Investigation of viscosity of construction mixtures applied for 3D printing

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Abstract. Additive technologies meet the requirements of the modern technological stage, which applies to all areas industrial activities. Construction using additive technologies is actively developing and this technology has been introduced into production. All this makes 3D concrete printing a relevant and promising direction for the modernization of the construction industry. Concrete mixtures with certain characteristics are used as the "ink" for the construction 3D-printers. In this paper, we investigate the rheological properties of mortars for construction. We also determined the effectiveness of the mechanical activation of the initial components of the mixture, evaluated its effect on the rheological properties and the setting time of the mortar. The optimal values of the dynamic shear stress for the compositions for construction 3D printing are calculated.

1. Introduction
Additive technologies are actively used in many areas of production (mechanical engineering, metallurgy, etc.), as they correspond to the modern technological stage requirements aimed at ensuring the rational use of energy and material resources. In construction, additive technologies have also found wide application, and at the moment concrete 3D printing is successfully developing and being applied in practice. This method of construction has several advantages compared with the traditional method. The layer printing of structures can significantly reduce construction time, material consumption, and labor and machine costs. During construction using additive technologies, there is practically no construction waste left, since only as much material is used as necessary. In addition, it becomes possible to create complex geometry and make the objects architecturally expressive using construction 3D printing [1, 2]. All this allows one to achieve significant results of construction, and makes concrete 3D printing one of the most popular research subjects in this area.

For the construction 3D-printers, concrete mixtures are used as “ink”. Their deformative properties should make it possible to grow the building structures layer by layer. The curing rate of the formed layer is the defining characteristics of the concrete mixtures for a 3D printer. The deformative properties of concrete mixtures are characterized by the rheological parameters [1]. In the construction industry, instead of the real rheological characteristics (viscosity, ultimate shear stress, etc.), some generalized indicators are used: conditional viscosity, texture of the binder, workability of mortar or concrete mixes, etc.

It has been proved that the mechanical activation of cement leads to an increase in the activity of cement at the early stages of hydration, as well as a reduction in the setting time [3]. The mechanical
activation of cement contributes to an increase in the strength of concrete in the initial periods of hardening. This is a necessary condition for the using of additive technologies in construction. Thereby, the development of the methods of dry building mix activation is the key to developing a technology for preparing concrete for additive technologies in construction.

In the present work, we carried out the mechanical activation of individual components of the dry building mixture. The influence of the mechanical activation of components on the rheology of the mortar was determined. We considered the flowability and consistency of cement paste as the rheological characteristics of concrete solutions that determine their applicability in 3D printers. Such properties as workability, i.e. the ability to fill the form, while maintaining its uniformity, viscosity, as well as the ultimate dynamic shear stress were investigated. Simultaneously with the rheological characteristics, the setting time of mortars was controlled.

2. Materials and Methods

In the experimental part of the work, we carried out the mechanical activation of the initial components of the concrete mixture. The following rheological parameters were determined: the flowability (using ring spreading, GOST 31356-2007) and the consistency of the cement paste by the Vicat apparatus with a plunger (GOST 310.3-76). When choosing the optimal composition of the initial building mix, it is necessary to take into account not only the rheological characteristics but also the speed of curing. For this purpose, the setting time of the concrete mixture was determined using the Vicat apparatus with a needle according to GOST 310.3-76. In another work, an additional calorimetric analysis was performed to assess the activity and rate of cement paste hydration [4].

2.1. Materials

Compositions of the initial dry building mixtures were selected based on the known experimental data [1, 5, 6, 7]. The following materials were used for the preparation of the mortar: 1) construction sand: 0–3 mm fraction; size modulus Ms = 3.15; origin location: Kaliningrad region; 2) Portland cement Eurocem super 500, CEM I 42.5H, Petersburg Cement; 3) polypropylene fiber: length of 12 mm, thickness of 35 μm, country of origin: Russia; 4) fly ash; 5) highly active metakaolin: white, country of origin: Russia, Chelyabinsk region, Synergo; 6) STACHEMENT 1267 hyperplasticizer based on polycarboxylates for the production of ready-mixed concrete: country of origin: Poland.

2.2. Methods

The initial components of the building mixtures were modified by mechanical activation in a Retsch EMax high-speed ball mill at speeds of 500 rpm, 750 rpm and 1000 rpm for 5 minutes. Corundum balls were used as the grinding media. A series of activations was carried out to study changes in the rheological properties of the mortars caused by the mechanical activation of the initial components. We carried out activations of the following components: 1) cement, 2) ash, 3) cement and ash, 4) cement and sand. Table 1 shows 13 testing compositions.

| Sample No. | Cement (g) | Sand (g) | Water (ml) | Fly ash (g) | Metakaolin (g) | Fiber (g) | Hyperplast. (ml) |
|------------|------------|----------|------------|-------------|----------------|-----------|-----------------|
| 1          | 150 (500 rpm) | 321.7    | 114.2      | 43          | 21.5           | 0.33      | 3.22            |
| 2          | 150 (750 rpm) | 321.7    | 99.2       | 43          | 21.5           | 0.33      | 3.22            |
| 3          | 150 (1000 rpm) | 321.7    | 99.2       | 43          | 21.5           | 0.33      | 3.22            |
| 4          | 150   | 321.7    | 104.2      | 43 (500 rpm) | 21.5     | 0.33      | 3.22            |
| 5          | 150   | 321.7    | 99.2       | 43 (750 rpm) | 21.5     | 0.33      | 3.22            |
| 6          | 150   | 321.7    | 99.2       | 43 (1000 rpm) | 21.5     | 0.33      | 3.22            |
2.2.1. **The ring flowability test.** The flowability of dispersed mixtures is determined using a ring test by the diameter of the mixture spread (mm) flowing out of the ring when the ring is removed (Figure 1). The auxiliary devices for testing are a ring of stainless steel with a polished inner surface with an inner diameter of 70 mm and a height of 50 mm, a 400x400 mm glass plate, and a metal ruler. The prepared mixture is placed within the ring installed in the center of the glass plate. The glass and the ring are pre-wiped with a damp cloth. The surface of the mixture is struck of flush with the top edge of the ring. After 10-15 seconds, the ring is lifted vertically to a height of 10-15 cm and set aside. Immediately after lifting the ring, the diameter of the mixture spreading is measured with a ruler in two mutually perpendicular directions with an error of ± 5 mm. Then, the average value of the results of the two measurements of the spreading diameter is calculated. The average is rounded off to whole mm. The test is repeated using a different mortar sample. The average value of the spreading diameter of the two mixture samples, rounded off to whole mm, is taken as the final result.

|   |   |   |   |   |   |   |
|---|---|---|---|---|---|---|
| 7 | 150 (500 rpm) | 321.7 | 99.2 | 43 (500 rpm) | 21.5 | 0.33 | 3.22 |
| 8 | 150 (750 rpm) | 321.7 | 99.2 | 43 (750 rpm) | 21.5 | 0.33 | 3.22 |
| 9 | 150 (1000 rpm) | 321.7 | 99.2 | 43 (1000 rpm) | 21.5 | 0.33 | 3.22 |
| 10 | 150 (500 rpm) | 321.7 (500 rpm) | 99.2 | 43 | 21.5 | 0.33 | 3.22 |
| 11 | 150 (750 rpm) | 321.7 (750 rpm) | 99.2 | 43 | 21.5 | 0.33 | 3.22 |
| 12 | 150 (1000 rpm) | 321.7 (1000 rpm) | 106.7 | 43 | 21.5 | 0.33 | 3.22 |
| 13 | 150 | 321.7 | 114.2 | 43 | 21.5 | 0.33 | 3.22 |

2.2.2. **The mortar consistency using the Vicat apparatus.** To determine the consistency of the mixture, the Vicat apparatus is used. The apparatus is equipped with a metal plunger with a diameter of 10 mm and a ring with an internal diameter of 65 mm and a height of 40 mm (Figure 2). After mixing the solution, the ring is filled quickly in one step. Then, the ring is shaken five or six times, while tapping the plate against a solid base. The surface of the mixture is struck off flush with the edges of the ring. The plunger of the device is brought into contact with the surface of the mixture in the center of the ring and the rod is secured with a locking device. Then the rod is quickly released and the plunger sinks freely into the mortar. The depth of plunger immersion is measured by the instrument scale in 30 seconds.

2.2.3. **The setting time using the Vicat apparatus.** The Vicat apparatus with a metal needle and a ring is used to determine the setting time (Figure 3). The needle of the device is brought into contact with the surface of the mixture, which had been prepared and placed into the ring. In this position, the rod is first secured a stopper and later released. The needle sinks freely into the mortar. The moment when the setting begins is determined with the free lowering of the needle. The needle is immersed in the mixture every 10 minutes. The ring is moved after each dive so that the needle does not fall into its original position. The time from the moment of water pouring to that when the needle is 2-4 mm above the plate is considered the initial setting time of the cement paste.

**Figure 1. Ring flowability test.**
3. Results and Discussion
The following table shows the results of the tests.

| Sample No. | Ring flowability (mm) | Vicat depth (mm) | Initial setting time (min) |
|------------|-----------------------|------------------|---------------------------|
| 1          | 70                    | 37               | 190                       |
| 2          | 72                    | 35               | 195                       |
| 3          | 72                    | 33               | 210                       |
| 4          | 70                    | 36               | 220                       |
| 5          | 71                    | 37               | 200                       |
| 6          | 71                    | 37               | 180                       |
| 7          | 71                    | 31               | 250                       |
| 8          | 71                    | 36               | 230                       |
| 9          | 70                    | 36               | 260                       |
| 10         | 70                    | 36               | 180                       |
| 11         | 70                    | 37               | 160                       |
| 12         | 70                    | 35               | 160                       |
| 13         | 73                    | 31               | 240                       |

The concrete mixtures are classified as non-Newtonian fluids. Therefore the value of concrete viscosity is not constant and depends at any time on the shear stress and the strain rate (the shear rate). In [8] the group of researchers derived a dependence that makes it possible to obtain the numerical characteristic of dynamic stress during shear on the basis of the experimental data obtained.

$$\tau_0 = \frac{3}{2\pi}Rh$$

Based on the value of the Vicat plunger depth and the radius of the plunger (5 mm), the following data were obtained:
### Table 3. Dynamic shear stress.

| Sample No. | h (mm) | \( \tau_0 \) (MPa) |
|------------|--------|-------------------|
| 1          | 3      | 0,032             |
| 2          | 5      | 0,019             |
| 3          | 7      | 0,014             |
| 4          | 4      | 0,024             |
| 5          | 3      | 0,032             |
| 6          | 3      | 0,032             |
| 7          | 9      | 0,011             |
| 8          | 4      | 0,024             |
| 9          | 4      | 0,024             |
| 10         | 4      | 0,024             |
| 11         | 3      | 0,032             |
| 12         | 5      | 0,019             |
| 13         | 9      | 0,011             |

The best results were observed for mortars with the intensive joint mechanical activation of sand and cement. Mechanical activation of fly ash was ineffective.

### 4. Conclusion

As applied in civil engineering, additive technology is a new research-intensive method to construct buildings and structures. The use of 3D printing in construction has several significant advantages, such as saving time and labor or the rational use of raw materials and finished materials. Although the principles for the development of building mixtures for 3D printers exist already, this topic requires a large amount of work. This work includes the creation of a regulatory framework and the development of a technology for mixtures production on an industrial scale.

The mechanical activation of components is one of the possible modifiers of dry construction mixtures used in additive technologies. Additional grinding of the components reduces the setting time of the mixture and increases the initial strength of the mortar. This paper investigated the effect of the mechanical activation of components on the rheological properties of the mixture. The compositions modified by activation have good workability; the mixture holds its shape well and does not spread (the ring flowability test). The initial setting time is also reduced. It was established that the joint activation of cement and sand was the most effective, as it reduced the initial setting time 1.5-fold compared with the non-activated composition. The values of the dynamic shear stress are determined by the Vicat apparatus. The calculations showed that for the compositions suitable for 3D printing, the values of dynamic shear stress were within 0.019-0.032 MPa.

A study into the rheological properties of mortars and into the rate of hardening makes it possible to evaluate whether the mortars can be used as “ink” for a 3D construction printer. The method of mechanical activation of the original cement-sand mixture can be applied to control effectively the rheological characteristics of concrete mixtures.

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