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Review

X-ray Computed Microtomography technique applied for cementitious materials: A review

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ABSTRACT

The main objective of this article is to present a bibliographical review about the use of the X-ray microtomography method in 3D images processing of cementitious materials microstructure, analyzing the pores microstructure and connectivity network, enabling tthe possibility of building a relationship between permeability and porosity. The use of this technique enables the understanding of physical, chemical and mechanical properties of cementitious materials by publishing good results, considering that the quality and quantity of accessible information were significant and may contribute to the study of cementitious materials development.

1. Introduction

The development of studies aimed at structuring the oil industry, more specifically in the area of in well cementation, is a fundamental for nations that have oil production as main generator of profits. Cementation is an extremely important work for the drilling and oil well completion phases, resulting in a major impact on the well productivity.

The formulation of cementitious materials are composed of two or more phases, with secondary phases denominated load or support and primary phase named array. There are three fundamental phases in the composites formation in the Portland cement matrix: cement paste, aggregate and slurry/aggregate transition zone. Even with solid appearance (rigid), these materials have an internal network with porous microstructure that may contain, collect or transport, in its indoor connectivity, gases or liquids.

The productive process itself promotes the formation of this essentially porous structure. To ensure workability, the amount of water used is greater than the required to promote the hydration of the cement grains. During the drying stage, this surplus water evaporates leaving void spaces in the structure. In addition, there is also an incorporation of air bubbles during the mixing process, what corroborates to this porosity.

Due to the variation of pore dimensions, from a few angstroms up to several micrometers, as well as their very irregular shapes, the techniques used to study these materials range from simple equipment and operations up to sophisticated ones (Campiteli, 1987).

There are several techniques that can be used for characterize the material structure void spaces. Some studies use physical principles as mercury porosimeter, nitrogen adsorption, densities relationship using helium and mercury pycnometer, etc.

Other approach is the use of the images, such as, optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray or Gamma-ray tomography and nuclear magnetic resonance.

According to Pessôa (2011), image analysis, enables to build a relationship between the microstructure of the material with its physical and mechanical properties, being this a tool for structure and morphology investigation in a microscopic scale.

The X-ray computed microtomography was developed based on the traditional tomography, however it was focused in small samples analysis. The literature shows that the studies regarding pore microstructure analysis of cementitious composites using this technique are recent. However, the results are favorable to its appliance in this kind of research.

The technique is capable to provide a variety of information about the sample microstructure, such as total pore volume, open pore volume (permeability), pores fragmentation index, fractal dimension, pores average size, presence of microcracks, among others.

Some studies have used the method of X-ray computed microtomography for processing digital images, like Gallucci et al. (2007), who developed a study of network connectivity and texture of pores in 3D images of cement slurries pastes with age of 1–60 days using Synchroton microtomography MS-X04SA. His study developed a quantitative and morphological investigation of pore and its network connectivity. Using an X-ray computed microtomography image processing

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technique with high-resolution 3D images, he was able to show a certain relationship between the pore networks in the microstructure of cement slurries as the spatial resolution was increased. The authors obtained good results using this technique, once that the quality and quantity of accessible information were significant, which may contribute to the study development of cementitious materials.

The word of Promentilla and Sugiyama (2010) is presented the application of X-ray microtomography to characterize the internal structure of mortars that were exposed to freezing-thawing action and the results suggest that the initial air voids follow a lognormal distribution with the highest population of modal size around 30–50 µm, irrespective of the type of mortar (four different types of mortars specimens were studied). Promentilla et al. (2009) also present another study on quantification of tortuosity in hardened cement pastes using synchrotronbased X-ray computed microtomography and their results show the feasibility of using synchrotron microtomography coupled with 3D image analysis and random walk simulation to measure the diffusion tortuosity that has a direct bearing on transport properties.

In their papers, Lu et al. (2006) used the computed microtomography technique to study the pore structure and permeability of Portland cement in concrete. The pore system distribution, size and pore connectivity were characterized. It is important to highlight that the study of the pores structure is recognized to be the key to the understanding of the physical, chemical and mechanical properties of cementitious materials. In the article, Bernardes et al. (2015), highlights the use of microtomography technique in the analysis of images is useful to investigate the relationship between the pore network of the samples and its permeability, once the connectivity between the pores is directly related with the fluids and Ionic species diffusion.

Through a study of igneous rocks using 3D images, Baker et al. (2012) applied the technique of X-ray microtomography at the Electra's laboratory located in Trieste (Italy). The authors reveal that the use of the X-ray tomography was most used on biomedical and clinical research in recent decades, only being acknowledged recently in the construction a three-dimensional digital map in the field of Geosciences. In addition to study of the rocks internal structure, morphology and texture of the components, the authors presented a literary review of research that used the principles and techniques of X-ray microtomography, showing how this method is effective and useful for scientific research.

The work of Cid et al. (2017) presents an estimation study of porosity and permeability in porous materials, based on the work of Baker et al. (2012). The parameters were described by the use of X-ray computed microtomography, which provided the opportunity to construct three-dimensional images with high resolution, enabling to perform an investigation of the void volume percentage. Promentilla et al. (2016) showed the evaluation of microstructure and transport properties of deteriorated cementitious materials from their X-ray Computed Tomography (CT) Images, finding indications suggest that as effective porosity increases, the geometric tortuosity increases and the permeability decreases. Correlation among these microstructure and transport parameters is also presented in this study.

Sugiyama et al. (2010) applied of synchrotron microtomography for pore structure characterization of deteriorated cementitious materials due to leaching, showing that Indications suggest that the deterioration of the cement matrix due primarily to the dissolution of portlandite decreases the diffusion tortuosity to a single digit as the degree of pore connectivity becomes larger at the submicron scale. Darma et al. (2013) highlights a study on demonstrates aplication of microfocus X-ray computed tomography of solute transport in cracked concret, getting results from microtomographic images suggest that the entire cracked space may not always be filled with the tracer.

In this way, the technique is capable to provide a variety of information about the microstructure of the sample, such as total pore volume, open pore volume (permeability), pores fragmentation index, fractal dimension, pores average size, presence of microcracks, among others.

The relationship between sample size and spatial resolution is a critical point when working with X-ray microtomography. A small size of sample is crucial for a high spatial resolution. However, working with small samples is a problem on representation, since the sample volume should contain the properties of the entire product, in order to obtain an acceptable estimative of the material overall properties. (Cnnudde et al., 2011).

Thus, the studies involving the analysis of the pore structure of cementitious matrix composites using X-ray microtomography have the most diverse sampling techniques. The main objective of this work is present a bibliographical review about the use of X-ray microtomography method to 3D image processing in cementitious materials or rock microstructure analysis of pores and its connectivity network, being also aimed to develop a possible relationship between permeability and porosity.

2. Materials and methods

2.1. X-ray microtomography technique in 3D image processing

The X-ray microtomography is a three-dimensional image processing technique that uses a series of radiographic images to reconstruct a map of X-ray absorption on the object in analysis. The method is identical to the computed axial tomography (CAT), used for medicine, however the microtomography achieves a much higher spatial resolution, since it combines a very strong brightness, a synchrotron monochrome radiation with high quality optical X-ray detection. A schematic illustration of a microtomography system is presented in Fig. 1. Variations of space expansion were made possible due to the use of different microscope lenses, which focus the X-ray image on the CCD detector.

Synchrotron-based microtomography was widely used in recent decades in order to study the porous structure of materials, among them the microstructure of pores in concrete and cement pastes. Applied to concrete, the microtomography has been applied to problems involving fractures (Bentz and Garboczi, 1991), sulfate attack (Navi and Pignat, 1996) and cement hydration. (Neto et al., 2011). The significance of each technique is on the ability to capture a high-resolution 3D internal structure, as seen in Fig. 3.

Then, the images produced using tomography are placed in a gray scale, where the intensity of the pixels is directly related with the object's density (Brun et al., 2010). An example of a two-dimensional image with a cross-section of the cement Portland microstructure is shown in Fig. 2. In this figure, it is possible to observe the characteristics of a hydrated cement slurry cement grains that did not react (white spots), pore space (black dots), transition zones surrounding aggregate, such as the change of density in the aggregate materials.

It must be emphasized that this is just one cross-section of hundreds that are generated in a single run. The Fig. 3 highlights the data nature in a 3D image, this image illustrates how a photography can be digitally sectioned according to an arbitrary spatial orientation based on the microtomography processed data.

2.2. Digital 3D processing

The digital processing built through the reading of the data generated by microtomography makes it possible to employ an extensive library of image analysis, for example, the library of the 3D PORE software, developed and used by the SYRMEP research group Synchrotron Light Laboratory of Elettra at Trieste (Italy). (Brun et al., 2010). The Fig. 3 was constructed by using the processing of the 3D PORE, in a sample of cementitious material. Through various basic image analysis, it is possible to distinguish solids from voids, as presented in Fig. 2 (2D) and in Fig. 3 (3D). Also using the analysis it is possible to obtain results of void spaces and the connectivity of these spaces, seeking to identify

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