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Chapter 20

Synthesis and Characterization of Gadolinium Oxide Nanocrystallites

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Abstract

Lanthanide oxide nanocrystallites have gained a lot of attention due to their diverse use for potential applications and for this reason it is very important to find a suitable preparation method that would be economically inexpensive and easy to implement. The chapter describes the preparation of gadolinium oxide nanocrystallites (nano Gd_2O_3) through thermal decomposition of a complex formed by $Gd(NO_3)_3{\cdot}6\ H_2O$ and glycine. Decomposition of the complex occurs at temperatures about (250 ± 10)°C. An ultrafine white powder of the gadolinium oxide nanocrystallites was obtained. The resulting nanocrystallites were characterized by X-ray powder diffraction analysis, which revealed the size of the gadolinium oxide nanocrystallites equal to 10 nm. The morphology of the gadolinium oxide nanocrystallites was examined by scanning electron microscopy. The elemental composition of the product was confirmed by EDS analysis.

Keywords: thermal decomposition, nanocrystallites, gadolinium oxide, XRD, EDS, SEM

1. Introduction

Nanomaterials are defined as materials with at least one direction usually in the range of 1–100 nm [1] and because these materials have different physical, chemical, and electrical properties in comparison with traditional bulk materials, they may be used for new products and applications and may also be incorporated into various industrial processes [2].

Lanthanide oxides have gained a lot of attention due to their diverse use for applications such as in the nuclear industry, electronics, lasers, and optical materials [3]. Gadolinium oxide

(Gd_2O_3) is the most researched of all the lanthanide oxide. A great deal of interest in gadolinium oxide exists because of its physicochemical properties, such as the crystallographic stability up to temperatures of 2325°C, high mechanical strength, excellent thermal conductivity, and a wide band optical gap [4]. Generally, nanoparticles of lanthanide oxides can be prepared using a variety of methods, such as homogeneous precipitation [5], thermal decomposition [6], combustion method [7], microemulsion techniques [8], hydrothermal crystallization [9], spray pyrolysis [10], sol-gel [11], sonochemical methods [12], and other methods [13]. Most often nanocrystallites of lanthanide oxides are prepared through calcination methods using a suitable precursor [14].

The aim of this work was the preparation of gadolinium oxide nanocrystallites through a thermal decomposition method and their subsequent characterization using a combination of techniques.

2. Experimental

2.1. Synthesis of gadolinium oxide nanocrystallites

Gadolinium oxide nanocrystallites (nano Gd_2O_3) were prepared by the thermal decomposition [15] of the complex formed by the salt $Gd(NO_3)_3 \cdot 6\ H_2O$ and glycine. An aqueous solution of $Gd(NO_3)_3 \cdot 6\ H_2O$ and glycine (NH_2CH_2COOH) with a concentration of 0.5 $mol \cdot dm^{-3}$ were mixed. The resulting complex was dried at 120°C and calcined at 600°C for 1 hour. Decomposition of the complex occurred at about of (250 ± 10)°C. Other components of the complex evaporated in the form of the following gases: N_2, CO_2, and H_2O. The following scheme illustrates synthesis of samarium oxide nanocrystallites:

$$Gd(NO_3)_3 \bullet 6H_2O + NH_2CH_2COOH \xrightarrow[N_2, CO_2, H_2O]{600^\circ C,\ 1hod} Gd_2O_3 \quad (1)$$

2.2. Characterization of gadolinium oxide nanocrystallites

X-ray powder diffraction analysis was performed using the X-ray diffractometer Ultima IV Rigaku (Rigaku, Japan), operated at 40 kV and 40 mA with CuKα radiation (reflection mode, Bragg-Brentano arrangement, scintillation counter). The XRD patterns were recorded in the 10–70° 2θ range with a scanning rate of $2^\circ \cdot min^{-1}$. The samples were placed in a ground glass depression in the sample holder and flattened with a glass slide. X-ray beam was demarcated by 2/3° divergence, 10 mm divergent height limiting, 2/3° scattering, and 0.6 mm receiving slits. Phase analysis was evaluated by database PDF-2 Release 2011. Graphics processing XRD patterns was made using OriginPro8. The Gd_2O_3 reflection of the (222) plane was used to determine crystallite size using the Scherrer formula [16]

$$L_c = \frac{K \bullet \lambda}{\beta \bullet \cos\theta}, \quad (2)$$

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where K is the factor of microstructure, λ is the wavelength of radiation, β is the full-width at half-maximum (FWHM), and θ is the diffraction angle.

Scanning electron microscope MAIA3 GMU (TESCAN)—ultra-high resolution SEM with Schottky field emission cathode—was used for electron micrographs. Images were taken using a combination of InBeam SE + Low-Energy BSE detector at 2.5 kV. Furthermore, the product morphology was also observed by scanning electron microscope Quanta FEG (FEI), and EDS analysis was performed using the APOLLO X analyzer (EDAX).

3. Results and discussion

The thermal decomposition of the gadolinium salt and glycine produced a white powder of gadolinium oxide nanocrystallites.

The obtained XRD pattern in **Figure 1** shows the single phase of Gd_2O_3 (data from JCPDS file No. 03-065-3181) with cubic crystal structure. Four major reflections of Gd_2O_3 were observed and correspond to the (222), (400), (440), and (622) crystal lattice planes. Other smaller reflections were assigned to the (211) and (431) planes, respectively. The crystallite size of reflection (222) was about 10 nm.

EDS spectrum (**Figure 2**) confirmed the presence of gadolinium and oxygen. Gold present in the EDS pattern is caused by that coated with Au thin layer.

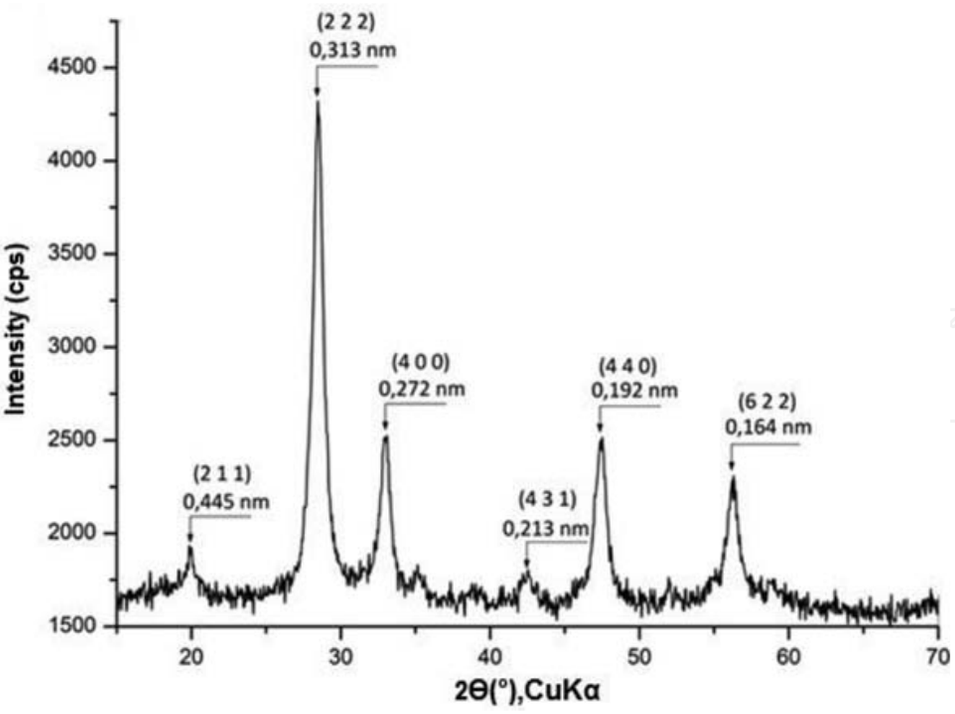

Figure 1. X-ray powder diffraction pattern of the prepared nanomaterial.

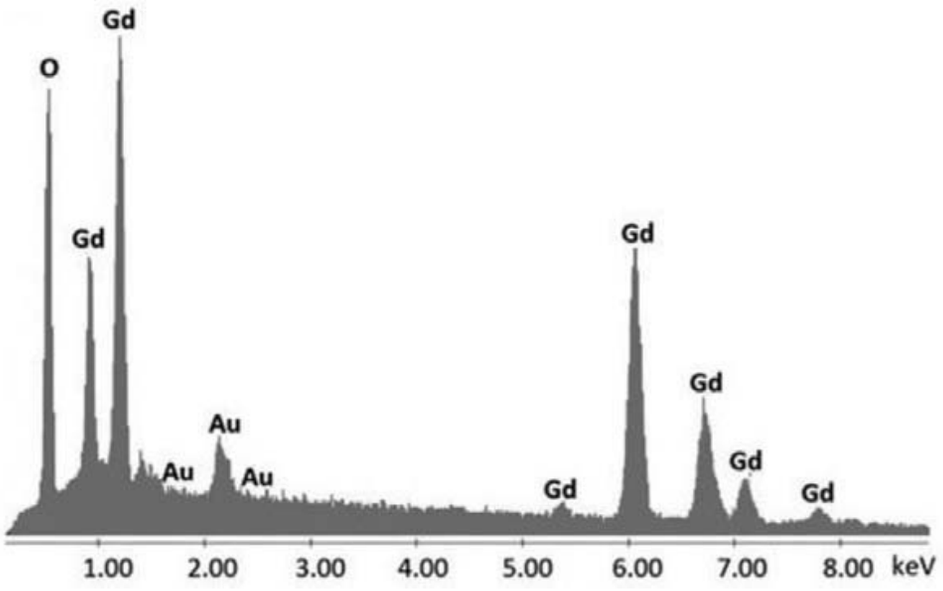

Figure 2. EDS spectrum of the sample.

The SEM images were taken in secondary electron modes (**Figure 3**). It can be seen that gadolinium oxide nanocrystals form aggregates which can be explained by the electrostatic forces. At lower magnifications, the network configuration of the material can be observed, and at the higher magnifications, it can be seen that the material appears as porous mousse with meso- and macropores.

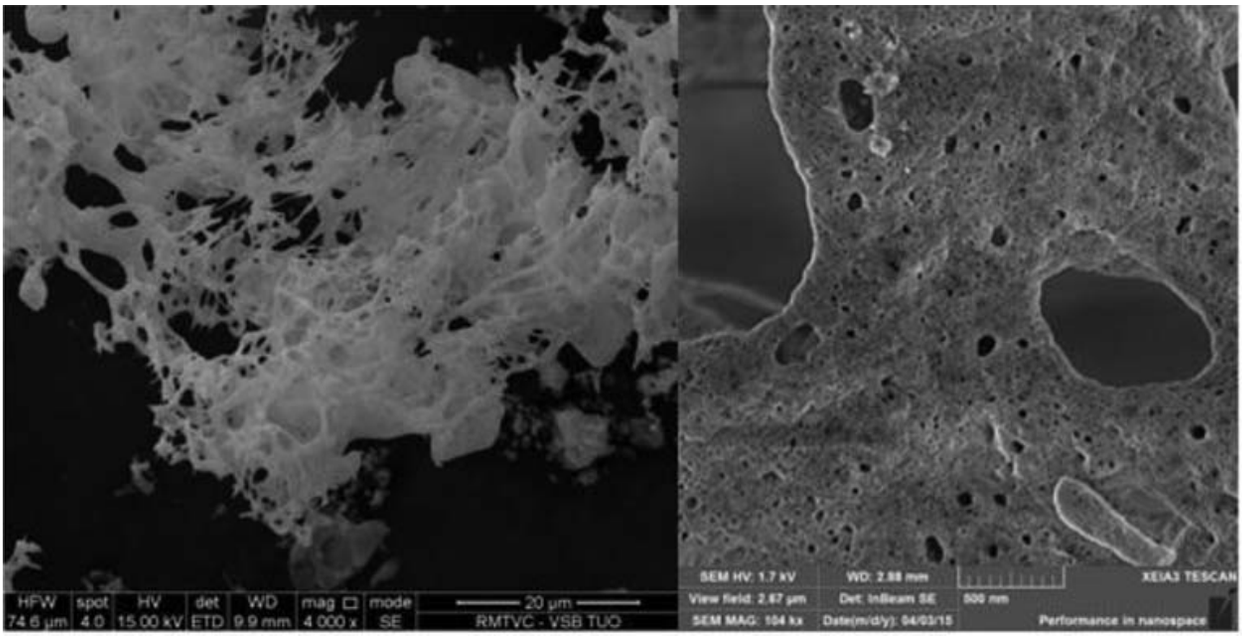

Figure 3. Examples of SEM images of the sample at different magnifications.

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4. Conclusions

Gadolinium oxide nanocrystallites with crystallite size of 10 nm were prepared by the thermal decomposition of $Gd(NO_3)_3 \cdot 6\ H_2O$ and glycine. Currently, increasing production and usage of nanocrystallites for various industrial applications may raise questions and concerns about their impact on human health and environment. Therefore, potential toxic effects of gadolinium oxide nanocrystallites prepared by the thermal decomposition method should be evaluated in future as well.

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