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Synthesis and characterization of low temperature (<800 °C) ceramics from red mud geopolymer precursor

Patrick N. Lemougna [a,b,*], Kai-tuo Wang [a], Qing Tang [a], Xue-min Cui [a,*]

[a] School of Chemistry and Chemical Engineering and Guangxi Key Lab of Petrochemical Resource Processing and Process Intensification Technology, Guangxi University, Nanning 530004, China

[b] Local Materials Promotion Authority, MINRESI/MIPROMALO, P.O. Box 2396, Yaounde, Cameroon

HIGHLIGHTS

- Compressive strength of up to 40 MPa was obtained from red mud geopolymer.
- Curing at 60 °C for weeks was essential for better strength development.
- Post heating to 700 °C increased the strength to 55 MPa and the mechanical stability.
- The use of sodium silicate instead of NaOH was essential for the material stability.

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ABSTRACT

This paper presents a way to valorize red mud for the production of potential structural materials, using geopolymer technology. Several compositions of red mud geopolymers were prepared with sodium silicate solutions. After sintering the red mud geopolymer products at 300–800 °C, their stability in water has been improved. The starting red mud was found to contain hematite, katoite, cancrinite and a few amount of diaspore, which hardly dissolved and participated in the geopolymerization reaction. However, the geopolymer gel formed was sufficient to bind the unreacted phases and form a high strength material with about 40 MPa after appropriate curing.

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1. Introduction

Red mud (RM) or bauxite residue is the major waste produced by the alumina refining industry where the Bayer process is used to extract alumina from bauxite ores [1–3]. The process involves the use of highly concentrated NaOH solution for the ore digestion at temperatures up to 240 °C and 1–6 atm pressure [3,4]. The production of 1 ton of aluminum metal produces as a by-product 4–5 tons of red mud [4]. Fresh RM slurry is usually transported to waste lakes for impoundments, followed by dewatering and drying to reduce its volume and maintenance costs [3]. The exact composition of the mud depends on the origin of bauxite and on processing conditions [5], but essentially consists of oxides and hydroxides of Fe, Al and Si along with some quantities of CaO, TiO_2, Na_2O, and is alkaline in nature [5,6]. The worldwide bauxite residue disposal areas contains an estimated 2.7 billion tons of residue, with an increase of approximately 120 million tons per annum, representing an environmental and economic problem [1–3,7]. To date, although enormous efforts have been made on RM treatment, recycling, and utilization [1,3,7–10], only a small fraction, most probably <5 wt% is being used in few countries in specific industrial processes such as cement production, the rest being stored [9]. An economical widely accepted technology for the recycle and reuse of RM has yet to be developed [2,3,9,10], hence the need of further researches to explore possibilities of valorizing red mud [1,10]. Among the utilization options, construction and building materials pose lower risk for implementation and the manufacture of geopolymers based on RM including controlled low strength materials were suggested as interesting area of research to be explored [1,2].

Geopolymers are a class of inorganic polymer materials which have attracted interest during the last decades due to their inter-

* Corresponding authors at: School of Chemistry and Chemical Engineering and Guangxi Key Lab of Petrochemical Resource Processing and Process Intensification Technology, Guangxi University, Nanning 530004, China.

E-mail addresses: lemougna@yahoo.fr (P.N. Lemougna), cui-xm@tsinghua.edu.cn (X.-m. Cui).

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esting physical, structural and thermal properties [11–15]. Most of previous reports on the use of red mud for the development of potential building materials using geopolymer synthesis have been limited to alkali-activated composites of red mud with other aluminosilicate materials [16–19], likely due to the difficulty of obtaining good structural properties from a pure RM geopolymer based system. Attempts to produce traditional ceramic materials showed a substantial inertia of RM up to 900 °C, resulting in a cost disadvantage need of high temperatures of 1000–1200 °C to obtain potential useful structural properties [7,8,20,21].

The present work investigates the possibility to produce inorganic polymers using red mud as the aluminosilicate precursor, and the effect of post heating of the products to produce low temperature ceramics. Several compositions of the red mud geopolymers were prepared by varying the modulus ($R = SiO_2/Na_2O$) of the activating solution from 1.6 to 2.2. The geopolymer samples were subjected to different curing regimes (sealed and humid) and the effect of post heating (200–800 °C) was assessed on the optimally formulated products. The starting red mud was also treated at some temperatures for comparative studies. The resulting products were characterized by X-ray diffraction, Scanning Electron Microscopy, Differential Scanning Calorimetry and Infrared spectroscopy. The wet and dry compressive strengths, the leaching test, water absorption and porosity were then performed to assess the suitability of the synthetized harmless products for potential structural applications.

2. Experimental

2.1. Materials

The red mud used in this study was from Guangxi Province, China. The specific surface area determined by the BET method was 8.04 m^2/g. The oxide composition determined by X-ray fluorescence is reported in Table 1.

The alkaline activating solutions with silica moduli ($R = SiO_2/Na_2O$) of 1.6–2.2 with 0.2 interval were prepared by dissolving solid sodium hydroxide in a commercial sodium water glass with R = 3.3. The alkaline activating 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 red mud, water glass with different moduli (R = 1.6; 1.8; 2.0 and 2.2), and some amount of deionized water. The details on the mix proportioning are presented in Table 2. The mixing process was performed for about 10 min, using an electric mixer at 600 rpm, up to obtaining a homogenous paste. The samples were then casted in cubic alloy molds of $20 \times 20 \times 20\ mm^3$, covered with a thin layer of plastic to facilitate the removal of the hardened paste upon curing. The alloy molds were vibrated on a vibration table for 2 min to remove air bubbles and sealed afterwards. The specimens were stored at 25 °C for 48 h, unmolded and kept for further 48 h sealed at 25 °C. After this period, the specimens were transferred sealed at 60 °C. Some of them were maintained sealed until the 7 and 28 days strength testing while others were progressively dried at 60 °C in the following manner: the first day, the samples were kept sealed; the second day, the plastic bag was open but the samples were maintained inside the bag and, the third day, the samples were removed from the plastic bag to the oven atmosphere. This low and long curing procedure was adopted to reduce the sensitivity of crack formation observed on the red mud. Part of the 7 days dried specimens was subjected to heat treatment from 200 to 800 °C at 100 °C interval, in an electric furnace, heating rate of 2 °C/min with a dwell time of 2 h at each temperature. The heating rate of 2 °C/min was adopted to reduce the sensitivity to form cracks, which was observed at higher heating rates, with negative effect on the mechanical properties. The temperature 60 °C was chosen for curing because relatively poor performances were observed during preliminary investigations on specimens cured at 25 and 40 °C. The post heating temperature range of 200–800 °C was chosen because it is below the usual sintering temperatures of traditional ceramics and below the temperature at which red mud without admixtures generally presents a substantial inertia [7,8]. On the basis of the phases identified by XRD, the pure red mud was also treated at 300 °C, 500 °C, 700 °C and 800 °C for X-ray comparative study.

2.3. Characterization method

2.3.1. XRD and FTIR analyses

The samples were powdered and examined by X-ray diffraction with a Rigaku Mini Flex 600 instrument with Ni-filtered Cu (Kα) radiation, a 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 5–80°. The powdered samples were also pressed into KBr pellets for FTIR analysis using a Thermo Scientific FTIR spectrometer.

2.3.2. TG/DTA analysis

TG/DTA analysis was performed with a simultaneous STA409PC TG/DTA measurement in air, at a constant heating rate of 5 °C/min. The sample was heated from room temperature to 1000 °C.

2.3.3. SEM/EDX analysis

Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX) were used to analyze the microstructure of the powdered red mud and the polished surfaces of the specimens with an S-3400N device (Japan Hitachi Limited Company). Specimens were impregnated using absolute ethyl alcohol, polished with SiC paper, and then coated with gold.

2.3.4. Compressive strength testing

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 7 and 28 days and for specimens post heated at 200–800 °C. The dry strength is for specimens after the curing or post heating treatment, and the wet strength is for these specimens immersed for 24 h in water. The values were determined as the average of three samples of each composition.

2.3.5. ICP analysis, water absorption and porosity

In the realization of the ICP analysis, the samples were soaked in deionized water for 96 h. The sample/water weight ratio was 1/3. The water was collected for analysis after 48 h, then renewed and collected again for analysis after an additional 48 h for some samples. This experiment was carried out for the samples treated

Table 1
Chemical composition of red mud (wt%).

Fe_2O_3	Al_2O_3	CaO	SiO_2	TiO_2	Na_2O	MgO	SO_2	K_2O	MnO
33.99	18.47	14.19	9.39	5.42	5.11	0.32	0.33	0.10	0.091

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