



Research paper

The corrosion of kaolinite by iron minerals and the effects on geopolymerization



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ABSTRACT

Iron-rich aluminosilicates with disordered structure (laterites) due to the corrosion of kaolinite by iron minerals were investigated as solid precursors for geopolymerization. The particle size distribution, B.E.T surface area, thermal activation, and chemical and mineralogical compositions were used to evaluate the reactivity of iron-rich laterites (35 wt.% of Fe₂O₃-FeO). The raw materials in the temperature range between 25 and 500 °C showed geopolymerization behaviour similar to that of metakaolin. At temperatures higher than 500 °C, the coarsening of particles and the decrease of B.E.T surface area correspond to an initial sintering of laterites explaining the poor polycondensation/geopolymerization and the decrease of strength of the final products. The increase of the temperature of calcination of raw laterites between 25 and 500 °C corresponds to a reduction of the setting time of geopolymer products. However, this variation of temperature did not significantly affect the flexural strength that remained between ~4 and ~6 MPa, confirming the possibility to produce sustainable matrices, with more energy saving, using highly corroded laterites.

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1. Introduction

Laterites and lateritic soils, described as Fe₂O₃(FeO)-Al₂O₃-SiO₂-H₂O matrices, are made from kaolinite in which a high proportion of Al³⁺ is replaced by Fe²⁺ or Fe³⁺ (Lyon Associates, Inc., 1971, Obonyo et al., 2014). This substitution induces important transformation in the structure of kaolinite with consequent reduction of the crystallinity and increase of the crystal layer. The disordered structure and the increase of the amorphous phase that results, enhance the vulnerability to chemical attack (Nahon, 1976; Trolard and Tardy, 1989; Kasthurba et al., 2008; Ngon Ngon et al., 2009; Pignatelli et al., 2014; Obonyo et al., 2014). In the tropical areas of the world, soils with kaolinite in the presence of iron minerals are progressively corroded to form hardened matrices generally called indurated matrices (Nahon, 1976; Trolard and Tardy, 1989; Kasthurba et al., 2008).

The induration of laterite is due to the development of sesquioxides (Fe₂O₃ and Al₂O₃) that precipitate, concentrate and crystallize under the fluctuation of humidity and temperature (Lecomte-nana et al., 2009; Obonyo et al., 2014). For Maignien (1958), indurated lateritic formations are typified by the predominance of hydrous alumina (gibbsite and boehmite), and the sesquioxides of iron (goethite and hematite) mixed in variable proportions with disordered or less crystallized kaolinites. They are generally contaminated to some extent by residual silica mixed with amorphous and quartz. The precipitation of ferrihydrite from Fe³⁺ solutions has been shown to be accelerated by interfaces such as those of silica or kaolinite particles. In the presence of kaolinite, the release of Al³⁺ species into solution and the precipitation of Fe³⁺ hydroxide were observed to be two simultaneous processes. The increase in iron accumulation led firstly to the production of nodule nuclides that progressively grow with the iron content and, finally, laterite concrete is formed with amorphous silica which acts as binder. Three factors can affect the induration of laterites according to Maignien (1958):

- (i) The composition and the extent of crystallization of the components: the higher the sesquioxide content, the greater the induration; hardness increases as iron content increases; the hardest laterites are also the least hydrated.

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Table 1
Chemical composition of raw materials.

Oxides (wt.%)	Laterite from Odza (LATOD)	Laterite from Eloumden (LATEL)
Fe ₂ O ₃	37.5	36.9
SiO ₂	25.1	27.7
Al ₂ O ₃	23.0	22.4
TiO ₂	0.908	0.733
V ₂ O ₅	0.150	0.120
P ₂ O ₅	0.148	0.170
Cr ₂ O ₃	0.227	/
LOI	12.7	11.6

- (ii) The arrangement of the various constituents: compact crusts are more indurated than loose crusts; homogeneous materials are harder than those whose components are segregated.
- (iii) The extent of ageing: older occurrences are frequently harder than recent occurrences of the same type of laterite.

The corrosion of the kaolinite and formation of lateritic concretes make the matrix of laterite appears into XRD patterns with similar disorder as that of metakaolin due to the important content of amorphous materials (Obonyo et al., 2014; Kasthurba et al., 2007). Previous works on lateritic soils for structural application have showed that, physical and mechanical properties can be improved through chemical activation which is affected by the mineralogical compositions, the degree of corrosion of raw materials as well as the curing conditions as humidity and temperature (Billong et al., 2009; Kamseu et al., 2013; Obonyo et al., 2014; Lemougna et al., 2014; Lassinantti Gualtieri et al., 2015; Nik Ab Aziz et al., 2015). These materials are available in tropical areas of the world, with nearly 67% found in the Cameroonian territory (Mbumbia et al., 2000) and are of great interest for the design of sustainable building materials through the chemical activation. Obonyo et al. (2014) showed that fraction of calcined laterites can be used for the nucleation of geopolymer gels that progress and extend the polycondensation to the major part of the matrix. The microstructure of the products shows compactness with fracture surfaces evidencing the cross linking between phases and particles with similar texture to that of high compact metakaolin geopolymer composites. The products of the geopolymerization of laterites showed stability in water already after 14 days and good stability after 90 days thus indicating the effective transformation and induration of the matrices.

In this project, we are investigating the optimum conditions in which fully indurated/corroded laterites can appear as alternatives to metakaolin with significant reduction of the temperature of calcination. This will make the geopolymer from laterites more green and sustainable to act as alternative for the reduction of the CO₂ emission that the cement industry is actually responsible (5–8% of worldwide CO₂ emission according to Gartner, 2004). Alkaline cements are an interesting alternative to Portland cement as they present similar or even better binding properties, low cost, and high ecological benefits. Most of the work reported in the literature has been based on the study of metakaolin, fly ash, and slag activation (Davidovits, 1991; Malhotra and Metha, 2005; Malhotra, 2010). Metakaolin is essentially an anhydrous aluminosilicate produced by the thermal decomposition of kaolin at temperatures above 550–650 °C (Kamseu et al., 2011). However, in the tropical areas it is easier to have laterites, generally at the surface, than struggle for clays for which exploitation will be detrimental to the environment due to their coverage in most cases by laterites or various types of soils (Trolard and Tardy, 1989). Since laterites should be locally sourced with negligible transport costs and environmental impact, having thermal efficiency, financial viability and low energy required in the geopolymerization process (Duxson et al., 2007), the products of the geopolymerization are classified as green, environmentally-friendly and sustainable. These requirements can be easily fulfilled with the

manufacturing of geopolymers based on laterites rather than metakaolin.

The present study investigates the feasibility of using untreated iron-rich laterite with kaolinite almost amorphous (highly corroded) as precursor materials for the synthesis of geopolymers via alkaline activation, and understanding the extent of calcination into the physico-chemical properties of the resulted binders. The raw laterites were submitted to several analyses such as surface area, ultrasound and particle size distribution, Differential Scanning Calorimetry simultaneously with the thermogravimetric (DSC/TG) analysis, X-ray diffraction (XRD) analysis and X-ray Fluorescence (XRF) analysis to validate their suitability as solid precursors for geopolymers. The resulted products of geopolymerization were characterized by determination of setting time, Fourier Transformed Infrared Spectroscopy (FTIR) analysis and Environmental Scanning Electron Microscopy (ESEM). Mechanical properties were assessed by the measurement of flexural strength and water absorption. The characteristics of laterite based geopolymers are interpreted correlating the microstructure and strength to the initial properties of raw materials.

2. Materials and experimental methods

2.1. Materials

Two iron-rich aluminosilicates (laterites) were collected from the localities of Odza and Eloumden, Central region, Cameroon. Both materials were dried in an oven at 105 °C for 24 h to remove moisture water. They were dry ground with a MMS (machine maccina smalto, MMS, Italy) for 6 h to obtain fine powders that were sieved through a 90 µm micron mesh prior to be used for analysis and characterization in cement paste preparation. Additionally, the raw materials were calcined at 500, 600, and 700 °C for 4 h at a heating rate of 5 °C/min in a programmable electric furnace. The choice of these two laterites was based on their level of concretisation (formation of aggregates) with consequent increase of structural disorder that means vulnerability in alkaline solution, as indicated in the Introduction (Obonyo et al., 2014).

Alkaline solution used as activator was prepared by mixing an 8 M solution of sodium hydroxide (NaOH, 99.9%, Sigma-Aldrich) with sodium silicate solution (SiO₂/Na₂O = 3.1, L.O.I = 60 wt.%). The sodium silicate solution was provided by Ingessil, Italy.

2.2. Characterization of starting materials

Table 1 reports the chemical compositions of each raw material as obtained by X-ray Fluorescence analysis (Thermo Fisher Scientific). The analysis and characterizations done on raw materials were carried out into the specimens ground immediately before the experimentation. The specific surface area of unheated and calcined laterites was determined according to the Brunauer–Emmet–Teller (B.E.T) method based on nitrogen absorption (on a Micromeritics GEMINI 2360 instrument). The particle size distribution (cumulative and density distribution) was determined with Malvern instruments Ltd. (Mastersizer 2000 Ver. 5.22). For the granulometry analysis, the experimentation was performed under ultrasonic condition to compare with those done without ultrasound. The objective was to investigate on the ability of iron-rich particles of both laterites to flocculate instantaneously.

Differential thermal analysis and simultaneous thermogravimetric analysis (DSC/TG) was performed on the two samples, using a Netzsch STA 429 CD unit in air atmosphere. The analysis was carried out on ground powders at a heating rate of 20 °C/min in platinum-rhodium crucibles using calcined alumina as reference.

Mineralogical analysis of the laterites was carried out with an X-ray powder diffractometer, XRD, (PW3710, Phillips) Cu K α , Ni-filtered radiation (the wavelength was 1.54184 Å). The radiation was generated at 40 mA and 40 kV. The analysis was performed on fine grains of ground

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