Extracting the pore structure features of cement based materials using 3D X-ray Computed Tomography

***M. Murugan*[1] *and Manu Santhanam*[2]**
[1]Research Scholar, Department of Civil Engineering, IIT Madras, India
[2]Professor, Department of Civil Engineering, IIT Madras, India
Mobile - +91-44-9962661506, Email ID – murugan88m@gmail.com

Abstract

3D X-ray Computed Tomography (CT) is a powerful tool in assessing the pore structure features of cement based materials. In this paper, X-ray CT testing were performed over different cement paste specimens incorporated with nanomaterials such as reduced graphene oxide (rGO), dry aluminium oxide nanopowder (n-Al_2O_3), and colloidal silicon dioxide nanoparticles (n-SiO_2) respectively. Such kind of non-destructive testing was conducted to determine the influence of nanomaterials on the pore refinement of cement paste. The acquired tomographic images were processed with the image analysis software and further analyzed to determine the distribution of pores/voids inside the cementitious matrix. In addition, the paper will also present the pore structure details of enhanced porosity concrete (EPC) specimen tested using X-ray CT.

Keywords: reduced graphene oxide, pore structure, enhanced porosity concrete and computed tomography

----

1. Introduction

Cement based materials are widely used for the construction of skyscrapers, hydraulic structures, radioactive waste disposals, dams, underground tunnels, offshore structures, highways and bridges [1]. The durability of such concrete structures is affected due to the migration/diffusion of deleterious ions (chloride and sulphate) into the cementitious matrix through its interconnected pore channels [2]. To resolve the aforementioned issue, finely grained micro (mineral based) additives such as Fly ash and GGBS (ground granulated blast furnace slag) are used as a partial replacement to cement in order to strengthen its pore structure [3]. There are investigations are under the way to use nanomaterials having high surface area such as nano silica, nano alumina, carbon nanotube, graphene nanosheet and nano Titania as a minor addition to further reduce the pore volume fraction in hydrated cementitious matrix [4, 5]. In contrast to the aforementioned non-permeable concrete applications, a special type named enhanced porosity concrete (EPC) is a highly permeable concrete medium which allows fluid to move under gravity through inter-linked pores/voids are used for different functions such as pavements, sidewalks, storm drains and shoulders in order to increase the ground water recharge by allowing the storm water [6-9].
3D X-ray computed tomography is a non-destructive technique which provides a 3-dimensional view in the form of a grey scale image due to the differential penetration of X-rays through the material [10-13]. To investigate on the pore structure features of the cementitious matrix,

Mercury intrusion porosimetry (MIP) are widely used [14]. However, the porosimetry studies are used to determine the quantity of gel pores (< 10 nm) within the cement hydrates and capillary pores (10 nm to 10 µm) that are formed during the hydration of cement [15]. The presence of larger pores/voids (> 100 µm) inside the cementitious system is accurately quantified only through analysing the CT images of the cement system [16]. In this paper, the pore structure features of two dissimilar cementitious systems (permeable and non-permeable) were determined by processing and analysing the CT image of those scanned objects.

2. Materials and methods

53 Grade ordinary Portland cement (OPC) conforming to IS 12269-1987 were used for both cement pastes and EPC mix. Table 1 shows the chemical composition of the cement.

Table 1 – Chemical composition of 53 grade ordinary Portland cement (conforming to IS 12269-1987) used in this study

Name of the compound	CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	Cl
% by weight	62.16	20.95	5.14	3.06	3.08	1.33	0.009

Firstly, the effect of three different nanomaterials such as reduced graphene oxide (rGO), dry aluminium oxide nanopowder (n-Al_2O_3) and colloidal silicon dioxide nanoparticles (n-SiO_2) on the pore refinement of cement paste were assessed in this study. The graphene nanosheets (rGO) used in this study is of size ranging between 5 nm to 1500 nm were extracted from natural graphite using Chemical Oxidation method. The other nanomaterials used in this work were dry aluminium oxide nanopowder (n-Al_2O_3) of average size less than 50 nm and silicon dioxide nanoparticles of average size 12 nm dispersed in deionized water (n-SiO_2) having surface area of 40 m^2/g and 225 m^2/g respectively were obtained from Sigma-Aldrich. Four different cement paste specimens of size 25 x 25 x 25 mm were prepared with a water to cement ratio of 0.32. One mix was kept as reference paste whereas the remaining three mixes were incorporated with different nanomaterials (rGO, n-Al_2O_3 and n-SiO_2) at concentrations such as 0.02%, 0.20% and 4.0% by weight of cement respectively. Polycarboxylic ether (PCE) based superplasticizer were used for effective dispersion of nanomaterials within the cementitious matrix. The specimens were demolded after 24 hours and next cured in limewater solution for a period of 28 days. For X-ray CT scanning, the samples of size 10 x 10 x 10 mm as shown in Fig. 1a were cut from the paste specimens (25 x 25 x 25 mm) using Diamond tipped-saw.

Secondly, the X-ray scans were performed over six number of enhanced porosity concrete specimens to determine the alterations in its pore structure. The EPC cylindrical specimens (100 mm dia. x 200 mm height) were prepared with a water to cement ratio of 0.32. For the preparation of EPC specimen, crushed granite aggregates ranging between 6.3 mm to 300 µm conforming to IS 383-1970 were used. The aggregates were carefully gap graded in order to allow water to permeate through the pore channels of such concrete system. To increase its workability, PCE based superplasticizer of 0.50% by weight of cement was added into the concrete mix. Further, the specimens were demolded after 24 hours and cured in tap water for a period of 7 days. For CT scanning, the EPC sample of size 40 mm x 100 mm as shown in Fig. 1a were cored out from the EPC cylindrical specimen of size 100 mm x 200 mm using core cutting machine. Fig. 1b and 1c shows the X-ray scanned 2D CT slice of cement paste and EPC sample.

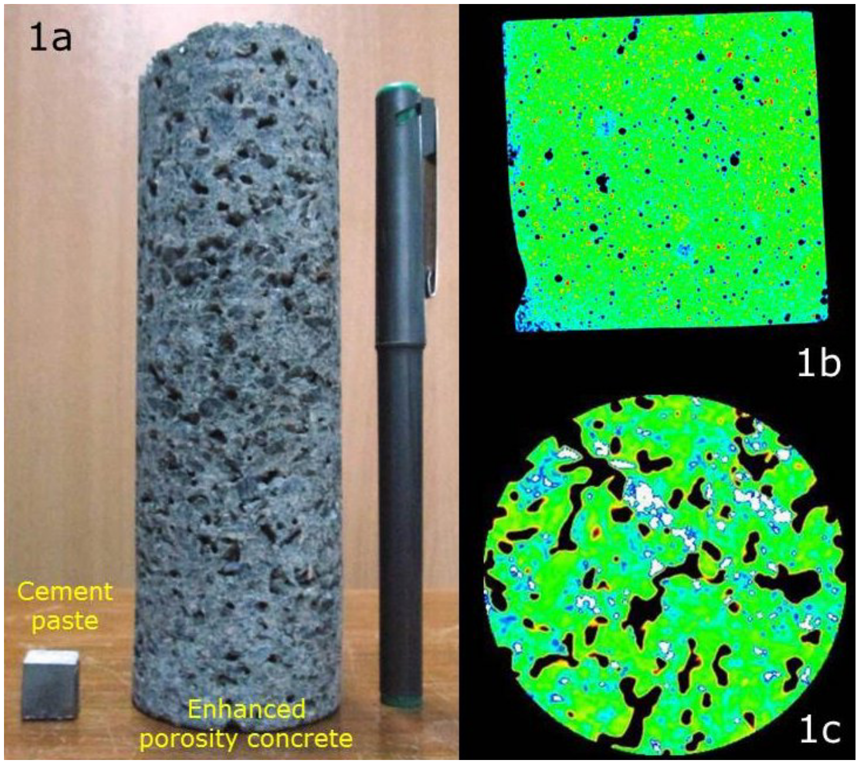

Fig. 1 – a. Cement paste and EPC sample scanned using X-ray CT, b-c. Pseudo-coloured 2D CT slice of cement paste and EPC sample

The CT scans were performed using GE phoenix v|tome|x s 3D X-ray CT testing machine which is available at the Centre for Non-destructive Evaluation (CNDE), Indian Institute of Technology Madras. The contrasting cementitious systems such as cement paste and EPC samples were scanned using an X-ray source generated at a voltage and current of 120 kV and 70 µA respectively. The scanning rate was maintained around 4 milliseconds per image. Caesium Iodide flat panel detector equipped inside the CT machine was used to convert the X-rays into light source. As a result of X-ray scanning, 2D radiographs were extracted with a data acquisition software named Phoenix datos|x CT. Further, the 2D data were reconstructed using the software named VGStudio MAX version 2.2 to obtain the 3D image of the scanned material.

3. Results and discussion

3.1 Analysing the CT images of the different cement paste samples

The presence of pores/voids inside the reconstructed CT image of the different cement pastes were visualized by adjusting its grey level value (GLV), with darker and brighter regions corresponding to lower and higher value of attenuation co-efficient. Fig. 2a-c presents the reconstructed and transparent CT image of the cement paste sample.

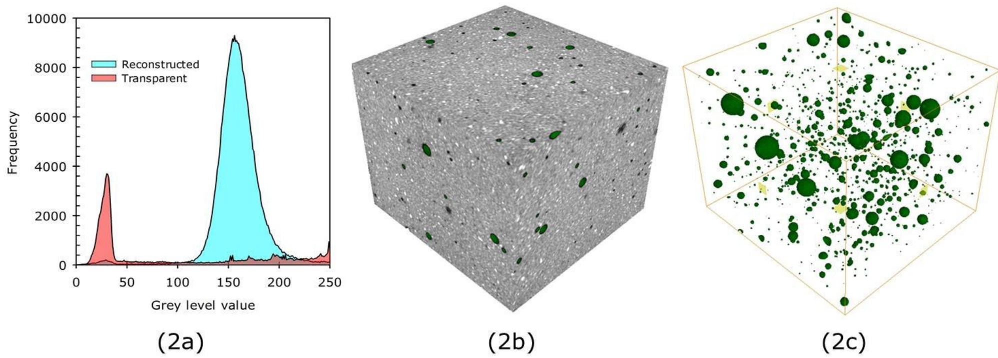

Fig. 2 – a. Adjusted GLV, b. Reconstructed and c. Transparent CT image of the cement paste sample

The 3D image reconstruction software VGStudio MAX version 2.2 is supplied with an add-on module named Porosity/Inclusion Analysis. The module operates with a wide range of algorithms allowing pores/voids to be located within the scanned object and further yields quantitative information about those voids/pores. By analysing the material with such module, detailed information about each individual defect (pores/inclusion) such as its volume, projected size (Feret's diameter) and position in the material are obtained. Additionally, colour coded 3D views of the analysed material are also obtained through such module. The only input data that are required to run the analysis are lower and upper bound of the pore/void volume which are expected to be present in the scanned material. In this study, the CT image of the different paste specimens were analysed using the porosity module. Fig. 3a-b presents the colour coded transparent 3D image of the pastes showing two different ranges of pore/void volume.

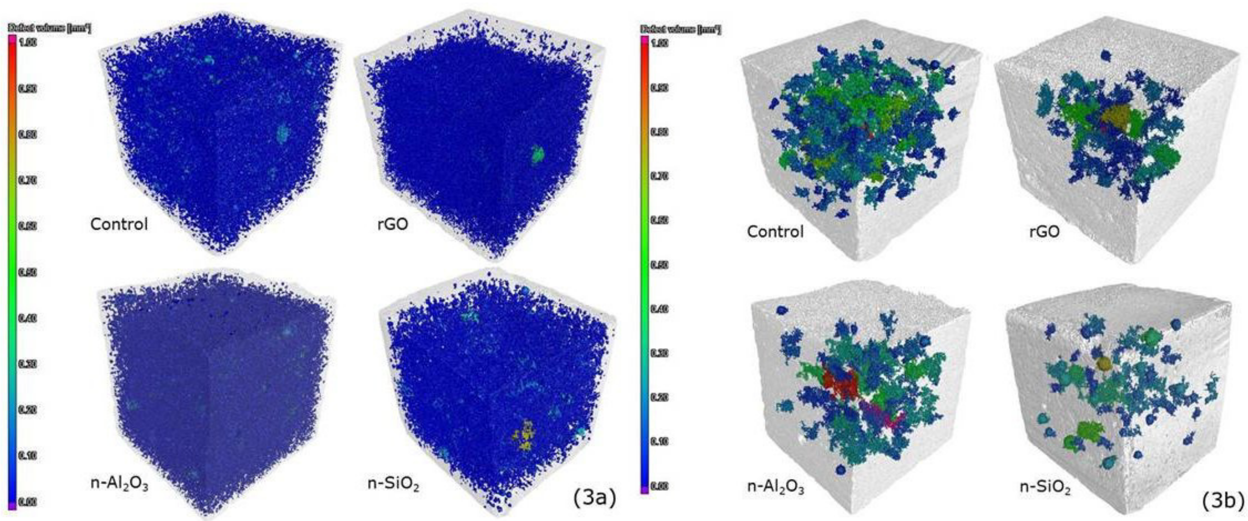

Fig. 3 – Colour-coded transparent 3D images of the different pastes having pore/void volume ranging between a. 0.01 to 0.75 mm^3 b. 0.07 to 0.75 mm^3

Further, the module provides the volumetric distribution of pores/voids inside the different pastes after analysis. The analysis results were depicted in the form of histogram which is presented in Fig. 4.

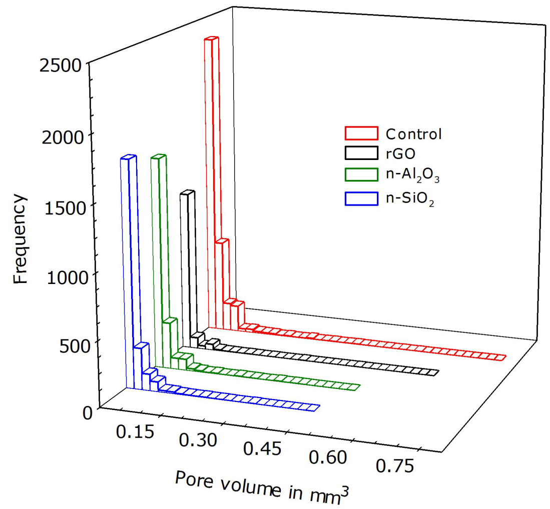

Fig. 4 – Histogram results representing the pore/void volume inside the different pastes analysed using Porosity Module of the software VGStudio MAX 2.2

The histogram results suggest that the addition of nanomaterials such as rGO, n-Al_2O_3 and n-SiO_2 in cement paste were found to reduce the porosity to a significant extent when compared to the reference paste. In particular, the addition of graphene nanosheets (rGO) in cement paste was found efficient in refining the pores when compared to other nanocomposites.

Analysing the CT images of the EPC samples

Fig. 5 shows the CT image of the EPC sample (of 40 mm x 110 mm) of six numbers scanned using X-ray CT and then analysed using an image analysis technique to determine the pore size distribution inside such concrete systems. Image Pro Premier 9.1 was the software used for analysis.

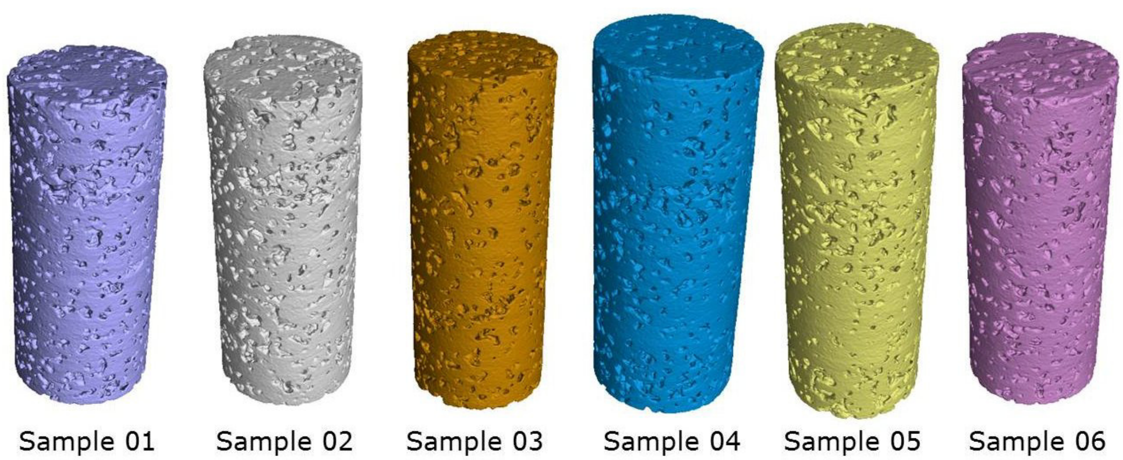

Fig. 5 – 3D CT image of six number of EPC samples scanned using X-ray CT

The reconstructed CT image of the EPC samples were sectioned into multiple slices (of around 130 numbers) and then processed using the tools such as auto calibration, batch processing, length and area measurement that are available within the software in order to determine the size of pores inside the cementitious system. To have an enclosed boundary, the actual diameter of the EPC slice considered for analysis were cropped and decreased (as 3.5 cm diameter). Fig. 6 shows an unanalysed and analysed CT slice of the EPC sample obtained from the image analysis software. The statistical data obtained from the analysis were converted into a histogram representing the distribution of pores/voids inside the concrete system as shown in Fig. 7.

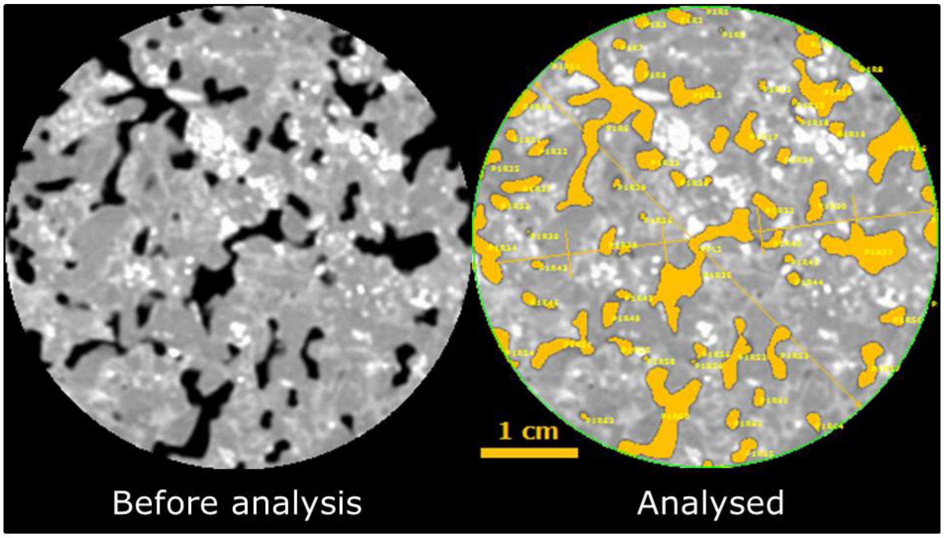

Fig. 6 – CT slice of the EPC sample before and after image analysis

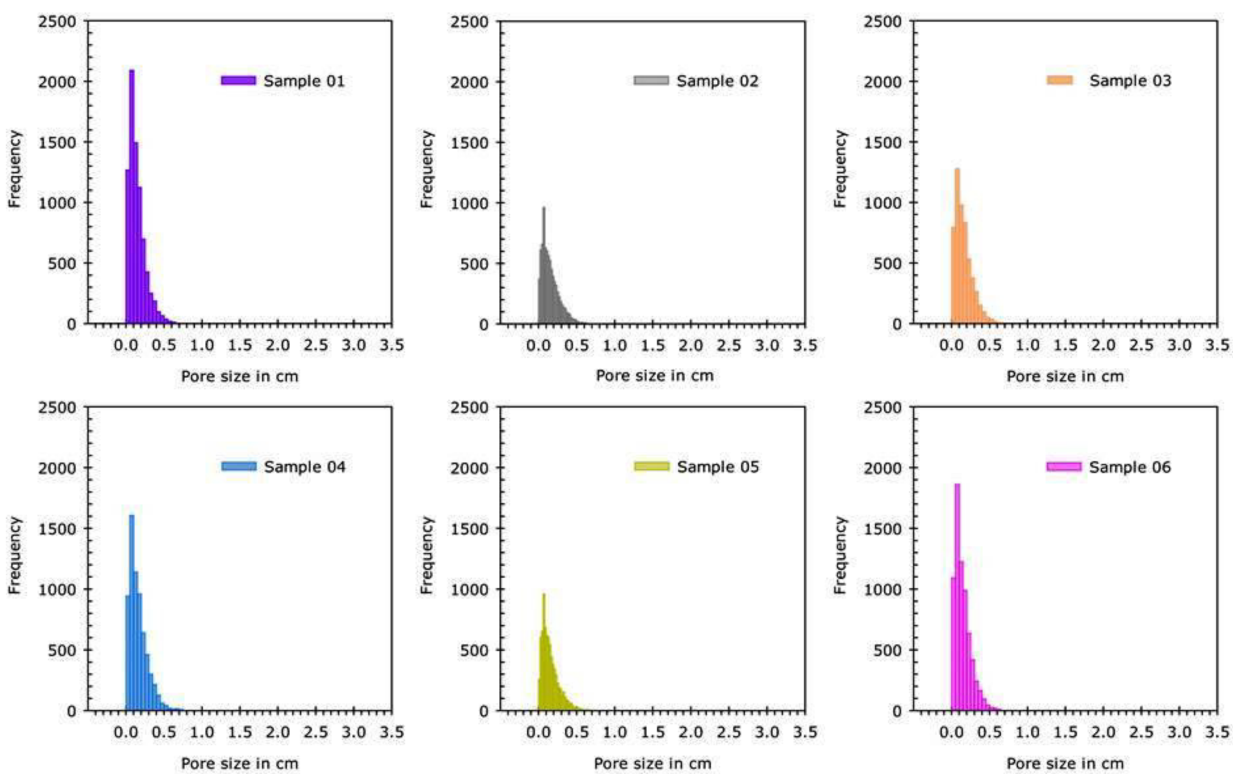

Fig. 7 – Histogram results representing the pore size distribution of the different EPC samples

From the results of image analysis technique, it was found that the size range of pores/voids that were accumulated inside the six EPC samples were in between 1 to 7 mm respectively. To control the water percolation rate of the EPC specimens, such results were found useful in redesigning the concrete mix.

Conclusions

In this paper, the pore structure features of cement paste and EPC sample were determined from the CT images of the scanned material. The key findings resulted from analysing the tomographic images with the help of image analysis technique are summarized as below:

1. The addition of nanomaterials such as rGO, n-Al_2O_3 and n-SiO_2 in cement paste were found to decrease the porosity to an appreciable extent when compared to control paste. Especially, the rGO incorporation in cement paste was found efficient than the other nanocomposites.
2. The 2D image analysis results suggest that the interconnected pores/voids inside the EPC sample were found to have a size range in between 1 to 7 mm respectively.

Acknowledgement

The authors thank Varun Kannaiyan, Project Officer, Centre for Non-Destructive Evaluation Facility, Indian Institute of Technology Madras for his help in scanning the X-ray tomographic images.

References

[1] Wan K, Li Y, Sun W. Experimental and modelling research of the accelerated calcium leaching of cement paste in ammonium nitrate solution. Construction and Building Materials. 2013;40:832-46.
[2] Claisse PA. Chapter 25 - Durability of concrete structures. In: Claisse PA, editor. Civil Engineering Materials. Boston: Butterworth-Heinemann; 2016. p. 259-74.
[3] Rodríguez-Camacho RE, Uribe-Afif R. Importance of using the natural pozzolans on concrete durability. Cement and Concrete Research. 2002;32(12):1851-8.
[4] Konsta-Gdoutos MS, Metaxa ZS, Shah SP. Highly dispersed carbon nanotube reinforced cement based materials. Cement and Concrete Research. 2010;40(7):1052-9.
[5] Ghafari E, Costa H, Júlio E. Critical review on eco-efficient ultra high performance concrete enhanced with nano-materials. Construction and Building Materials. 2015;101, Part 1:201-8.
[6] Neithalath N. Extracting the performance predictors of Enhanced Porosity Concretes from electrical conductivity spectra. Cement and Concrete Research. 2007;37(5):796-804.
[7] Sumanasooriya MS, Neithalath N. Pore structure features of pervious concretes proportioned for desired porosities and their performance prediction. Cement and Concrete Composites. 2011;33(8):778-87.
[8] Kayhanian M, Anderson D, Harvey JT, Jones D, Muhunthan B. Permeability measurement and scan imaging to assess clogging of pervious concrete pavements in parking lots. Journal of Environmental Management. 2012;95(1):114-23.
[9] Fletcher TD, Andrieu H, Hamel P. Understanding, management and modelling of urban hydrology and its consequences for receiving waters: A state of the art. Advances in Water Resources. 2013;51:261-79.
[10] Stock SR, Naik NK, Wilkinson AP, Kurtis KE. X-ray microtomography (microCT) of the progression of sulfate attack of cement paste. Cement and Concrete Research. 2002;32(10):1673-5.
[11] Farber L, Tardos G, Michaels JN. Use of X-ray tomography to study the porosity and morphology of granules. Powder Technology. 2003;132(1):57-63.
[12] Rattanasak U, Kendall K. Pore structure of cement/pozzolan composites by X-ray microtomography. Cement and Concrete Research. 2005;35(4):637-40.
[13] Promentilla MAB, Sugiyama T, Hitomi T, Takeda N. Quantification of tortuosity in hardened cement pastes using synchrotron-based X-ray computed microtomography. Cement and Concrete Research. 2009;39(6):548-57.

[14] Zhou J, Ye G, van Breugel K. Characterization of pore structure in cement-based materials using pressurization–depressurization cycling mercury intrusion porosimetry (PDC-MIP). Cement and Concrete Research. 2010;40(7):1120-8.
[15] Panesar DK, Francis J. Influence of limestone and slag on the pore structure of cement paste based on mercury intrusion porosimetry and water vapour sorption measurements. Construction and Building Materials. 2014;52:52-8.
[16] Wan K, Xue X. In situ compressive damage of cement paste characterized by lab source X-ray computer tomography. Materials Characterization. 2013;82:32-40.