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Preparation and characterization of foamed geopolymers from waste glass and red mud

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HIGHLIGHTS

- Foamed geopolymers were obtained by re-using two waste (glass cullet and red mud).
- As liquid component NaOH solution or the filtrate obtained by red mud slurry dewatering was used.
- The foaming process of sodium silicate (aluminate) hydrates determine important volume increase.
- Open pores with a wide range of sizes (1–100 μm) were present.
- The obtained foamed geopolymers are promising for thermal and sound insulation.

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ABSTRACT

New foamed geopolymer materials were successfully synthesized from two waste i.e. red mud and cullet soda-glass, by thermal treatment at 600–800 °C for 1 h. The geopolymers were obtained by the alkali activation with sodium hydroxide solution or the liquid part of red mud slurry (filtrate) of waste glass (cullet) with/without red mud admixture.

Thermal treatment of these geopolymers, at temperatures between 600 and 800 °C determined an important volume increase, due to a foaming process specific for sodium silicate (aluminate) hydrates, the main components of these materials. When red mud substitutes 25% of brown glass, softening and partial melting of material occurs at higher temperatures (due to the presence of complex refractory crystalline phases with Al content); for these compositions the increase of volume due to foaming process is noticed at higher temperatures.

Open pores with a wide range of sizes (1–100 μm) were assessed by scanning electron microscopy; this highly porous microstructure, formed by the bubbling and coalescence of bubbles, is specific for foam materials. The compressive strength values of foamed geopolymers are in the range 2.1–8.6 MPa, and as expected, decrease with the increase of open porosity.

1. Introduction

Large amounts of inorganic waste are produce yearly allover the word. Majority of them are back-filled therefore recycling and re-use of inorganic industrial and municipal waste (such as glass cullet) is an important environmental issue. Red mud slurry, waste resulted in bauxite processing plants, has a major environmental impact due to its high alkalinity and its generation in huge amounts, estimated at 200 million tons per year worldwide [1]. In order to reduce the environmental impact of red mud storage as well as open new opportunities for its re-use, its neutralization to a pH value around 8 is generally performed [1]. Among the multiple uses of red mud, the most promising is for building material production i.e. cement, bricks, glass ceramics or geopolymers [1,2].

Geopolymers or alkali activated cements are materials that can be produced from two precursors i.e. a solid component (aluminate silicate source) and alkaline activators (caustic alkalis or alkaline salts). Numerous types of aluminate silicate sources can be used for geopolymers synthesis, the most common being metakaolinite, slag and fly ash. The use of highly alkaline red mud in combination with slag or fly ash permits not only its neutralization [3] but also the development of geopolymeric materials for construction and building industry [1,4,5]. Chen and co-workers [6] reported also the possibility to recycle red mud in combination with fly ash for foamed ceramics production, using sodium silicate as foaming

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agent. Guo and co-workers [7] reported also the synthesis of glass ceramic foamed materials based on fly ash and red mud with calcium carbonate as foaming agent. This type of foamed ceramics had good performance for thermal and sound insulation being considered energy saving materials.

Geopolymers can be obtained also by the alkali activation of cullet soda-glass with sodium or potassium hydroxide solutions and curing at 40–60 °C [8]. In our previous work, binding materials were synthesized starting from waste glass with/without red mud admixtures by alkaline activation with NaOH solution [9]. The main reaction products formed in these systems are sodium silicate hydrates or/and sodium silicate aluminate hydrates with amorphous to crystalline morphologies. The presence of sodium silicate hydrates or/and sodium silicate aluminate hydrates in the hardened geopolymer is likely to act as a foaming agent therefore it should be possible to produce foamed materials without the use of other foaming agents.

Therefore, in this study we assess the possibility to produce foamed materials by the thermal treatment of geopolymers resulted by the alkali activation of waste glass (cullet) with/without red mud admixtures. We present also results regarding the synthesis and thermal treatment of geopolymers obtained by alkali activation of waste glass and red mud mixture with the liquid part of red mud slurry (filtrate). To our best knowledge no information are presently available regarding the synthesis and properties of this type of foamed geopolymers.

2. Materials and methods

2.1. Materials

Waste glass (cullet) from glass bottles production plant was used as solid component for the synthesis of geopolymers. Brown glass cullet was milled for 10 h in a ball mill.

Table 1
Composition of alkali activated binders.

Sample	Solid component (w.%)		Liquid component
	Brow glass (B)	Red mud (R)	
B_N5	100	–	NaOH 5 M
BR25_N5	75	25	NaOH 5 M
BR25_RF	75	25	Red mud slurry filtrate (RF)

Red mud sludge received from alumina plant was dewatered by filtration. The liquid part represents around 32%. The filtrate (RF) has the pH of 12.96 and a conductivity of 21.82 mS/cm. The density of this liquid is 1.015 g/cm^2. The filtrate was titrated with HCl 0.1 N up to a pH value of 7 in order to determine it's acid neutralization capacity (ANC) [10]. The ACN was 3 moles H^+/l.

The solid part (R) was dried at 100 °C, up to constant mass, in order to determine its moisture content – 36%. This material was calcined at 1000 °C and the value of loss on ignition was 8%. The dried red mud (R) was disaggregated in a ball mill for 5 min. The density of R powder determined by pycnometric method was 1.69 g/cm^3.

Brown glass powder (B) or mixtures of glass powder and red mud (Table 1) were used for the preparation of geopolymers. The activator solution was NaOH 5 M aqueous solution (N5) or the filtrate (RF) resulted in red mud slurry processing. The liquid to solid ratio was of 0.3.

For the preparation of paste specimens the solid component was mixed with alkali activator solution; the resulted material was cast in cubic (l = 20 mm) molds and vibrated for 2 min. The specimens were cured in the mold (covered with cling film) at 60 °C the first 24 h, then de-molded and cured at 20 °C in humid air (R.H. 85%) up to 7 days. After that the specimens were subjected to thermal treatment at different temperatures (between 400 and 800 °C), for 1 h, in an electric oven. The heating rate was 10 °C/min and the specimens were slow cooled in the oven until next day. Each thermal treatment experiment was carried out in duplicate.

2.2. Methods

Chemical compositions of waste glasses powder (B), was assessed using the chemical and analytical procedures stipulated in Romanian standards 5771/1-11/89 [11] similar with ASTM C196-92 (2011).

Elemental compositions of red mud (R) and filtrate (RF) were assessed by X-ray fluorescence spectrometry.

The glass dilatograms were recorded with dilatometer in temperature range: 20–800 °C with a heating rate of 10 °C/min.

The particle size distribution of the brown glass powder and red mud were assessed with a laser particle seizer.

The mineralogical compositions of dried red mud (R), alkali activated materials (after curing 24 h at 60 °C and 6 days in air – see § 2.1) and after thermal treatment at 400 °C, 600 °C and 800 °C, were assessed by X-ray diffraction (XRD). The XRD spectra were obtained using a monochromatic CuKα radiation (λ = 1.5406 Å), range 2θ from 5° to 50°.

SEM and EDX analyses were performed on paste specimens coated with Ag.

Thermal analyses were performed in the temperature range: 20–1000 °C with a heating rate of 5 °C/min.

Apparent density and open porosity of pastes, before and after thermal treatment, were measured using the liquid saturation method under vacuum [12]; as working liquid was used xylene (C_8H_{10}, ρ = 0.866 g/mL).

The weight losses of thermally treated pastes were calculated with the formula:

$$\Delta W = [(W_a - W_b)/W_b] \cdot 100\ (\%) \tag{2.1}$$

where W_b = specimen's weight before thermal treatment; W_a = specimen's weight after thermal treatment.

Table 2
Elemental composition of red mud (R) and filtrate (RF).

Materials	Element (w.%)										
	Fe	Na	Al	Ti	Si	Ca	Cr	P	S	K	Other elements
R	37.81	25.49	17.39	5.97	5.59	2.64	0.25	0.18	0.17	0.06	4.28
RF	–	6.32	2.65	–	0.03	–	–	–	–	0.05	90.95

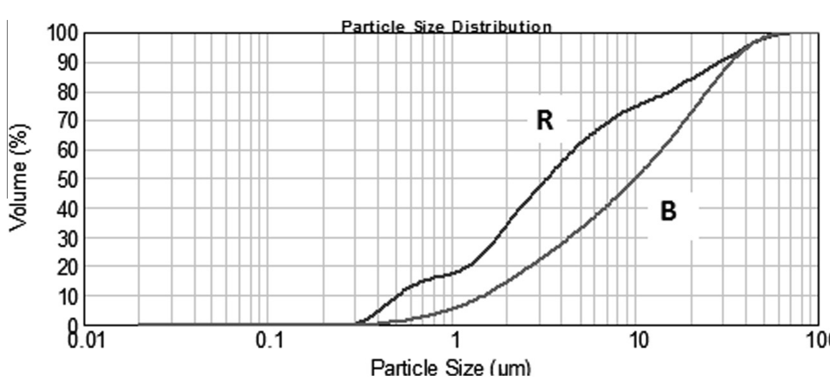

Fig. 1. Particle size distribution of brown glass (B) and red mud (R).