Applicability of X-ray computed tomography for concrete cellular structure analysis

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Abstract. Nowadays, the researchers of materials science area, use direct and indirect methods such as microscopy, porosimetry, etc. for studying structural characteristics of materials. X-ray computed tomography is among one of the modern and widely used research analytical methods that provides 3D images of solid materials without any preliminary preparation, such as crashing of sample, and violation of its structural integrity. To demonstrate the potential possibilities of X-ray computed tomography, in this research matrices with cellular structure using portland cement-based cellular concrete was studied as an example. The study showed that the pore structure of cellular concrete is dominated by capillary pores with a diameter of up to 200 μm. The majority of pores did not exceed 1.6 mm in diameter, that formed during the foaming process. The calculated average size of the volumetric distribution of air voids was 0.95 mm. About 80% of large pores of a cellular concrete specimen with an average size of about 1 mm determines high porosity of the composite which is consistent with its average density values. The study of interpore structure partition using X-ray computed tomography allows for evaluation the difference in thickness from 10 μm to 0.6 mm in the zones of “confluence” of large pores. The porosity of cement matrix, including individual pores with sizes from ≈30 to 250 μm, was about 16.5%. The cement matrix is dominated by the products of cement hydration with capillary and “gel” pores. There are few nonreacted cement particles that are evenly distributed throughout the volume of the composite. To obtain more complete information about the structure of cellular concrete or any other composite, it is necessary to perform complex studies applying not only X-ray computer tomography technique, also scanning electron microscopy for evaluation of chemical analysis to identify mineral phases present and correlate them with absorption intensity of X-ray radiation on tomographic images.

1. Introduction
The performance characteristics of any material are predetermined by its structure. Construction materials are no exception, especially, portland cement-based concretes. Concrete is a composite material and is a complex system consisting of several components. Its properties are determined not only by physical and mechanical characteristics of cementitious binder, aggregates, and mineral additives, but
also by their mutual arrangement and the resulting microstructure, since the phase and structure formation of the hardening matrix of the composite occurs at the micro- and even nano-level. Since construction materials are capillary-porous bodies, their performance characteristics largely depend on the amount and type of pores. At the same time, in case of structural and frost-resistant concretes, while casting using technological methods, the porosity is reduced. In other cases, like cellular concrete, on the contrary, foaming additives are introduced. This research is focused to study the structure of cellular concrete. These lightweight composites are characterized by a cellular structure with a large number of air pores evenly distributed throughout the volume, which are formed as a result of the effect of gas or foaming agents. They are effectively used in the construction of building envelopes. The main goal of increasing the porosity of concrete is to lighten the structure and improve the thermal insulation properties [1-3]. Nowadays, direct and indirect methods for studying structural characteristics are commonly used by scientists. Direct methods include microscopic examinations such as optical and scanning electron microscopy (SEM). Indirect methods are methods for measuring porosity which include mercury porosimetry, water absorption as well as calculation methods based on hydrostatic weighing [2, 4–7]. All these classical methods provide information only about total content of pores and individual components in the resulting material, but nothing about their actual size or spatial distribution. The main drawback of the currently used methods in materials science is the lack of 3D information. With 2D photographs obtained with a microscope, it is possible to estimate the qualitative and quantitative pore distributions and such parameters as the volume fractions of individual phases locally, i.e. at certain points. To see the full picture, in this case, is quite laborious. Also, for many analyses, in particular, for electron microscopy and porosimetry, samples of construction materials are destroyed, and then properly prepared: dried and exposed to high vacuum in the case of SEM. Optical microscopy of bulk objects requires preparation of slices and thin sections [8]. With regard to cellular concrete as a material with low strength, the structure of the slice can be distorted under mechanical action. To avoid these problems, scientists more and more are willing to use other research methods of experimental physics. One of them is the method of X-ray computed tomography (X-ray CT).

Tomographic methods enable to produce 3D images of materials without any preliminary preparation, such as grinding or drying [9]. In addition, it is a non-destructive analytic method. The principle of the method is based on a 3D computational reconstruction of a sample from 2D projections. The larger the number of projections, the higher the resolution of the elements of the reconstructed volume [10]. The obtained image of the cross-section of the sample, using X-ray CT, is a map of the distribution of the absorption coefficient of X-ray radiation. With volumetric reconstruction, a spatial distribution was obtained. Lighter areas on the projection correspond to higher values of the coefficient, i.e. these are areas of a denser structure and vice versa. Accordingly, the tomographic images show differences in the density of the substance at each point on 2D projections and in the volume of the sample during its spatial reconstruction. Thus, it is possible to visualize the details within the sample framework and analyze the structure without destroying it. Over the past two decades, materials science has taken a big step in this research direction. The modern development of science and technology has contributed to the creation of X-ray tomography, which allow visualizing the internal structure of various materials with a spatial resolution of 1 μm or less [11]. An analysis of the literature on the use of X-ray CT in building materials science allowed to identify the basic direction of research, which is directly related to cellular concrete. These are the followings questions of studying porosity: determining the size of pores, their distribution by size, and the volume of the composite [12–14]. The use of the method of X-ray tomography pursues the goal of 3D modeling of the studied cellular concrete, and associate the results obtained with indicators of physical and mechanical properties of composites such as strength, heat conductivity, etc. to predict the behavior of the material during exploitation [13, 15–17]. Despite a fairly large number of studies of concrete with various structures and functional purposes, the X-ray CT method seems to be relatively "young". Its application for the problems of building materials science will make it possible to make many discoveries and expand knowledge about the formation of the structure of composites. This work shows the potential possibilities of using X-ray CT to study cellular concrete structures using the example of cement-based cellular concrete.
2. Materials and methods

2.1. Materials
The research object of this study was a specimen of cellular concrete with an average density of 500 kg/m$^3$ produced with portland cement CEMI 42.5N, according to the Russian Standard 31108-2020 and protein foaming agent Biofoam. The studied cellular specimen was an irregular shaped fragment with the size of about 13.3 × 8.2 × 8.5 mm.

2.2. Methods
X-ray CT was performed on a SkyScan 1172 high-resolution microtomograph (Bruker, Belgium) at the Soil Institute named after V.I. V.V. Dokuchaeva (Moscow). The specimen was scanned with rotation around the vertical axis at an angle of 360° with a step of 0.3°. The spatial resolution (voxel) of the reconstructed 2D sections was 3.366 μm. 3D reconstruction of the structure was performed using the specialized NRecon-software. The determination of pore size on 2D projections was carried out with the Zeiss ZEN program. Image processing by removing the black background was carried out using the Magic Wand tool with the Corel Photo-Paint program. The distribution of the X-ray absorption coefficient was recorded as grayscale values (from 0 (black) to 255 (white)). Measurement of the gray color intensity and analysis of the distribution of shades over the cross-sectional area were performed with the Image J software.

3. Results and discussions
One of the main advantages of X-ray CT over other research methods is the provision of 3D information about the structure of solid-phase samples. Therefore, the first potential application of this method for the analysis of cellular concrete is a 3D reconstruction of the entire sample or part of it (Fig. 1 a). The resulting 3D model can be moved, rotated, and scaled for a more detailed study of the structure. In addition, using specialized software, it is possible to look at the specimen "from the inside" by cutting off parts of the model or making it transparent. This enables selection and visualization in the entire volume of the specimen of the specific components and structural elements, such as mineral additive particles, fibers, air pores, inhomogeneities, etc. Note, that these fragments should differ in density from the surrounding matrix. For example, in the studied specimen of cellular concrete, the distribution of particles in the volume of the specimen with the highest density, probably nonreacted cement grains, is shown (Fig. 1 b).

![Figure 1. Reconstructed 3D image of a cellular concrete specimen (a) and visualization of the densest particles in the specimen matrix (b).](image-url)
formed during foaming, as well as capillary pores of the cement matrix and air voids from the heterogeneity of the initial mixture in the interpore partitions (Fig. 2). It is rather difficult to differentiate the pores by size, but the size boundary was chosen conventionally at the level of 250 µm (pore diameter). Thus, the diameter of the purposefully obtained pores was from 250 µm to 1.6 mm, and the pore partition contains pores from ≈30 µm to 250 µm. However, the lower-dimensional limit is also rather arbitrary, since the X-ray CT has a limited spatial resolution. Cement paste is characterized by the presence of smaller capillary pores and “gel pores” in C-S-H-products of cement hydration [18]. Detection of these pores can be realized by scanning electron microscopy. The complex application of X-ray CT and SEM will provide an opportunity to obtain more complete information about the structure of any composite, including cellular concrete.

![Figure 2](image_url)

**Figure 2.** Reconstructed cross-section of a cellular concrete specimen with a cellular structure (left) and an enlarged fragment of the structure, including interpore partitions with capillary pores (right).

Another structural part of cellular concrete is interpore partitions, that primarily determining its strength characteristics. As mentioned above, they are also porous. Their thickness varies significantly from ultra-thin to 10 µm, and less, to about 600 µm in the "confluence" zones of large pores (Fig. 2). Analysis of the reconstructed section of the cellular concrete specimen enables to determine the pore size distribution (Fig. 3). In terms of the frequency of occurrence, capillary pores with a diameter of up to 200 µm are clearly predominant (Fig. 3, a). This is consistent with the fact that the calculated average pore size was 126 µm (Table 1). The number of larger "heat-insulating" pores is significantly less (less than 5% for each individual fraction). But the larger the pore diameter, the less frequent it occurs and the larger its volume. In this regard, the volumetric pore size distribution was calculated (Fig. 3b). In this case, voids with a diameter of 800–900 µm, 1.1–1.2 mm, and 1.5–1.6 mm (the largest pores) have the maximum representation. The noted capillary pores up to 200 µm account for only slightly more than 1%. The calculated average pore size here was 0.95 mm (Table 1).

![Figure 3](image_url)

**Figure 3.** The pores size distribution in a cellular concrete specimen: a - by the frequency of meeting; b - by volume.
As mentioned above, the experimentally obtained image of the specimen and the reconstructed 2D section are a grayscale map of the X-ray absorption coefficient. Based on this, the volumetric contents of the pores and cement matrix were quantified by analyzing a number of sections. This is another one-potential application of the X-ray CT for the study of the structure of cellular concrete. For the calculation of porosity, a value of 50 units was chosen as the upper threshold for a shade of gray that would describe the presence of pores. An analysis of the interpore space was also performed in order to determine the content of nonhydrated particles of cement and its hydration products. For this, the boundary values of gray between the components were set in a similar way (Fig. 4).

Table 1. Characteristics of the pore structure and interpore partition in the studied cellular concrete specimen.

| Parameter                        | Value     | Standard deviation | Variation coefficient, % |
|----------------------------------|-----------|--------------------|--------------------------|
| Pore content, %                  | 80.01     | 1.67               | 2.09                     |
| Average pore size, mm            |           |                    |                          |
| by frequency                     | 0.126     |                    |                          |
| by volume                        | 0.956     |                    |                          |
| Interpore partition              |           |                    |                          |
| Volume percentage, %             |           |                    |                          |
| pores                            | 16.50     | 2.77               | 16.79                    |
| cement paste + pores             | 71.74     | 1.59               | 2.22                     |
| Products of cement hydration     | 10.51     | 2.25               | 21.40                    |
| nonreacted cement particles      | 1.26      | 0.39               | 31.10                    |

The predominance in the cellular concrete specimen of large pores with an average size of about 1 mm determines its high porosity - 80% (Table 1). This is consistent with the experimentally measured average density of 500 kg/m³. There is a good correlation of the results throughout the sample volume. The correlation coefficient was 2.09%. This is an indication of the accuracy of X-ray CT as a measurement method.

Figure 4. Differential and integral distribution of pixels as an interpretation of the X-ray absorption coefficient in the form of shades of grey in the cross-section of a specimen obtained by X-ray CT. The study of the interpore partition showed that the content of nonreacted cement grains was 1.26%, only (Table 1, Fig. 4). This indicates a high degree of cementitious binder hydration. The result
obtained can be associated with the distribution of particles with the highest density in the cellular concrete specimen (Fig. 1 b) and with a fairly uniform structure of the cement paste. The porosity of the cement matrix, calculated using the data on the distribution of individual pores with sizes from ≈30 to 250 µm, was 16.5%. The proportion of cement hydration products was only 10.51%. However, taking into account that they mainly contain "gel" and capillary pores, it is logical to consider them together with a porous cement paste with lower grayscale values. In this case, they account for 82.25%. It is worth noting that the resulting component ratios are very approximate, as the gray cut-off values were tentatively determined. To obtain more accurate and reliable results, it is necessary to perform complex studies using not only X-ray CT, but also scanning electron microscopy with the possibility of chemical analysis using an energy dispersive spectrometer to identify and clearly distinguish the phases present and correlate them with the intensity of X-ray absorption in tomographic images.

4. Summary
This research work presents the results of studying the structure of cement-based cellular concrete, obtained using X-ray CT. This example shows the potential application of this method in the study of cellular concrete, as well as other composites. The first potential application of X-ray CT for the analysis of cellular concrete is the creation of a 3D model of a specimen to obtain information about the volumetric structure: visualization of specific components and structural elements, such as mineral additive particles, fibers, air pores, irregularities, etc. This feature is one of the main advantages of X-ray CT vs. microscopy. Another potential application of X-ray CT is to obtain quantitative structural data by easier way, vs. microscopy or porosimetry. The study of the pore structure of cellular concrete showed that the sample is quantitatively dominated by capillary pores with a diameter of up to 200 µm, while the main volume is occupied by pores formed during foaming, with an average size of 0.95 mm. The porosity of the specimen, obtained from the quantitative analysis of the distribution of shades of gray for several 2D reconstructed sections, was 80%. This is in agreement with the average density of cellular concrete. The study of the structure of interpore partitions allows establishing that their thickness varies from 10 µm to 0.6 mm. Analysis of images allows to establish their porosity which was 16.5%. The cement matrix is dominated by the products of cement hydration with capillary and "gel" pores. There are few nonreacted cement particles and they are evenly distributed throughout the volume of the composite.

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