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Letter to the Editor

Synthesis of calcium hexaaluminate ($CaAl_{12}O_{19}$) via reverse micelle process

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ABSTRACT

This letter describes the synthesis of $CaAl_{12}O_{19}$ powders using micro reactors made of Igepal CO520/water/cyclohexane microemulsions. Characterization of the powder was done by DTA-TGA, X-ray diffraction, Scanning electron microscopy, Fourier Transform Infrared Spectroscopy. The XRD results show that the hexagonal $CaAl_{12}O_{19}$ powders have been obtained at 1200 °C for 2 h. The SEM examination shows that the hexagonal $CaAl_{12}O_{19}$ has plate-like grain morphology with most of the grain took the form of hexagonal platelets with-developed faces. The FTIR spectra show the lower frequency bands are assigned to AlO_6 octohedra and AlO_4 tetrahedra in $CaAl_{12}O_{19}$.

1. Introduction

Calcium hexaluminate, $CaAl_{12}O_{19}$ (hibonite) has the magnetoplumbite crystal structure [1] in which the calcium occurs in cleavage planes located between blocks of Al_2O_3 spinel structure containing 20 octahedral Al and two tetrahedral Al per unit cell. The remaining two Al atoms per unit cell are associated with the cleavage plane, and are suggested to be in irregular five-coordinated environments. The behavior of Al in this phase during its structural evolution is therefore of interest for the unusual Al environments in both amorphous and crystalline aluminates. Calcium hexaluminate, $CaAl_{12}O_{19}$, has shown promise as an interface phase in fiber-reinforced ceramic oxide composites because of its preferred basal-plane cleavage properties [2]. Calcium hexaluminate, $CaAl_{12}O_{19}$, is an oxide-based alternative to carbon and boron nitride for fiber coating in ceramic–matrix composites in oxidizing environments at temperatures > 1000 °C, but its synthesis as single phase powder requires high temperature treatments [3]. MacKenzie et al. [4] prepared calcium hexaaluminate by Pechini method and shows magnetoplumbite structure formed in an exothermic reaction in which the large Ca ion migrates into the mirror planes between the spinel blocks. Singh et al. [5] prepared Cr^{3+} activated $CaAl_{12}O_{19}$ phosphors by combustion process. Evidence for the existence of Cr^{3+} ions in the doped lattice is presented. Costa et al. [6] prepared a new turquoise blue ceramic pigment on the basis of nickel-doped hibonite. Nie et al. [7] reported the Cr^{3+} ion as a co-dopant to modify the unpractical Photon cascade emission properties of Pr^{3+} in $CaAl_{12}O_{19}$ phosphors. Among all the chemical processes that were developed for the preparation of fine ceramic powders and for producing a wide array of metals and metal oxide compounds [8–10] the microemulsion processing involving reverse micelles has been demonstrated as a superior method [11] in terms of being able to deliver homogeneous and nanosized grains of a variety of oxides. This aqueous method uses readily available inexpensive, and easily handled precursors of $Ca(NO_3)_3$ and $Al(NO_3)_3$, and eliminates the extra handling requirements that usually associates with moisture sensitive precursors. In recent years, microemulsion method has been studied widely and has been key technique to synthesize oxide nanoparticles owing to the products which has a characteristic of well dispersed, controlled size and narrow size distribution. However, there is no literature available on the synthesis of Calcium hexaaluminate ($CaAl_{12}O_{19}$) by reverse micelle process. In the present work, we prepared pure $CaAl_{12}O_{19}$ for the first time based on Igepal CO520/water/cyclohexane reverse microemulsion route at 1200 °C for 2 h.

2. Experimental

Fig. 1 shows the flow chart for the preparation of $CaAl_{12}O_{19}$ by reverse micelle process. Calcium nitrate ($Ca(NO_3)_2{\cdot}4H_2O$),

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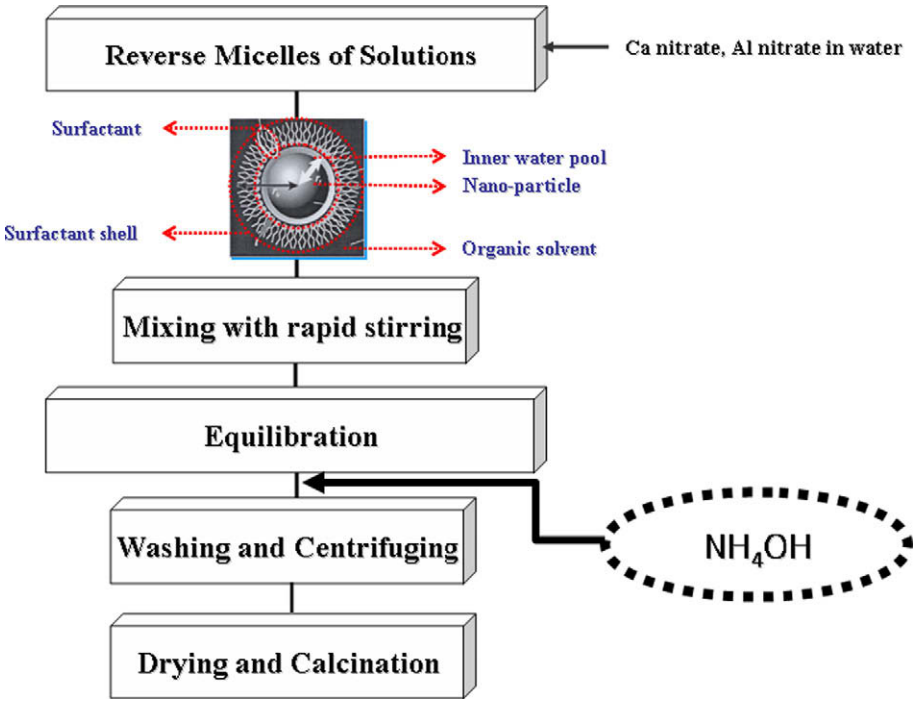

Fig. 1. Flow chart for the preparation of $CaAl_{12}O_{19}$ powders by reverse micelle processing.

aluminum nitrate ($Al(NO_3)_3{\cdot}9H_2O$ were used as the precursor of calcia and alumina. Cyclohexane (Sigma Aldrich) was used as organic solvent. Reverse microemulsion solution was prepared by mixing 40 mL of nonionic surfactant poly(oxyethylene) nonylphenyl ether (Igepal CO-520, Aldrich Chemical Co., USA), 100 mL of cyclohexane and 13.2 mL of mixed aqueous solution (Ca:Al = 1:2). The water/surfactant (R) was maintained at 8. The microemulsion was mixed rapidly, and after 5 min of equilibration, 6 ml of NH_4OH (28%) (Dae Jung chemicals, Korea) was injected into the microemulsion. The microemulsion was then centrifuged to extract the particles, which were subsequently washed by ethanol to remove any residual surfactant. The phase identification of calcined powders was recorded by X-ray diffractometer (Philips X'pert MPD 3040). The morphology of the calcined powder was observed by Scanning electron microscopy (SEM) operating at an accelerating voltage of 30 kV. The Fourier transform infrared spectra (FTIR) were measured on a Nicolet Impact 410 DSP spectrophotometer using the KBr pellet method.

3. Results and discussion

Thermal behavior of the precursor powders determined by DTA/TG in oxygen atmosphere up to 1400 °C at a heating rate of 10 °C/min. is shown in Fig. 2. Decomposition started below 150 °C with a weight loss of 5.35% corresponds to adsorbed water. In the temperature region between 150–600 °C, the main decomposition occurs with a weight loss of 40.7%. The thermal decomposition behavior is associated with endothermic and exothermic effects in the DTA curve. It may be inferred that endothermic peak between 200–300 °C represents the decomposition of $Al(OH)_3$. Previous workers [12–14] have reported $Al(OH)_3$ decomposition takes place at 300,

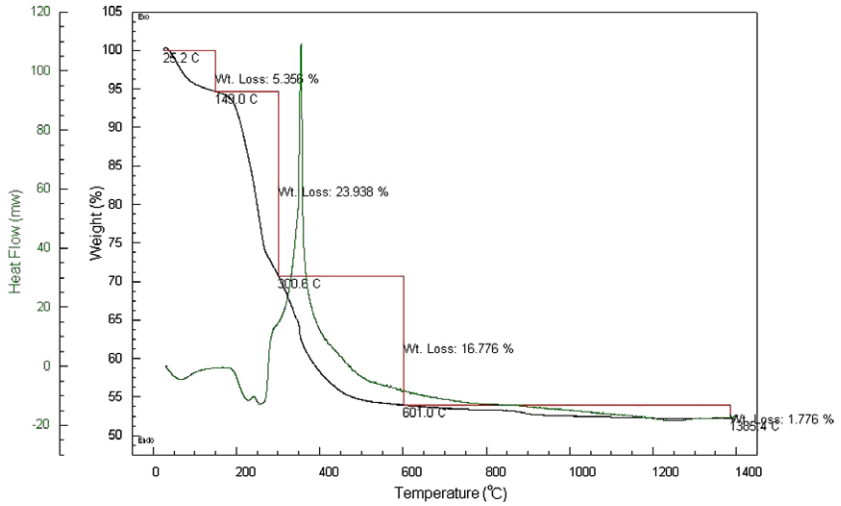

Fig. 2. DTA and TGA studies of as-synthesized precursor.