Micro CT analysis of geopolymer composites

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There are many situations when you need information about the internal structure of the materials. Computed Tomography (CT) is a non-destructive method of evaluating the internal structure, which was originally used for medical examination of the human body. Today it is already used in many different fields, where the aim is the examination of the internal structure of the material. Geopolymer is a term for all inorganic polymeric materials which are prepared from alumino-silicate materials by geopolymerization in alkaline environment at normal temperature and pressure. Computed tomography (CT) is an advanced method that allows the study of the structure based geopolymer composites with short fibers or nanoparticulate reinforcement.

Keywords: computerized tomography (CT), non-destructive method, internal structure, geopolymer

1 Introduction

Still growing industry produces large quantities of waste materials, such as water, oil, solvent and solid waste (ash, glass, stone dust, spoil, etc.). The main problem of the environment is becoming the disposal of of these wastes. The use of waste materials in the production of cement, mortar, concrete and other materials intended for construction leads to a reduction in production rates of a specific product. Another benefit is energy savings and reduction of greenhouse gases CO_X which are produced during the manufacture of cement. The use of geopolymers leads to a reduction in consumption of raw material sources, protect the environment and reduce the environmental damage caused by mining and use of materials for the cement production. Another benefit is an improvement of mechanical properties, durability of the product and its microstructure. [1]

The microstructure of various materials has a profound effect on the physical and mechanical properties and thus on their durability. There are many cases when we need information about the internal structure of materials to obtain by a non-destructive method. Computer X-ray microtomography (micro - CT) allows a non-destructive 3D imaging of the internal microstructure of materials and it also allows to analyze the length and size of the object, its shape, orientation, porosity, etc. inside the material. [2]

2 Geopolymer

The term geopolymer was first introduced in 1979 by the French Professor Joseph Davidovits. Geopolymer is a polymeric material having a chemical composition similar to the natural zeolite, but containing an amorphous structure, resembling the structure and properties of ceramics. [3, 4] This material was synthesized and cured at ambient temperature and pressure. [5, 6] It consists of two components - the source materials and the alkaline liquids. Source materials for geopolymers based on aluminosilicates should be rich in silicon and aluminum, such as natural aluminosilicate, slag, fly ash, ash from rice husks, stone powder, etc. Selection of the source material for production of geopolymers will depend on factors such as availability, price, type of application and the specific requirement of the final user.

Study of geopolymer composites shows geopolymers as a promising new material with environmentally sustainable properties. [7, 8, 9] It is also considered as a new material for coatings and adhesives, a binder for fiber composites and a new kind of cement for production of concrete. [3, 10, 11]

Geopolymers have many advantages in comparison with Portland cement:

- plentiful raw material resources [5],
- saving energy and protecting the environment [3, 5] - treatment of natural alumino-silicates at relatively low temperature (600 to 800°C),
- simple preparation technique [5] - can be synthesized by mixing the alumino-silicate with an alkaline solution, then it can be toughen at room temperature,
- high heat resistance (up to 1000°C), [11] low shrinkage and low thermal conductivity [11, 12],
- good volume stability, good resistance to acids and salt solutions [11, 13],
- extremely good durability and high compressive strength [5, 11, 14].

3 Experimental equipment

Desk micro-tomograph device SkyScan 1272 (see Figure 1) is able to non-destructively analyze and visualize the structure of textile materials, composites, skeleton and soft tissues from the fields of medicine, materials from the field of geology, electronics, etc. The device scans the object in the form of 2D images, which can be converted with the help of a special program (reconstruction software) to 3D object. The resolution of the device is up to 0.35 micron, the maximum size of a tested material can be 75 mm in diameter and 70 mm in length. [2]

Fig. 1 *Micro-tomograph device SkyScan 1272 used for non-destructive 2D a 3D X-raying of composites and textile structure*

Computer tomograph SkyScan 1272 is equipped with:

- Closed, air-cooled X-ray tube with voltage of 20 – 100 kV, 0 – 250 μA (10 W max),
- 16 Mpx detecting CCD camera,
- a motorized sample holder for precise positioning of the sample to the radiation source and a CCD camera,
- 2 control computers with LCD monitors
- software for complete 2D and 3D quantitative analysis, for morphometry (shape measurement) and densitometry (measurement of the optical densities of processed photographic records) for realistic 3D visualization of object of the research etc. [2]

4 Results of experiment

For the preparation of samples and realization of the experiment following materials were used: cement - a commercially available powder component cement Baucis L160, sodium hydroxide - commercially available geopolymer activator Baucis L160, Australian garnet with a particle size of 20-50 μm (Fig. 2).

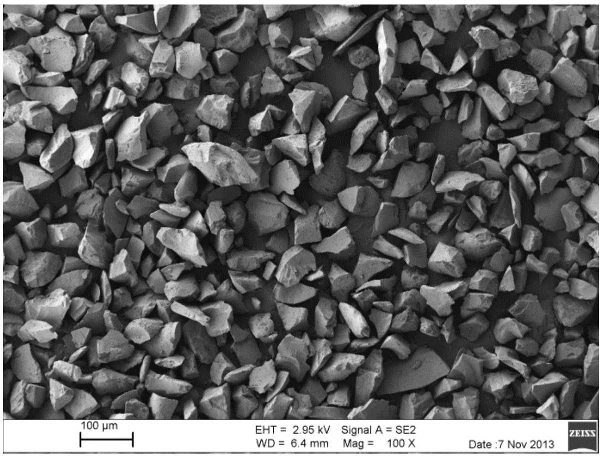

Fig. 2 *Detail of particles of Australian garnet (acquired by SEM)*

Cement Baucis L160 is distinguished by fast toughen, 40-50 % of the final strength achieved during the first day. Time of the solidification of the binder mixture is in the range from 140 to 240 minutes, solidification is activated by sulfate activator.

Preparation of test specimens with garnet filler proceeded as follows:

- Mixing, the cement activator for 10 minutes,
- Weighed amount of garnet was added carefully, as it was desirable to mixed the garnet into the mix of activator and cement thoroughly,
- Mixing of the mass for next 20 minutes.

For sample preparation were used moulds with dimensions of ø 12.5 mm, made of polypropylene. To non-destructive imaging of internal microstructure of the geopolymer, six samples were prepared: a pure geopolymer at 20°C and after heating to 700°C, geopolymer filled with 5% of the garnet at 20°C and after heating to 700°C and geopolymer filled with 15 % of the garnet at 20°C and after heating to 700°C. In Figures 3, 4 and 5, there are compared studied samples and shown the changes in the structures at a temperature of 20°C and after heating to 700°C.

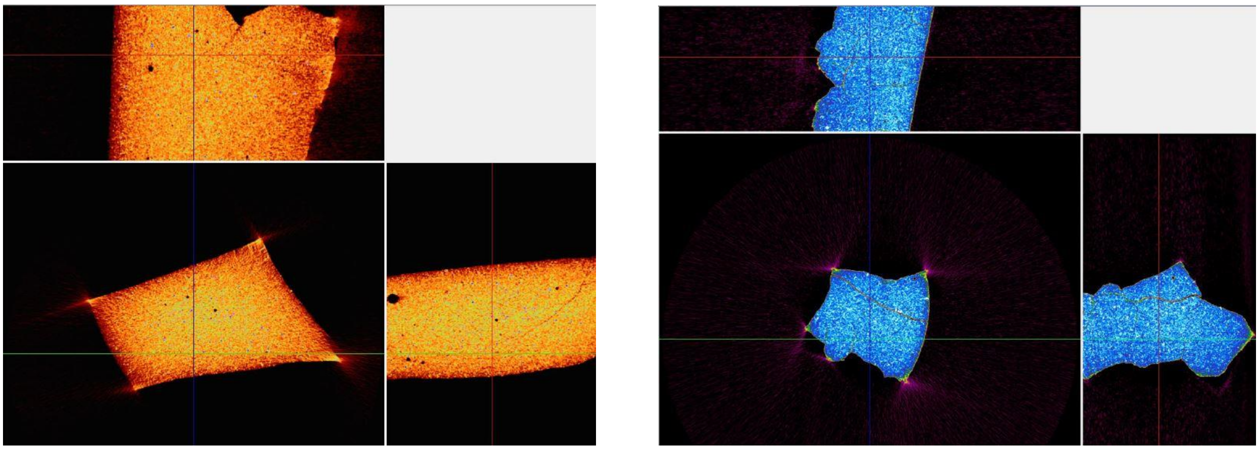

Fig. 3 *Comparison of the internal structure of the pure geopolymer at 20°C (left) and after heating to 700°C (right)*

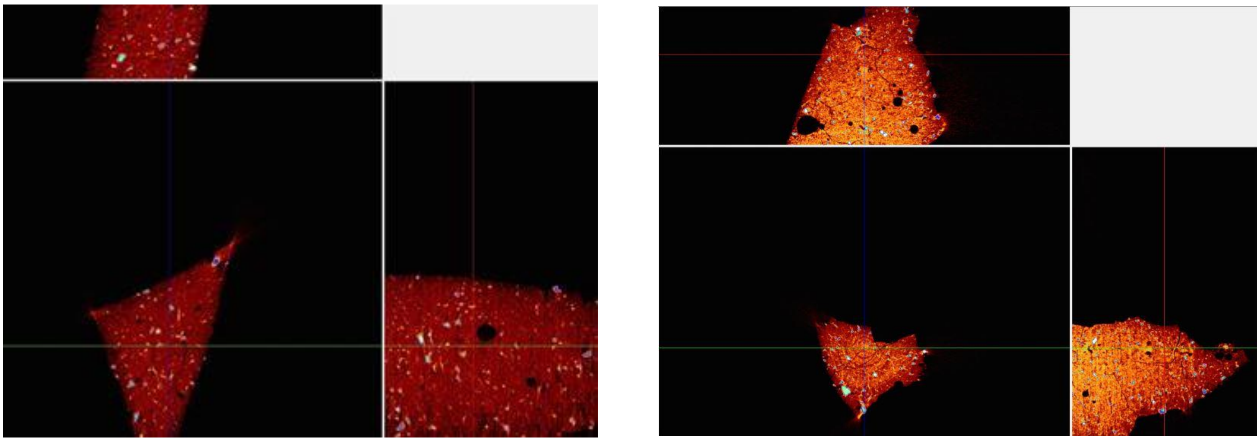

Fig. 4 *Comparison of the internal structure of the geopolymer filled with 5% garnet at 20°C (left) and after heating to 700°C (right)*

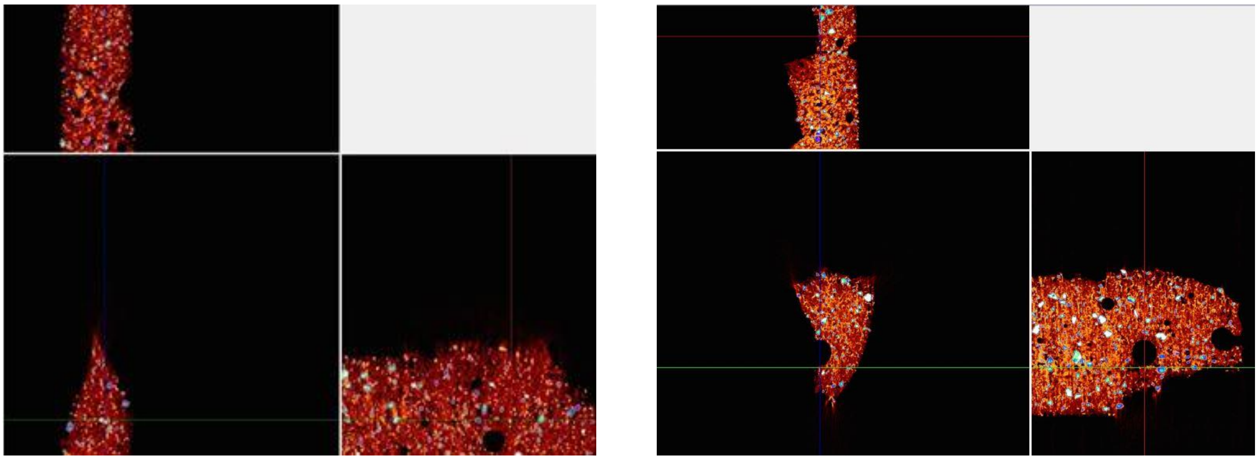

Fig. 5 *Comparison of the internal structure of the geopolymer filled with 15% garnet at 20°C (left) and after heating to 700°C (right)*

The same conditions of computed tomography scanning were used for all investigated samples. The goal of this study was to observe and compare the changes in the structures of the various samples and track the distribution of added particles of garnet ´s fillers into the geopolymer material. Table 1 describes the conditions of scanning by computed tomography.

Table 1 Conditions of scanning of samples by computed tomography

X-ray tube voltage [kV]	*electric current [μA]*	*exposure [ms]*	*resolution [μm]*	*scanning time [min]*
70	*142*	*886*	*9.9*	*112*

Figures 6, 7 and 8 show the distribution of added garnet Picture by micro-CT at 20°C and after heating to 700°C.

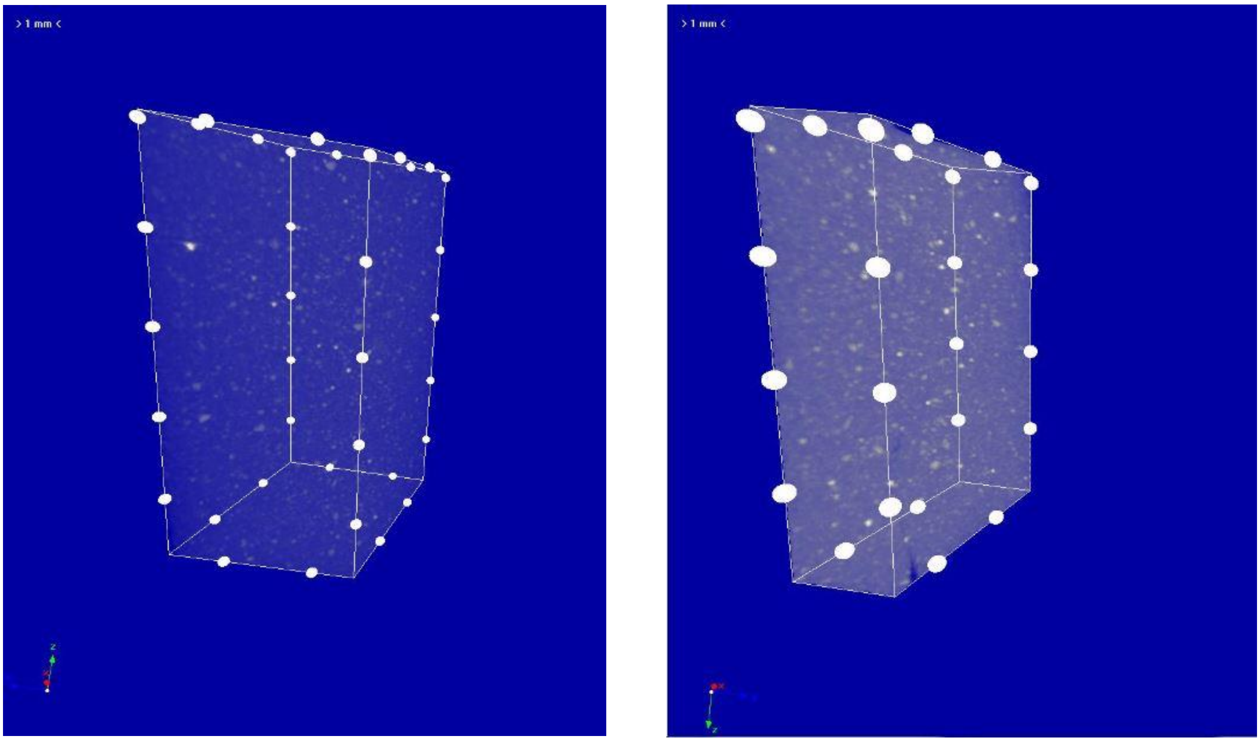

Fig. 6 *Distribution of particles imaged by micro - CT in pure geopolymer at 20°C (left) and after heating to 700°C (right)*

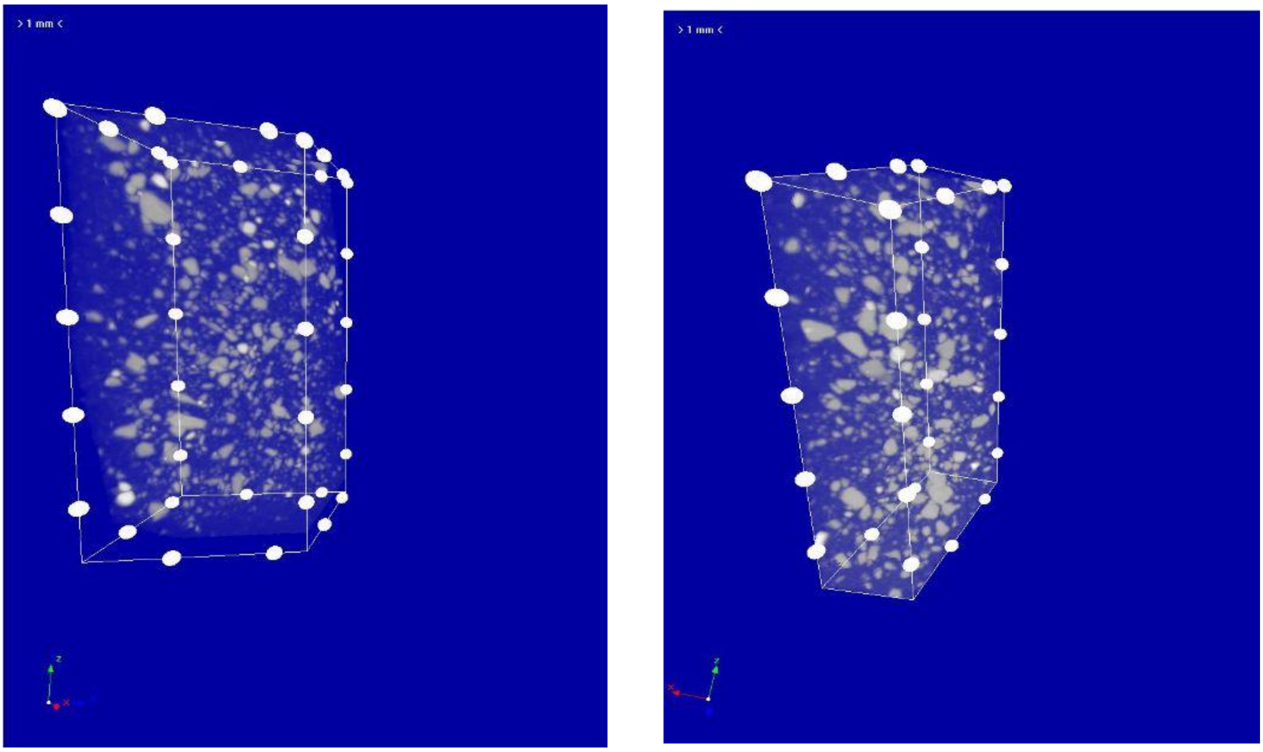

Fig. 7 *Distribution of particles imaged by micro - CT in geopolymer which was filled with 5% of garnet at 20°C (left) and after heating to 700°C (right)*

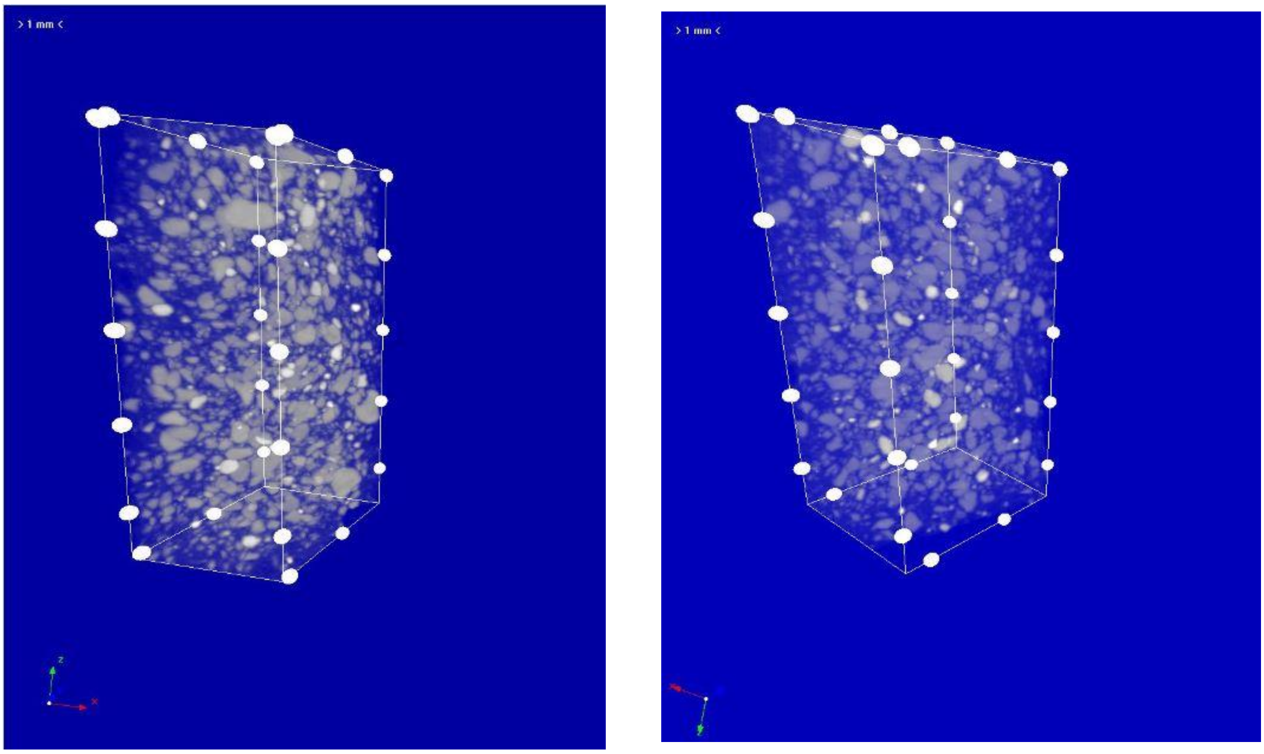

Fig. 8 *Distribution of particles imaged by micro- CT in geopolymer which was filled with1 5% of garnet at 20°C (left) and after heating to 700°C (right)*

After scanning by micro-tomograph SkyScan 1272, saved data can be processed by the software and provide information about the total porosity of the studied samples. For each sample, the analysis of tested materials has been carried out three times and from these measurements the average value has been calculated. In Table 2 are shown the values of total porosity and the volume fraction of particles in the tested samples of the garnet is shown in Table 3.

Table 2: The total porosity of the tested samples with a total volume of 6.06 mm³

Tested sample	*Temperature 20°C*		*Temperature 700°C*	
	Porosity [%]	*Average porosity [%]*	*Porosity [%]*	*Average porosity [%]*
Pure geopoplymer	*0.14*	*0.15*	*1.87*	*1.99*
	0.11		*2.13*	
	0.20		*1.98*	
Filled geopolymer with 5% of garnet	*3.87*	*2.89*	*3.43*	*3.89*
	2.10		*4.92*	
	2.71		*3.33*	
Filled geopolymer with 15% of garnet	*16.12*	*10.95*	*7.45*	*6.57*
	6.76		*7.49*	
	9.97		*4.78*	

Table 3: The volume fraction of particles of garnet in the investigated samples with a total volume of 6.06 mm³

Pure geo-poplymer	*Temperature 20°C*			*Temperature 700°C*		
	Volume fraction of particles [mm³]	*Average volume fraction of particles [mm³]*	*Average volume fraction of particles [%]*	*Volume fraction of particles [mm³]*	*Average volume fraction of particles [mm³]*	*Average volume fraction of particles [%]*
Filled geopolymer with 5% of garnet	*0.098*	*0.126*	*2.21*	*0.14*	*0.16*	*2.69*
	0.16			*0.15*		
	0.12			*0.20*		
Filled geopolymer with 15% of garnet	*0.53*	*0.37*	*7.07*	*0.53*	*0.50*	*9.04*
	0.33			*0.51*		
	0.26			*0.48*		

The values of porosity are different due to the heterogeneity of geopolymer system. During the analysis by computed tomography the area of interest is selected with a given number of cuts saved during scanning. For this area the parameters listed in Table 2 and 3 can then be selected. The cause of this variation may be air bubbles occurring in the investigated volumes.

5 Conclusion

Computed tomography (CT) is advanced method that enables high-quality view of the internal structures of various objects. By CT method it is possible to measure the distribution of particles, investigate the relative content of components in different areas of cut or technological visible defects in various stages of production.

Experiment shows that when passing through the different materials X-rays are weakening. The extent of absorption is smaller or larger depending on the different densities of the material and depends primarily on the properties of the examined material. Radiation absorption coefficient expresses the ability of different materials to absorb X-rays. This is the reason of visibility of garnet particles added as filler to geopolymer composites.

Micro-CT analysis allows a nondestructive testing of materials and obtains information about the distribution of particles in a specific volume. Specific software can determine the porosity of the pure geopolymer and geopolymer filled with 5 % and 15 % of garnet.

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