



Auto-combustion processed high alumina cement and its implementation as bauxite based low cement castables

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Abstract

The present work involved solution combustion synthesis of high alumina cements from their metal nitrate precursors, using urea as an organic fuel. Combusted aluminium nitrate, calcium nitrate and urea complexes leads to formation of high alumina cement at temperature as low as 800 °C. The prime cementing phases observed were CA, CA₂ and C₁₂A₇. The second stage of work evaluates, the role of this high alumina cement (HAC) on bauxite based low cement castables. The effect of micro-fine Cr₂O₃ and MgO additions on the sinterability and thermo-mechanical strength of these castables was also investigated. Phase identification and microstructural evaluation of the sintered castables confirms spinel and corundum phase formation. Addition of nano structured cements in refractory castables improved the thermo-mechanical properties to a significant extent.

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1. Introduction

Iron and steel making industries being the chief consumer of refractories have made major improvements in the quality and consistency of their products [1]. Demand for structural and clean steel in this century has increased manifold which in turn prompted for better refractories [2–4]. A significant advancement in refractory technology is hence, continually required to meet the service conditions. Evolution of monolithic for a refractory construction in the present day scenario shows a remarkable increase, due mainly to certain advantages they offer, especially in comparison with shaped articles [5–7]. The properties of these castables have been exceedingly better than bricks with comparable chemistries. With the changes in technology it is now necessary to overcome the limitation of conventional castables (15–20% of calcium aluminate cements), due to its low hot strength, especially when molten slag and metal are present [9]. The most noticeable improvement in monolithic refractory is low cement/low moisture castable development [8]. In the new monolithic refractories technology,

low and ultra-low cement castables have an ever-decreasing amount of high alumina cement and its corresponding water for placement. Low cement and low water improves high temperature properties. As CaO forms eutectic by slag dissolution, therefore, it became obvious to reduce the CaO content of the castables which meant reducing the cement and water content. To meet the required service condition, this is how the concept of low cement castables developed [10]. There has been a rapid development in the use of low cement castables in many industries such as non-ferrous metals, cements, petrochemicals and of course in iron and steel industry [11]. These give excellent results in reheating furnaces, ladle linings, soaking pit bottoms, blast furnace troughs. The superior properties of the low cement castables helped the iron and steel makers to reduce their consumption enormously and hence their operating cost.

Solid state synthesis of calcium aluminates require high temperatures and full conversion is not guaranteed. Moreover, it is difficult to obtain a compositionally homogeneous product with this method. In recent years, combustion synthesis has attracted a great deal of attention to produce superfine, unagglomerated, multicomponent crystalline ceramic without intermediate decomposition or calcination [12]. This method

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exploits an exothermic, rapid and self-sustaining chemical reaction. Its key feature is that the reaction itself provides the heat required to drive the chemical reaction without any external source [13–15].

Commercial high alumina cements are known to contain various phase viz., CA, CA₂, C₁₂A₇ and α-alumina whose hydration behavior is interdependent and not yet fully understood [16–19]. The mono calcium aluminate (CA) imparts high mechanical strength and refractoriness to the cement, where rapid setting is mostly associated with C₁₂A₇ which dissolves rapidly [20–22]. Cement with high amount of CaO has many drawbacks when used as hydraulic binders in refractory castables. Their excess water demand during casting process, porosity due to drying and also in their low refractoriness when in contact with SiO₂.

There has been enormous amount of work in the field of solution derived combustion of calcium aluminates, however there is lack of studies which focus on their utilization as castables. Present work opens new doors by implementation of prepared cement as monoliths and their hot properties. In the first part, the work that follows describes the synthesis of high alumina cements (CA, CA₂ and C₁₂A₇) by the auto combustion reaction of redox mixtures of the corresponding water-soluble nitrate salts with urea. For reference study, a comparison with commercial high alumina cement is reported herein. In the second stage of work, formulation as well as characterization of bauxite castables and prepared cement as the binder is discussed to assess and achieve a good quality of low cement castable. Together, the aluminates and the micro fillers are responsible for the hydraulic setting of the castable. With packing density maximized, the low cement castables are characterized by their low porosity, high density and exceptional hot strength; with enhanced erosion, corrosion, and spalling resistance [23–25].

2. Experimental procedure

2.1. Materials

The starting raw materials A.R. grade aluminum nitrate Al(NO₃)₃ · 9H₂O, calcium nitrate Ca(NO₃)₂ · 4H₂O, urea (NH₂)₂CO, chromium oxide (Cr₂O₃) magnesium oxide (MgO) powder and Chinese bauxite were supplied by Loba Chemie Pvt. Ltd., Mumbai India. The calcined Chinese bauxite that was used in castable formulation in the present study contained approximately 88.60%, 4.78%, 1.58%, 4.0%, 0.26%, 0.08% and 0.70% by weight Al₂O₃, SiO₂, Fe₂O₃, TiO₂, CaO, Na₂O and others, respectively, as specified by the supplier (Shiva Minerals, Rourkela, India).

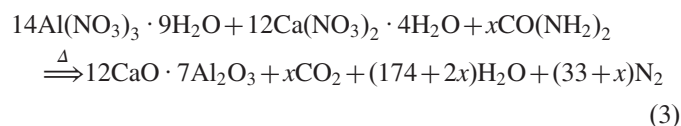
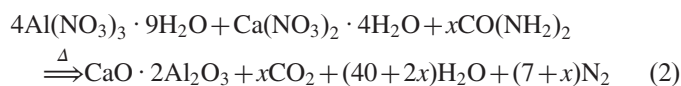
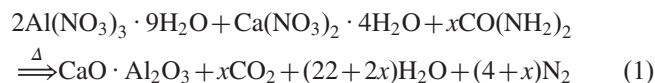
2.2. HAC powder preparation

Combustion synthesis or self-propagating high temperature synthesis is a versatile method used for the synthesis of a variety of solids. The method makes use of a highly exothermic reaction between the reactants to produce a flame due to spontaneous combustion which then yields the desired product or its precursor in finely divided form. In order for

combustion to occur, initial mixture of reactants should be highly dispersed and has must have high chemical energy. Even if the desired product is not formed immediately after combustion, the fine particulate nature of the product facilitates its formation on further heating. Reaction times are very short since the desired product results soon after the combustion.

The auto-combustion synthesis technique consists of bringing a saturated aqueous solution of the desired metal salts and a suitable organic fuel to boil, until the mixture ignites and a self-sustaining and rather fast combustion reaction takes off, resulting in a dry, usually crystalline oxide powder. While redox reactions such as this are exothermic and often lead to explosion if not controlled, the combustion of metal nitrates and urea mixtures usually occurs as a self-propagating and non-explosive exothermic reaction. The large amounts of gases formed can result in visible flame, which can reach temperatures in excess of 1000 °C.

Aluminum nitrate Al(NO₃)₃ · 9H₂O and calcium nitrate Ca(NO₃)₂ · 4H₂O were weighed and mixed on a laboratory scale to wt ratio of 7:3 and 8:2 of Al₂O₃/CaO, which were denoted as HAC70 and HAC80 high alumina cements, respectively. For complete dissolution of the salts, the solution was kept on a hot plate at around 60 °C with simultaneous stirring until a clear transparent solution was obtained. To this precursor solution urea was poured keeping a cement precursor/fuel ratio of 1:1. The prepared gel was air-dried in an oven at 80 °C to complete dryness. Finally, after the solution was converted to a dried gel powder it was calcined, for 15 min, in air at 500 °C, and then quenched to room temperature. During the first few minutes of the calcinations process, ignition took place with a rapid evolution of large amounts of gases. Therefore, only small portions of the gels were calcined. The synthesis technique use the heat energy released by the redox exothermic reaction at a relatively low ignition temperature between metal nitrates and urea. The process lasted for a relatively short time, so that the particles could be maintained to the nanometer scale. Further high alumina cements nano composite powders were heated at 800 and 1000 °C in a platinum dish using a SiC muffle furnace for a soaking period of 2 h. After heating the HAC powder was stored in desiccator. The probable chemical reactions are given below:



2.3. HAC powder characterization

X-ray diffraction patterns were determined using a Rigaku portable XRD machine (Rigaku, Tokyo, Japan). Calcined powders

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