



## Research paper

## Effect of slag and calcium carbonate addition on the development of geopolymer from indurated laterite



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## ABSTRACT

This paper reports the development of inorganic polymers from laterite, for a potential use at least as non-load bearing building materials. The effect of blending laterite with Ground Granulated Blast Furnace Slag (GGBS) and calcite ( $\text{CaCO}_3$ ) was investigated. Laterite was substituted by calcite at 2 to 20% in mass and by GGBS at 5 to 50% in mass. X-ray diffraction, Fourier Transform Infrared spectroscopy and differential thermal analysis evidenced the presence of kaolinite, hematite, and quartz minerals in the laterite. Sodium silicate solutions of modulus ranging from 1.6 to 2.2 were used to prepare inorganic polymers from laterite that had been thermally treated at 700 °C. The strength increased with the reduction of the modulus of the activating solution, achieving a 28 days maximum compressive strength of 36 MPa at 25 °C. The substitution of laterite by calcite was found to have little effect on the compressive strength meanwhile the substitution of laterite by slag was beneficial for strength development, mainly from 20% substitution, achieving a 28 days compressive strength of 65 MPa at 50% slag. Mercury Intrusion Porosity and Scanning Electron Microscopy analysis evidenced a better refinement in microstructure with slag addition in the system. The results of compressive strength were found to be in agreement with ASTM C216–17 standard for building bricks, suggesting a potential interest for the valorization of laterite and blended laterite/slag in the development of at least non-load bearing building materials, using geopolymer technology.

## 1. Introduction

Lateritic soils are formed in the tropics through weathering processes that favor the formation of iron, aluminum and titanium oxides. These processes break down silicate minerals into clay minerals such as kaolinite and illite, giving rise to a variety of physical properties. Lateritic soils are widely distributed throughout the world in regions with high rainfall, but especially in the inter-tropical regions of Africa, Australia, India, South-East Asia and South America, where they generally occur just below the surface of grasslands or forest clearings (Kasthurba et al., 2007; Lemougna et al., 2014a; Maignien, 1966). The specific property of laterites to harden (indurate) on exposure to the atmosphere is due to the precipitation, concentration and crystallization of constituent sesquioxides ( $\text{Fe}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$ ), as a result of desiccation. The degree of induration varies ranging from almost loose coherent mass which can be broken by hand, to the most hardened blocks difficult to break with a hammer (Kasthurba et al., 2007;

Maignien, 1966). Many laterites were reported to contain significant amount of residual kaolinite (Billong et al., 2009; Goure-Doubi et al., 2014; Gualtieri et al., 2015; Obonyo et al., 2014). However, despite the fact that laterites have been found to be a potential source of aluminosilicates for inorganic polymers (geopolymers) synthesis, very little has been reported on the development of geopolymers from laterites (Davidovits, 2013; Gualtieri et al., 2015; Kaze et al., 2017; Obonyo et al., 2014), in comparison to other natural minerals like kaolinite or substitute raw materials like fly ashes from coal combustion. Additionally, the composition of laterites can vary widely (Gualtieri et al., 2015; Maignien, 1966), hence, different behavior can be expected if used as raw material for geopolymers synthesis. Geopolymers are a class of inorganic materials which have received a burgeoning interest during the last decades due to their excellent physical, mechanical and thermal properties (Cui et al., 2008; Davidovits, 2008; He et al., 2016; Tang et al., 2015; van Deventer et al., 2012; Wang et al., 2015; Yan et al., 2016). Another important advantage associated with the

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development of geopolymer materials for building applications is the relatively low CO<sub>2</sub> footprint in comparison to Ordinary Portland Cement (OPC). Actually, the production of one ton of OPC or OPC blends releases about 0.6 to 1 ton of CO<sub>2</sub>, mainly related to the decarbonation of limestone involved in the production of clinker and its high production temperature of about 1400–1500 °C (Kavitha et al., 2016; van Deventer et al., 2012; Wianglor et al., 2017). According to the 2012 report on environmental outlook to 2050 from the Organization for Economic Co-operation and Development (OECD), the global production of OPC contributed for 5–8% of global anthropogenic CO<sub>2</sub> in 2010 (Kajaste and Hurme, 2016). In order to reduce these emissions, some investigations have also been conducted to assess the pozzolanic activity of some laterites and similar low grade kaolinic clays for their use as supplementary cementitious materials (Alujas et al., 2015; Ambroise et al., 1985; Pera et al., 1998).

Beside laterite, Ground Granulated Blast Furnace Slag (GGBS) is an industrial by-product material generated from the manufacturing of pig iron. It is well known for its high long-term strength and resistance to deterioration under severe environmental conditions as well as its environmental advantage due to lower CO<sub>2</sub> emissions and energy consumption in the development of cementitious materials (Puertas and Fernandez-Jimenez, 2003; van Deventer et al., 2012; Shin et al., 2016). In fact, GGBS can be used as supplementary cementitious material for OPC or raw material for geopolymer synthesis and, optimally formulated GGBS based geopolymer was suggested to release 80% less CO<sub>2</sub> than OPC (van Deventer et al., 2012).

A synergetic use of two starting natural materials or two aluminosilicate sources has often been applied to optimize the properties of geopolymers (Borges et al., 2016; Habert et al., 2011; Yip et al., 2008). The addition of a moderate amount of calcium carbonate to metakaolin based geopolymer was found to be able to affect the structure and properties (Yip et al., 2008). An appropriate addition of slag, which is also a calcium containing material, to metakaolin or fly ash geopolymer, was also often observed to lead to products with optimum properties (Borges et al., 2016; Habert et al., 2011). From a chemical point of view, the mixing of a poor Ca aluminosilicate precursor such as laterite with a rich Ca aluminosilicate precursor such as slag in geopolymer synthesis may also lead to an appropriate mixture of sodium (alumino) silicate hydrate (N-A-S-(H)) gel and calcium (alumino) silicate hydrate (C-A-S-(H)) gel, which is beneficial for the mechanical performance (Provis and Bernal, 2014).

The present study investigated the development of inorganic polymers from indurated laterite, for a potential use at least as non-load bearing building materials. Low amount of activating solution was used as this can reduce the potential production cost and minimize the environmental concern of the process (Habert et al., 2011; Lemougna et al., 2016). The effect of calcite (2–20%) and slag (5–50%) addition on the final properties was investigated. The products were characterized by X-ray diffraction, Infrared Spectroscopy, Thermogravimetry analysis, Mercury Intrusion Porosity and Scanning Electron Microscopy. The wet and dry compressive strength were performed to assess the suitability of the products for building applications.

## 2. Experimental

### 2.1. Materials

The laterite used in this study was from Olembe, a locality situated in the north of Yaounde, in Cameroon. The sampled laterite was in the form of indurated nodules of about 2–15 mm difficult to break by hand, but easy to break with a hammer. The material was ball milled and calcined at 700 °C for 2 h. The ground granulated blast-furnace slag used in this study was provided by the Chengde Group Company, Beihai Guangxi, P.R. China. The oxide composition of the laterite and slag determined by X-ray fluorescence (XRF, PANalytic Axios, using the fusing method) is reported in Table 1. The particle size distribution of

the laterite, slag and calcium carbonate, determined with an OMEC POP 6 Laser particle size analyzer is presented in Fig. 1. Calcium carbonate was of AR laboratory type, 99% CaCO<sub>3</sub>.

The alkaline solutions with silica modulus R (R = molar ratio of SiO<sub>2</sub>/Na<sub>2</sub>O) of 1.6 to 2.2, with 0.2 interval, were prepared by dissolving appropriate amount of solid sodium hydroxide in a commercial liquid sodium water glass with R = 3.3. The alkaline solutions were sealed and stored for a minimum of 24 h prior to use.

### 2.2. Specimens preparation

The preparation of the fresh mixture was performed by mixing calcined laterite and sodium silicate with different moduli (R = 1.6; 1.8; 2.0 and 2.2), with R representing the molar ratio of SiO<sub>2</sub>/Na<sub>2</sub>O of the sodium silicate solution. This has led to a Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub> molar ratio of 0.54 to 0.70. These ratios were kept low in order to minimize the use of alkaline reagent. The details on the mix proportioning are presented in Table 2. The addition of 2 to 10% with 2% interval and 20% of calcite on one hand, and 5 to 25% with 5% interval and 50% of GGBS on the other hand, was also investigated. The mixing process was performed for about 10 min, using an electrical mixer at 700 rpm. The homogeneous pastes were then casted in cubic metallic molds of 20 × 20 × 20 mm<sup>3</sup>, covered with a thin layer of plastic to facilitate the removal of the hardened pastes upon curing. The molds were vibrated on a vibration table for 2 min to remove air bubbles and sealed afterwards. The specimens were cured at 25 °C for 28 days prior to characterization.

### 2.3. Characterization method

#### 2.3.1. XRD and FTIR analyses

The samples were powdered, front-loaded in standard sample holder and examined by X-ray diffraction with a Rigaku Mini Flex 600 instrument with a monochromator detector and Ni-filtered Cu (Kα) radiation, step size of 0.02°, operated at 40 kV and 15 mA, with a dwell time of 0.5 s and a 2θ range of 5 to 70°; Divergence slit (DS): 1.250°; Anti-scatter slit (SS): 1.250°; Receiving slit (RS): 0.3 mm.

The powdered samples were also pressed into KBr pellets for FTIR analysis using a Thermo Scientific NICOLET 6700 FT-IR spectrometer. For each spectrum, 32 scans with a resolution of 2 cm<sup>-1</sup> were used in absorbance mode, scan range 1600–400 cm<sup>-1</sup>, on pellets made with 1 mg of sample and 100 mg KBr.

#### 2.3.2. SEM/EDS analysis

Scanning electron microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS) were performed to analyze the polished surfaces of the specimens with an S-3400N device (Japan Hitachi Limited Company), with an accelerating voltage of 15 kV. Specimens were impregnated using absolute ethyl alcohol, polished with SiC paper, and then coated with gold.

#### 2.3.3. TG/DTA analysis

TG/DTA analysis was performed with a simultaneous NETZSCH STA 449F3 TG/DTA measurement under nitrogen atmosphere, at a constant heating rate of 5 °C/min. The sample was heated from room temperature to 1000 °C.

#### 2.3.4. Compressive strength and mercury intrusion porosity test

Compressive strength testing was performed on specimens using a DNS100 universal testing machine. The displacement rate used was 0.5 mm/min. The test was performed on specimens cured for 28 days. The values were determined as the average of three samples of each composition. For the wet compressive strength, the specimens were immersed for 48 h in deionized water prior to testing. The standard deviation of the replicate specimens was used as the error bar in Figures. Mercury Intrusion Porosity test was performed using a Pore

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