Structure and composition of innovative porous composite material

Veselin Petkov, Rositza Dimitrova, Luben Lakov
Institute of Metal Science, Equipment and Technologies with Hydro- and Aerodynamics Centre "Acad. A. Balevski",
Bulgarian Academy of Science, Sofia, Bulgaria
veselin.petkov@ims.bas.bg, rossy@ims.bas.bg, silicatchim@mb.bia-bg.com

***Abstract**: The subject of this article is a study on the composition and structure of innovative Porous Composite Material (PCM). The latter is obtained from powders of glass waste and heat-treated rice husks. The PCM is non-flammable, non-combustible and has high heat and sound insulation properties. The structural elements made of it are lighter and stronger than the ones made of foam glass. It is expected that PCM is to find wide application in construction and as an absorbent of heavy metals and oil from polluted water.*

***Keywords**: HEAT INSULATION, SOUND INSULATION, ABSORPTION, NON-FLAMMABLE, NON-COMBUSTIBLE, STRONG, LIGHT-WEIGHT*

1. Introduction

Household, construction and industrial glass waste as well as rice husks are dumped worldwide in huge quantities – more than 130 million tons per year [1]. Many research teams in different countries work on methods to utilize these two types of waste materials. Rice is one of the most consumed crops in the world, with China being the largest consumer. Rice husks are a by-product of rice production. Their use as a source of energy is only a partial solution to the problem of their utilization. The valuable ingredient, in the seemingly useless residue after heat treatment of rice husks, is the silicon dioxide, whose content in the resulting ash varies from 90 to 97 weight %. It is important to note that the silicon dioxide is in an amorphous (active) form and with a large specific surface area [2 & 3].

2. Prerequisites and means for solving the problem

Different technologies have been developed for the production of light-weight ceramic materials in the form of bricks and blocks for the utilization of powders from glass waste and rice husks [4]. Another common use is in the production of boards, panels and chips (we have all heard of the Silicon Valley...). Of no less ecological importance is the use of rice husk powder for the production of silica used in construction as a component of cements or for the production, under certain conditions, of silicon carbide [5].

3. Solution of the examined problem

The innovativeness of the porous composite material created by us stems from its higher strength compared to foam glass as well as its non-flammability, non-combustibility and high thermal and sound insulation performance. Besides as construction material, PCM is also valuable as an absorbent of heavy metals and oil from polluted waters. Regarding the broader scientific importance it can be pointed out that the current research contributes to solving a long-standing problem namely utilization of huge amounts of waste materials coupled with the production of useful new material. From a scientific point of view, PCM's composition and structure are of particular interest hence the current study.

4. Results and discussion

4.1. Optical microscopy (OM)

In Fig. 1a and Fig. 1b images from PCM sample examination using an optical microscope are presented. The high degree of porosity is clearly visible (low magnification image – Fig. 1a). The main part of the pores have sizes ranging from 200 to 600 µm. It is worth noting the presence of micro-pores in the walls separating the main pores (see low magnification image – Fig 1b). The dimensions of the micro-pores are mainly in the range of 5 to 50 µm. As a result of the above observations it is concluded that PCM has a pronounced cellular structure. The difference between PCM's structure and the structure of cellular foam glass is the formation of micro-pores in the cell walls of PCM. These micro-pores effectively increase the specific surface area of the material thus further stimulating its absorbing ability by creating "breathing" cell walls.

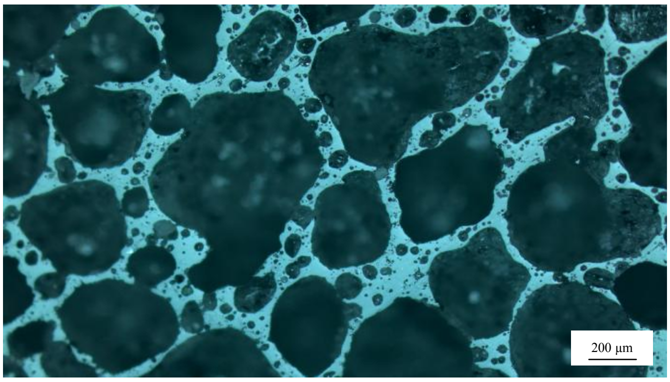

***Fig. 1a.** Low-magnification OM image of PCM sample*

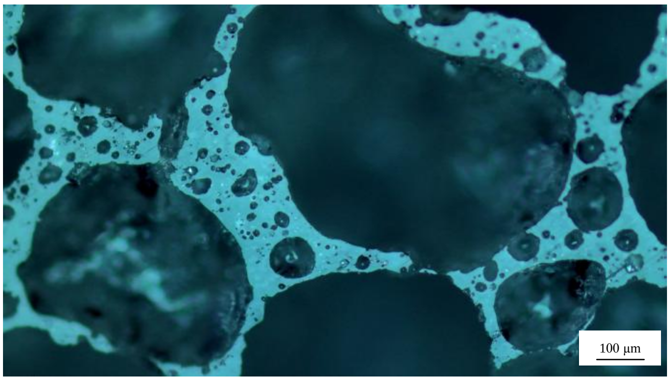

***Fig. 1b.** High-magnification OM image of PCM sample*

Images of the same sample taken with a stereo optical microscope (Fig. 2a and Fig. 2b) give a better spatial picture of PCM's cell structure. The pores are formed as a result of the combustion of the foaming agent, in this case, the carbonized biomass from the rice husks remaining after the heat treatment. The large foaming particles form the large pores and the small ones form the micro-pores in the cell walls.

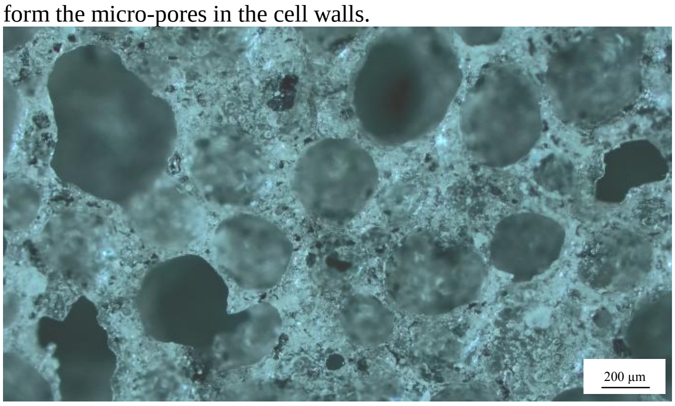

***Fig. 2a.** Low-magnification stereo OM image of PCM sample*

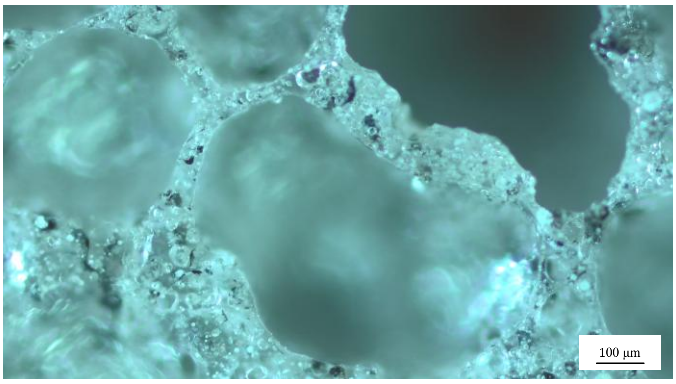

Fig. 2b. *High-magnification stereo OM image of PCM sample*

4.2. Scanning electron microscopy (SEM)

In Fig. 3a and Fig. 3b images from PCM sample examination using a scanning electron microscope are presented. The images confirm the results of the study with optical microscopes, namely: a high degree of porosity and presence of micro-pores in the walls separating the main pores. The dimensions of the main part of the two types of pores are also confirmed with Fig. 3b showing in detail a micro-pore.

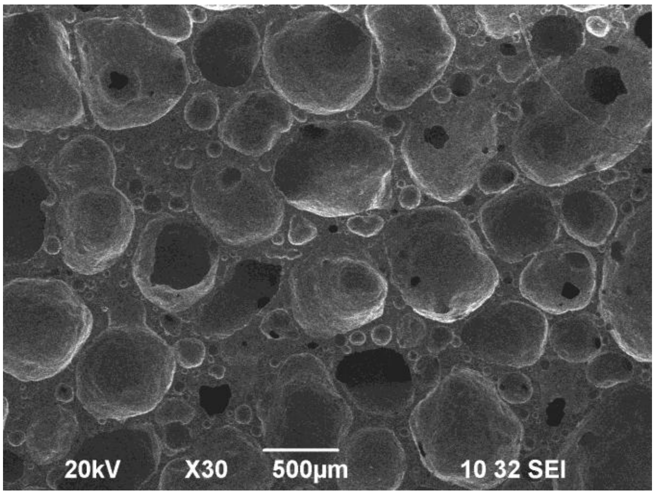

Fig. 3a. *Low-magnification SEM image of PCM sample*

The high level of porosity of the material is a prerequisite for its low thermal conductivity and high sound insulation capacity; these two properties will be the subject of another study.

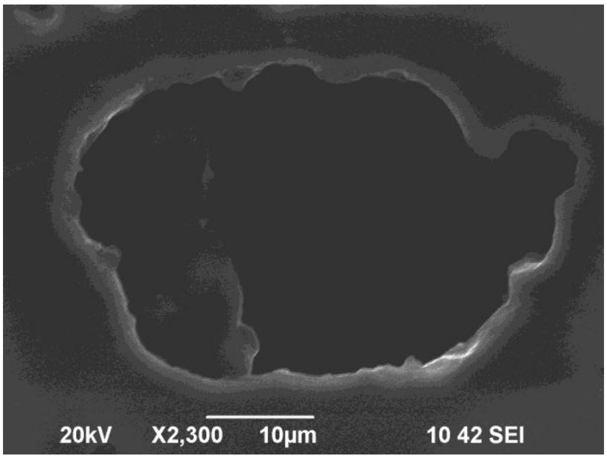

Fig. 3b. *High-magnification SEM image of PCM sample*

Table 1 shows the average results of chemical analyses (EDS) in five randomly selected sample points. The presence of 4 oxides was detected, namely: SiO_2, Na_2O, CaO and MgO. The high content of SiO_2 is due to its accumulation from the two raw materials – powders of waste glass and active amorphous silica from heat-treated rice husks.

Table 1. Chemical composition of PCM sample (EDS analysis)

Element	Atomic %	Weight %
Si	20,72	29,44
Na	7,41	8,84
Ca	2,69	5,49
Mg	1,77	2,26
O	67,41	53,97

4.3. Three-dimensional micro Computed Tomography (3D micro CT)

A PCM sample with a roughly parallelepiped shape (approximate dimensions: 15 x 12 x 9 mm) was examined using a 3D X-ray micro-tomographer “SkyScan 1272” from Bruker (*see last page). Fig. 4 shows the sample at the start of the examination, the so-called preview or projection.

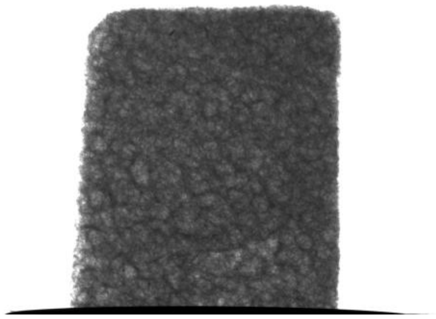

Fig. 4. *PCM sample preview/projection*

After initial settings, the following radiation parameters were selected:

- resolution: 2452 x 1640 pixels;
- pixel size: 10,8 µm;
- no filter;
- distance from sample to X-ray source: 200,4 mm;
- source voltage: 50 kV;
- source current: 200 µA;
- radiation step: 0,2˚;
- 360˚ radiation (full rotation of the sample).

The sample radiation (examination) with the selected parameters had a duration of 02h 38m 28s.

After completion of the radiation, the so-called “3D reconstruction” was performed by processing the obtained graphic images with NRecon software (v. 1.7.4.2). After that CTVox software (v. 3.3.0r1403) was used to visualize the 3D models in different spatial orientations (shown in Fig. 5a – 5d). PCM’s highly porous structure is clearly visible in all orientations; now and then some of the pores merge to form channels such as the one that can be seen in Fig. 5c.

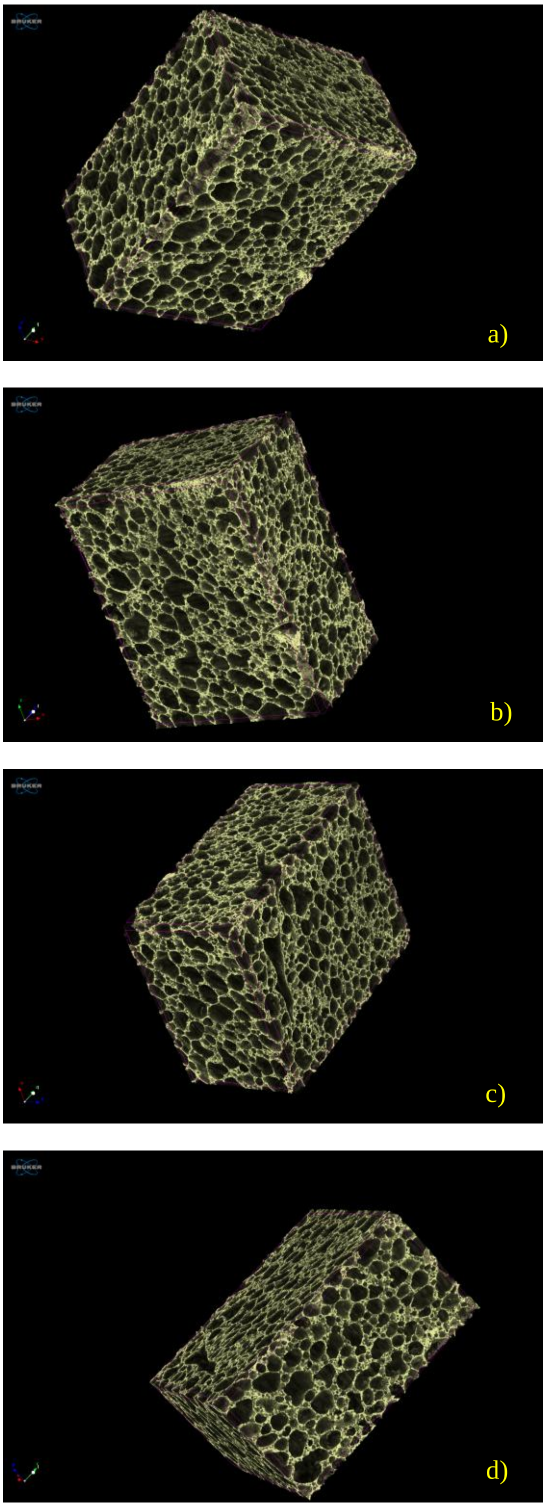

Fig. 5. *3D views of PCM sample*

The software has a number of useful functionalities; two of them are demonstrated in Fig. 6 and Fig. 7. The first functionality allows the operator to make a spherical section with a variable size in any part of the sample – so-called “cutting”. This is demonstrated in Fig. 6 (a & b) by making sections in two corners of the sample thus revealing the inner structure in depth customizable by the operator.

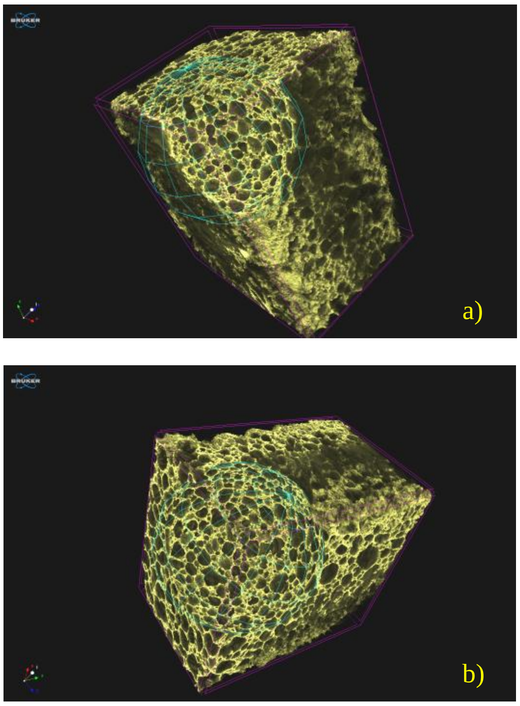

Fig. 6. *Spherical sections in two corners of PCM sample*

Another functionality allows the operator to insert a sphere in any part of the sample and then remove all surrounding material thus leaving visible only the part which is inside the sphere – so-called “clipping”. This functionality is demonstrated in Fig. 7 (a & b).

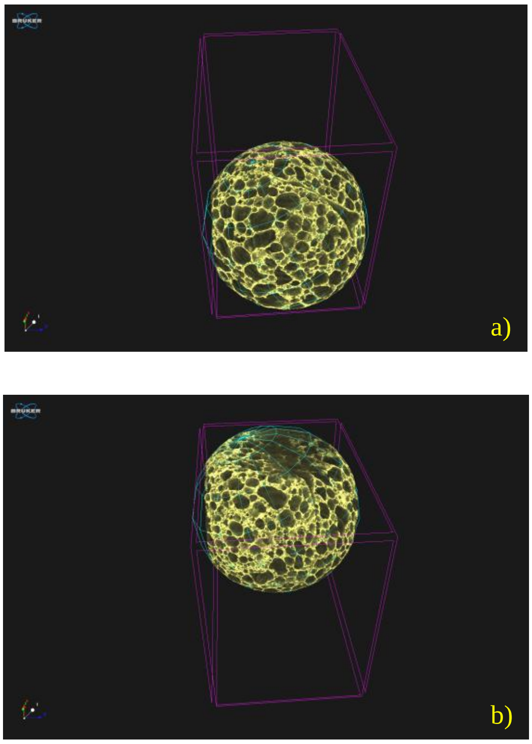

Fig. 7. *Spherical inserts in two parts of PCM sample*

Using CTAn software (v.1.18.8.0) additional processing of the 3D model was performed in order to obtain quantitative characteristics of the sample. The porosity of the sample was calculated and the results show that the total porosity is 80,45 %, with the percentage of closed pores being only 0,15 % while the percentage of open pores being 80,30 %.

5. Conclusions

As a result of the conducted research it was established that the studied porous composite material has a pronounced cellular structure with a very high degree of porosity (80,45 %) and almost all of its pores are open. This is a prerequisite for very low density and high heat and sound insulation capacity. The last two properties will be the subject of another study and when quantitatively determined will open the door for wide application of the material in construction especially in the renovation of buildings as well as in the production of light-weight concrete and new composite mixtures. Due to the high specific surface area it can be expected that PCM will also find application as an absorbent of harmful substances from various fluids (for example, of heavy metals and oil from polluted water).

6. References

1. L.A. Lyubchev, I.G. Markovska, International symposium “Ecology 93”, Sept. 9-11, 1993, Burgas , Bulgaria, 32

2. I. Javed, S.H. Tajwar, S. Shafaq, M. Zafar, M. Kazmi, 2009 “ Characterization of Silica from Sodium hydroxide Treated Rice Husk”, Journal of Pakistan Institute of Chemical Engineers, 37, pp. 97-101

3. D. Yordanov, Z. Tsonev, G. Yordanova “ Characterization of Silica material from Magnesium and Lead Oxide Treated with Agriculture waste, Industrial Technologies, Vol. II (1) 2015

4. G. Marahlev, Amer., J. Appl. Sci. 2 (4) 2005, 778

5. B.I. Ugheoke, E. O. Onche, O. N. Namessan, G. A. Asikpq, Leonardo Electronic Journal of Practices and Technologies, 9 (2006), 167

*3D X-ray micro-tomographer “SkyScan 1272” from Bruker is purchased under Project BG05M2OP001-1.001-0008 “National Center for Mechatronics and Clean Technologies” funded by the EU Operational Program “Science and Education for Smart Growth” 2014-2020