Study of strength kinetics of sand concrete system of accelerated hardening

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Abstract. Methods of calorimetric analysis are used to study the dynamics of the hydration processes of concretes with different accelerator contents. The efficiency of the isothermal calorimetry method is shown for study of strength kinetics of concrete mixtures of accelerated hardening, promising for additive technologies in civil engineering.

1. Introduction
Nowadays the use of additive technologies in construction industry is one of the most actual subjects of research. Growth of interest in such technologies in construction is conditioned by a number of factors: high level of production automation, improvement of production quality, acceleration of creation processes, possibility of optimization of CAD models, reduction of production wastes [1]. These factors represent a great interest for the application of additive technologies in civil engineering and use of 3D prototyping in place of the traditional methods of construction.

The main problem for the use of additive technologies in construction is the materials used, since the ordinary concrete does not meet the requirements of 3D printing productivity [2]. Special composition for building printing should harden quickly enough to withstand the weight of subsequently deposited layers without deformation. Composites that harden for a long time slow down the process of erecting the building and cannot meet the requirements of 3D printing productivity [1]. There is a need to select and determine the optimal composition of accelerating additives to reduce the setting time of the concrete mixture.

A large number of recent works on the control of setting time show the relevance of this subject of research. Shakor P [3] uses a printed composition based on a mixture of ordinary Portland cement (32.2%) and high aluminate cement (67.8%) with the addition of a lithium carbonate accelerator (4.5%). Lithium carbonate provides hardening acceleration, high early strength and good adhesion. Kazemian A et al [4] examine the effect of the accelerating additive based on calcium chloride on the workability of the concrete mixture for the building 3D printer. It is noted that increasing the dose of accelerator from 2% to 3% has no significant effect on the loss of workability of concrete in the first 90 minutes after the preparation of the mixture. In addition to the accelerators, retarders of setting time are also widely used to maintain the concrete mixture in the liquid state for the time necessary for the extrusion of the material. Retarders and accelerators of the setting time are added at different stages of production [5]. Sulphoaluminate cement could be used as an accelerator. In the work [6] a mixture of ordinary Portland cement (93%) with a sulphoaluminate cement additive (7%) is studied as a working
material for a building 3D printer. The use of sulfoaluminate cement reduces the start and the end of setting. In the same study, a calorimetric analysis of cement paste was performed to determine the effect of the addition of sulfoaluminate cement on the hydration process. The total heat of the mixture is close to the heat release of pure Portland cement, with the exception of reduction of the induction period, with leads to an acceleration of hardening.

One of the effective methods for estimating the kinetics of cement hydration is the isothermal calorimetry method [8-10]. In this study, the efficiency of the isothermal calorimetry is evaluated to determine the optimum concentration of the accelerating additive.

2. Materials and Methods
To evaluate the strength properties of sans concrete, mechanical tests were carried out on beam samples made from compositions with different contents of the accelerating additive. The flexural tensile strength and compressive strength were measured in the first 18 and 24 hours after the samples were made. At the same time, the calorimetric analysis of the studied compositions was performed.

2.1. Materials
Four compositions of sand concrete with different accelerator contents were used. For the preparation of sand concrete the following materials were used: 1) sand for construction: fraction 0-3 mm, size modulus M с=3.15, mining: Kaliningrad region; 2) Portland cement Eurocem 500 super, CEM I 42.5 H, LLC "Petersburg cement"; 3) hardening accelerator of concrete and mortars "SPEED Vincent polyline", component: calcium chloride 25-35%.

Calculation of the concrete composition was made in accordance with GOST 26633-2015 using the "Recommendation on the selection of heavy and sand concretes" [7]. The accelerator was used in dosages of 1L, 2L and 3L for 50 kg of cement, respectively, which for water/binder ratio W/B=0.5 corresponds to 4%, 8% and 12% of water volume. The consumption of materials per 1m³ of concrete mixture is given in table 1. The samples were hardened in a chamber of normal hardening at a temperature of 20±1 °C and an air humidity not less than 90%.

| No. of composition | Cement (kg/m³) | Sand(kg/m³) | Water (kg/m³) | Accelerator (L) |
|--------------------|----------------|-------------|---------------|-----------------|
| 1                  | 459.6          | 1470.7      | 229.8         | 0               |
| 2                  | 459.6          | 1470.7      | 220.6         | 9.2             |
| 3                  | 459.6          | 1470.7      | 211.42        | 18.38           |
| 4                  | 459.6          | 1470.7      | 202.23        | 27.57           |

2.2. Mechanical testing
To carry out mechanical tests for flexural tensile strength and compressive strength, control beam samples measuring 40×40×160 mm were made (GOST 310.4-81). Strength tests of samples aged 18 and 24 hours were conducted using servo-hydraulic testing machine ToniPrax by ToniTechnik Company.

2.3. Calorimetric analysis
Calorimetric analysis of the four test compositions was carried out in accordance with GOST 310.5-88 using an 8-channel isothermal calorimeter TAM Air at a constant temperature 20°C inside the measuring channel. Standard ampoules of 20 ml were used for the analysis. Dry cement weighing 6 g was placed in an ampoule, then, using a special device, 3g of liquid (water + accelerator of the necessary concentration) were added to the ampoule and thoroughly mixed with a hand-bladed stirrer. Thus, water/binder ration of 0.5 was maintained, which was used for the preparation of samples for mechanical testing. Immediately after adding liquid and stirring, the ampoules with the mortars were sealed and placed in a calorimeter for analysis.
3. Results and Discussion

3.1. Mechanical testing

The test results are shown in Table 2.

Table 2. Results of mechanical tests.

| No. of composition | Age (hours) | R_{tens} (MPa) | R_{comp} (MPa) | Age (hours) | R_{tens} (MPa) | R_{comp} (MPa) |
|--------------------|-------------|----------------|----------------|-------------|----------------|----------------|
| 1                  | 18          | 0.63           | 4.09           | 24          | 1.41           | 8.57           |
| 2                  | 18          | 0.63           | 3.88           | 24          | 0.94           | 6.48           |
| 3                  | 18          | 0.94           | 6.66           | 24          | 1.56           | 10.95          |
| 4                  | 18          | 0.86           | 5.53           | 24          | 1.02           | 7.56           |

Dynamics of the tensile strength $R_{tens}$ and compressive strength $R_{comp}$ of the test samples are shown in figures 1 and 2 by the dependencies of the strength limits on the setting time. Figures 3 and 4 show the tensile strength and compressive strength dependencies on the concentration of hardening accelerator.

Figure 1. Graph of tensile strength growth.

Figure 2. Graph of compressive strength growth.

Figure 3. Dependence of $R_{tens}$ on the accelerator concentration.

Figure 4. Dependence of $R_{comp}$ on the accelerator concentration.

Analyzing the graphs in figures 3 and 4, we conclude that there is a nonlinear dependence of the strength on the accelerator concentration. There is an optimum value of the accelerator concentration,
which ensures a rapid increase in the strength of concrete without loss of final strength characteristics. In this study, composition No.3 can be considered as optimal.

3.2. Calorimetric analysis

Isothermal calorimetry allows to accurately measure the heat flow change in time. Due to isothermal conditions during the measurement, it is possible to detect all the exothermic reactions associated with the hydration of fresh cementitious materials [8]. The heat flow graph and heat release graph of the test samples are shown in figures 5 and 6 respectively.

![Heat flow graph](image)

**Figure 5.** Heat flow graph.

Peaks on the graph of heat flow indicate the passage of the main hydration reaction of cement, or so-called period of acceleration of reactions [9]. The higher is the peak, the more intense is the reaction. The analysis of the heat flow curves in figure 5 shows the increased heat dissipation rate at the early stage of hydration in samples No.2 and No.3 in relation to samples No.1 and No.4. This indicates a real acceleration of the hydration process of cement when the accelerating additive is injected.

Analysis of the heat release curves, obtained using the isothermal calorimetry method, allows to evaluate the dynamic characteristics of the hydration processes of compositions with and without an accelerator [10]. According to the summary heat release shown in figure 6, the accelerating effects of additives in the control ages of the test compounds were calculated. The results of the calculations are presented in table 3.

**Table 3.** Determination of the accelerating effect.

| Total heat energy (J/g) | Composition No.1 | Composition No.2 | Composition No.3 | Composition No.4 |
|------------------------|------------------|------------------|------------------|------------------|
|                        | Age (hours)      | Age (hours)      | Age (hours)      | Age (hours)      |
|                        | Acceleration (%) | Acceleration (%) | Acceleration (%) | Acceleration (%) |
| 56.8                   | 18               | 17               | 16.3             | 9.4              |
|                        |                  |                  |                  |                  |
| 82.2                   | 24               | 22.6             | 22.2             | 22.4             |
|                        |                  |                  |                  |                  |
According to the data in table 3, we conclude that the greatest accelerating effect from the introduction of the additive is observed in the composition No.3.

The graphs in figures 1, 2 and 5 show that one of the composition is the most active in the strength set at the early stages of hardening (18 and 24 hours) compared to others, and the data in table 3 confirm this.

The hardening of a concrete mixture is a highly exothermic physicochemical process. Calorimetric tests allow us to investigate the dynamics of thermal processes. The results of the evaluation of the strength characteristics and the thermodynamics of the hardening process correlate well with each other. This allows us to conclude that the method of isothermal calorimetry is applicable for studying the strength kinetics of sand concrete systems of accelerated hardening.

4. Conclusion
This paper presents the results of the study of the strength kinetics of sand concrete systems of accelerated hardening. The samples of sand concrete with different contents of the accelerating additive were subjected to mechanical strength tests at the age of 18 and 24 hours after manufacturing. Also, the calorimetric analysis of concrete mixtures with different accelerator contents was carried out. According to the results of mechanical tests, the composition with the best strength parameters at the early stages of hardening was determined. This allows us to state that there is an optimum value of the accelerator concentration, which ensures a rapid increase in the strength of concrete without loss of final strength characteristics. Calorimetric analysis confirmed the accelerating effect of the additive at the stage of cement hydration. According to the heat release graph, the accelerating effect of the additives was calculated. The composition with the optimum accelerator concentration was determined, this composition corresponds to the results of mechanical tests.

Analysis of the results of mechanical and calorimetric tests showed that the method of isothermal calorimetry is applicable for studying the strength kinetics of sand concrete systems of accelerated hardening. It becomes possible to specify the direction of searching for methods of nondestructive testing of the degree of materials strength setting in the printing process by the methods of additive technologies in construction.
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