



## Research paper

# Mechanical and microstructural properties of metakaolin-based geopolymer cements from sodium waterglass and phosphoric acid solution as hardeners: A comparative study



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## ABSTRACT

Sodium waterglass with mass ratios  $\text{SiO}_2/\text{Na}_2\text{O}$  and  $\text{H}_2\text{O}/\text{Na}_2\text{O}$  equal to 1.5 and 10, respectively was prepared from commercial silica fume as a silica source. The phosphoric acid solution with molar concentration 10 M was prepared by dilution of commercial phosphoric acid in distilled water. The ATR-infrared spectrum of alkaline hardener shows the presence of  $\text{SiQ}^0$ ,  $\text{SiQ}^1$  and  $\text{SiQ}^2$  units suggesting a more depolymerized solution. While the acid hardener contains  $\text{H}_2\text{PO}_4^-$  due to the deprotonation of commercial  $\text{H}_3\text{PO}_4$  indicating that the molar concentration 10 M contained an appropriate amount of water necessary for the workability. The formation of  $\text{H}_2\text{PO}_4^-$  is appropriate for the second step of geopolymerization. Metakaolin-based geopolymer cements were obtained by adding each fresh hardener to metakaolin. The results show that the compressive strength of phosphate-based geopolymer cement is 93.8 MPa while the one of alkali-based geopolymer cement is 63.8 MPa. The difference of the compressive strength could be related to the formation of berlinite ( $\text{AlPO}_4$ ) in the structure of phosphate-based geopolymer cement which acts as a filler and reinforces the structure and therefore the compressive strength of the specimen. However, the microstructures of both geopolymer cements are homogeneous and compact structure. It was typically found that phosphate-based geopolymer cement has a higher compressive strength compared to the one of alkali-based geopolymer cement. It is important to note that the hardening of metakaolin-based geopolymer cement from phosphoric acid solution required an energy gradient while metakaolin-based geopolymer cement from sodium waterglass hardens at room temperature like regular Portland cement. The purpose of this work was to compare the mechanical and microstructural properties of metakaolin-based geopolymer cements obtained in the same condition using sodium waterglass and phosphoric acid solution as hardeners. Due to their higher mechanical properties, phosphate-based geopolymer cement could be used for the construction of roads and bridges and geopolymer cements from alkaline medium could be used for building the houses.

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## 1. Introduction

Geopolymer is a concept, or a science or a technology that we used to prepare geopolymer cements, mortars or concretes. Geopolymer cement is a new class of binder system which is generally obtained in an alkaline medium using sodium or potassium water glass. This inorganic polymer is essentially a mineral chemical compound consisting of repeating units such as poly(sialate)  $-\text{Si}-\text{O}-\text{Al}-\text{O}-$ , poly(sialate-siloxo)  $-\text{Si}-\text{O}-\text{Al}-\text{O}-\text{Si}-\text{O}-$ , poly(sialate-disiloxo)

$-\text{Si}-\text{O}-\text{Al}-\text{O}-\text{Si}-\text{O}-\text{Si}-$ , created through a process of geopolymerization at room temperature like regular Portland cement. According to Davidovits (2011), geopolymer cement can be obtained by different two routes such as in alkaline medium ( $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Li}^+$ ,  $\text{Ca}^{++}$ ,  $\text{Cs}^+$ , etc.) and in acidic medium with phosphoric acid or humic acids. The product obtained using the phosphoric acid solution as a hardener is called poly(phospho-siloxo)  $-\text{Si}-\text{O}-\text{P}-\text{O}-\text{Si}-\text{O}-$ . This inorganic polymer is obtained after curing the fresh samples for 24 h in an oven at about 60 °C (Cao et al., 2005; Perera et al., 2008; Louati et al., 2016a, 2016b). Geopolymer cement prepared in an alkaline medium have good mechanical properties (Davidovits, 1991), chemical properties (Palomo et al., 1999), thermal behavior (Elimbi et al., 2014; Tchakouté et al., 2016), etc. Recent studies have shown that metakaolin phosphate-based geopolymer cements possess high mechanical properties. For examples, Cao et al. (2005)

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investigated the molar ratios Si/P between 0.13 and 0.63 and obtained metakaolin phosphate-based geopolymer cement with a maximum strength of 55 MPa. [Perera et al. \(2008\)](#) performed a thorough study on the geopolymerization conditions and obtained a material with compressive strength range of 140–146 MPa. Other researchers such as [Louati et al. \(2014, 2016a,b\)](#) used the Tunisian raw material for producing metakaolin phosphate-based geopolymer cements. They obtained materials with a maximum strength of 59 and 37 MPa. In our point of view, the comparison of the mechanical and microstructural properties of metakaolin-based geopolymer cements from sodium waterglass and phosphoric acid solution as reactive ingredients which are prepared in the same conditions could be very interesting.

The main objective of this work was to use sodium waterglass and phosphoric acid solution as hardeners for producing metakaolin-based geopolymer cements. The mechanical and microstructural properties of both geopolymer cements have been studied using infrared spectroscopy, X-ray diffractometry (XRD), scanning electron microscope (SEM) coupled with energy dispersive X-ray spectroscopy (EDX) and compressive strength.

## 2. Materials and experimental methods

Kaolin used in this study was extracted from Bomkoul in the Littoral region of Cameroon. This raw material was crushed in a ball mill with a porcelain jar and microspheres of higher-grade alumina as grinding medium. The obtained sample was passed through a 90 mesh sieve to get powder which was calcined at 700 °C in a programmable electric furnace (Nabaertherm, Mod\_LH60/14) for 4 h with a heating and cooling rate of 5 °C/min to obtain metakaolin. The physico-chemical characterization of kaolin was already studied by [Elimbi et al. \(2011\)](#). The chemical composition of this kaolin is given in [Table 1](#). Commercial phosphoric acid (H<sub>3</sub>PO<sub>4</sub> 85%, puriss. p.a., Reag. ACS, Reag. ISO, Reag. Ph. Eur.) was used to prepare acid reactive ingredient or acid hardener. Commercial silica fume, SiO<sub>2</sub>, xH<sub>2</sub>O (Merck, N<sup>o</sup> 10 279-57-9) was used to prepare the alkaline hardener.

The acid solution was prepared by mixing distilled water and commercial phosphoric acid, in order to get the reactive ingredient or hardener with molar concentration 10 M, denoted PS4. The alkaline solution was prepared by adding commercial silica fume to NaOH pellets and mixed with distilled water. The assembly was treated for 1 h at 100 °C using a magnetic stirrer to enhance the dissolution of amorphous silica from silica fume in order to obtain the reactive ingredient with the molar ratios SiO<sub>2</sub>/Na<sub>2</sub>O and H<sub>2</sub>O/Na<sub>2</sub>O kept constant at 1.5 and 10, respectively. The obtained sodium waterglass was denoted as NSF. The molar ratios SiO<sub>2</sub>/Na<sub>2</sub>O was chosen according to the findings work of [Gao et al. \(2014\)](#) which reported that the most convenient molar ratio SiO<sub>2</sub>/Na<sub>2</sub>O in the hardener necessary to prepare geopolymer cements with higher mechanical strength is 1.5. According to [Davidovits \(2011\)](#), the alkaline solution with a molar ratio SiO<sub>2</sub>/Na<sub>2</sub>O equal to 1.5 could classify as a merely user-friendly hardener. The aforementioned hardeners, i.e. NSF and PS4 were allowed to mature in the laboratory at room temperature for 24 h prior to use in order to dissolve and dilute completely silica and commercial phosphoric acid, respectively.

Both geopolymer cements were prepared by adding each hardener gradually to metakaolin in a porcelain mortar and mixing mechanically for 5 min, obtaining the specimens GPS4 and GPWG. GPS4 and GPWG correspond to geopolymer cements from phosphoric acid solution and sodium waterglass as hardeners, respectively. The liquid/solid mass

ratio for both specimens was kept constant at 0.80. The fresh geopolymer cements were poured into cylindrical PE-containers (20 mm in diameter and 40 mm in height), which were closed in order to hinder the evaporation of water during the setting. The specimens were kept at room temperature for 24 h but GPS4 does not consolidate compared to GPWG. In order to make a strict comparison, the both geopolymer cements were cured at 60 °C in an oven for 24 h. After 24 h in an oven, the obtained geopolymer cements (GPS4 and GPWG) were demoulded and conserved at room temperature for 28 days before compressive measurement.

The comparison of the mechanical and microstructural properties of geopolymer cements from alkaline and acid reactive ingredients were evaluated by measuring the compressive strength. It was measured using a manually driven testing machine (ENERPAC P392, USA). Eleven samples were tested for each composition and the mean values for the closet ten were determined. After compressive strength measurement, the fragments of each specimen were collected. One part of these fragments was used to observe the morphological changes. The other parts were crushed and the obtained powders were used to measure X-ray diffractometry and infrared spectroscopy. XRD patterns were taken using CuK $\alpha$  radiation between 5 and 80° in 5 h in steps of 0.02° (Bruker D8). Infrared (IR) absorption spectra were recorded by the KBr method (200 mg KBr, 1 mg sample, Bruker Vertex 80v, 2 cm<sup>-1</sup>, 32 scans). Fragments from the mechanical testing, after gold coating and drying, were used for microstructure observations using a JEOL JSM-6390A scanning electron microscope (SEM) coupled to microanalysis by energy-dispersive X-ray analysis (EDX) with an acceleration voltage of 30.0 kV.

In order to understand the different between both hardeners on the depolymerization of metakaolin, the IR spectra absorption of hardeners PS4 and NSF were measured on a Bruker Vertex 80v Fourier transform spectrophotometer using the attenuated total reflection (ATR) method. The IR spectra were recorded over a range of 700–3800 cm<sup>-1</sup> at a resolution 2 cm<sup>-1</sup> with 32 scans.

## 3. Results and discussion

### 3.1. Characterization of starting materials and PS4, NSF

The XRD pattern of metakaolin shows the X-ray amorphous phases in the range between 2 $\theta$  = 14 and 30° centered around 22° ( $\theta$ ) with quartz, anatase, and hematite as crystalline impurities ([Fig. 1](#)). The presence of hematite in MK is confirmed by the red color of MK, whereas; the one of silica fume ([Fig. 2](#)) is completely amorphous owing to the presence of a broad hump structure with higher intensity between 2 $\theta$  = 14 and 34° centered around 22° ( $\theta$ ).

[Fig. 3](#) presents the IR spectrum of metakaolin (MK) and indicates the lowest absorption band at 1620 cm<sup>-1</sup> which are attributed to the O—H bending vibrations of water molecule bonds. The peak at 1076 cm<sup>-1</sup> was assigned to asymmetric Si—O—Al and Si—O—Si. The Si—O observed at 800, 685 and 466 cm<sup>-1</sup> indicate the presence of quartz and amorphous silica. The signal of quartz appears often at 515, 696 and 797 cm<sup>-1</sup> ([Tchakouté et al., 2015](#)). This slight difference could be related to the low quartz and high amorphous silica content in MK and the band at 515 cm<sup>-1</sup> is masked or superimposed by one of 546 cm<sup>-1</sup>. The absorption band at 546 cm<sup>-1</sup> could be related to the presence of Al<sup>VI</sup>—O—Si vibration modes. The infrared spectrum of silica fume is illustrated in [Fig. 4](#). This infrared spectrum shows the band at 806 and 1103 cm<sup>-1</sup> which are assigned to asymmetric stretching of the SiO<sub>4</sub>

**Table 1**  
Chemical composition (wt.%) of kaolin.

Oxide	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	TiO <sub>2</sub>	MgO	Na <sub>2</sub> O	CaO	P <sub>2</sub> O <sub>5</sub>	MnO	LOI
Kaolin	41.46	31.47	7.65	0.51	1.50	0.65	0.69	0.15	0.09	0.06	15.76

LOI: Loss on ignition at 1000 °C.

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