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Characterization and microstructure observation of sintered red mud–fly ash mixtures at various elevated temperature

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ABSTRACT

This study investigated the characterization and microstructure observation of sintered red mud–fly ash mixtures at various elevated temperature. The detailed analysis of the phase evolution during sintering process of red mud and fly ash as the function of sintering temperature were explored. Phase evolution and microstructure observation were carried out by X-ray diffraction method (XRD) and scanning electron microscope (SEM) method. Thermal analysis was carried out by thermogravimetric and differential thermal (TG-DTA) analysis. The mineralogical evolution revealed that quartz and goethite originates from the parent composition of red mud and fly ash. Microstructure observation revealed the transformation of globular grains towards elongated grains during the sintering process. Mass loss around 11% is observed in the thermal analysis patterns due to the dehydration, hydroxide-oxide phase transitions from iron (primary phase) and aluminum (secondary phase). The phase transition, evolution and dissolution as the function of sintering temperature were correlated as the function of temperature and the amount of fly ash content. The disappearance and appearance of the different phases confirms the decomposition and formation reaction suggested by thermal analysis.

1. Introduction

Red mud and fly ash is considered as major industrial hazardous wastes that causes environmental problems and generate multiphase problems in the society. The utilization of red mud is realistically a significant problem in the alumina industry (Samal et al., 2013). The integrated utilization of red mud has thus been intensively investigated, especially in terms of construction materials like cement, land fill etc. (Wanchao Liu et al., 2014). The waste materials obtained from the alumina industries and thermal power plant need necessary treatment and confined disposal to manage them properly (Borges et al., 2011).

In an earlier article the development of sintered ceramics using red mud was reported (Ines Ponsot et al., 2015). The sintering processes are energy intensive and very expensive. The energy intensive problem can be overcome by the addition of additive to the basic raw materials. The sintering additive reduced the energy consumption, but created new unknown phases with impurities and porosity in the ceramics. Similarly, in the thermal power plants, fly ash is generated as a major industrial waste. Fly ash has been used as one of the additive in concrete materials production (Mahdi et al., 2015; Alaa M. Rashad, 2015). To utilize both these wastes together without any chemical stabilizer, an investigation was undertaken by Liu et al. (2007) to develop a process for the manufacture of building materials using conventional sintering method. The characterization of fly ash in the basis of chemical composition analysis was mostly focused by areas of multi-Technique application for waste material detection and soil remediation strategies (Belviso et al., 2011). Those investigations, focused in the development of red mud with fly ash mixtures in order to obtain marketable ceramics materials, have some benefits, e.g., saving of resources such as; raw material and energy, improving the quality of the product, and finally reducing the cost due to use of the waste materials in the process (Yang and Xiao, 2008).

Pontikes et al. (2009) have studied the ceramics derived from the red mud using different sintering additives. In the previous work, the suitability of red mud with fly ash for the development of paving blocks has been evaluated (Anuj and Kumar, 2013).

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Neutralization of alkaline medium of red mud by adding fly ash different quantity has been studied earlier with focus more on pH of the medium (suchita et al., 2013). Although previous researcher mention about the suitable sintering temperature 950–1250 °C, but in nowhere it has shown clearly about in detail specific sintering temperature and the evolution of microstructure and phases. Water absorption and mechanical properties showed that specimens fired at their respective optimum sintering temperature met the requirement values established by different Indian Standards for bricks, tiles and making paving blocks. Based on these preliminary analyses, the investigation conclude that studied red mud and fly ash mixtures can be used as secondary raw materials for the production of bricks, tiles and blocks by sintering method. The properties of the building materials such as abrasion strength, stability and corrosion resistance depend on the phase constitution of red mud, sintering time, temperature and post sintering composition. The mineral undergoes phase transformation and relationships between reactions, transformations and mineral phases needed to be studied.

In this article distinguished way of approach with more focus on characterization, feasibility of reaction and possible phase generation has been studied in general to broad areas of topic cover from raw materials to application. The interphase of processing is more important in the terms of possible microstructural evolution as the function of sintering temperature and possible phase detection using XRD. This intermediate step controls the final product and possibility of feasibility of reaction process and production. This paper aims on the originality of the microstructure evolution with respect to sintering temperature. This type of work has not published anywhere. Although researcher described this part secondarily not primarily the main subject area of concern. In this article it's more focus on experimental investigation of phase change as well as morphological evolution of red mud–fly ash composite as the function of specific sintering temperature. This article will open up more fundamental knowledge and possibilities of the intermediate sintering process and the respective feasible reaction steps for the utilization of red mud–fly ash composites.

In the sintering process, a series of different chemical reactions and physical transformations take place during sintering. As a result a mechanically weak green body is transformed into a strong product. The technological properties of the fired material are strongly dependent on both mineralogy and microstructure and their knowledge will lead to optimization of the firing process and properties of the end products. The effect of sintering temperature has a significant role on the densification and porosity of ceramics. Therefore the phases and their contribution for the final product characteristics need to be studied. The purpose of this work was to investigate the mineralogical evolution in the microstructure of the product as a function of sintering temperature. A correlation was established between the various phases of the microstructure of ceramic fired product as function of their optimum sintering temperature.

2. Experimental

2.1. Raw materials

Red mud (slurry) material was provided from the Bharat Aluminum Company Ltd. (BALCO), Kobra in Madhya Pradesh, India. The plant imports its bauxite from Amarkantak/Phulkanahar mines near to it. Red mud sample was dried in oven at 100 °C for 24 h. Fly ash samples were supplied from one of the thermal power plants of M/S Calcutta electric supply company, Calcutta, India. The red mud powder is well mixed with fly-ash using an agate motor. Pellets were prepared from the sample (red mud and red mud + fly ash) using water as the binder with pressure 2.5 ton with pellet thickness 0.5 cm with diameter 2.5 cm. The quantity of fly ash varies from 10, 20 wt. % in the red mud. The chemical compositions and the mineral phases of the red mud and fly ash are given in Table 1. Iron makes up one-third of the content of the red mud, and since the entire elements exist in oxides, iron oxides (Fe_2O_3) forms more than other component of the red mud. Next to iron, residual aluminum, silica and sodium form the major components. In case of fly ash, silica in the oxides form (SiO_2) is the major component, which is more than half of the component. The aluminum is the next major component consists of one-third part of the fly ash. Both elemental components of red mud and fly ash are suitable for ceramics in terms of the silicate and alumina forms.

2.2. Sample preparation

The red mud was ground to a size of less than 150 μm (−100 mesh) was mixed with 0, 10 and 20 wt% fly ash. The sample was compacted in a stainless steel die by an uni-axial hydraulic press under a pressure of 50 MPa. The pellets were dried in air at room temperature for 48 h followed by drying in an air convection oven. The oven was heated at a rate of 2 °C/min up to 120 °C, for 5 h. Sintering of the samples in a static argon atmosphere was carried out at various isotherms in the range 1000–1100 °C in a graphite resistant furnaces for a period of 2 h. The furnace was heated to a pre-determined temperature and was allowed to achieve temperature equilibration. The pressed samples of red mud and red mud with fly ash mixtures were placed inside the graphite hearth for sintering. After sintering, the furnace was cooled for 2 h and then the sintered samples were removed from the furnace. The schematic presentation of the process is displayed in Fig. 1. The sintered samples were characterized with various techniques like DTA, XRD and SEM.

Table 1
Chemical and mineralogical composition of red mud and fly ash.

Constituent red mud	Composition (mass percent)	Constituent fly ash	Composition (mass percent)
SiO_2	8.31	SiO_2	59.9
Al_2O_3	22.36	Al_2O_3	29.2
Fe_2O_3	34.27	Fe_2O_3	3.8
TiO_2	17.13	TiO_2	2.7
CaO	1.73	Alkaline oxides	0.3
Na_2O	6.12	Alkaline earth oxides	3.1
LOI	9.38	P_2O_5	1.4
		Others	0.2
Mineralogical phases	Quartz (SiO_2), goethite ({FeO(OH)} Pervoskite $CaTiO_3$	Hematite (Fe_2O_3), Calcium aluminum silicate, $Ca_2Al_2(SiO_4)(OH)_8$	