Journal of Physics: Conference Series

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To cite this article: G Bumanis and D Vaiiukynien 2023 *J. Phys.: Conf. Ser.* **2423** 012031

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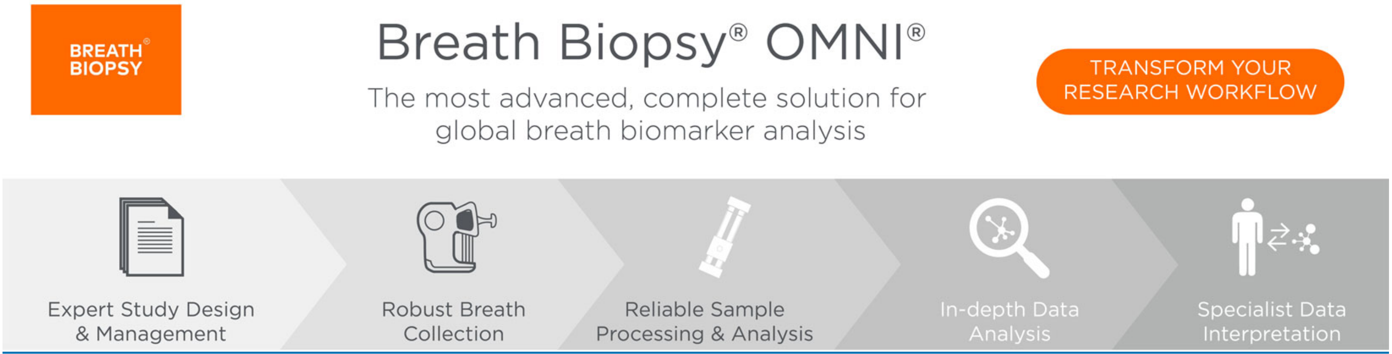

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Lightweight porous geopolymers from waste red brick precursor and synthetic foaming admixture

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Abstract. Geopolymers are associated with a high potential to replace traditional Portland cement. Up to now, research has been ongoing on alternative precursors with wide availability, low cost, and low environmental impact. Attention is paid to ashes, industrial by-products, and construction and demolition waste. Sophisticated materials with high porosity and improved physical or mechanical properties are developed to enhance the application of geopolymers. In this paper, the waste red brick is used as a precursor to produce highly porous geopolymers. Here, porous geopolymers on waste brick precursor and synthetic foaming agent were developed for the first time. Red waste brick was ground to powder particles and activated with NaOH solution. A porous structure was obtained by a pre-foaming technique where geopolymer slurry was mixed in the foams. Physical and mechanical properties were evaluated depending on mixture composition and foaming agent content. Porous geopolymers with densities from 240 to 1200 kg/m^3 were obtained. The pore structure of obtained materials was characterized. Reduced density and characteristics of red clay precursor summarised in low compressive strength which was from 0.04 to 0.4 MPa.

1. Introduction

Geopolymers have been proven to be suitable cementitious materials to replace traditional mineral binders in different applications in civil engineering. There are efforts to (i) substitute Portland cement binder to produce geopolymer concrete [1], [2], (ii) to use geopolymer material as advanced sorbent material in water treatment systems in order to remove pollutants such as heavy metals or other hazardous substances [3], [4], (iii) catalytic material in biogas production to control and improve biogas digestion process [5], and others. Therefore, geopolymer research focuses on developing dense and strong load-bearing materials, where durability and strength are the most important characteristics. The other direction is dedicated to developing highly porous geopolymer materials. Low density gives improved thermal characteristics, which is essential in civil engineering. In different absorption and filtration processes, the open structure of geopolymers is important. High porosity allows to reach higher process efficiency as the extra surface of geopolymers is available [6]. There are different methods elaborated to produce highly porous geopolymers. Direct foaming, the addition of blowing agents, mechanical frothing, or direct gas insertion is possible [7]. Stabilizing agents are used to maintain the green structure of geopolymers until curing of geopolymers is complete [8]. Material with different density can be obtained from various precursor geopolymers. Fly-ash geopolymer alkali activation with sodium waterglass solution and H_2O_2 pore foaming agent lead to highly porous material with density of 239 kg/m^3 and compressive strength of 0.8 MPa and ultra-low thermal conductivity for such mineral materials of 0.0744 W/mK [9]. Al powder foaming agent incorporated in fly ash-based geopolymers resulted in 403 kg/m^3 and a strength of 0.9 MPa [10]. Different waste types, such as tailing from various industries, have been examined as geopolymer precursors [11]. Construction and demolition waste, as well as secondary by-products from various industries, is also possible to use in the production of

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geopolymers. There are reports that red brick waste ground to powder particles are suitable for alkali activation, to obtain geopolymer binder with the strength of 30 MPa [12], but there is little effort on the production of lightweight geopolymers based on red clay precursor.

In this research development and testing of highly porous red brick powder based geopolymers are examined with different mixture compositions and foaming admixtures.

2. Materials and methods

Red brick production waste was used as a precursor to produce geopolymers. Red brick waste sand with a fraction 0/2 mm was ground with collision milling in disintegrator to a fineness of 2515 cm^2/g, with d_{50} of 24.64 µm. The properties of the precursor were described in the previous manuscript [13]. Alkali activation was done with 7M NaOH solution. Sodium hydroxide (Tianye Chemicals Ltd., China) with 99 % purity was used. Anion surface-active substance with stabilizing and functional agents PB-Lux synthetic foaming admixture was used to prepare foamed samples with the pre-foaming technique. 35% H_2O_2 solution was used to prepare foamed geopolymer samples with a chemical aeration technique. Prismatic specimens with dimensions of 60x60x180mm were prepared and tested. Mechanical properties of at least three geopolymer samples with dimensions of 20x20x20mm were tested on a Zwick Z100 universal testing machine with a testing speed of 0.5 mm/min. Macrostructure was observed by Veho Discovery DX-3 digital microscope. The microstructure was determined using Hitachi Tabletop TM3000 Electron Microscope.

2.1. Mixture composition

Red brick clay-based geopolymer is used as a base binder to produce porous geopolymers. In previous papers published by authors, raw materials and activation techniques were described. The strength of reached up to 29 MPa[13]. Two mixture series with different foaming admixtures and precursor compositions were prepared. Pre-foaming method was elaborated in the first series with red brick waste, activated with 7M NaOH solution and also with a Portland cement addition (table 1). The foaming admixture dose was from 0.05 to 0.5 and a constant water amount to prepare foams was used. Portland cement was used to stabilize the foam structure and give additional strength to foamed geopolymer and avoid pore collapse during the curing. Cement partially replaced brick precursor. Since the cement has finer nature in comparison to brick powder (2515 cm^2/g for a brick powder to 3487 cm^2/g for Portland cement [13]), additional water was added to the mixture, so the foamed structure is possible to cast. The second series were foamed with H_2O_2. Different H_2O_2 content was selected for the same amount of paste (table 2). 0.5 to 1 liter of foamed geopolymers were prepared for each mixture.

Table 1. Mixture composition with synthetic foaming admixture (weight ratios).

	Foams		Geopolymer binder			
Composition	H_2O	Pblux	Brick powder	7M NaOH	Cement	H_2O
P1	25	0.1	167	80	-	-
P2	25	0.3	167	80	-	-
P3	25	0.5	167	80	-	-
PC1	50	0.05	150	75	30	42
PC2	50	0.1	150	75	30	42
PC3	50	0.3	150	75	30	42
PC4	50	0.5	150	75	30	42

Table 2. Mixture composition of porous geopolymers foamed with H_2O_2 admixture (weight ratios)

	Foams	Geopolymer binder	
Composition	H_2O_2, ml	Brick powder	7M NaOH
H1	1	167	80
H2	2	167	80
H3	3	167	80

2.2. Mixing procedure and curing

For the first series with a synthetic foaming agent pre-foaming method was used. The mixing procedure included the preparation of geopolymer paste (figure 1a), then the preparation of foams was obtained by mixing foaming admixture with water and mixing it for 2 minutes (figure 1b). Then geopolymer paste was gradually inserted in the foams with simultaneous mixing until homogenous mixture composition was obtained (Figures 1c and 1d). Then the mixture was cast in prismatic molds and covered with plastic film. For the second series, geopolymer paste was prepared and then H_2O_2 admixture added to the paste and mixed throughout. Afterwards, the mixture was cast in prismatic mold and after blowing and volume increase of paste it was placed in a curing chamber. Curing conditions were at 80°C for 24h.

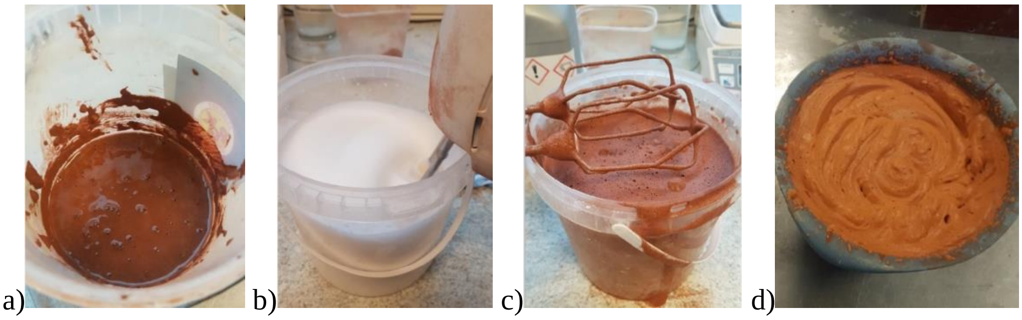


Figure 1. Foam preparation and casting process.

3. Results

3.1. Mixture stability

Using synthetic foaming admixture Pblux and H_2O_2, trial geopolymer mixes were prepared and observations made. Stabilizing additives such as SDS and PC (10% of the weight of brick precursor) were also applied. Additive importance was attributed to the foam stability. In the beginning, all foams collapsed before or after heat treatment. The deviation in mixture composition and overdose of foam water, alkali solution, or foaming admixture may lead to various failures in porous structure formation. In figure 2a the overdosing of H_2O_2 admixture leads to a rapid expansion of geopolymer paste and that is followed by the collapse of foam structure and that is resulting with dense geopolymer paste. High foaming agent content or excess water may lead to good and flowable foam geopolymer, while the foams are not stable and might set down or lead to a total collapse of geopolymer foam structure (figure 2b). Cement additive gives a more sophisticated and stable foam structure, while shrinkage and reduction of foamed material volume is still possible (figure 2c). Overdose of cement addition and high foaming agent content may lead to a rapid foaming reaction and set of foams, without the possibility of casting the sample (figure 2d).

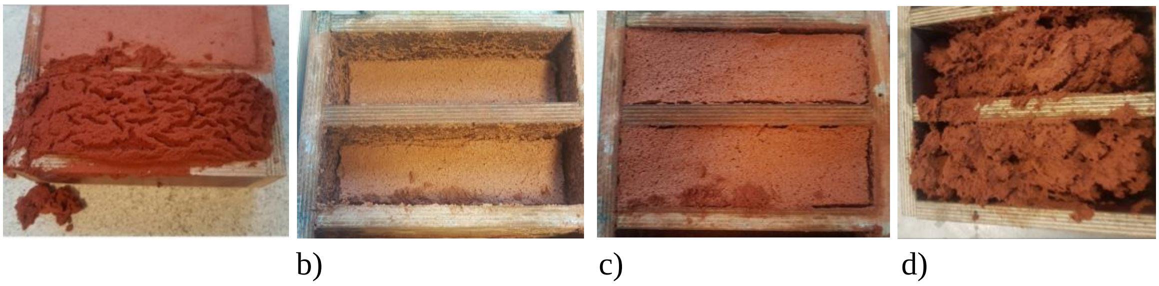


Figure 2. Failure modes in the preparation process of foamed geopolymers. a) Rapid foam blowing and collapsing. b) foam collapse after mixing and casting. c) shrinkage during drying and curing; d) rapid blowing reaction and setting of geopolymer foams.

Figure 3 is given molded samples of given geopolymer mixtures. The cement additive gives light color comparing to intensive orange to brown for samples only with red brick precursor. Samples prepared with pre-foaming technique is possible to cast and handle samples, while for samples foamed with H_2O_2 need steady state to entrap the air bubbles and avoid collapse of foamed geopolymer structure. In this case, the sample in the mold was not possible to level. The water content in the mixture plays a significant role. On the one hand, water is needed to obtain high foam volume and increase the total volume of geopolymer mixture. Water also gives workability as extra water ensures that foams can be casted by pouring and no extra leveling is needed. No entrapped air gets into the mixture and homogenous material is obtained. On the other hand, extra water increases porosity, reduces alkali concentration in activator and extra fluidity gives more probability of foamed system collapse. The proper water content can be controlled by additives like Portland-cement or gypsum, or chemical admixture as sodium dodecyl sulphate (SDS).

Figure 3. Casted samples of foamed geopolymers. a) without additives, b) with Portland cement additive, c) with H_2O_2 foaming admixture.

3.2. Macro and microstructure of foamed geopolymers

The macrostructure of porous geopolymers made with H_2O_2 is given in figure 4. The increase of H_2O_2 content in the mixture composition made the structure of geopolymer more porous. The pore size was from 0.2 to 1.5mm. A more homogenous pore structure was obtained with higher H_2O_2 content. The difficult part of the use of H_2O_2 as foaming admixture is associated with the stability of fresh foams. Most trial mixes showed delayed and weak blowing or rapid reaction with higher H_2O_2 dosage with the problems of stabilizing geopolymer structure until curing of the material is completed.

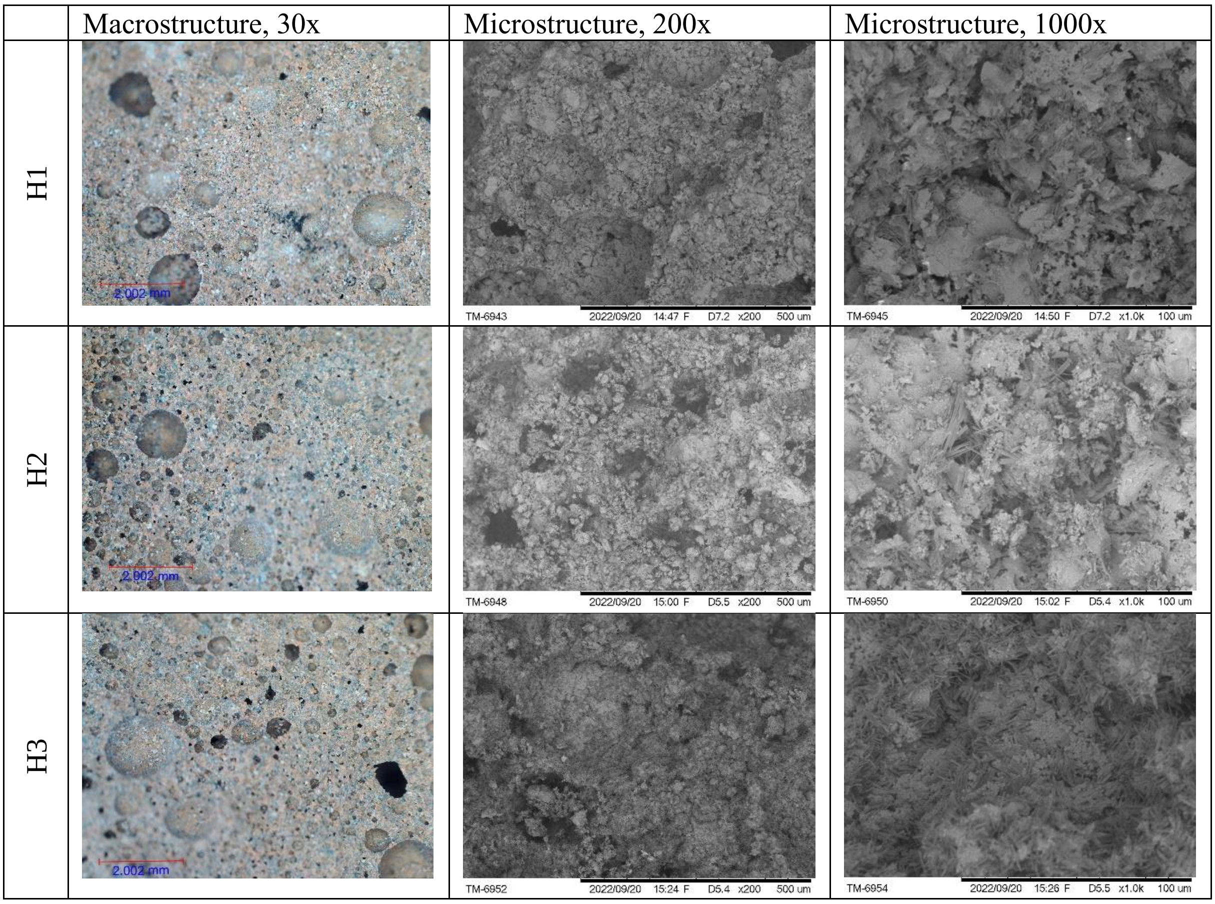


Figure 4. Macro and microstructure of foamed geopolymers with H_2O_2.

The macro and microstructure of porous geopolymers based on pure geopolymer paste and foaming agent is given in figure 5. Selected mixture compositions give a highly porous material structure with large and well-formed pores. The amount of foaming admixture did not impact the macro-appearance dramatically. Most of pores are in size from 0.2-0.5mm. In microstructure of hardened geopolymers separate raw material particles can be identified. Smaller amount of foaming admixture gives larger pores and the pore structure is less dense. By the increase of foaming admixture, pore size reduces. High foaming admixture content allows to introduce finer and more stable foams. Smaller micropores with size from 5 to 20 µm, remaining also larger pores. Under the magnification of 1000x, clay crystalline structure is still visible and also geopolymerisation products can be identified.

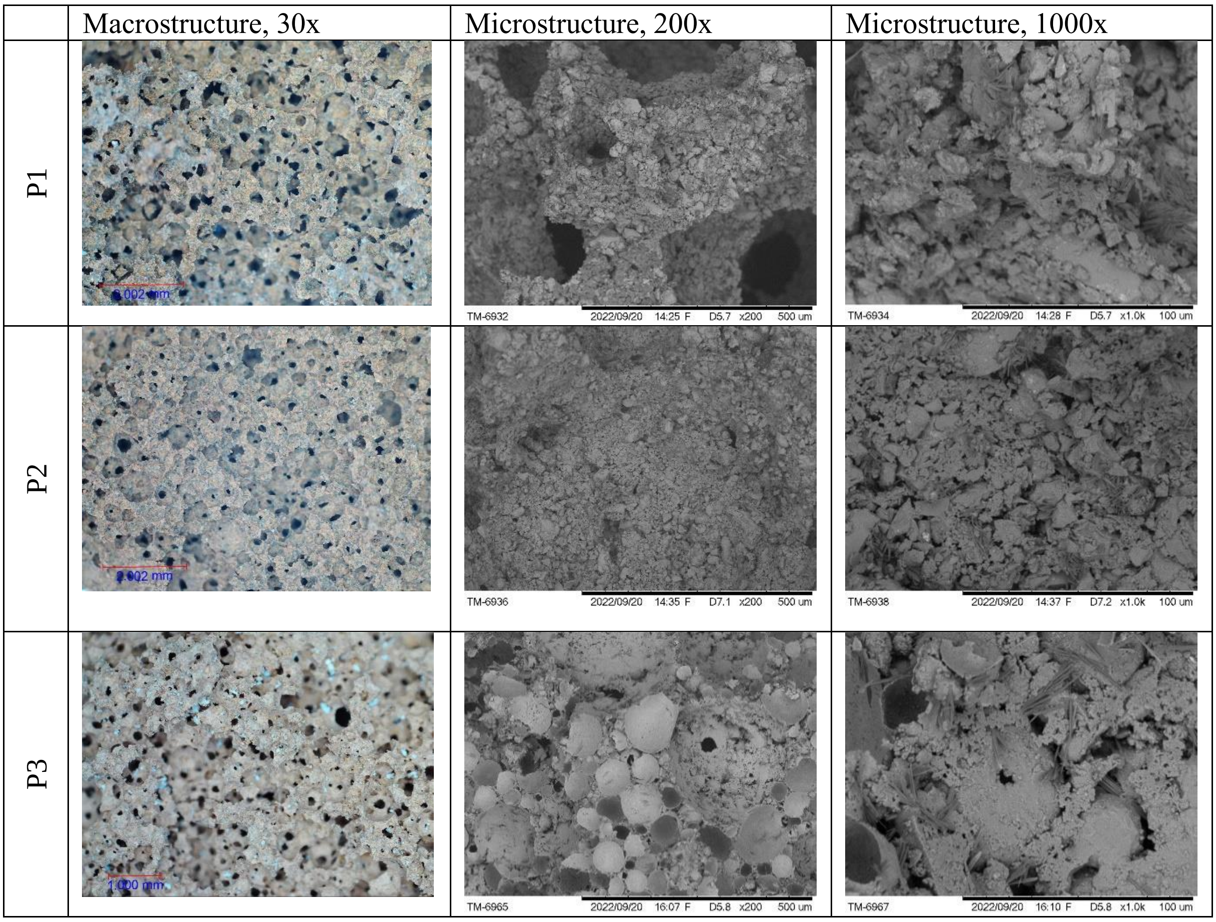


Figure 5. Macro and microstructure of porous AAM based on pure geopolymer paste

Foamed geopolymer structure with Portland cement additive is given in figure 6. The incorporation of Portland cement not only affected the visual appearance of the samples but allowed to obtained completely different pore structure for porous geopolymers. The increased fineness or different mineralogical characteristic to that of alkali-activated clay allowed to obtain much sophisticated pore structure. Macropores were hardly visible even for mixture composition with high foaming admixture content. Specific characteristics of cement allowed to reduce foaming admixture and still obtain highly porous material. The use of foaming admixture in the rate from 0.05 to 0.5 produced fine pore structure with macropores from 0.1-0.3 mm, while the microstructure indicate large number of micropores which were present between larger pores (see magnification 200x and 100x). Higher foaming admixture content made interlayer structure between pores more porous, which later affected both density and compressive strength of the materials. For cured materials with Portland cement additive, material was subjected to severe cracking and disintegration of sample pore structure. The overdose of Portland cement and foaming admixture lead to samples with low strength.

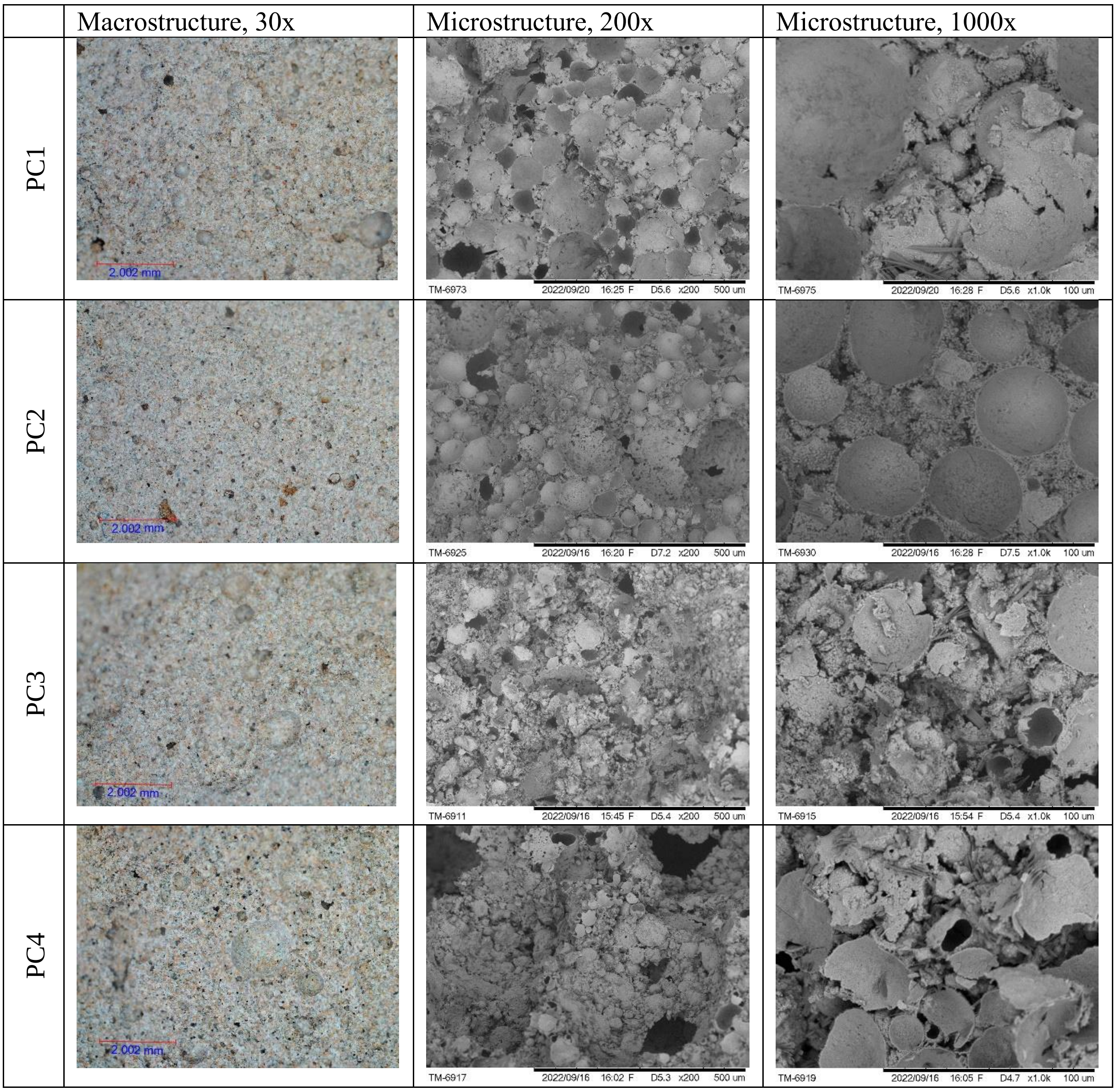
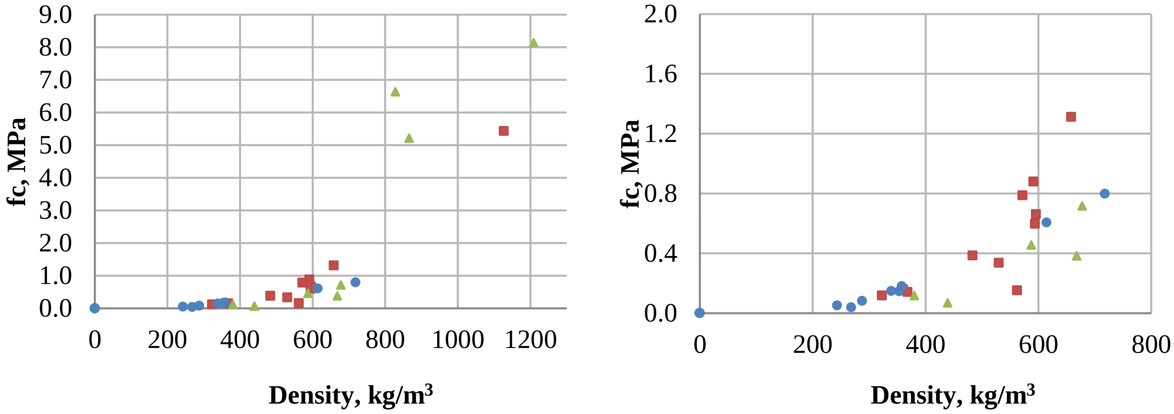


Figure 6. Foamed geopolymer structure with Portland cement additive and synthetic foaming admixture

3.3. Physical and mechanical properties

The relationship between density and compressive strength of obtained geopolymers is given in figure 7. Figure 7a represents both porous geopolymers with low density and samples which were defective (with collapsed pore structure). This represents how the strength of geopolymer binder increases if pore volume reduces. With density as high as from 827 kg/m^3 to 1209 kg/m^3 gives compressive strength from 5 to 8 MPa. By the density below 718 give significant strength reduction and for such foamed geopolymers strength is below 1.3 MPa. Samples with the H_2O_2 foaming admixture was with the highest density as it was not possible to obtain stable foam structure. Density from 827 to 1209 kg/m^3 and strength from 5.2 to 8.1 MPa was obtained.

Samples prepared with pre-foaming technique and pure geopolymer paste give bulk density from 439 to 678 kg/m^3 and the strength of such material was from 0.1 to 0.7 MPa (Figure 7b). Cement addition allowed to obtain much lighter mixtures with low density. Mixture composition with 0.1 to 0.3 parts of foaming admixture allowed to obtain density from 243 to 288 kg/m^3. Unfortunately, such low density even with a cement addition did not allow to reach good compressive strength results. Compressive strength of such materials was 0,04 to 0,08 MPa. Samples with cement addition with density from 323 kg/m^3 to 596 kg/m^3 allowed to obtain lightweight geopolymer with compressive strength from 0,12 to 0.88 MPa.



a) b)

Figure 7. Physical and mechanical properties of porous geopolymers based on red brick powder precursor. a) geopolymers with density up to 1200 kg/m^3, b) for geopolymer with density up to 800 kg/m^3.

4. Conclusions

Porous geopolymers can be obtained by chemical foaming method and pre-foaming method using synthetic foaming admixtures. Chemical foaming proved to be problematic as during gas release, the geopolymer can go through rapid foaming process and there might be problems to keep the porous structure of green geopolymer until it hardens at elevated temperatures. In this research pre-foaming technique was more efficient method to obtain ultra-lightweight geopolymers. The Portland cement addition allowed to obtain geopolymers with much finer pore structure comparing to samples without Portland cement additive. Pore size can be obtained both in macro scale (0.1-0.5mm) and even in microscale when pore size was in range from 5 to 20 μm. Density of geopolymers obtained in this research was from 243 to 865 kg/m^3. The low density was followed by low compressive strength – from 0.04 to 0.08 MPa which might lead to problems with such material handling. Density from 323 kg/m^3 to 596 kg/m^3 gives more reasonable strength of the material (0,12 to 0.88 MPa). Production of porous geopolymers gives a challenge to carefully select mixture composition and ratio between foams and geopolymer paste to obtain durable material with proper physical and mechanical properties.

Acknowledgments

This research is funded by the European Social Fund under the No 09.3.3-LMT-K-712 “Development of Competences of Scientists, other Researchers and Students through Practical Research Activities” measure.

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