FOAMING GEOPOLYMERS PREPARATION BY ALKALI ACTIVATION OF GLASS WASTE

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Foamed geopolymer (inorganic polymers materials) were successfully produced from alkali activated glass waste powder, after thermally treated at temperatures between 500 and 700ºC for 1 hour. These geopolymers were synthesized by mixing the mixed color glass waste powder with (potassium hydroxide and sodium silicate) solutions. Thermal treatment of these new materials at temperatures ranging from 500ºC to 700ºC recorded an important volume increase 18-41%, during to a foaming process specific for sodium or potassium silicate (aluminate) hydrates. For these compositions and due to foaming process, the increase of volume is noticed at 600ºC and partial melting occurs at 700ºC. The formation of glass foams leading to large changes in volume associated with different sizes of open porosity. This method for creating a foaming geopolymers represents a novel reuse of the waste glass in engineering applications as thermal and sound insulations coupled with low cost and environmental benefits.

Keywords: *glass waste, foamed geopolymers, alkali activators, thermal treatments*

1. Introduction

Today a wide variety of industrial and municipal waste was re-used and recycled. These represent an important research field due to the economic, environmental and legal sides [1]. Glass can be utilized and recycled considered to one appealing choice that could significantly contribute to the avoiding or at least decreasing of environmental pollution [2-4].

The European Union produce in excess of 80%soda-lime glass as container glass and flat glass [2,5,6]. Through investigations has been done on the recycle and usage of waste glass from various industries such as construction industry, which was considered as eco-friendly solution for running waste glass [2,6-8].

In cement and concrete production, glass waste can be inserted as aggregate or supplementary cementitious material, therefore, this enhancement addition to further develop the characteristics of sustainability of construction industries [9-11]. In Portland cement concrete mixture, glass bottle cullet can be used to replace a portion of aggregate with some restrictions by some specific affairs like the reaction of alkali silica or bond strengths between glass aggregate and cementitious matrix [10,11]. Geopolymer materials fabrication is one possible applications of other usage for this type of glass waste as solid component basis.

Geopolymers (inorganic polymers), is defined and coined by the scientist Davidovits [12-14].This new materials can be fabricated from natural or synthetic aluminosilicate minerals or industrial aluminosilicate byproducts/wastes (such as: metakaolin, fly ash, slag, red mud،glass, perlite, sand, rice husk ash, clay, or a combination of them) as solid component; mixed with an aqueous solution containing reactive ingredients (potassium/sodium hydroxide, phosphoric acid, potassium/sodium silicate etc. [13,15-23]. According to Davidovits definition, these new materials (geopolymers) as 3D framework structures in which tetrahedral $[SiO_4]^{4-}$ and $[AlO_4]^{5-}$ units are connected with alkali ions balancing the negative framework charge [12-14].

Also, these geopolymers can be synthesized from the alkali activation of soda-glass (cullet) with sodium or potassium hydroxide solutions and curing at 40–60ºC [17]. A previous work [24], new materials (geopolymers or inorganic polymers) was synthesized starting from glass waste powder with/without red mud admixtures by alkaline activation with NaOH solution. In these frameworks; the main reaction products created are sodium silicate hydrates and/or sodium silicate aluminate hydrates with amorphous to crystalline structures. In the hardened geopolymer products; the entity of sodium silicate hydrates and/or sodium silicate aluminate hydrates is most probably to act as a foaming agent; therefore it should be possible to manufacture foamed materials without usage of other foaming agents.

Foams materials (very low density), can be produced by using glass as a portent [25,26].

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Table 1

The oxide composition of mixed glass powder MGP

Sample	Oxide composition (%)									
	SiO_2	Na_2O	CaO	Al_2O_3	Fe_2O_3	MgO	K_2O	TiO_2	SO_3	LOI
Mixed colors glass powder (MGP)	76.71	15.75	4.96	0.83	0.55	0.58	0.12	0.08	0.02	0.36

Table 2

The composition of geopolymers studied

Sample	Solid component	Liquid component	Liquid/Solid Ratio
MGP-K5	Mixed color glass powder	KOH solution (K5)	0.3
MGP-S	Mixed color glass powder	Sodium silicate solution(Na_2SiO_3) (S)	0.3

During the glass foams production; some treatment parameters must be considered and these variables can mutate the product formation. These parameters are: heating rate, amount of foaming agent, time and final temperature of sintering [27-30]. Geopolymer foams have been the focus of optimistic research in the field of porous inorganic materials because of their unparalleled combination of good physical properties correlated with good thermal and chemical stability, excellent mechanical properties [30-34], low CO_2 emission and low energy consumption in their manufacture [35,36].

The possibility to create foamed materials was assessed in this work by thermally treated of geopolymers which was synthesized from the alkali activation of glass waste (cullet). We exhibit for the first time in this research results regarding the thermal treatment behavior of geopolymers fabricated from the glass waste powder (MGP) by alkali activation with KOH 5M and sodium silicate solutions (37%) of mixtures of waste glass.

2. Materials and method

2.1. Materials

Mixed color glass cullet (from a glass bottles production plant) was crushed and grinded in a laboratory ball mill for 15h in order to achieve a fineness with specific surface area of 3035 cm^2/g, examined by Blaine method. Table 1 presents the oxide compositions of mixed color glass powder (MGP). The average particle size (D0.5) is 41.778 µm and was assessed by laser granulometry.

Two types of alkali activated materials (geopolymers) were prepared based on the mixed color glass waste powder MGP as shown in Table 2. The activator solutions were: i) KOH 5M aqueous solution (K5) and ii) sodium silicate solution (S) with a density of 1.534g/cm^3 and 37% concentration.

2.2.Methods

For paste specimens preparation; mixed color glass powder (MGP) as solid component was mixed with alkali activator solution 5M KOH and 37% sodium silicate solutions, the resulted pastes were casted in a cubic molds with dimensions of (20x20x20 mm) and vibrated for 2-3 minutes. The paste specimens were covered with cling film and cured in the electric oven at 60 ºC for 24 h, then demolded and cured at 20ºC in humid air (R.H.85%) up to 7 days.

The specimens after curing (1day at 60ºC + 6 days at 20ºC) were undergo to thermal treatment at various temperatures 500, 600 and 700ºC for 1 h in the electric oven with heating rate was 10ºC/min and these specimens were cooled slowly in the same oven until next day. These thermal treatment experiments were done twice for each degree of temperature.

The volume changes of the pastes were determined by thermally treatment of specimens was estimated from the formula (1):

$$\Delta V = [(Va-Vb)/Vb] \times 100\ (\%) \quad (1)$$

where: Vb and Va; specimen‘s volume before and after thermal treatment respectively.

The mass changes of the pastes were determined by the thermal treatment of specimens and calculated from the formula (2):

$$\Delta M = [(Ma-Mb)/Mb] \times 100\ (\%) \quad (2)$$

where: Mb and Ma; specimen‘s mass before and after thermal treatment respectively.

Chemical (oxide) composition of soda lime silicate mixed color glass powder (MGP) was assessed by using the analytical technique X-ray fluorescence spectrometry (SHIMADZU XRF-1800).

Mineralogical compositions were assessed for MGP-K5 and MGP-S pastes by using X-ray diffraction analysis type SHIMADZU XRD-6000 using a monochromatic CuKα radiation (λ = 1.5406 Å) for the XRD patterns in the range of 2θ from 5 to 80°.

Blaine specific surface area of mixed color glass powder MGP was examined bya method described in SR EN 196-6, 2010 [37].

Particle size distributions of mixed color

glass powder MGP were assessed by MASTERSIZER 2000 (MALVERN) laser particle size analyzer.

Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray spectroscopy (EDS) analyses were carried out for paste specimens coated with Au(30nm), using type TESCAN-Vega III microscope.

3.Results and discussion

In order to evaluate the thermal behavior of the studied geopolymers paste specimens (MGP-K5 and MGP-S); firstly they were cured for the first 24 h in an oven at 60ºC then those samples were left up to 7 days at 20ºC. After that they were thermally treated at temperatures of 500, 600 and 700 ºC for 1 h. Finally the specimens were cooled slowly in the same oventill the next day.

Figures 1 and 2, presents the visual appearance of paste specimens, before and after thermal treatment.

From Fig.1, it can be observed the MGP-K5 geopolymer paste specimens treated at 500 and 600ºC has a orderly structure with some cracks at 500ºC and exhibits a significant expansion (swelling) at 600ºC.The partial melting (self-glazing) recorded at temperature 700ºC, thus shape deformation and increase in volume of the specimens (see Fig.3).

Figure 2, illustrated MGP-S geopolymer paste specimens before and after thermal treatment temperatures at 500, 600 and 700 ºC for 1 h; it can be seen the expansion and deformation in the structure with increases the temperatures of thermal treatment. At 500ºC, the paste specimen present's cracks and partial melting; the specimen presents the shape deformation and swelling at600ºC. At 700°C one can observed the increase of the porosity. In general, the volume change values (calculated from formula 1) for both geopolymers were increases with the increase of thermal treatment temperatures (due to a foaming process) (see Fig.3); from our results one can noticed a sharp increase in volume for MGP-S paste at 700ºC as compared with MGP-K paste at the same thermal treatment temperature.

The increases in volume after thermally treated for the pastes are due to the occurrence and different rates of two processes:

- Dehydration of sodium (aluminate) silicate hydrates and the conversion with gas generation [23,38].
- "Softening and melting" phenomenon of material.

The mass changes (mass loss ΔM%) as shown in Fig.4, were calculated assessed for the cubic pastes with the formula (2). It can be seen that the mass loss recorded for MGP-K5 and MGP-S pastes after thermal treatment at temperatures ranging from 500-700ºC were comprised between 0.46-13.14%.The smaller mass loss (0.46%), recorded at 500ºC for MGP-K5 is most probably due to a existence of a lower quantity of sodium or potassium silicate aluminate hydrates formed in this composition[39].

The higher value of mass loss 8.01% and 13.14% recorded at 600ºC for MGP-K5 and MGP-S respectively (see Fig.4), indicates the presence a higher amount of sodium/potassium silicate (aluminate) hydrates in this composition. For MGP-S paste specimens, mass loss recorded (12.41-13.14%) after thermal treatment at 500, 600 and 700 ºC. These losses in mass are important and at

Fig.1 -MGP-K5 paste specimen's images before and after thermally treated at 500,600 and 700ºC/1h.

Fig.2 - MGP-S paste specimen's images before and after thermally treated at 500,600 and 700ºC/1h

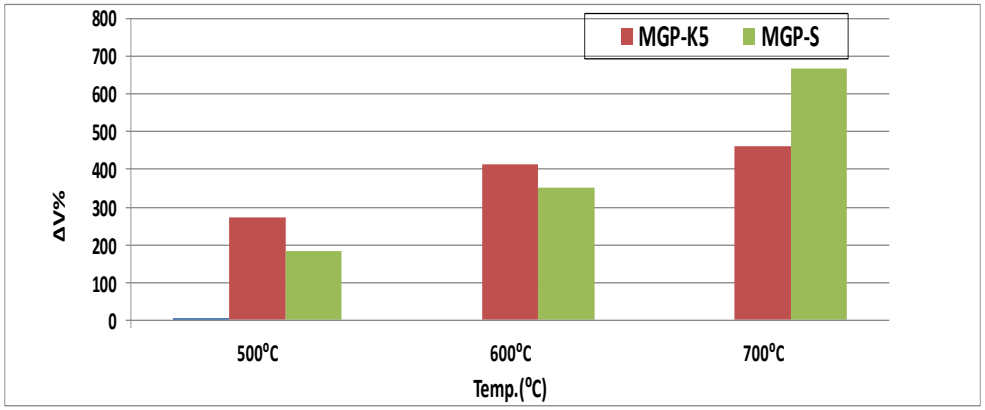


Fig.3 -Volume changes of MGP-K5 and MGP-S paste specimen's after thermally treated at 500,600and700ºC/1 h.

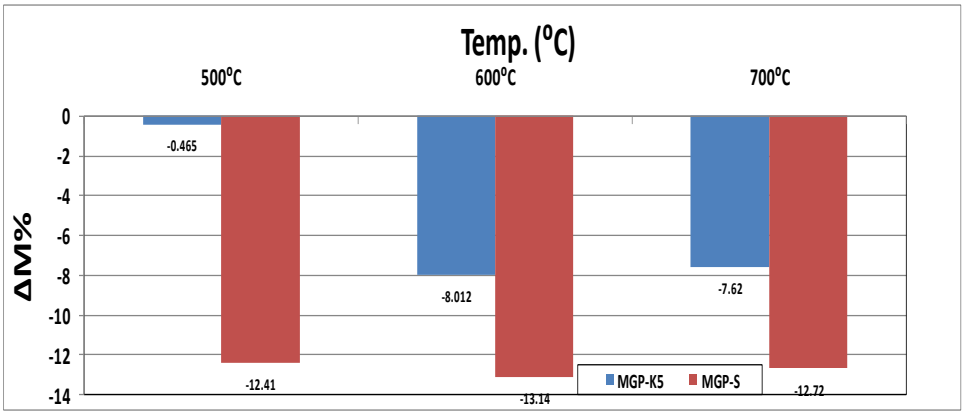


Fig.4 - Mass change of MGP-K5 and MGP-S paste specimen'safter thermally treated at 500,600and700ºC/1 h.

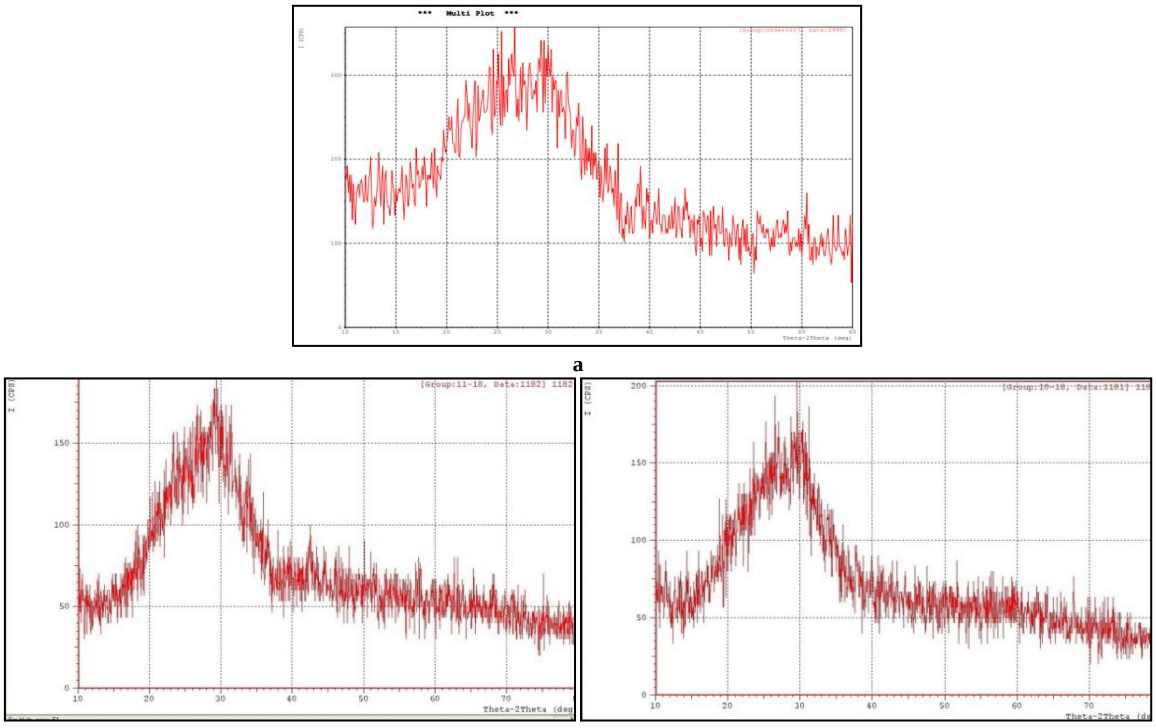


Fig. 5 - XRD patterns of a: MGP, b: MGP-K5 pastes, c: MGP-S pastes (cured 1 day at 60°C and up to 7 days at 20°C).

most due to the transformation and dehydration of sodium silicate (aluminate) hydrates which represents the foaming of the material [23].

Both (MGP-S and MGP-K5) pastes, the mass loss recorded at 600ºC is due to the release of gases (foaming) and volume increase observed forthese specimens Figs.3 and 4.

X-ray analysis of mixed glass powder (MGP)

and geopolymers pastes (MGP-K5 and MGP-S) cured 1 day at 60°C then curedup to 7 days at 20°C are presented in Fig.5. Mixed glass powder as anticipated, have a glassy structure (see the wide peak between 2θ = 20–35); the same shape of XRD pattern recorded for the alkali activated materials suggests the formation of geopolymers as reaction products with very low crystalline (gel like).

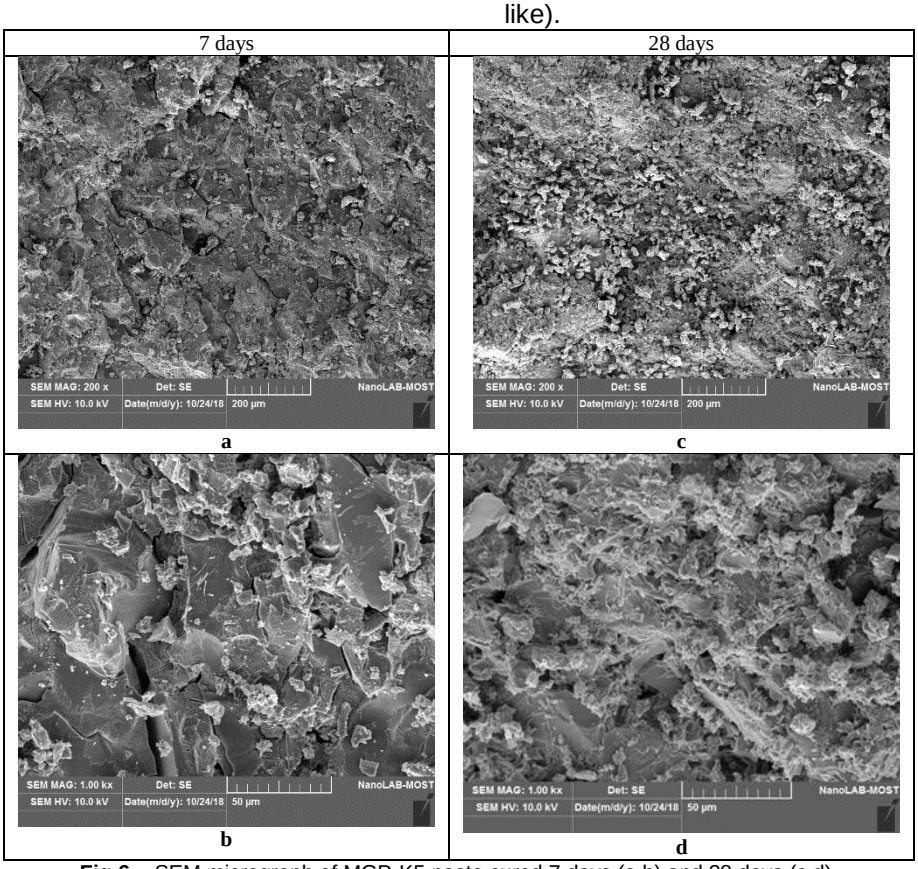


Fig.6_ - SEM micrograph of MGP-K5 paste cured 7 days (a,b) and 28 days (c,d).

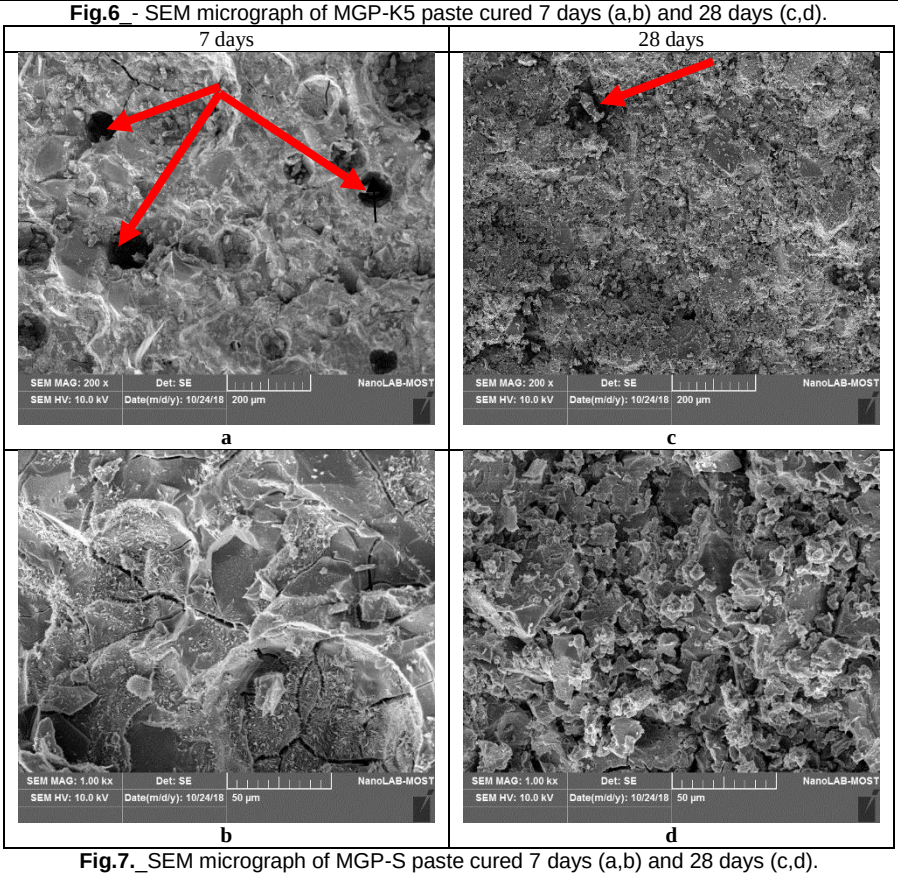


Fig.7._SEM micrograph of MGP-S paste cured 7 days (a,b) and 28 days (c,d).

Figures 6 and 7, present the SEM images and the microstructure of geopolymer pastes (MGP-K5 and MGP-S).

For MGP-K5 one can assess in the SEM macrographs from Fig.6, glass grains included in a continuous matrix formation most probably by the hydrates of potassium silicate aluminate [24].

All pastes produced by alkali activation of mixed glass powder with sodium silicate solution MGP-S observed pores with different sizes in the micrographs (see arrows Fig.7a & c). These round pores result essentially by air detention during the mixing of paste.

The EDS analyses for geopolymer paste prepared (MGP-K5 and MGP-S) are presented in Figs. 8 and 9 respectively. From these figures, it can be confirmed the presence of Si, Na ,Ca, K.

For both pastes MGP-K5 and MGP-S, the existence of Si and Na in a higher quantity in the binding matrix at most the presence of alkali (aluminate) silicate hydrate.

The microstructure of paste specimens MGP-K5 and MGP-S after thermal treatment at 500, 600 and 700°C was assessed by SEM, in fracture as shown in Figs.10 and 11 respectively.

The SEM images (Fig.10 a & b), of MGP-K5 pastes after thermally treated at 500°C presents an increase of the internal porosity; pores with irregular shapes and sizes in the range between 66- 201 µm are appears in this specimen. This significant increase of the porosity is due to the transformation and dehydration of potassium silicate (aluminate) hydrates obtained by activation of glass powder with KOH solution. One can also notice the increase of pores size 99-657 µm in the paste thermally treated at 600°C (Fig.10 c & d); for the paste after thermally treated at 700°C there are present large pores size 222-2644 µm, in addition to small round pores in the walls of the larger pores size (Fig.10 e & f).

Figure 11, present the SEM micrographs of the MGP-S paste specimens after thermally treatment at 500-700°C. The size (diameter) of the pores is in the range of 46-605 µm, 76-958 µm and 141-1335 µm respectively. The increase of temperature of thermal treatment from 500 to 700°C has an important and clear effect on the microstructure of the foams. Both large and small size of round pores, were formed by the dehydration of hydrated compounds [23,40]. The further increase in the size and non-uniform of pores indicates that the activation of the foaming process was generated by the thermal treatment.

The high value of open porosity and pores inter connectivity suggest that these new synthesized materials are appropriate for filtration process [7], usage in sound absorption [41] and other engineering applications. Furthermore, they could be utilized as fireproof insulation (lightweight), the interior walls or used as ceiling flagstones in building.

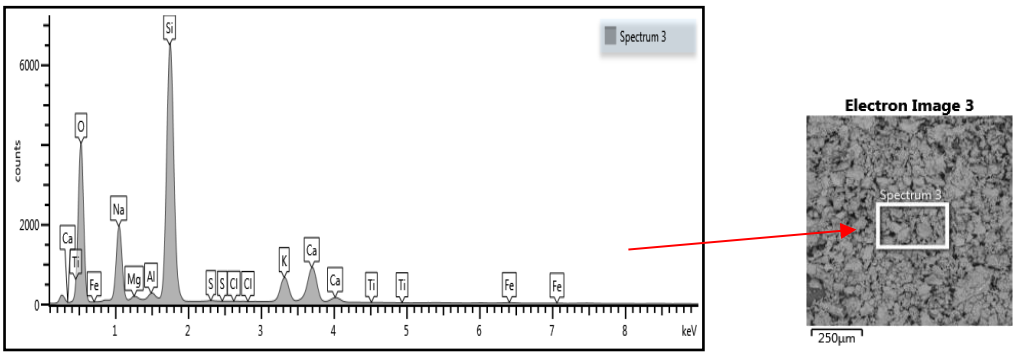


Fig.8 - SEM micrograph and EDS for MGP-K5 paste cured 24 h at 60°C and up to 7 days at 20°C.

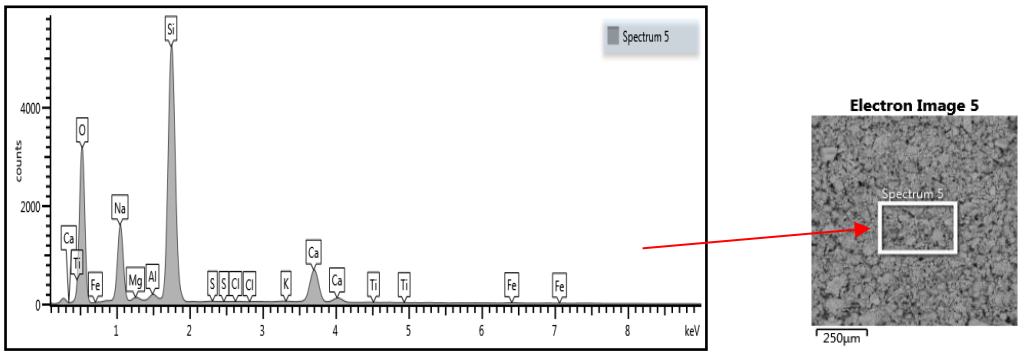


Fig.9 -SEM micrograph and EDS for MGP-S paste cured24 h at 60°C and up to 7days at 20°C.

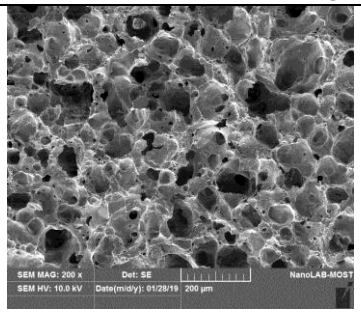
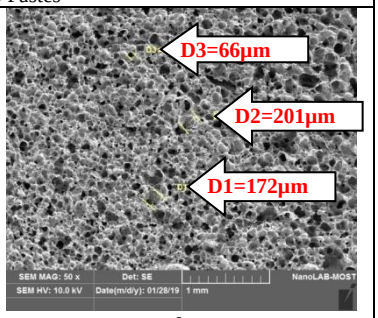
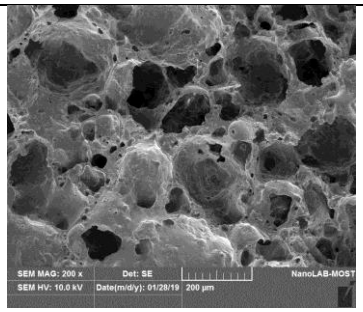
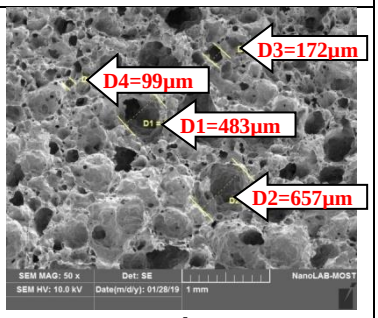
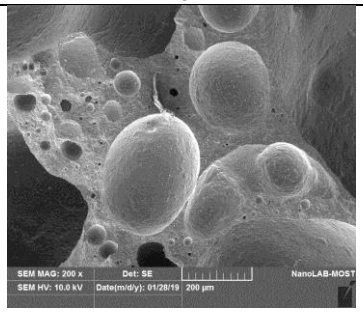
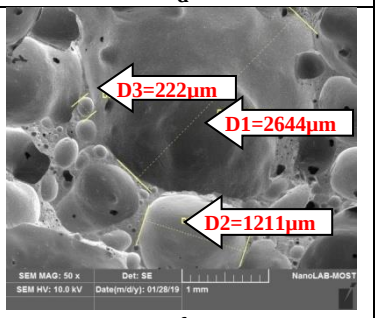
Temp.	MGP-K5 Pastes	
500°C	 a	 b
600°C	 c	 d
700°C	 e	 f

Fig.10 -SEM images of MGP-K5 paste specimens after thermally treated at temperatures 500,600 and 700°C (on fracture surface).

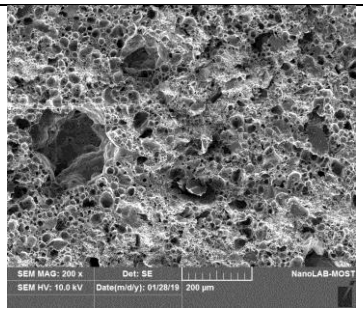
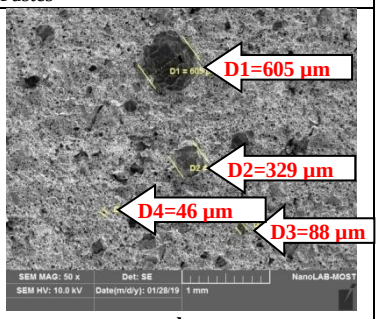
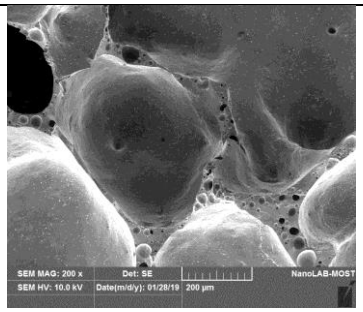
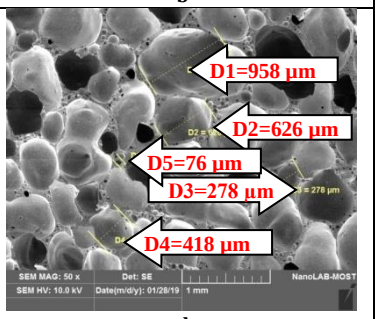
Temp.	MGP-S Pastes	
500°C	 a	 b
600°C	 c	 d

Fig. 11 continues on next page

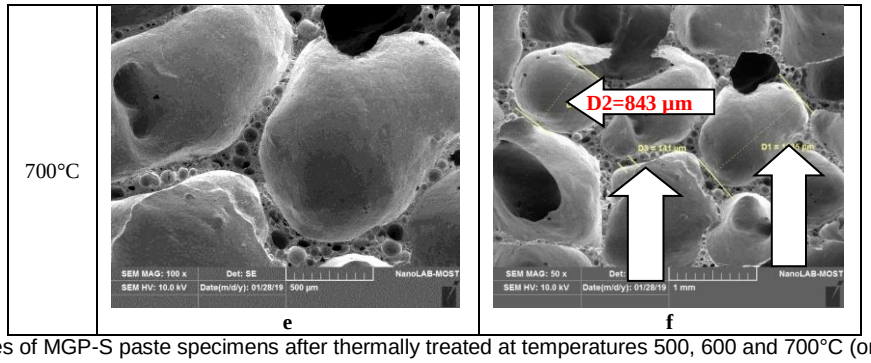


Fig.11 -SEM images of MGP-S paste specimens after thermally treated at temperatures 500, 600 and 700°C (on fracture surface).

4. Conclusions

In this work, is highlighted the possibility to reuse the glass waste (mixed color glass cullet) in order to obtain foamed geopolymers. The studied geopolymers were manufactured using the glass waste powder "as solid component" and potassium hydroxide (5M) and sodium silicates solutions 37% concentration"as liquid components".

The resulted geopolymers were thermally treated at temperatures in the range between 500°C and 700°C and a significantincrease in volume was recorded according to a foaming process particular for sodium/potassium silicate (aluminate) hydrates, the main components of these materials.

Regarding foamed geopolymers synthesis (increasing volume, increasing size of porosity and light weight), the results obtained so far, are promising; these materials could be use in engineering applications such as thermal and sound insulations, with low cost, light energy saving materials, and environmental benefits.

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