



Synthesis and characterization of GdVO₄ nanostructures by a tartaric acid-assisted sol–gel method

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Abstract

GdVO₄ nanoparticles were prepared by a sol–gel method employing tartaric acid as a chelating agent. Effects of calcination temperature and mole ratio of total metal ions to tartaric acid on the products were investigated. The products, characterized by XRD, FESEM and TEM, were tetragonal GdVO₄ nanoparticles with diameters of about 20–30 nm. TG and DSC data showed weight loss and phase formation due to evaporation and decomposition processes. FTIR presented the stretching band of VO₄³⁻ tetrahedrons and Raman analysis revealed the vibrational modes of the GdVO₄.

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1. Introduction

Gadolinium orthovanadate (GdVO₄) has been widely used as a catalyst [1] and host material for rare earth ions used in luminescent displays [2,3] because of its chemical stability and its high thermal conductivity. GdVO₄ also has large emission and absorption cross sections, suitable for laser materials [4,5]. Conventionally, GdVO₄ was prepared by a solid-state method [4–7] based on simply mixing of solid reagents. However, poor mixing of the starting materials could provide low reproducibility and high reaction temperature was needed in order to achieve a highly pure GdVO₄ powder. Sol–gel [2,3] and sonochemical methods [1,8] have been developed to prepare GdVO₄ nanostructures at a significantly lower temperature. These methods also provided a molecular level mixing of the starting materials, which led to better chemical homogeneity of the final products in small particle sizes and narrow size distribution.

In this study, a tartaric acid assisted sol–gel method was applied to synthesize the GdVO₄ nanoparticles. Tartaric acid used in the present research functioned as a chelating agent and additional fuel during the combustion of the precursors. The effects of calcination temperature and mole ratio of total metal ions to tartaric acid on purity, crystallinity and particle size of the GdVO₄ powder were investigated.

2. Experimental procedure

1.25 mmol of Gd(OAc)₃ (Sigma-Aldrich, Co., 99.9%) and 1.25 mmol of NH₄VO₃ (Ajax Finechem, 99.5%) were separately dissolved in deionized water and mixed into a homogeneous solution. Then, tartaric acid (Carlo Erba Reagent, 99.0%) was slowly added into the solution. After that, the solution was heated and stirred at 80 °C until a gel was formed. The gel was subsequently dried in an oven at 80 °C for 24 h to get tartrate precursor and, later, calcined at 300–700 °C for 3 h, resulting in yellow powders. To study the effect of the tartaric acid content, four powders were prepared using M:TA mole

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ratios of 1:1, 1:2, 1:3 and 1:4, following by the calcination at 500 °C.

Thermal decomposition behavior of the precursors was analyzed by thermogravimetry (TG, Perkin-Elmer TGA7) and differential scanning calorimetry (DSC, NETZSCH STA 409 PC/PG), with a heating rate of 10 °C min⁻¹ from room temperature to 800 °C under a flowing nitrogen atmosphere. The products were characterized by X-ray diffraction spectroscopy (XRD, Rigaku Miniflex II) with CuK α radiation ($\lambda=1.54059\text{\AA}$). Vibration modes of the powders were determined by Fourier transform infrared spectroscopy (FTIR, Bruker TENSOR27) and Raman spectroscopy (Jobin Yvon Horiba, T64000). Particle sizes and morphologies were determined by a field emission scanning electron microscope (FESEM, JEOL JSM-6335F) operated at 15 kV of accelerating voltage and transmission electron microscopy (TEM, JEOL JEM-2010) operating at 200 kV. Specific surface areas were measured by the Brunauer–Emmett–Teller method (BET, Autosorb 1 MP, Quantachrome).

3. Results and discussion

Fig. 1(a) shows the TG–DSC curves of the tartrate precursor (M:TA=1:1). The TG curve shows continuous weight loss, in

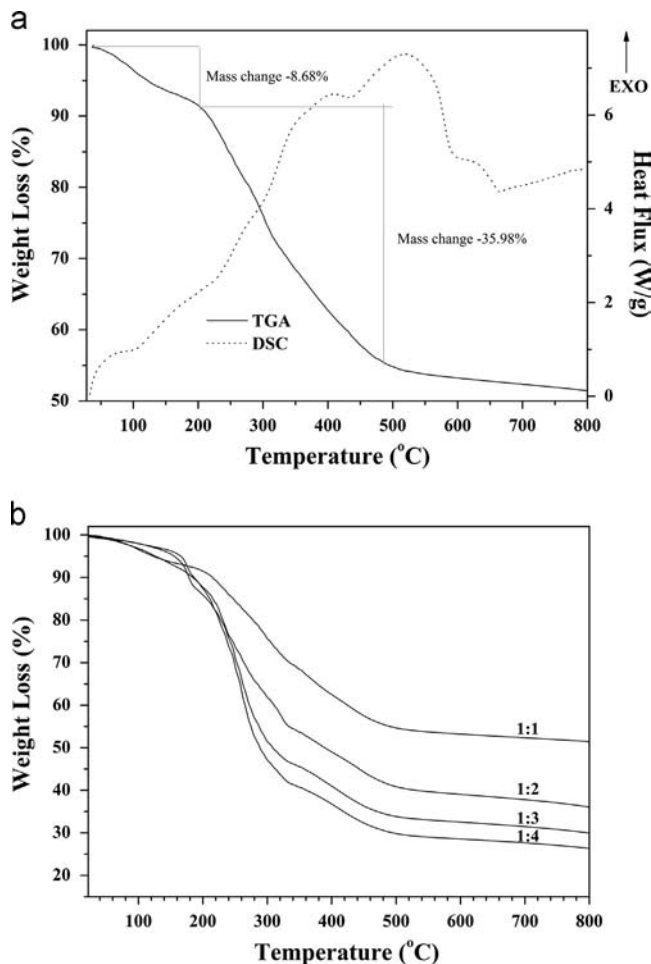


Fig. 1. (a) TG–DSC curves of the tartrate precursor (M:TA=1:1) and (b) TG curves of the tartrate precursors (M:TA=1:1–1:4).

three stages from ambient temperature to 800 °C. The first stage, from 25 °C to 180 °C conforming to a small endothermic peak at about 100 °C in the DSC curve, indicates the evaporation of surface-adsorbed water and the dehydration of hydroxyl groups [9,10]. The second stage, from 180 °C to 490 °C accompanied by an exothermic peak at about 400 °C in the DSC curve, is the decomposition/combustion of tartrate ions, acetate ions and other residual organic compounds [3,10]. The final stage, noticed between 490 °C and 600 °C as accompanied by a large exothermic peak at about 520 °C observed in the DSC curve, shows that the crystallization of the GdVO₄ took place. Fig. 1(b) shows the TG curves of the tartrate precursors at different M:TA mole ratios. It can be seen that the percentage of weight loss tended to increase with an increasing of the tartaric acid contents. The faster decomposition of the precursors with higher tartaric acid contents could be due to the self-igniting property of the carboxylic acid [11,12]. This reveals that the acid functioned as a fuel supplier

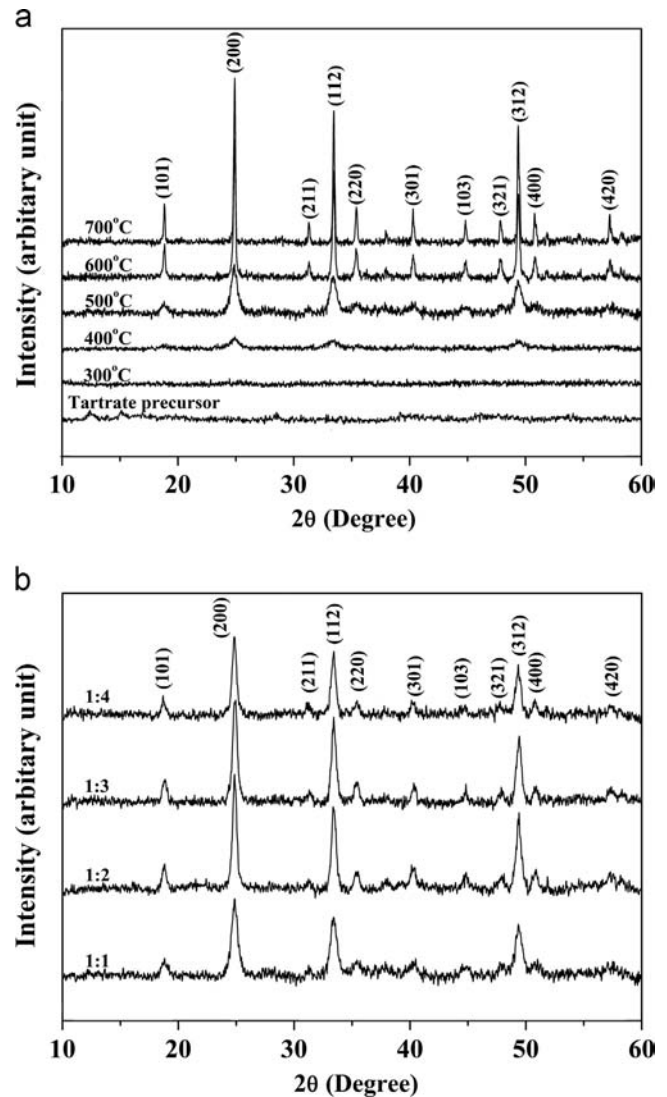


Fig. 2. XRD spectra of (a) the tartrate precursor (M:TA=1:1) and the precursors calcined at different temperatures and (b) the powders prepared using M:TA=1:1–1:4.

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