



Partial replacement of volcanic ash by bauxite and calcined oyster shell in the synthesis of volcanic ash-based geopolymers



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HIGHLIGHTS

- Bauxite and calcined oyster shell were used as partial replacements of volcanic ash.
- Replacements were weakly dissolved in alkaline medium.
- Low percentage of bauxite reduced efflorescence.
- Low percentage of calcined oyster shell lessened setting time.

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ABSTRACT

Volcanic ash is an aluminosilicate material with generally variable chemical and mineralogical compositions from one deposit to another. When combined with suitable alkaline solution, its powder can be converted into geopolymer whose characteristics such as setting time and compressive strength are moderate. The present study investigates the effects of bauxite and calcined oyster shell powders as partial replacement for volcanic ash in order to enhance certain characteristics of resulted geopolymers. Depending on the nature of the concerned materials (volcanic ash, replacements and geopolymers), chemical and mineralogical analyses, Fourier transform infrared spectroscopy, X-ray diffractometry, visual aspect, durability, apparent porosity, initial setting time, linear shrinkage and 28 days compressive strength were performed. The replacements were weakly dissolved in alkaline medium due to their high crystalline phase contents. However, replacement of calcined oyster shell or up to 20% mass of bauxite lessened the setting time. For a given age of geopolymers, any quantity of replacement lowered linear shrinkage and about 10% mass of bauxite was enough to reduce efflorescence. Immersion of specimens in 3% solution of sulfuric acid for 28 days led to mass loss of less than 2.5% with surfaces that seemed to remain structurally intact. There was weak increase of 28 days compressive strength for geopolymers with small quantity of replacement. Conversely, great quantity of replacement produced the decrease of 28 days compressive strength. Anyhow, in geopolymer synthesis of volcanic ash, partial replacement by bauxite is a promising way of reducing efflorescence while calcined oyster shell is recommended for reducing of setting time.

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1. Introduction

The term “geopolymer” was coined by Davidovits in the 1970s, originally referring to chemical reaction between metakaolin and alkaline medium to form inorganic aluminosilicate polymers [1,2]. These materials are a class of inorganic polymers formed by chemical reaction between alkaline solution and aluminosilicate powder. Geopolymer framework is the result of aluminosili-

cate gel whereby silicon and aluminium are tetrahedrally bonded through sharing oxygen atoms. The basic monomer unit carries a negative charge due to tetrahedral groups AlO_4 which are charge-balanced by alkali metal cations such as K^+ or Na^+ [3]. There is wide range of reactive aluminosilicate powders such as fly ash, metakaolin or certain industrial wastes available for the production of geopolymers. For industrial propose, metakaolin which is the most effective aluminosilicate to produce geopolymers is of weakly focus because of energy consumption required for calcination of kaolin. The use of aluminosilicates such as fly ash or slag to produce geopolymers has been widely convincing [4–10] and is of

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high ecological benefit and low cost. The search for other alternative low cost and easily available aluminosilicate materials such as volcanic ash to produce geopolymers is up-to-date and an increasing number of studies are based on them [11–18]. This raw material is widely available in countries with past or present volcanism [19]. Samples of volcanic ash from two deposits in Cameroon were recently utilized to produce geopolymers at ambient temperature [15]. Among them, geopolymer specimens from one deposit exhibited great efflorescence, high linear shrinkage, weak 28 days compressive strength along with long setting time, while the products of the other deposit showed good enough properties [15]. There are numerous studies reporting beneficial effects of adding Al-rich or Ca-rich compounds to aluminosilicates during the synthesis of geopolymers. Kamseu et al. [20] showed that addition of quartz or fine alumina to metakaolin enhances the thermal stability of resulted geopolymers for high-temperature applications. Bondar et al. [11] observed an increase of amount of reactive phase when utilizing a natural pozzolana as aluminosilicate. Conversely using an additive rich in alumina did not improve the characteristics of resulted geopolymers [11]. Huang and Han [21] investigated the effect of α -Al₂O₃ during the geopolymerisation of fly ash and reported that addition of an appropriate quantity (5% mass) increased both compressive strength and surface area of resulted products. Tchakouté et al. [14] reported improvement of 28 days compressive strength of volcanic ash-based geopolymers when amorphous alumina was used as additive. Kani et al. [22] showed that extent of efflorescence was efficiently reduced by addition of high-alumina cement during the synthesis of natural pozzolana-based geopolymer. Djobo et al. [18] reported that addition of metakaolin (up to 25% mass) to a poorly reactive volcanic scoria led to geopolymers with improved 28 days physical and mechanical properties. Elsewhere, a certain number of authors [10,23–26] showed that addition of Ca-rich compounds (CaCO₃, Ca(OH)₂ and CaO) improved characteristics such as setting time and mechanical strength of geopolymers. This is likely the result of possible formation of C-A-S-H or C-S-H gel and coexistence of C-A-S-H/N-A-S-H or C-S-H/N-A-S-H gels within the matrix of samples and the beneficial effects of all of these were the increase of physical and mechanical properties of obtained geopolymers.

Cameroon possesses large deposits of bauxite such as the one located at Minim-Martap (Adamawa Region) with rich content of gibbsite [27]. Also, oyster shell originating from local fishermen at Mouanko (Littoral Region, Cameroon) is a by-product mainly composed of calcite and up to now, it is weakly valorized. The aim of this work is to investigate the possible benefits of bauxite and calcined oyster shell as partial replacements for improvement of certain characteristics of volcanic ash-based geopolymers.

2. Materials and experimental methods

2.1. Materials

Volcanic ash denoted as Z_C was previously used as aluminosilicate raw material for the synthesis of geopolymers cured at ambient temperature [15]. Except MgO and CaO whose amounts (% mass) were greater than 6, the major oxides in Z_C were SiO₂, Al₂O₃ and Fe₂O₃ (Table 1) and its mineralogical composition was made up of anorthoclase, diopside, hematite, maghemite, muscovite and nepheline (Fig. 1a). Bauxite and oyster shell originated respectively from Minim Martap (Adamawa-Region, Cameroon) and Mouanko (Littoral-Region, Cameroon). For use purpose, small fragments of bauxite were dried at 105 °C for 24 h in an electrical oven (Heraeus, type VT 5042 EK), ground for 6 h in a planetary ball mill and then sieved to 80 μm. Fragments of Oyster shell were firstly calcined at 500 °C in a programmable electric furnace (Nabertherm, Mode LH 60/14) for 2 h at a heating rate of 5 °C/min in order to remove organic glaze, ground for 6 h in a planetary ball mill and then sieved to 80 μm. In bauxite, the major oxides were Al₂O₃ (% mass of 58.10), Fe₂O₃ (% mass of 5.54) and TiO₂ (% mass of 2.40) and gibbsite was the main mineral associated with minor ones such as boehmite, goethite, kaolinite, corundum, maghemite and anatase (Table 1 and Fig. 1b). The chemical composition of oyster shell

Table 1
Chemical composition of the raw materials (% mass).

Oxides	Z _C	CH	BX
SiO ₂	41.36	0.30	1
Al ₂ O ₃	15.41	0.19	58.10
Fe ₂ O ₃	12.88	0.10	5.54
TiO ₂	3.04	–	2.40
MnO	0.2	–	–
MgO	6.45	–	–
CaO	7.88	74.73	0.08
K ₂ O	0.90	–	–
Na ₂ O	2.22	0.57	–
SO ₃	–	0.11	0.07
P ₂ O ₅	0.48	–	0.17
Cr ₂ O ₃	0.03	–	0.06
ZrO ₂	–	–	0.08
Cl ⁻	–	0.12	0.14
SrO	–	0.32	0.05
ZnO	–	0.07	0.03
As ₂ O ₃	–	0.27	0.30
V ₂ O ₅	–	–	0.08
LOI	9.31	23.23	31.67
Total	100.16	100.01	98.77

consisted of CaO as the main oxide (Table 1) and calcite was the identifiable crystalline phase (Fig. 1c) which may be consistent with its great LOI (% mass of 23.23). Bauxite and calcined oyster shell powders were respectively denoted as BX and CH.

Mixture of sodium silicate (bulk density \approx 1365 kg/m³) with composition (% mass) of 28.7 (SiO₂), 8.9 (Na₂O), 62.4 (H₂O) and solution of sodium hydroxide (12 M) was used as alkaline solution. The molar ratio SiO₂/Na₂O of the alkaline solution was 1.45 and sodium hydroxide solution was obtained by dissolving soda pellets of 99% purity in distilled water. Before to be used, the hot alkaline liquid was stored for at least 24 h at ambient temperature of the laboratory.

2.2. Geopolymer paste samples

To get geopolymer pastes, powder of volcanic ash and replacements were mixed in a Hobart mixer (M & O model N50-G) for 3 min and other 3 min were used to add alkaline liquid in the mixer. In order to get good workability, the liquid to solid mass ratio (L/S) was maintained at 0.40. Pastes were prepared by replacement of 0, 10, 20 and 30% mass of volcanic ash by BX or CH and both the composition and designation of resulted mixes were given in Table 2. For determination of 28 days compressive strength or linear shrinkage, fresh geopolymer pastes were molded in cylindrical PVC molds (diameter 31 mm, height 62 mm). Once molded, they were vibrated for 5 min on an electrical vibrating table (M & O, type 202, N° 106) in order to remove entrapped air bubbles and then covered with thin film of polyethylene for 24 h at ambient atmosphere of the laboratory (24 \pm 5 °C) to avoid water evaporation before demolding.

2.3. Experimental methods

Chemical analysis of bauxite and calcined oyster shell were carried out by ICP-AES via a Perkin Elmer- Optima 7000DV device and crystalline phases determination of BX, CH and geopolymers (cured at ambient atmosphere of the laboratory (24 \pm 5 °C) and aged 28 days) was obtained thank to a Bruker D 4 advance diffractometer, operating on the CuK α radiation between 5 and 70° (2 θ). Fourier transform infra-red spectroscopy (FTIR) was carried out on powders of Z_C, BX, CH and geopolymers (cured at ambient atmosphere of the laboratory (24 \pm 5 °C) and aged 28 days) using a Bruker Vertex 80v device operating in absorbance mode (4000–400 cm⁻¹). Initial setting time was determined on fresh geopolymer pastes using Vicat apparatus according to the EN 196-3 standard [28]. Linear shrinkage was measured on cylindrical geopolymer samples at 7, 14, 21 and 28th days according to ASTM C 157 standard [29]. Test on durability consisted of determination of mass change of specimens which were initially cured at ambient atmosphere of the laboratory, aged 28 days and immersed for 28 other days in a 3% solution of sulfuric acid (referred to ASTM C 267 standard [30]) and 28 days compressive strength was determined on geopolymer cylinder samples cured at ambient atmosphere of the laboratory (24 \pm 5 °C) using an electro-hydraulic press (M & O, type 11.50, N° 21) which operated at an average rate of 3 mm/min until the sample failed. Extent of efflorescence on geopolymers consisted of observing visually conical samples aged 90 days which were exposed at ambient atmosphere of the laboratory (24 \pm 5 °C). Determination of apparent porosity was done on cylindrical samples cured at ambient atmosphere of the laboratory (24 \pm 5 °C) and aged 28 days thank to NF P 18 – 554 standard [31].

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