



Novel simple solvent-less preparation, characterization and degradation of the cationic dye over holmium oxide ceramic nanostructures

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Received 26 January 2015; received in revised form 28 January 2015; accepted 3 April 2015

Available online 14 April 2015

Abstract

Pure holmium oxide ceramic nanostructures were prepared via a new simple approach. Nanostructures were synthesized by heat treatment in air at 600 °C for 5 h, utilizing $[\text{Ho L}(\text{NO}_3)_2]\text{NO}_3$ ($\text{L} = \text{bis}-(2\text{-hydroxy-1-naphthaldehyde})\text{-butanediamine Schiff base ligand}$), as precursor, which was prepared via a solvent-free solid–solid reaction from different molar ratios of holmium nitrate and Schiff base ligand. The as-prepared nanostructures were characterized by field emission scanning electron microscopy (FESEM), thermo-gravimetric analysis (TGA), X-ray diffraction (XRD), energy dispersive X-ray microanalysis (EDX), transmission electron microscopy (TEM), UV–vis diffuse reflectance spectroscopy and Fourier transform infrared (FT-IR) spectroscopy. It was found that the calcination temperature and molar ratio of holmium nitrate and Schiff base ligand have significant and key effect on the morphology and particle size of the holmium oxide. To investigate the catalytic properties of as-obtained holmium oxide nanostructures, the photocatalytic degradation of rhodamine B as cationic dye under ultraviolet light irradiation was performed.

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Keywords: C. Optical properties; Ceramic; Holmium oxide; Nanostructures; Catalytic properties

1. Introduction

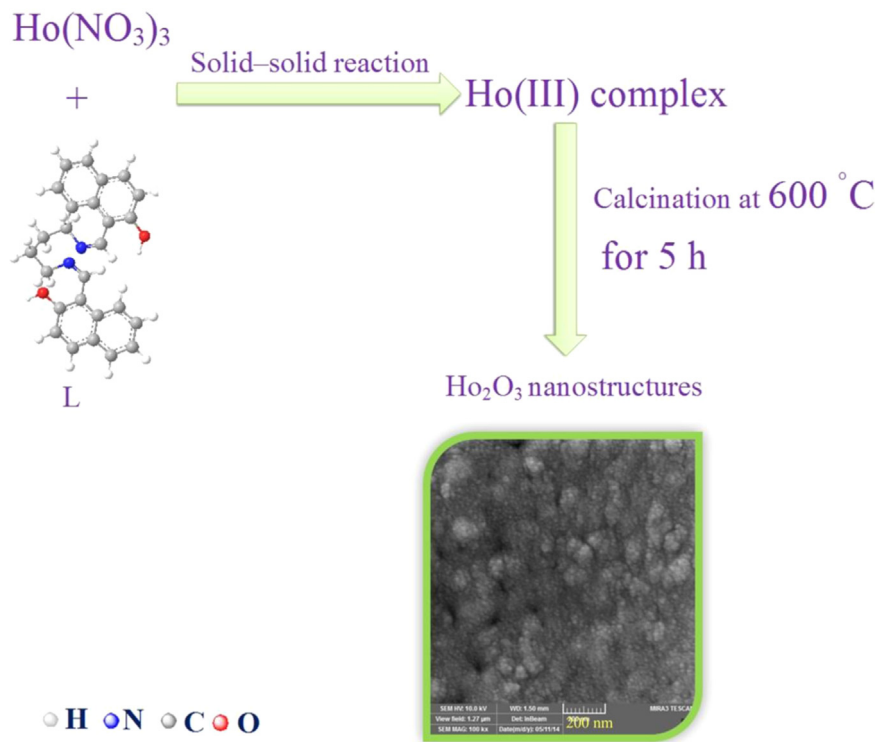
In recent years, the preparation of nanostructured materials has drawn tremendous attention owing to their wide range of interesting, significant and unique properties and as well as potential applications [1–5]. One of these nano-scale materials with specific and unique optical and electrical properties is holmium oxide (Ho_2O_3). Holmium oxide belongs to the family of rare earth metal oxide. It has been widely investigated owing to its several applications in the pyrolysis catalysts and wavelength-calibration instruments [6,7]. Up to now, holmium oxide has been prepared by the thermal decomposition of holmium nitrate at 660 °C [8], holmium carbonate and carbamide-containing complex of holmium at 700 °C [9,10] and oxalate at 740 °C [8].

Several nanostructured metal oxides have been prepared via the thermal decomposition of the organometallic molecular route as a proper and favorable synthetic method. This approach is well

known as versatile, reliable, facile and cost effective synthetic technique to the preparation of nanometer-scale materials with high purity and uniform morphology and does not need special apparatus, complicated procedures and severe reaction conditions, which can limit the large-scale production of the nanomaterials. On the other hand, solvent-free synthetic techniques have drawn tremendous attention in the field of green chemistry, and different solvent-less synthetic techniques for organic compounds have been developed. From the viewpoint of green chemistry, we succeeded in preparing rare earth metal complexes by solvent-free solid–solid reactions, and here we present the new preparation of $[\text{Ho L}(\text{NO}_3)_2]\text{NO}_3$ ($\text{L} = \text{bis}-(2\text{-hydroxy-1-naphthaldehyde})\text{-butanediamine Schiff base ligand}$) via facile solvent-free reactions utilizing different molar ratios of holmium nitrate and Schiff base ligand, and then $[\text{Ho L}(\text{NO}_3)_2]\text{NO}_3$ was used for synthesizing Ho_2O_3 ceramic nanostructures via a thermal treatment route (at lower temperature). To the best of our knowledge, investigations on the preparation of holmium oxide ceramic nanostructures by utilizing $[\text{Ho L}(\text{NO}_3)_2]\text{NO}_3$ have not been reported to date.

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Scheme 1. Schematic diagram of the preparation of Ho_2O_3 micro/nanostructures.

Table 1
Preparation conditions of holmium oxide samples.

Sample no.	Precursor	Molar ratio of Ho (NO_3) ₃ · 6H ₂ O/Schiff base ligand (L)	Calcination temperature (°C)	Figure of SEM images
1	Ho(III) complex	1:1	500	5a
2	Ho(III) complex	1:1	600	5b and 6b
3	Ho(III) complex	1:1	700	5c
4	Ho(III) complex	1:0.5	600	6a
5	Ho(III) complex	1:2	600	6c
6	Ho(III) complex	1:3	600	7a
7	Ho(III) complex	1:4	600	7b
8 ^a	Ho (NO_3) ₃ · 6H ₂ O	–	600	9

^aBlank test, in the absence of Schiff base ligand (L)

2. Materials and methods

2.1. Materials and characterization

In this investigation, 2-Hydroxy-1-naphthaldehyde, holmium nitrate ($\text{Ho}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$), 1,4-diaminobutane, methanol, ethyl acetate and chloroform, were bought from Merck

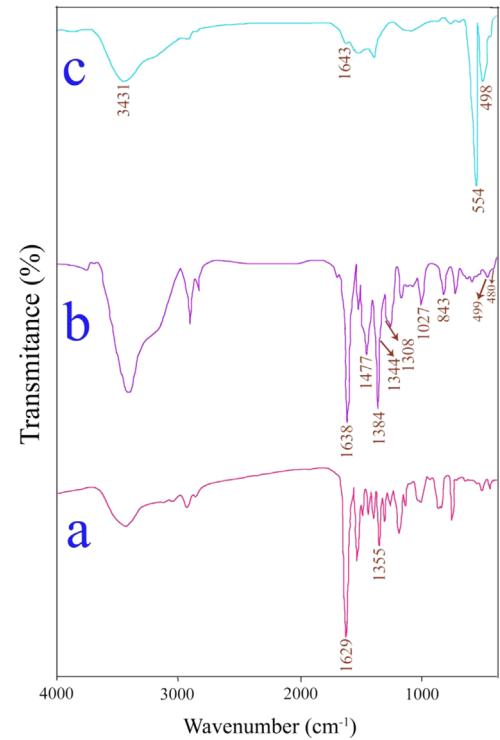


Fig. 1. FT-IR spectra of (a) Schiff base ligand (L), (b) Ho(III) complex and (c) Ho_2O_3 nanostructures obtained from Ho(III) complex in molar ratio of 1:1 by thermal treatment at 600 °C for 5 h.

Co. and were utilized as received. The electronic spectra of the Ho_2O_3 were obtained on a Scinco UV–vis scanning spectrometer (Model S-4100). Thermogravimetric-differential thermal

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