alumina cement.

Dash lines in the graph correspond to the Rtt of the investigated compositions of corresponding types of fine-grained concrete mixture laid without interruption, i.e. in one phase. The test results of the fine-grained concrete mixture based on Portland cement indicate that a break between laying has a negative impact on adhesion. Rtt naturally and predictably decreases almost linearly with an increase of time duration between the laying. For example, with a break of 30 minutes Rtt decreased slightly, but with a break of 60 minutes adhesion decreased more than twice.
Fig. 3. The effect of break duration between laying of horizontal layers of fine-grained concrete mixture on adhesion (splitting strength Rtt) for a mixture based on Portland cement (PC) and the one based on a modified expanding gypsum - alumina cement.

Fine-grained concrete mixture based on expanding gypsum - alumina cement showed itself in unexpected way. Under affect of 40 minutes delay between laying the adhesion strength dropped sharply to a level of 35% in comparison with the check composition. In this case, as well as with composition based on Portland cement, an increase in the break duration leads to a linear change in Rtt. But the principal difference is that the adhesion strength of composition based on the modified expanding gypsum - alumina cement increases with the increase of time duration between placements of layers. With a 2-hour break it drops to about 50% in comparison with the check sample.

The phenomena of a sharp (avalanche-like) drop in the adhesion strength with insignificant break duration between laying, and its growth with the further increase of the break period is associated with the peculiarities of intrinsic expansion deformations of the composition under test. Apparently, the expansion deformations are particularly active during the first hour after mixing with water. Therefore, applying the second layer during the period of active intrinsic deformations of the first layer leads to a drop in the degree of solidity of the layers. With a long break, the phase of active intrinsic deformations is almost completed up to the moment of application of the second layer, which leads to an increase in adhesion strength.

In order to assess the degree of solidity in the vertical joints sample beams were fabricated. A plastic partition was temporarily installed in the middle of the mold. Molds were filled in two phase. At first one half of the sample-beam was filled, then, after the required aging period, the partition was removed and the second half of the mold was filled. Thus, the vertical cold joint turned out to be in the center of the beam. A comparative evaluation of adhesion degree in a vertical joint was carried out on the bending tensile strength of the samples (Rt). The test results are shown in Figure 4.
The effect of break duration between laying of horizontal layers of fine-grained concrete mixture on adhesion (tensile strength $R_t$) for a mixture based on Portland cement (PC) and the one based on a modified expanding gypsum-alumina cement.

The patterns of change in adhesion that were obtained for vertical joints are not fundamentally different from those of horizontal joints. During the time interval considered the relationship is linear; sufficiently reliable approximation $R^2$ is at least 0.8. The fine-grained concrete based on Portland cement shows a linear decrease in the adhesion strength with an increase in the break duration between the placement of layers. The fine-grained concrete based on the modified expanding gypsum-alumina cement shows a sharp (more than 4 times in comparison with the check sample) drop in $R_t$ after a 40-minute break, and then a slow linear increase in strength over the entire time interval.

When implementing additive manufacturing for industrial purposes, breaks between the laying of adjacent layers in continuous concreting seldom attain duration of 30–40 minutes or more. As a rule, this time interval is much shorter and depends on the performance of the plant and the configuration of the designed structure.

### 3.2 Flexural strength

The regression equation describing the effect of variables on the flexural strength ($R_f$) is as follows:

$$Y_3 = R_f = 3.5 - 0.33x_1x_2 + 0.45x_1x_3 - 0.32x_2^2 - 0.3x_3^2$$

As it is seen from the equation, the average value of $R_f$ is 3.5 MPa.

In order to build up and analyze the response surfaces of the function, one of the factors ($X_1$) was fixed at the average variation level. The resulting response surface is shown in Figure 5.
In fig. 5 (at $X_1 = 0$) the domain of the function straddled the positive extremum (peak), which is located in the center of the domain of definitions of the variables. Considering the central position of the extremum, the influence of the other variables is similar: if $X_2$ or $X_3$ value deviates from the center to the right or to the left along the respective axes, the flexural strength decreases. Maximum value is achieved at medium dosages of redispersible powder and superplasticizer.

Thus, in terms of increase of the flexural strength, the dosages of water-holding additive, redispersible powder and superplasticizer should be taken at an average level, i.e. the optimum content of the components in coded form is as follows:

$$R_{fl} = \text{max when } X_1 = 0, X_2 = 0, X_3 = 0.$$  

### 3.3 Compression strength

The regression equation describing the effect of variables on the compression strength ($R_c$) is as follows:

$$Y_4 = R_c = 10.67 - 0.83x_1 - 0.62x_2 - 0.69x_3^2$$  

Therefore, it may be noted that all three factors presented in the equation have a significant impact on $R_c$. The average level of compression strength was about 11 MPa. Here we should pay attention to the slight difference between the flexural strength and compression strength of the developed mortar at the age of 7 days. It is assumed that if the $R_{fl}/R_c$ ratio is greater than 0.35, then the hardened mortar has a high crack resistance. In our case, $R_{fl}/R_c = 0.32$ on average, which indicates a rather high resistance to the development of cracks during the structure formation.

In order to build-up and analyze the response surfaces of the function, one of the factors ($X_1$) was fixed at the average variation level. The resulting response surface is shown in Figure 6.
The range of variation of $R_c$ over the whole area of variation of the three factors is quite wide: from 8 to 12 MPa. This reflects a significant effect of dosages of additives on the objective function. As is the case with flexural strength, we strive to increase the result parameter here. The maximum $R_c$ is achieved in the graph of Figure 6 (at $X_1 = -1$) and approximately equals to 12.3 MPa.

The effect of the other variables on the objective function can be described by using detailed analysis of the corresponding response surface. So the increase of the content of redispersible powder content (factor $X_2$) in the composition over the entire range of variation leads to decrease in compression strength. It can be explained by the air-entraining effect of the redispersible powder and the consequent decrease in $R_c$.

The dosage of the superplasticizer has a less definite effect. Maximum strength is achieved in the center of the variation range (at $X_3 = 0$). Deviation along the $X_3$ axis in any direction from the center leads to a decrease in the objective function.

Thus, in the term of increase of the compression strength, we should strive to decrease redispersible powder content and water-holding additive. As for the dosage of superplasticizer, it should be taken at an average level. The optimum content of the components in coded form is as follows:

$$R_c = \text{max} \quad \text{when} \quad X_1 = -1, X_2 = -1, X_3 = 0.$$

### 3.4 Effect of factors under consideration on adhesion in horizontal joint

The regression equation describing the effect of variables on the adhesion strength in a horizontal joint is as follows:

$$Y_5 = R_{ad} = 0.512 - 0.044x_1x_2 - 0.034x_1x_3 - 0.056x_2x_3 + 0.035x_1^2 \quad (3)$$

As it follows from the equation, the average level of $R_{ad}$ in a horizontal joint is 0.51 MPa, which is a fairly high value. Individual effect of each variable factor turned out to be insignificant in the experiment. However, the combined impact is strong. A detailed analysis of this effect was carried out by using graphic method. In order to build up and analyze the response surfaces of the function one of the factors ($X_2$) was fixed at the upper level of variation. The result response surface is shown in Figure 7.
Considering the type of surface, we can conclude that it is a hyperbolic paraboloid. The range of variation of $R_{ad}$ over the entire variation area of the three factors is quite wide: from 0.42 to 0.62 MPa. This indicates a significant impact of dosages of additives on the objective function. It is notable that the increase in the content of the redispersible powder (i.e., with an increase in the value of $X_2$), the average adhesion strength increases and reaches a maximum value at $X_2 = +1$. This conclusion is consistent with the well-known notions of role that redispersible powder plays in mortar. It is considered to be an adhesive additive. Considering the appropriate graph, we can draw conclusions about the influence of factors $X_1$ and $X_3$ on $R_{ad}$.

It is apparent from the graph that the function reaches maximum with a simultaneous decrease in the content of water holding additive and superplasticizer to the minimum in the variation range. This can be attributed to the known retarding effect of water-holding additives on the strength development of binders containing cement. The reason for the retarding effect of the water-holding additive on the adhesion strength under the experimental conditions is not so obvious. It requires a deeper analysis. However, it can be assumed that this is an intrinsic feature of expanding gypsum-alumina cement, since constructive and destructive processes occur simultaneously in expansive sulfoaluminate cements during the structure formation. As a result, the water sensitivity is very high and can be shifted by various factors from one condition or another.

Thus, from the point of view of increasing the strength of adhesion in a horizontal seam ($R_{bt}$), one should strive with a decrease in the EC and SP content while simultaneously increasing the RPP dosage. The optimum content of the components in encoded form is as follows:

$$R_{bt} = \text{max when } X_1 = -1, X_2 = +1, X_3 = -1.$$  

Thus, in terms of increase of the adhesion strength in a horizontal joint, we should strive to decrease the content of water-holding additive and superplasticizer while increasing redispersible powder dosage. The optimum content of the components in coded form is as follows:

$$R_{ad} = \text{max when } X_1 = -1, X_2 = +1, X_3 = -1.$$  

It follows from the above analysis that there is a combination of optimum dosages of all components for each result parameter ($Y_i$), and they are not the same. From a technological perspective, it is necessary to choose one option that would ensure obtaining of result parameters that meet the specified requirements. It should be taken into account that all the parameters have different practical significance. In our case, considering the purpose of the
composition being developed, the result parameters were ranked as follows: $Y_1 = 1$, $Y_2 = 2$, $Y_3 = 3$, bearing in mind that the more significant the result parameter, the higher the value of its rank. The average optimum was calculated with regard to the rank of result parameters and individual optimum combinations of the result variable values. Results of the calculations are presented in Table 3.

Table 3. Desired content of the components with regard to significance of the result parameters.

| $Y_i$ | Optimum factors $X_i$ | Rank |
|---|---|---|
|   | $X_1$ | $X_2$ | $X_3$ |
| $Y_1$ | 0 | 0 | 0 | 1 |
| $Y_2$ | -1 | -1 | 0 | 2 |
| $Y_3$ | -1 | +1 | -1 | 3 |
| Average optimum | -0.83 | +0.17 | -0.5 | - |
| Dosage | 0.089% | 2.13% | 0.17% | - |

4 Conclusions

1. The data obtained indicate that the break and its duration in laying of horizontal or vertical layers affects the solidity degree of the concrete structure constructed by using additive technology. When Portland cement is used as a binder, the adhesion strength of the layers decreases linearly as the break duration between placement of layers increases. If the break lasts for 30 minutes, $R_{rt}$ decreases by 14%, $R_t$ - by 27%. If the break lasts for 90 minutes, $R_{rt}$ decreases by 72%, $R_t$ - by 68%.
2. Based on the experiments, analyzing and selecting of the optimum ratio of components, the composition of fine-grained concrete regarding the chemical additives for the purpose of Construction 3D printing was specified: water-holding additive Mecellose 24502 - 0.089% by weight of mineral components, adhesive additive VINNAPAS 7220 - 2.13% by weight of mineral components, as well as dry superplasticizer based on polycarboxylate ethers Melflux 5581F - 0.17% by weight of expanding gypsum - alumina cement.

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