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Reverse precipitation synthesis and characterization of CeO_2 nanopowder

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ABSTRACT

In the present work, CeO_2 nanopowder was synthesized via a reverse precipitation method using $CeCl_3{\cdot}7H_2O$, NH_4OH and sodium dodecyl sulfate as raw materials. The effect of thermal treatment on the crystal growth, surface area and chemical bonds of the powder was discussed. The structural evolutions and morphological characteristics of the nanopowder were investigated using X-ray diffractometery, transmission electron microscopy, scanning electron microscopy, differential thermal analysis and Fourier transform infrared spectroscopy. The results showed that CeO_2 with an average particle size of 45 nm is formed. The BET surface area increased to ≈41 m^2/g, with increasing calcination to 300 °C; then, it decreased following calcination at higher temperatures. The particles sintered and agglomerateed together after 500 °C. The activation energy for CeO_2 nanocrystallite growth during calcination was calculated to be about 14.6 kJ/mol.

1. Introduction

Ceria (CeO_2) is a fluorite-structured ceramic material that does not show any known crystallographic change from room temperature up to its melting point (2700 °C) [1].

In recent years, nanocrystalline cerium oxide (CeO_2) particles have been extensively studied owing to their potential uses in many applications, such as UV absorbents and filters [2], buffer layers with silicon wafer [3], gas sensors [4], catalysts in the fuel cell technology [5], catalytic wet oxidation [6], engine exhaust catalysts [7], NO removal [8], photocatalytic oxidation of water [9], etc. CeO_2 nanopowders have been reported to be synthesized by different techniques such as: hydrothermal [10], mechanochemical [11], sonochemical [12], combustion synthesis [13], sol–gel [14], semi-batch reactor [15], microemulsion [16] and spray-pyrolysis [17]. Compared with these methods, homogeneous precipitation method is one of the most promising techniques because of the inexpensive starting materials, a simple synthesis process and commonly available apparatus. For example, Zhou et al. [18] produced CeO_2 particles of about 4 nm from cerium nitrate and ammonia. Chen and Chen [19] prepared CeO_2 particles from cerium nitrate with hexamethylenetetramine, whereas Li et al. [20] used ammonia carbonate and diethylamine as the precipitate agents. Yamashita et al. [21] produced CeO_2 particles from cerium chloride and sodium hydroxide with the presence of hydrogen peroxide under various pH conditions from 6 to 12. Uekawa et al. [22] obtained 7–9 nm CeO_2 particles starting from cerium nitrate in the polyethylene glycol solution. Chen and Chang [23] prepared nanocrystalline CeO_2 particles with different shapes and sizes by two-stage non-isothermal precipitation using cerium (III) nitrate and ammonia. Yuejnan et al. [24] produced high surface area nano-CeO_2 using surfactant CTAB, $Ce(NO_3)_3$ and NaOH as precipitation agent. Although CeO_2 nanoparticles prepared by the precipitation technique have been extensively studied but most of the previous reports were focused on direct precipitation of ceria without using a dispersant.

In the present work, a CeO_2 nanopowder was synthesized via a reverse precipitation method using $CeCl_3{\cdot}7H_2O$ and NH_4OH as precipitation agent. Sodium dodecyl sulfate (SDS), as a common anionic surfactant, was selected as dispersant to reduce agglomeration.

2. Experimental procedure

CeO_2 nanopowder was prepared by a reverse precipitation method using $CeCl_3{\cdot}7H_2O$ (Merck, purity > 99.5%), NH_4OH (Merck, purity > 99%) and SDS (Sigma–Aldrich). $CeCl_3{\cdot}7H_2O$ and SDS were dissolved in deionized water. The molar ratio of Ce^{4+} to SDS was 2. The solution was added drop-wise to NH_4OH while keeping a constant pH value of 8.5 by adding extra-ammonia solution. The resultant synthesis precipitate was washed with deionized water and dried at 80 °C for 24 h. The dried precipitate was calcined for 2 h in a tube furnace at different temperatures.

The crystalline structure of the powders was determined by X-ray diffraction (Philips pw 3710) with Cu Kα radiation. The average crystallite size (d) of the powder was estimated from the Scherrer equation (Eq. (1)):

$$d = \frac{0.9\lambda}{\beta_{sample}\cos(\theta)} \quad (1)$$

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