ELSEVIER

Ceramics International 31 (2005) 831–838

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Physicochemical characteristics of alumina gel in hydroxyhydrogel and normal form

T. Meher, A.K. Basu, S. Ghatak*

Central Glass and Ceramic Research Institute, Non-Oxide Ceramic Section, 196 Raja SC Mullick Road, Kolkata 700032, India

Received 19 April 2004; received in revised form 6 June 2004; accepted 14 September 2004
Available online 31 December 2004

Abstract

Aluminium hydroxide as a powder precursor for making advanced ceramic materials such as yttrium aluminium garnet (YAG) was prepared from aluminium nitrate and ammonium hydroxide by two different procedures and was analyzed for determining its physico-chemical characteristics. It was observed that through the end products were essentially $Al(OH)_3$, they differ in their physico-chemical properties. Rate constants evaluated by thermal analysis were compared and the differences observed were explained in terms of structural parameters by FTIR and related analysis.

Keywords: Hydroxyhydrogel; Alumina gel; Aluminium hydroxide; YAG precursor

1. Introduction

Alumina is a versatile material used as refractory, engineering ceramics material, abrasive and in various other applications where chemical inertness coupled with its high hardness and abrasiveness is of primary importance. In addition to this, alumina is also used in combination with other materials such as MgO, SiO_2 and Y_2O_3 to produce versatile materials, like magnesium aluminates spinel [1], mullite [2] and yttrium aluminium garnet (YAG) [3]. Preparation of later types of materials like YAG requires high degree of purity and homogeneity in the raw materials, i.e. Al_2O_3 and Y_2O_3 [4]. This is possible only in wet interaction method. Alumina in wet mixing process is generally derived from the aluminium hydroxide, which exists in several modifications (gibbsite, bayerite, boehmite and diaspore). The structure of the aluminium hydroxide in all its forms consists of stacked double layers of oxygen atoms in which all the cations are located in the octahedral coordination in the interstices [5]. The packing of oxygen ions inside the layer can be either hexagonal or cubic, whereas the symmetry for each hydroxide is determined by the distribution of hydrogen. The relative distances between hydroxyl groups, both within and between the layers, have been suggested to control the mechanism of dehydration for the particular hydroxide [6]. This structure of aluminium hydroxide depends on the aluminium hydroxide precursors which in turn may be influenced by the aluminium salts used as the starting reagents as well as on the procedure by which the precursors have been prepared [7]. This aluminium hydroxide precursor often results in alumina gel. Alumina gel has been prepared by sol–gel technique [8–10], as proposed by Yoldas, using aluminium alkoxide as precursors. One of the major advantages of the sol–gel processing is that the properties of the alumina can be altered by manipulating any of the processing steps during precursor formation. For example: the resultant gel structure can be altered by parameters that affect the kinetics of the reaction. These are the kinetics of hydrolysis and polymerization. They are in turn affected by: (a) starting compound and host media [7], (b) dilution, water alkoxide ratio [11], (c) catalyst and (d) temperature [12–15]. The resultant gel structure ranges from all the varieties of aluminium trihydroxides to monohydroxide [15,16], crystalline, more or less amorphous or even superamorphous [17].

* Corresponding author. Fax: +91 33 2473 0957.
E-mail address: sghatak@cgcri.res.in (S. Ghatak).

0272-8842/$30.00
doi:10.1016/j.ceramint.2004.09.011

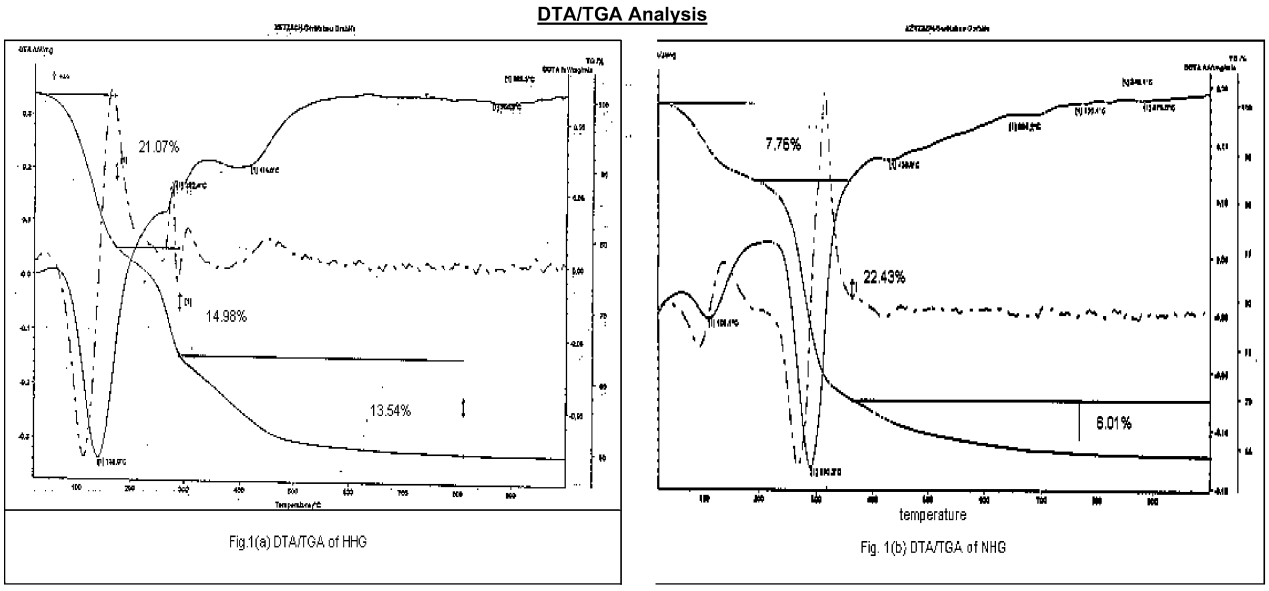

Fig. 1. (a) DTA and TGA of HHG sample and (b) DTA and TGA of NHG sample.

In the present work aluminium hydroxide is prepared through two different sequences of chemical ingredients addition. The products obtained, $Al(OH)_3$ in both the cases, were characterized to get an idea about the overall chemical reactivity of $Al(OH)_3$ formed by two different routes.

2. Experimental

A saturated solution of $Al(NO_3)_3{\cdot}9H_2O$ was added to ammonia in one case (this process led to HHG) and in another 1:1 (v/v) ammonia was slowly added (this process led to NHG). The pH of the hydroxide precipitation was adjusted in the value of 4.5–5.5 for complete precipitation of the specific cations as hydroxides. The gel like masses so obtained was aged overnight for complete reaction. The extraneous insoluble impurities were removed by washing with water and the precipitate was dried at (100 ± 10 °C). The characteristics of the powder precursors prepared by both routes were examined by DTA, TGA, kinetics studies, FTIR spectroscopy. DTA,TGA and kinetics studies were conducted using Libratherm TGA instrument (no. PID-300/25). FTIR spectroscopy was done by FTIR spectrometer (Perkin–Elmer, model no. 1615) on the heat-treated samples to identify the nature of the bonding.

3. Results

Dynamic equilibrium studies on the sample hydroxyhydrogel (HHG) and normal gel (NHG) studied in the temperature range 300–1000 °C revealed the existence of several peaks indicating the existence of H_2O in the main network structure in different forms. This observation is partly supported by the existence of corresponding DTA peaks as shown in Fig. 1(a) and (b). The loss of water took place in three distinctly different stages and the values are given in Table 1. From Table 1 it appears that HHG samples and NHG samples have different characteristics as the net water content of HHG sample is more than that of NHG

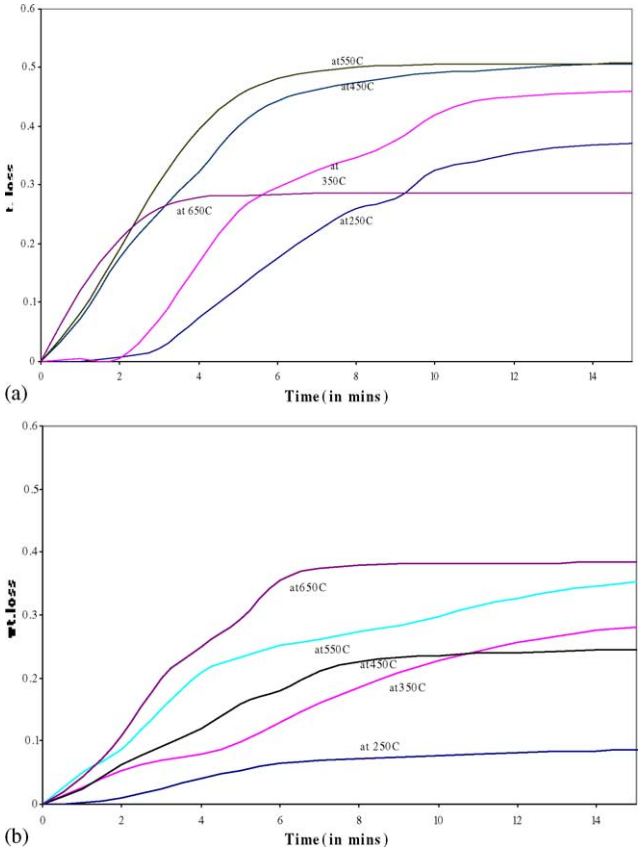

Fig. 2. (a) Weight loss vs. time for HHG sample and (b) weight loss vs. time for NHG sample.