Experimental studies of the evolution of structural and mechanical characteristics of cement stone in the process of hydration

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Abstract. An experimental approach is proposed to study the evolution of structural and mechanical characteristics of cement stone in the process of hydration. This approach is based on the use of computed tomography methods and the determination of the strength at different times of the setting of concrete compositions. It is shown that modifying additives of calcium formate and redispersible polymer powder provide an increase in strength at the initial setting time. In this case, the highest strength value 30 minutes after mixing is achieved for a composition with 2% content of redispersible polymer powder by weight of cement. The highest density is demonstrated by compositions with additives of redispersible polymer powder. A developed experimental approach, combining a non-destructive computed tomography method and a direct assessment of the compressive strength of samples of multicomponent concrete of various compositions, makes it possible to effectively evaluate the effect of modifying additives on the properties of modified concrete.

1. Introduction

The existing standard methods for studying the internal structure of concrete are based on the use of microscopy. By layer-by-layer analysis of sample sections, it is possible to determine the location and nature of the pores, as well as the quality of the formed spatial bonds in the cement stone. However, this technique has a number of significant drawbacks, which include not only the laboriousness in performing operations for preparing sections of samples, leading to their destruction, which makes it impossible to quickly study the evolution of the structure of individual samples at different stages of hydration. Classical methods for controlling the structure of concrete cannot be used in studies of changes in the structure of materials at the early periods of setting, and obtaining a clear and complete picture of the location of pores, voids and cracks using these methods is impossible [1]. The development of non-destructive control methods is an urgent task of studying the process of structure formation of concrete. The use of X-ray computed tomography for these purposes allows examining the complete structure of the material without subjecting the sample to mechanical destruction.

The use of various instruments based on computer X-ray tomography in modern materials science is becoming one of the main methods for studying the structure of inhomogeneous materials due to a large number of advantages. This method makes it possible, for example, to completely eliminate mechanical stress on the samples. The software helps not only to visually characterize the location of individual phases and pores in the samples, but also to distribute their dimensions [1, 2]. A similar result of studying the structure cannot be provided by any of the methods used for controlling the
structure of heterogeneous materials. For example, the analysis of pore sizes in multicomponent concrete samples modified with nanoparticles, as well as the nature of their distribution in the material, made it possible to conclude that the additives also have a positive effect on the microstructure of cement stone, making it denser [3]. Experience shows that from the point of view of research using computed tomography methods, in addition to analyzing the effect of modifications on the properties of a composition, a set of survey parameters should also be taken into account [2, 4].

In addition to studying the structure formation of concrete at the stage of working out the mixture in laboratory conditions, computed tomography is also actively used by researchers in the analysis of concrete samples that have been subjected to operation. By means of computed tomography, it is possible to determine the nature of destruction throughout the volume of the material, the magnitude of crack opening. Researchers from Japan conducted a series of experiments related to a detailed study of the features of visualization of images from samples without any damage to the obvious presence of cracks in them as a result of operation [5]. In addition to the visible nature of the propagation of changes in the volume of the material, it was also revealed how various kinds of kinks relate to the value of the absorption coefficient of X-ray radiation.

2. Materials and methods

The following materials were used to prepare the concrete mix: 1) Portland cement (PC) Eurocem super 500, CEM I 42.5H, Petersburg Cement; 2) Sand: unwashed construction, fractions: 1.25 mm and 0.315 mm; origin location: Kaliningrad region; 3) Silica fume (SF): wastes of the metallurgical industry, country of origin: Poland; 4) Highly active metakaolin (HAM): white, country of origin: Russia, Chelyabinsk region, Synergo; 5) Polypropylene fiber: length of 12 mm, thickness of 35 μm, country of origin: Russia; 6) STACHEMENT 1267 hyperplasticizer based on polycarboxylates for the production of ready-mixed concrete: country of origin: Poland; 7) Calcium Formate (CF); 8) Redispersible polymer powder (RPP). Material consumption is shown in Table 1.

| Components                        | Concrete mix number |
|-----------------------------------|---------------------|
|                                   | 1       | 2       | 3       | 4       |
| Cement (kg)                       | 545.3   | 545.3   | 545.3   | 545.3   |
| Sand (kg)                         | 1168    | 1168    | 1168    | 1168    |
| Water (l)                         | 416.6   | 381.9   | 381.9   | 381.9   |
| Silica fume (kg)                  | 156     | 156     | 156     | 156     |
| Metakaolin (kg)                   | 78      | 78      | 78      | 78      |
| Calcium formate (kg)              | -       | 10.4    | -       | -       |
| Redispersible polymer powder (kg) | -       | -       | 10.4    | 5.2     |
| Fiber (kg)                        | 1.2     | 1.2     | 1.2     | 1.2     |
| St. 1267 (l)                      | 11.7    | 11.7    | 11.7    | 11.7    |

In the course of the study, samples of concrete mixtures of a cubic shape with a size of 20x20x20 mm were made, for each mixture the setting times and compressive strength were determined at the early stages of hardening [6]. The dry components were mixed by hand until smooth, then polypropylene fiber was added, pre-divided into individual fibers. Next, the plasticizer was diluted in the required volume of water, after which the dry mixture was mixed. The concrete mixture was placed in molds treated with a lubricant and carefully compacted to extract excess air, which, in particular, could get into the mixture when it was stirred. After the moment of mixing with water, the countdown began until the moment of strength tests. The samples were tested for compressive strength at the ages of 30, 45, 60, 75 and 90 minutes. To carry out strength tests, a universal testing machine INSTRON ElectroPuls E1000 was used - a benchtop machine with Bluehill software and a wide range of speeds for simulating real conditions.
The internal structure and the nature of the destruction of the samples were analyzed using X-ray computed tomography on a YXLON Y. Cheetah instrument.

The shooting process consists in the selection of parameters for the location of the ray tube and the detector relative to each other. It is this setting that significantly affects the level of contrast and color intensity in future images. The shooting area can be further corrected in relation to the location of the sample in the tomograph camera. After the completion of the sample survey, the results are loaded into a graphics station, using the software “Volume Graphics Studio” of which it is possible to build a 3D model of the material and analyze specific structural elements throughout the entire volume of the material under study, as well as additionally adjust the image parameters.

3. Results
The results of testing the compressive strength of a series of concrete specimens of various compositions in the early stages of hardening are presented in Table 2. All reported results have an error within ± 10%.

Table 2. The strength of the compositions at the initial stages of hardening.

| Concrete mix number | Strength value, MPa |
|---------------------|---------------------|
|                     | 30 min   | 45 min | 60 min | 75 min | 90 min |
| 1                   | 0.06     | 0.08   | 0.10   | 0.11   | 0.13   |
| 2                   | 0.08     | 0.10   | 0.13   | 0.17   | 0.16   |
| 3                   | 0.12     | 0.17   | 0.17   | 0.17   | 0.20   |
| 4                   | 0.09     | 0.13   | 0.14   | 0.17   | 0.18   |

Various kinetics of the strength gain of the samples under study are observed. The kinetics of setting concrete of composition No. 3, which has an inflection point, is of interest for further research. The typical structure of the samples for the 90th minute of setting is shown in Figure 1.
In the case of concrete, the following component categories can be distinguished with respect to the grayscale in the resulting images. Light gray represents the cement matrix and dark gray represents the aggregate. Based on the level of X-ray absorption by the material, pores, cracks and voids are black. For any section of the samples represented by the obtained 3D model, individual components of the cement stone, cracks and pores are clearly observed.

For a series of images of ordinary sections of samples of each of the compositions, the following indicators were determined: the number of voids, their average size, their percentage of the total cross-sectional area of the sample, as well as the total area and the minimum value of the pore diameter from the obtained sample. The averaged indicators of both porosity and other values in the series for each of the compositions are presented in Table 3.

**Table 3. Average structure characteristics of samples.**

| Structure characteristic | Concrete mix number |
|--------------------------|---------------------|
|                          | 1       | 2       | 3       | 4       |
| Porosity (%)             | 6.37    | 4.65    | 3.45    | 2.84    |
| Number of voids (pcs)    | 407     | 369     | 277     | 372     |
| Occupied area (mm²)      | 24.8    | 18.98   | 14.34   | 11.7    |
| Average size of voids (mm²) | 0.06 | 0.05   | 0.05   | 0.03    |
| Minimum diameter (mm)    | 0.15    | 0.15    | 0.14    | 0.12    |

**4. Discussion**

All compositions modified with RPP and CF additives have a general tendency to increase the strength at the initial setting time compared to the control sample. The highest strength value 30 minutes after mixing is achieved for composition No. 3 with 2% of RPP.

For a more accurate interpretation of the results of the influence of additives on the initial composition of the studied composition, it is undoubtedly necessary to consider the physical and mechanical properties of these samples after they reach the design strength at the age of 28 days.

For composition No. 4 with 1% RPP content, the characteristics related to the dimensions of voids and porosity has the lowest value among all the samples presented. Composition No. 1 (without the addition of RPP and CF) has the worst performance among all the studied compositions. However, the average void size is only slightly higher than that for compositions using calcium formate and 2% RPP. Among the three compositions with chemical additives, the one with 1% RPP content has the best performance. Despite the fact that the total number of recognized particles in the fourth
composition is greater than in the third, the voids occupy a smaller area and have a smaller size, which indicates a lower probability of the presence of large pores in the material volume.

Thus, the experimental approach to study the evolution of the structural and mechanical characteristics of cement stone during hydration, based on the use of computed tomography methods and determining the strength at different times of setting of concrete compositions, makes it possible to effectively evaluate the effect of modifying additives on the properties of multicomponent concretes.

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