Method for calculating the porosity of cement composite materials using X-ray computed tomography

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Abstract. X-ray computed tomography is a promising method for non-destructive testing of the internal structure of materials. The article provides an overview of the use of computed tomography to study the structure of cement-based composite building materials. Computed tomography of four samples of fine-grained concrete mixture was performed: basic composition and compositions with different contents of additives of redispersible polymer powder and calcium formate. A method for calculating the porosity in these samples using a constructed 3D model and special software is presented.

1. Introduction
Among the composite materials based on cement, concrete occupies a special place as the most widespread and versatile material for construction. The study of its properties is not only an interesting task for researchers, but also opens up more opportunities for the entire construction industry in matters of the economic component of construction production, the introduction of new technological approaches into practice, increasing the variability of architectural forms and structural characteristics of the building.

Despite the increase in the share of concrete production and its widespread use for the construction of various objects, the issue of material quality is still the most important. However, the structure is a key aspect that affects both the quality of the material and all the basic physical and mechanical properties. In this connection, the development of methods for studying the structure and a detailed study of the results of the interaction of material components with each other is one of the important tasks in building materials science.

The existing methods for determining and visualizing the internal structure of concrete are based on the use of electron 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 spatial bonds formed in the cement stone. However, this technique has a number of significant drawbacks, which include not only the laboriousness in performing operations to prepare samples for research and their destructive nature, but also the lack of the possibility of an operational study of the structure at various stages of hydration. In addition, this method makes it possible to study the structural features of only one specific area, and not the entire material as a whole, which significantly reduces the level of reliability of the results.
The development of non-destructive control methods is the most urgent task in the study of 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. Due to the small number of studies related to the features of visualization of the structure and further interpretation of the results of imaging multicomponent concretes using computed tomography, this area is very promising for study.

In the field of studying building materials, the use of computed x-ray tomography is gaining more and more popularity. The number of scientific articles in specialized journals on building materials science in the current year and over the past few years shows how much computed tomography of concrete structures is a relevant and promising topic for study [1-10]. If in the inspection of buildings and structures the method of computed tomography is already widely used to determine corrosion [1] and the location of steel elements of reinforced concrete structures [2], then in building materials science the use of such a method of non-destructive testing has much wider application [3]. In [1], it is shown that using X-ray tomography, it is possible to track the development of destruction of steel reinforcement inside the body of a reinforced concrete structure, and it is also calculated that the loss in mass of steel reinforcement and an increase in the width of internal cracks has a linear relationship.

Of particular interest is the detection and assessment of fatigue damage in concrete. Most often, for this type of monitoring, stepwise loading of concrete samples is used and X-ray images of the samples are obtained after each loading step. Thus, it is possible to trace the origin and nature of the propagation of cracks in concrete [4]. The work [5] determines the change in the nature of destruction in the process of hydration of the cement matrix, i.e., at the early stages of hardening of fine-grained concrete mixture. The images of not-yet-hardened fractured samples clearly show the transition from plastic to brittle destruction.

In addition to studying the propagation of internal cracks in concrete, X-ray computed tomography makes it possible to track the distribution of certain additives in a concrete sample. Work [6] is devoted to determining the location of the coarse aggregate in the material and the dependence of the workability of fresh concrete on the segregation of the coarse aggregate. Using computed tomography, the authors managed to obtain images of individual grains of coarse aggregate throughout the entire volume of a concrete sample. Correlations were found between aggregate gradation, aggregate grain spacing, its distribution, and workability of concrete. These studies can be useful with the correct selection of the size of the aggregate grains and the workability of the concrete mixture, so that the aggregate grains do not precipitate when the mixture is too flow. In [7], the method of computed tomography is used to determine the spread of polypropylene fibers throughout the volume of the material.

X-ray tomography is the most effective method of non-destructive testing, which is used not only to track damage in concrete, but also to study the processes of self-healing of concrete, since the assessment of crack healing and the characteristics of voids can be determined visually. The standard visual 3D structure test, although simpler and faster to perform, provides only minimal information compared to quantitative microstructure analysis to determine self-healing efficiency. This type of volumetric analysis is critical to researchers as it allows the overall healing efficiency of the entire sample to be assessed rather than focusing on localized areas that are mechanically tested [8].

As for the distribution of pores and voids in the structure of cement composite materials, in this case, the computed tomography method has all the advantages, since it does not damage the structure of the sample under study and makes it possible to calculate the quantitative characteristics of the pores throughout the sample volume, and not only on a flat section of the material surface. This is especially valuable for industrial enterprises producing blocks of foam concrete. X-ray tomography of foam concrete samples gives a visual characteristic of pore formation in the material, it is possible to track open or buried porosity, chains of pores [9], which are formed in the process of swelling, which ultimately affects the frost resistance and moisture absorption of foam concrete blocks, and, accordingly, the quality of the products.
Comparison of the porosity of cement materials based on ordinary Portland cement and geopolymer cement is given in [10]. The article shows that the number and shape of pores in cement compositions can change over time, which is confirmed by X-ray images. Portland cement concrete turns out to be less durable than geopolymer concrete, since the distribution of pores in geopolymer concrete is more uniform and does not change over time, while pores of increasing size are found in ordinary Portland cement concrete.

The study of the structure using computed tomography shows its greatest efficiency when using this method in combination with any other tools and approaches to analysis. To correctly interpret the results, it is often necessary to use software that allows not only visualizing images, but also understanding the behavior of the material in its 3D model. The nature of computer modeling, of course, differs in the types of tomography and sometimes requires additional knowledge of this kind of instruments. However, the relationship with the technique of shooting the object and the end result can significantly increase precisely due to the visualization of the 3D model. The information obtained affects both the conclusions regarding the behavior of the material and the spatial distribution of its particles, and the process of adjusting the boundary conditions to achieve more reliable results [11].

2. Materials and methods
An YXLON Cheetah EVO X-ray tomograph in SMT configuration was used as a testing machine to determine the structure of the samples (Figure 1). This setup allows obtaining high-resolution images, as well as layer-by-layer analysis of the internal structure of the material by non-destructive methods. After completing the survey of the sample, its 3D model is loaded into the program for processing virtual planes "Volume Graphics Studio", where you can analyze specific structural inclusions throughout the entire volume of the material under study, as well as additionally adjust the image parameters.

To analyze the pore structure of a cement-based composite material, 4 compositions were selected with different contents of additives that could change the structure of the material. Based on the analysis of modern approaches to the development and modification of cement-based compositions in order to study their structure [5, 12], the basic consumption of components per 1 m³ of mixture was determined. The other 3 compositions contain different percentages of additives of redispersible polymer powder and calcium formate, which change the physical and mechanical properties of the mixture [13, 14], and, accordingly, its structure (Table 1). These additives are widely used in dry mortars, plaster mortars, building adhesives to improve adhesion, to accelerate the setting of cement stone.
Sample cubes of 20x20x20 mm in size were made from the freshly mixed mixture, 3 samples for each composition. It is possible to study such samples in a tomograph by attaching an additional attachment to the sample, since the dimensions of the cube are large for direct attachment to the rotating stage of the tomograph.

### Table 1. Component consumption per 1 m³

| Components                          | Composition number |
|-------------------------------------|--------------------|
|                                     | 1 control          | 2 CF 2% | 3 RPP 2% | 4 RPP 1% |
| Water/cement                        | 0.67               | 0.67    | 0.67     | 0.67     |
| Cement M 500 42.5N, kg/m³           | 520.83             | 520.83  | 520.83   | 520.83   |
| Sand (0.63-0.315 mm), kg/m³         | 1116.9             | 1116.9  | 1116.9   | 1116.9   |
| Water, l/m³                         | 347.2              | 347.22  | 347.22   | 347.22   |
| Silica fume, kg/m³                  | 149.3              | 149.3   | 149.3    | 149.3    |
| Metakaolin, kg/m³                   | 74.6               | 74.6    | 74.6     | 74.6     |
| Calcium formate (CF), kg/m³         | -                  | 10.4    | -        | -        |
| Redispersible polymer powder (RPP), kg/m³ | -          | -      | 10.4    | 5.2      |
| Polypropylene fiber, kg/m³          | 1.04               | 1.04    | 1.04     | 1.04     |
| Hyperplasticizer Stachement 1267, kg/m³ | 11.11           | 11.11   | 11.11    | 11.11    |

The effectiveness of the use of computed tomography increases with the use of additional tools for analysis and interpretation of the results. As part of this work, in addition to analyzing the layers modeled in "Volume Graphics Studio", we will consider the joint use of this software with additional image processing functions available in "ImageJ". The main task of this stage in the analysis of the structure of the samples under study is the numerical determination of the nature of propagation and pore size, as well as the identification of the possibility of calculating the porosity of the material. The method for calculating the listed parameters is initially based on a sample of images of ordinary sections without any specific deviations using the "Volume Graphics Studio". Then, each of the images is loaded into "ImageJ" and the type of image is changed to make it easier to find the color gradations of black and white. In accordance with the obtained scale of the dimensional ruler in the image, this value is set in "ImageJ" as the main one for all subsequent measurements in order to interpret the value from pixels in the metric system (Figure 2).

![Figure 2. Setting the scale of correspondence between the value in pixels and mm](image)

**3. Results**

X-ray images of each of the four samples are shown in Figure 3 (a-d).

![Images of the internal structure of the samples](image)
The frequency of the color distribution in the image can be traced using the histogram that analyzes this indicator. Each pixel of the image corresponds to one or another color in black and white gradation. This histogram shows how often a particular color appears in the image. Based on these data, the range of correspondence of numerical values from 0 (absolutely black) to 255 (white) with their color interpretation is revealed (Figure 4).

To calculate the content of pores and voids in the material, it is necessary to determine what value is the limiting value for all dark areas of the image. That is, to what numerical expression of color gradation the selected areas still correspond to voids and not to other components of the sample. The "ImageJ" toolkit also helps to visualize the selection. Based on the histogram, one can only assume that the selected range of values meets the necessary criteria, however, taking into account the differences in the level of contrast, as well as other features of the image, the selection may turn out to be incorrect. In addition, concrete is a multicomponent material, which undoubtedly affects the correct search for the required limits (Figure 5).

The complexity of the selection of boundary conditions also lies in the fact that pores of small size in color value can correspond to dark areas of poorly mixed silica fume, as a result of which an error appears in the sampling results. Further, the fulfillment of the specified conditions allows us to display
only the image of pores and voids without the volume of the material itself. However, at this stage, some voids can form chains, which will significantly interfere with the correct interpretation of the results of counting their number. To avoid this, the software tools must use a command that separates adjacent particles. The resulting final image, which meets all the sampling criteria, can be used to calculate various values (Figure 6).

Figure 6. Image of pores and voids obtained after applying boundary conditions

It is worth noting that not all sample images have the same cutoff range. The peculiarities of the contrast level settings, as well as the location of the selected row section, significantly affect the interpretation of the results. For example, during the selection of the boundary conditions of black and white gradation for a sample with 1% RPP content, the common condition 0-90 did not fit (Figure 7).

Figure 7. Comparison of conditions 0-90 and 0-75

This range erroneously selects the material around the voids, despite the fact that structurally excess selected particles belong to the reacted bulk of the material. While lowering the boundary conditions contributes to a more reliable interpretation of the propagation of pores and voids within the material.

Further analysis consisted in calculating the dimensional characteristics based on the obtained images of exclusively pore structure. For a series of images of ordinary cross-sections of samples of each of the four compositions, the following parameters 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. Since a dimensional scale for converting the number of pixels to the metric system was previously set for the program, all indicators were calculated in millimeters. For each composition, the indicators were determined from the images
of three images. The averaged results of calculating the revealed characteristics for each of the four compositions are presented in Table 2.

| Table 2. Average structure characteristics of samples |
|------------------------------------------------------|
| **Index** | **Composition number** | 1 control | 2 CF 2% | 3 RPP 2% | 4 RPP 1% |
|-----------|------------------------|-----------|--------|--------|--------|
| Porosity, % | control | 6.37 | 4.65 | 3.45 | 2.84 |
| Number of voids, pcs | CF 2% | 407 | 369 | 277 | 372 |
| Occupied area, mm² | RPP 2% | 24.8 | 18.98 | 14.34 | 11.7 |
| Average void size, mm² | RPP 1% | 0.06 | 0.05 | 0.05 | 0.03 |
| Minimum diameter, mm | | 0.15 | 0.15 | 0.14 | 0.12 |

For the composition with 1% RPP content, the characteristics related to the dimensions of the voids and porosity has the lowest indicators of all the samples presented. The control sample has the worst performance among all the studied compositions. However, the average void size is only slightly higher than that for composition with calcium formate and 2% RPP. Of the three compositions with chemical additives, the best performance has the composition with 1% RPP content. 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 corresponds to a lower probability of the presence of large inclusions of pores in the volume of the material.

4. Discussion

The work investigated the specificity of the use of X-ray computed tomography as a non-destructive method for studying the internal structure of cement-based composite materials. The following tasks were solved:

1. A review of studies in the field of application of computed tomography as a method of non-destructive testing for construction materials science was made.
2. Approaches to determining the internal structure of samples using a tomograph were developed taking into account the conditions necessary to obtain reliable results.
3. A method for calculating the structural units of the material has been developed.

Further research will be aimed at studying the strength characteristics of these materials in order to develop methods for a comprehensive assessment of the structure and properties of cement-based materials. Development of approaches to determining the internal structure of concrete, as well as a method for calculating this characteristic, in combination with strength studies, will prove the effectiveness of using X-ray computed tomography as a method of non-destructive testing.

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