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Synthesis and characterization of geopolymer binders based on local materials from Burkina Faso – Metakaolin and rice husk ash

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HIGHLIGHTS

- Characterization of geopolymers based on materials local from Burkina Faso.
- Local calcined clay and rice husk ash were used.
- Improving the mechanical properties of geopolymers by adding 5% of rice husk ash.
- Rice husk ash and the temperature improved the polymerization.

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ABSTRACT

Geopolymer binders constitute an environmental friendly alternative of Portland cement, wellknown for its contribution to the emission of greenhouse gases. In this article, we study the possibility of valorizing local materials in Burkina Faso, namely metakaolin and rice husk ash, in geopolymer binders synthesis using sodium hydroxide solution.

The study focused on the influence of the addition of rice husk ash and the curing temperature on the mineralogical, microstructural, physical and mechanical properties of geopolymer binders, through X-ray diffraction, Fourier transform infrared spectroscopy, scanning electron microscopy, weight loss, apparent density, porosity accessible to water and compressive strength. Three types of geopolymer binders were synthesized in this study: geopolymer binders based on metakaolin alone (AN) and two other binders containing 5% (BN) and 10% (CN) of rice husk ash respectively.

The results obtained show the formation of new mineral phases and an improvement of the mechanical strength with the addition of rice husk ash and with curing temperature. There is also a high porosity for all synthesized geopolymer binders. These results are presented and discussed in terms of SiO_2/Al_2O_3 ratio and degree of polymerization.

1. Introduction

In Burkina Faso, cement is the most commonly used binder in modern construction although it is not the most suited in the context of hot tropical climate. The importation of the raw material (clinker) necessary for its production makes it very expensive and inaccessible to the majority of the population. To that problem of cost must be added environmental problems caused by the production (typically, emission of one ton of CO_2 for one ton of cement produced [1]). However, Burkina Faso, like other countries in the sub-region of West Africa has enormous resources in the form of clay materials (kaolinite) which are untapped except for a few artisanal applications (pottery, ceramics) and road construction. One way of giving economic value while respecting the environment, to this clay potential could be the synthesis of geopolymer binders based on clays for the stabilization of earth bricks. Geopolymers emerged in the 1970s [1] and can be synthesized at slightly elevated temperatures using the alkaline activation of aluminosilicates obtained from clays: calcined clays, natural minerals, industrial wastes or a mixture of two or more of these materials [2]. Geopolymers, as new binders, offer a more environmentally friendly alternative to cementitious materials and so are becoming increasingly used in the field of Civil Engineering [3]. The stabilization

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of earth bricks by this type of binder will also provide a solution to the major problem of instability of these bricks in rainy periods, while reducing the environmental pollution caused by the cement industry and lessening housing problems.

Specifically, the synthesis of a geopolymer generally involves materials containing amorphous silica (SiO_2) and alumina (Al_2O_3), alkali hydroxide and alkali silicate. The use of alkali silicate in addition to the alkali hydroxide is known to accelerate the activation of aluminosilicates and improve properties [1] but makes the material more expensive than cementitious ones. Temperature is also a reaction accelerator in geopolymerization and significantly affects mechanical strength. However, treatment at too high a temperature has been shown to lead to cracks and to have a negative effect on the chemical and mechanical properties of the material [4]. Given the cost of alkali silicate and energy (necessary to produce high temperature), as well as the negative impact of high temperature, it would be advantageous to have access to a local source of amorphous silica that could replace alkali silicate in the synthesis of geopolymer, followed by heat treatment at moderate temperatures.

Usually, a large number of minerals, agricultural by-products and industrial by-products are used as raw materials for geopolymer synthesis: natural aluminosilicates (kaolin) [5], metakaolin [1,6–8], and fly ash or silica fume [9,10], to mention but a few. In our study, two aluminosilicates from Burkina Faso were used for such synthesis. Metakaolin, which is a calcined clay from Burkina Faso, and rice husk ash, which is generated mainly by gasification of rice husk. Rice husk is an agricultural by-product comprising 40% cellulose, 30% lignin and 20% silica [11,12] and its ash is composed of more than 90% silica, most of which is amorphous although a minor amount is poorly crystallized. The amorphous content depends on the temperature and the duration of the calcination process [13]. Rice husk ash is used for three reasons. Firstly, as an economical alternative to alkali silicate due to its high amorphous silica content; secondly, to improve the physico-mechanical properties of geopolymer as demonstrated in [14]; and finally, to valorize an agricultural by-product from Burkina Faso. Based on the studies by Görhan et al. [9], a sodium hydroxide solution of 12 M was appropriate to activate our aluminosilicates.

The objective of this work was the synthesis of geopolymer binders based on local materials from Burkina Faso at moderate temperature. More specifically, our aim was to show the influence of partial substitution of metakaolin by rice husk ash (in order to increase the SiO_2/Al_2O_3 molar ratio) and the role of curing temperature on the properties of geopolymer binders. The characteristics of the synthesized products were determined by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR spectroscopy), scanning electron microscopy (SEM), weight loss, apparent density, porosity accessible to water and compressive strength. Some results from these parameters are presented and discussed in terms of the SiO_2/Al_2O_3 ratio and the degree of polymerization.

2. Materials and experimental methods

2.1. Materials

The metakaolin (MK) used in this work is the result of a heat treatment of kaolin (K) sampled in a kaolin quarry in Burkina Faso (Saaba – 12°22′46″ N; 1°24′38″ W and 317 m altitude). Kaolin (K) was calcined for 3 h at 700 °C at a heating rate of 10 °C/min [15,16]. The rice husk ash (RHA) was obtained by 3 hours' mineralization at 550 °C of carbon residue resulting from rice husk gasification. The rice husk was sampled from rice grown in Burkina Faso (Bagre – 11°28′26″ N, 00°32′07″ W and 233 m altitude). The heat treatments were carried out using a muffle furnace (HERAEUS MR 260 E) and powders were then crushed by means of a RETSCH-MS 100 crusher and sieved at 100 μm. Sodium hydroxide (12 M) was used as the alkaline solution, obtained by dissolving NaOH crystals in distilled water. The granular NaOH had a purity of 99%.

2.2. Experimental methods

2.2.1. Sample preparation

The aluminosilicate powders were homogenized using a mixer for 5 min. Then, the geopolymer paste was obtained by mixing alkaline solution with aluminosilicate powders in a weight ratio (alkaline solution/powder) of 0.8. This weight ratio allowed good workability of the paste. The powder-solution mixture was carefully homogenized a second time using an electric mixer (HOBART) for 10 min. The paste (Fig. 1) obtained was used to make prismatic specimens ($4 * 4 * 16\ cm^3$). The weight ratios of Na_2O/binder and water/(binder + Na_2O) were 0.2 and 0.5 respectively. Neither alkali silicate nor additional water was used during the preparation of the paste. The water quantity considered in the evaluation of the water/(binder + Na_2O) ratio was that used upstream for the preparation of the alkaline solution.

Casting was implemented with the Controlab Perrier shock table (2 times 60 shots) to expel the air trapped by particles of the materials. Thus, three formulations of geopolymer samples were synthesized:

- AN: aluminosilicate powder (metakaolin) + NaOH
- BN: aluminosilicate powders (95% metakaolin and 5% rice husk ash) + NaOH
- CN: aluminosilicate powders (90% metakaolin and 10% rice husk ash) + NaOH

The molds were covered with a polyethylene film in order to prevent water evaporation during the paste setting and hardening (Fig 2a). They were left at the ambient temperature of the laboratory, estimated at 30 °C, for 7 days before being unmolded. They then underwent a heat treatment (60 and 90 °C) for 7 days [17] using a laboratory oven with natural convection (Memmert 15U). After the 14 days of curing, the geopolymer samples (Fig 2b) were subjected to several characterization tests including microstructural and chemical characterization and physical and mechanical properties.

2.2.2. Microstructural and chemical characterization

A mineralogical study was made by X-ray diffraction (XRD) using a Siemens D5000 diffractometer, in Bragg-Brentano configuration with cobalt radiation (Co Kα, λ = 1.789 Å). The anode voltage

Fig. 1. Geopolymer paste.