



# Geopolymerization of glass- and silicate-containing heated clay



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## HIGHLIGHTS

- Na-Chabazite and gels formed in the cured alkali activated amended heated clay.
- The neoformation processes involved metakaolinite and glass or Na-silicate derivatives.
- The properties of the geopolymers and the operating factors were related by using polynomials.
- The suitable factors for bricks manufacturing from the cured materials were evaluated.

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## ABSTRACT

The microstructures of cured alkali-activated glass-modified heated clay and sodium silicate-containing heated clay were investigated by using X-ray diffraction, Fourier transform infrared spectroscopy, thermal analysis and scanning electron microscope. Moreover, the effects of the  $\text{SiO}_2/\text{Na}_2\text{O}$  ratio, ageing time and curing temperature and their mutual interactions on bending strength and water absorption of the cured alkali-activated materials were assessed by using the response surface methodology (RSM). It was found that the cured materials were composed of gels, neoformed crystalline phases (Na-chabazite, sodium carbonate) together with starting constituents (illite, quartz, metakaolinite, glass). The zeolite/gel essentially neoformed from metakaolinite derivatives. Sodium carbonate was the product of samples carbonation. A part of the gel formed from waste glass and sodium silicate derivatives respectively. The RSM results showed that the weights of the effects of the experimental factors on the measured properties of the materials obtained were well predicted by using polynomial models, and the  $\text{SiO}_2/\text{Na}_2\text{O}$  ratio was the most influencing factor. The effects of the factors studied were discussed in relation to the microstructure characterization. Also, the results showed that cured alkali activated glass-amended heated clay could be suitable for bricks manufacturing.

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## 1. Introduction

In concentrated alkaline solutions, siliceous materials (e.g., fly ash) and aluminosilicates (e.g., metakaolinite) could lead to the formation of three-dimensional structures of inorganic polymers (geopolymers), considered as promising eco-friendly cementitious materials [e.g., 1]. With the use of sodium hydroxide as activator and aluminosilicates as source materials, the frameworks of geopolymers should be composed of linked tetrahedral units of aluminate and silicate.  $\text{OH}^-$  ions provided by the base used plays a key role in the depolymerization of the structure of the aluminosilicate, whilst  $\text{Na}^+$  ions contributes to the neoformation of zeolites and to the offset of the charge deficit [2–4].

Geopolymerization could be also realized with sodium silicate solution. In this case, the degree of polymerization of the dissolved species was proven to be dependent on the ratio of the amount of  $\text{SiO}_2$  to that of  $\text{Na}_2\text{O}$ . The use of excessive silica may result in a premature solidification of the geopolymer-forming paste, and consequently it leads to the decrease of the rate of the geopolymerization reaction [3].

Referring to some authors [4], higher geopolymerization rates were obtained with mixtures of  $\text{NaOH}$  and  $\text{Na}_2\text{SiO}_3$  solutions. In the use of such a mixture, sodium hydroxide was presumably involved in the dissolution of the source materials, while  $\text{Na}_2\text{SiO}_3$  acted as binder [5].

The geopolymerization processes depend on, among others, the chemistry of the activator, the source material, the curing temperature and ageing time [6–9]. Dehydroxylated clay minerals, such as

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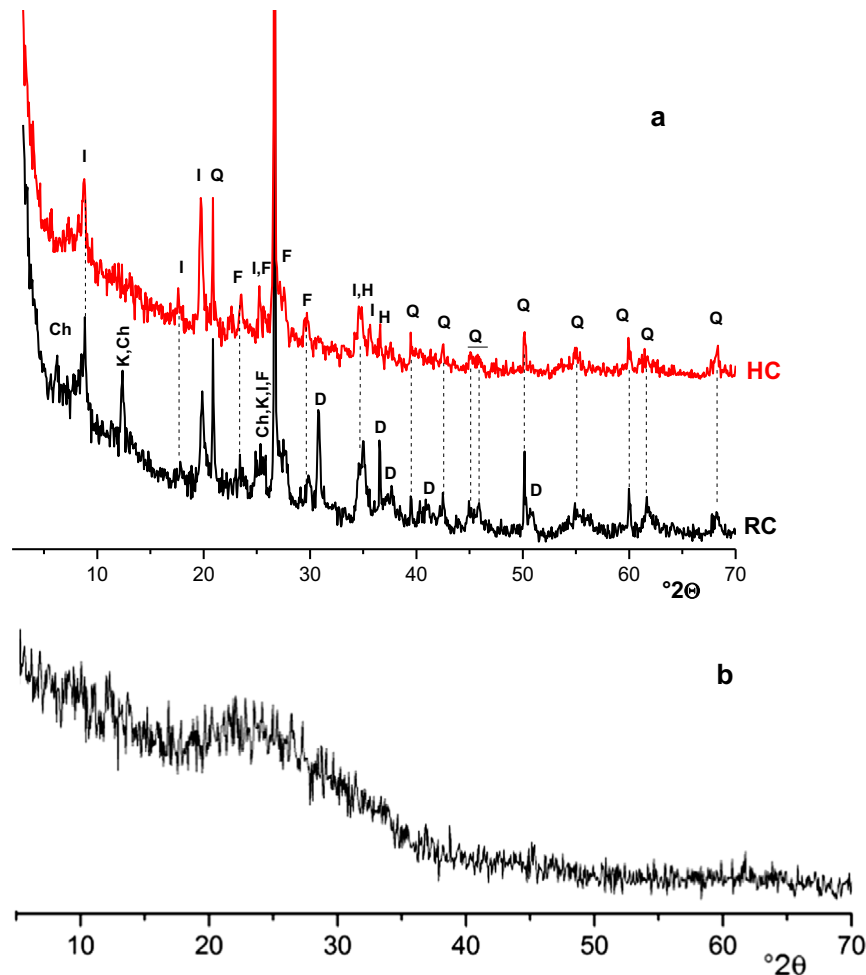
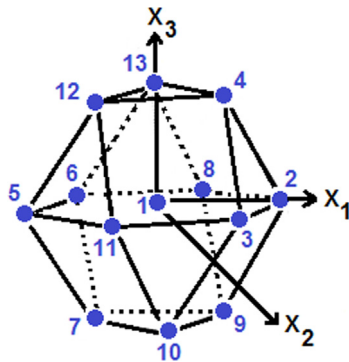
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**Table 1**

Chemical compositions (wt.%) of the clay and waste glass used.

	SiO <sub>2</sub>	Na <sub>2</sub> O	CaO	MgO	Al <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	L.O.I
Clay	51.4	1.0	3.1	3.3	21.7	3.5	4.0	0.6	10.7
Waste glass	68.7	20.9	4.4	3.7	1.4	0.4	0.1	–	0.2

\* Loss on ignition at 1000 °C.

**Fig. 1.** X-ray diffraction patterns of the raw (RC) and heated (HC) clay (a), and the waste glass used (b). I: illite (PDF# 43-0685); Ch: chlorite (PDF# 73-2376); K: kaolinite (PDF# 83-0971); Q: quartz (PDF# 05-0490); F: K-feldspar (PDF# 76-0831); D: dolomite (PDF# 84-1208); H: hematite (PDF# 85-0987).**Fig. 2.** Spatial distribution of the planned experiments listed in Table 2.

metakaolinite, are convenient source materials for geopolymer synthesis. Metakaolinite and NaOH and/or sodium silicate were extensively used for geopolymers production [e.g., 10]. The use of

sodium silicate resulted in good mechanical/physical properties of the geopolymer. But, because of its adverse effect on the environment [11], an intense research activity has been developed in order to find out alternative efficient activators.

In strong alkaline solutions, glass could be the subject of dissolution, and gives rise to various siliceous forms [12], which could be used as an alternative substitute of sodium silicate in the elaboration of geopolymers [13]. As soda-lime glass, wastes of glass windows could be used in conjunction with aluminosilicates for geopolymers preparation. In fact, recently waste glass together with fly ash [13] or metakaolinite [14] was used for geopolymers synthesis. However, to the best of our knowledge no attention was paid to the geopolymerization of alkali activated glass-modified raw clays or heated clays. Moreover, very little attention has been paid to the evaluation of the simultaneous effects of SiO<sub>2</sub>/Na<sub>2</sub>O molar ratio, ageing time and curing temperature on the mechanical/physical properties of raw clay-based geopolymers.

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