

A technical note on the production of zirconia and zircon brick from locally available zircon in Bangladesh

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Abstract

In this study, the potentiality of Bangladesh zircon as a raw material for zirconia and zircon brick production was examined. Decomposition of zircon ($ZrSiO_4$) into zirconia (ZrO_2) was attempted by using caustic soda (NaOH) as the decomposition agent. 5–10 μm size zircon, 5–30 wt.% NaOH, and decomposition temperatures ranging from 1300 to 1500 °C were investigated. X-ray diffraction method using Mo $K\alpha$ radiation was used in order to find out the extent of decomposition of zircon into zirconia. Zircon bricks of 0.06 mm \times 0.06 mm \times 0.025 mm shaped were made under a pressure of 15 MPa. Carboxy methyl cellulose (CMC) and Fe_2O_3 were used as a binder. With increasing temperature and percentage of NaOH, decomposition of zircon increases. Crushing strengths and density of these bricks could be found as high as 53.36 MPa and 3210 kg/m^3 , respectively. Bangladesh zircon can be used successfully for preparation of refractory bricks for high temperature use.
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Keywords: Zircon; Zirconia; Decomposition; NaOH; Temperature; Crushing strength; Density

1. Introduction

In recent years, zirconia (ZrO_2) has attracted considerable attention as a high-technology material for industrial applications because of superior mechanical, thermal, electrical, chemical and optical properties. For the preparation of ZrO_2 powder, the production routes of commercially viable processes normally use the economically available natural sources, namely, baddeleyite (ZrO_2) and zircon ($ZrSiO_4$). Of the two, zircon is more important because of its wide availability as beach sand deposits [1]. The processing of zirconia ore (baddeleyite) is more difficult because it contains a great amount of silica. A commercial process consists in melting zircon with coke, in a sufficient amount to reduce silica, and iron in an electric furnace, with a simultaneous addition of calcia in order to obtain stabilized zirconia as the final product of the operation. A by-product is ferro-silicon, formed from reduced silica and added iron metal [1]. Zircon bricks are also used in aluminium remelting furnaces, calcium phosphaste fertilizer furnaces and making crucibles for platinum melting.

Dense zircon silicate bricks are installed in glass melting furnaces as soldier bricks in the melting and refining areas of borosilicate glass tanks. In electrical tanks, they are installed in the bottom and as rear lining. Owing to the low electrical conductivity at high temperatures, so-called electrode bricks for electric heating are also manufactured out of dense zircon silicate minerals. Casting nozzles may also be made of zirconium silicate, and in Japan, such bricks are used in steel ladles [2].

Zircon is one of the most important among the heavy minerals occurring in the beach sand along the coastal belt of Bangladesh. The reserve of zircon in Bangladesh coast is roughly estimated to be more than one and half a million tonnes [3]. Zircon is a valuable mineral used as a raw material in various industries including foundry, ceramics, and refractories. In Bangladesh approximately 300 tonnes of zirconia flour is consumed every year in ceramic industries; the entire amount being currently imported. However, zircon occurring in the Bangladesh coast has so far not been utilized.

Previous study by the same authors fully characterized zircon beneficiated at the Cox's Bazar Pilot Plant of Bangladesh Atomic Energy Commission [4–6], particularly to find out its impurity content and state of impurity and ways to remove the impurities from Bangladesh zircon. They investigated that zircon sand as

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extracted at the Beach Sand Exploitation Center, Cox's Bazar is found to be very suitable for steel casting. Use of Bangladesh zircon is found to eliminate various surface defects in cast steel spares. In order to explore the full potentiality of Bangladesh zircon in ceramics and refractories, the objective of the present work is to investigate the suitability of Bangladesh zircon as a raw material for the production of zirconia and refractory bricks. The outcome of this study is expected to provide a scientific basis for the use of Bangladesh zircon in ceramics and refractory industries.

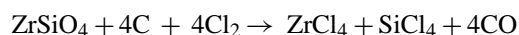
2. Some commercially available methods for the production of zirconia from zircon

2.1. Precipitation and calcination technique

Pure zirconia can be prepared from a fava [7] ore in the following way: finely ground ore is treated with concentrated sulfuric acid in the proportion 1:2. Most of the practically uncombined zirconium oxide is dissolved as zirconium sulphate, silica, and zircon remaining unattacked. The strongly acidic mixture is diluted with water, and the solution is filtered. The filtrate is concentrated again, and the solution is neutralized, whereby a precipitate of the basic zirconium sulphate is formed. The precipitate is filtered off, and washed. It can be decomposed to free zirconia by calcination, or it can be dissolved, precipitated as zirconium hydrate, and calcined [7].

2.2. Chlorination and thermal decomposition technique

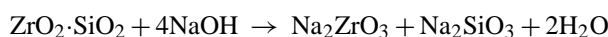
A higher purity grade of zirconia can be prepared in another way: The ore mixed with coke, in an amount sufficient to bind the total oxygen of the ore, and the mixture is heated at 800–1200 °C in a shaft kiln with a silica lining to a red heat or fluidized bed, the following reaction occurs [8]:



Chlorine gas is introduced into the hot mixture, whereby a complete chlorination of the ore takes place. Zirconium tetrachloride, iron trichloride, titanium tetrachloride, and so on, are formed. Silica is not substantially attacked in this operation. All the chlorides formed are volatile, and sublime out of the shaft. The sublimate is conducted into water, where soluble chlorides and oxychlorides are formed. Zirconia tetrachloride, after being distilled, is selectively condensed at 150–180 °C when the major impurities, i.e. chlorides of silicon, iron, titanium and aluminium are separated. After hydrolysis with water, a solution of zirconium oxychloride or zirconyl chloride (ZrOCl_2) is obtained. The ZrOCl_2 solution is used for the preparation of ZrO_2 powders. By recrystallization in strong hydrochloric acid the oxychloride is obtained in a pure state and in a good yield. It can be calcined immediately to zirconium oxide, or dissolved in water and precipitated with ammonia as zirconium oxide hydrate. After calcination, a heavy pure white zirconia powder is produced.

2.3. Alkali oxide decomposition technique

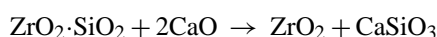
It is well known that zircon is decomposed at high temperature in reducing media. The decomposition temperature, however, is lowered when the silicate is mixed with fluxing agent such as NaOH [9], Na_2CO_3 [10], and $\text{CaO} + \text{MgO}$ [11]. The degree of decomposition depends on parameters like particle size of zircon, alkali/zircon molar ratio, temperature and time of decomposition, etc. By careful control of the process parameters, zircon decomposition either with NaOH or Na_2CO_3 , the following reaction products are obtained:



Leaching with water of the decomposed mass removes the soluble sodium silicate and simultaneously hydrolyzes sodium zirconate to hydrated zirconia. The impure hydrated zirconia is dissolved in concentrated HCl, HNO_3 or H_2SO_4 at 80 °C for obtaining a zirconyl chloride, nitrate or sulphate solution, respectively. These solutions are then purified either by precipitation as basic zirconium sulphate or by crystallization as the corresponding zirconium salt [12].

2.4. Lime fusion technique

When a mixture of ZrSiO_4 , and calcia (in different proportions) is heated at various temperatures, calcium zirconium silicate, calcium zirconate and calcium silicate are formed [13]:



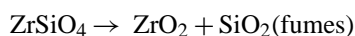
The second reaction is more suitable because CaSiO_3 can be removed by leaching with HCl at 90 °C followed by treatment with NaOH at 15 °C.

2.5. Plasma decomposition technique

When zircon particles are introduced into a stable argon plasma reactor at a temperature range of 600–1500 °C and quenched rapidly, dissociation of zircon occurs with the formation of the constituent oxides, i.e. ZrO_2 and SiO_2 [14]. The degree of dissociation depends on a number of process parameters, e.g. the particle size of zircon, flow rate of argon gas, arc current, feed rate, etc. The decomposed mass is treated with NaOH or H_2SO_4 .

2.6. Thermal dissociation technique

Zircon can be thermally dissociated in an arc furnace or an electric reactor [14] when heated above 1750 °C (followed by rapid cooling); into its constituent oxides and the chemical reaction that takes place is essentially as follows:



ZrO_2 and SiO_2 are recovered separately as discussed above.

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