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Study on the development of inorganic polymers from red mud and slag system: Application in mortar and lightweight materials

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

- Inorganic polymers from red mud (RM) and slag system were investigated.
- Better reactivity and faster setting were obtained for higher contents of slag.
- Up to 50% of RM in the system led to a 7 days strength of 54 MPa at 25 °C.
- Sand and H₂O₂ addition led to lightweight and mortar products of 10–70 MPa.
- The results are of interest for the conversion of red mud and slag by-products.

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ABSTRACT

The conversion of red mud into valuable products remains an important issue to be addressed. Inorganic polymers from red mud and slag system were investigated. Slag was substituted at 25, 50 and 75% by red mud in the system. Sodium silicate solutions of modulus ranging from 1.6 to 2.2 were used and the samples were cured at 25, 40 and 60 °C. Slag was amorphous phase while red mud contained some crystalline phases, mainly hematite, crancrinite and katoite. A better reactivity, faster setting and lower shrinkage were obtained for higher contents of slag, with a modulus of sodium silicate solution of 2.0. The composition made of 50% red mud and 50% slag achieved a 7 days compressive strength of 54 MPa at 25 °C. The use of standard sand and hydrogen peroxide allowed the formation of mortar and lightweight materials with reduced mechanical properties, but still of interest to address both issues of sustainable use of resources and CO₂ reduction in construction.

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1. Introduction

The term “geopolymer” was coined by Davidovits in 1978 to describe inorganic polymers with tridimensional structures formed by low temperature polycondensation of aluminosilicates [1,2]. These materials have received a burgeoning interest during the last decades due to their excellent physical, mechanical and thermal properties [2–9]. The development of geopolymer materials for building applications was reported to be less CO₂ footprint in comparison to the production of Ordinary Portland Cement (OPC). Actually, the production of one ton of OPC releases about

1 ton of CO₂ and the total OPC production is currently contributing to about 5–7% of global anthropogenic CO₂ [7–9]. Besides, optimally formulated geopolymer cement was found to be able to reduce by 80% the CO₂ associated to OPC cement production industry [7]. Another important advantage of geopolymers is the opportunity to valorize a wide range of secondary raw materials, including slag and red mud, the usage of which can contribute to the sustainability of non-renewable natural resources such as limestone and clay for building industry. Additional environmental benefits associated to the absence of surface-excavated operations required in the case of natural resources were also reported [10–12]. Hence, the successful development and use of building materials from industrial by-products such as slag and red mud is expected to contribute to both sustainable development and CO₂ reduction of OPC industry, the production of which has passed 4 billion tons in 2015 [13].

* 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 (P.N. Lemougna).

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

Slag (ground granulated blast furnace slag) 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 CO₂ emissions and energy consumption in the development of cementitious materials [12,14]. Beside slag, red mud (RM) is a waste product produced in huge amounts from aluminum industries. It is reported that 1.5–1.6 tons of RM is generated per ton of alumina produced, most of it stored in landfill areas or on-site waste lakes [11]. The mineral and chemical compositions of red mud depend on the quality of bauxite, and to a lesser degree, the processing parameters [15,16]. The treatment and utilization of huge red mud wastes has been a major challenge for the alumina industry throughout the world. Among the valorization options, the development of structural materials has been reported as a potential area of interest to be explored [17–20]. However, despite the difference in chemical and mineralogical composition of red mud from different origins, many types of red mud were found not to be reactive enough for geopolymer synthesis and as consequence, were generally mixed with more reactive aluminosilicate materials, mainly fly ashes and metakaolin [11,15,21–23]. Potential structural materials based on red mud combined with silica fume, metakaolin or fly ash were reported [11,22–26], but very little has been reported on the development of red mud/slag system. Pan et al. [27] observed that a poorly rich Al red mud, blended with slag, can present acceptable strength and chemical resistance after alkali activation. Considering the divergence of red muds from different locations [16] and the limited information on the mix proportioning or curing temperature in this previous report, further research is needed to explore the possibilities of red mud valorization. Furthermore, an economical widely accepted technology for the recycle and reuse of red mud has yet to be developed, despite the increasing environmental pressure associated to its worldwide annual generation, now surpassing 150 × 10⁶ t [19,21].

In building engineering, the use of binders or concretes of lower densities is beneficial in terms of structural load-bearing, acoustic and thermal insulation. Adding foaming agents is a common way of reducing the self-weight of binder pastes. Foaming agents generate artificial pores in hardened pastes and reduce their specific gravities. However, the foamed binder pastes are not suitable as load bearing building materials, due to their lower strength, but are efficient for nonstructural applications such as thermal insulation or soundproofing [28,29].

The present study investigated the conversion of red mud and slag into geopolymer materials for potential building applications. Many compositions were prepared by combining slag and red mud with an introduction of up to 75% of red mud in the system. Sodium silicate solutions with a silica modulus ranging from 1.6 to 2.2 were used and the geopolymer slurry was cured at 25–60 °C. The resulting geopolymers were characterized by X-ray diffraction, Fourier Transform Infrared Spectroscopy, Scanning Electron Microscopy, Electric conductance, setting time, linear shrinkage and compressive strength. Finally, an application in the development of mortar and lightweight materials was assessed.

2. Experimental

2.1. Materials

The red mud used in this study was from Guangxi Province, China. The material was dried at 105 °C for 24 h and sieved in a 144 μm sieve. The specific surface area determined by the BET method was 8.04 m²/g. The ground granulated blast furnace slag used in this study was provided by the Chengde Group Company, Beihai Guangxi, PR. China. The specific surface area determined by the BET method was

1.44 m²/g. The oxide composition of the slag and red mud determined by X-ray fluorescence and their particle size information are respectively reported in Tables 1 and 2.

The alkaline activating solutions with silica modulus ($R = \text{SiO}_2/\text{Na}_2\text{O}$) 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. Specimen preparation

The preparation of the fresh mixtures was performed by mixing slag, red mud, water glass of different moduli ($R = 1.6; 1.8; 2.0$ and 2.2), and some amount of deionized water. The mixing process was performed for about 10 min, using an electric mixer at 600 rpm. The homogenous pastes were then casted in cubic alloy molds of 20 × 20 × 20 mm³, covered with a thin layer of plastic to facilitate the removal of the hardened pastes upon curing. The alloy molds were vibrated on a vibration table for 2 min to remove air bubbles and sealed afterwards. The specimens were cured either at 25 °C, 40 °C and 60 °C for 7 days prior to characterization. Some specimens were maintained at 25 °C for 28 days for compressive strength test.

For the preparation of slag/red mud/sand geopolymer composites, some amount of standard sand ISO 679: 1989 was added (1/4, 2/4, 3/4 ratios of sand mass/slag + red mud mass). Lightweight materials were obtained by adding H₂O₂ (AR grade; 30% H₂O₂) in the mixture made with 50% slag and 50% red mud. The percentages of H₂O₂ were 0.5; 0.75; 1.00 and 1.25% of the dry mass of red mud and slag. The details on the mix proportioning are presented in Table 3.

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. SEM/EDS and optical 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). Specimens were impregnated using absolute ethyl alcohol, polished with SiC paper, and then coated with gold. Optical analysis of the composite slag red mud geopolymer containing sand was performed using an Olympus optical microscope of type SZ61.

2.3.3. Electric conductance and setting time analysis

Geopolymer slurry was prepared and casted into a 40-mm diameter × 20 mm height cylindrical mold. Two parallel copper electrodes were placed in the slurry and connected to an impedance equipment to record the electric conductance change of the slurry. The experiment was performed at 25 °C and frequency: 1.0 kHz; voltage: 1.0 V. The change on the electric conductance of the slurry was recorded for 5 h as follows: 1.0 min interval during the first hour, 5 min interval during the second hour and 15 min interval during the last 3 h. The setting time test was performed according to CNS786 and ASTM C191-01 standard test methods, using the Vicat apparatus.

2.3.4. Compressive strength, linear shrinkage and bulk density

Compressive strength testing was performed on 20 × 20 × 20 mm³ 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. For the wet compressive strength, the specimens were immersed for 24 h in water prior to testing. The values were determined as the average of three (03) samples of each composition, and the standard deviation of the three replicate specimens was used as the error bar in Figures. The linear shrinkage was expressed as the percentage of sample size reduction after 3, 7 and 28 days. The bulk density of the cubic samples was determined using the Archimedes principle.

3. Results and discussion

3.1. Phases composition

The phase composition of the synthesized products was investigated using XRD and FTIR analyses.

Fig. 1 presents the XRD results of the pure slag (a), alkali silicate activated slag (b), pure red mud (c) and alkali activated red mud/slag with 50% red mud and 50% slag (c), prepared with a sodium silicate solution of $R = 2.0$. Slag was almost amorphous while the

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