



Novel poly(ethyleneglycol)-assisted synthesis of praseodymium oxide nanostructures via a facile precipitation route

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Abstract

The effect of the polymeric capping agent on the morphology and size of praseodymium oxide precipitated from praseodymium nitrate in the presence of ethylenediamine (en) as precipitator was investigated. En was used as precipitator to fabricate praseodymium oxide nanostructures for the first time. The products have been successfully synthesized in the presence of poly ethylene glycol (PEG) as capping agent via an improved precipitation route. The effect of amount of en on the morphology and size of praseodymium oxide was investigated. It was found that morphology and size of the products could be greatly affected by this parameter. The as-produced nanostructures were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), ultraviolet–visible (UV–vis) spectroscopy, photoluminescence (PL) spectroscopy, Fourier transform infrared (FT-IR) spectroscopy, and energy dispersive X-ray microanalysis (EDX). To investigate the catalytic properties of praseodymium oxide nanostructures, the photocatalytic degradation of 2-naphthol was performed.

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1. Introduction

In the past decade, much attention has been focused on the preparation and characterization of nanomaterials owing to their interesting properties and potential applications [1–4]. One of these nanometer-scale materials is praseodymium oxide. It was found that praseodymium oxide is an important rare earth metal oxide because of its specific optical and electrical properties [5,6] as well as potential applications, including ceramic pigments, catalysts, promoters and stabilizers in combustion catalysts, oxygen-storage components, and materials with higher electrical conductivity [7–11]. Praseodymium oxide forms a homologous series with a number of stoichiometrically defined oxides: $\text{Pr}_n\text{O}_{2n-2}$, with $n=4, 7, 9,$

$10, 11, 12, \dots$ Among these oxides, Pr_6O_{11} is the most stable form at ambient temperature and pressure [12]. Up to now, very limited number of methods have been employed to synthesize praseodymium oxide nanostructures, such as, thermal decomposition [13], molten salt [14], hydrothermal [15], electrochemical [16], precipitation [12], and electrospinning [17]. The development of a simple effective route for synthesizing Pr_6O_{11} nanostructures is of great importance to the potential investigations of its physical and chemical properties. It is good to know that properties of nanomaterials depend on their particle size and shape [18–20]. Therefore, exploring suitable methods to synthesize praseodymium oxide nanostructures and controlling their particle morphology and size is important.

Herein, Pr_6O_{11} nanostructures are synthesized via a simple precipitation route in the presence of PEG and ethylenediamine (en). The precipitation method is an appropriate synthesis process for preparation of many inorganic nanomaterials. This method is facile, convenient and cost effective synthetic

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procedure and provides an effective way to the fabrication of uniform nanostructures. To our knowledge, it is the first time that en is utilized as precipitator for the synthesis of praseodymium oxide nanostructures and the effect of its concentration on the morphology and size of praseodymium oxide via a facile precipitation method in the presence of PEG is investigated.

2. Materials and methods

2.1. Materials and characterization

In this investigation, praseodymium nitrate ($\text{Pr}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$) (PN) as praseodymium precursor, ethylenediamine (en), liquor ammonia solution containing 25% ammonia, poly ethylene glycol 600 (PEG 600) were purchased from Merck Company and were used without further purification. Fourier transform infrared spectra were recorded using KBr pellets on an FT-IR spectrometer (Magna-IR, 550 Nicolet) in the 400–4000 cm^{-1} range. Powder X-ray diffraction (XRD) patterns were collected with a Philips diffractometer using X'PertPro and the monochromatized $\text{Cu K}\alpha$ radiation ($\lambda=1.54 \text{ \AA}$). Microscopic morphology of products was visualized by a Hitachi s4160 Japan scanning electron microscope (SEM). The energy dispersive spectrometry (EDS) analysis was studied by XL30, Philips microscope. Transmission electron microscope (TEM) images were obtained on a JEM-2100 with an accelerating voltage of 60–200 kV equipped with a high resolution CCD Camera. Room temperature photoluminescence (PL) was studied on a Perkin Elmer (LS 55) fluorescence spectrophotometer. The electronic spectra of the samples were taken on a Scinco UV–vis scanning spectrometer (Model S-4100).

2.2. Synthesis of praseodymium oxide nanostructures

Praseodymium oxide nanostructures were prepared by simple precipitation method. In a typical procedure, 1 mmol of en was dissolved in 20 ml of distilled water and then was drop-wise added to 20 ml solution containing 1 mmol of PN

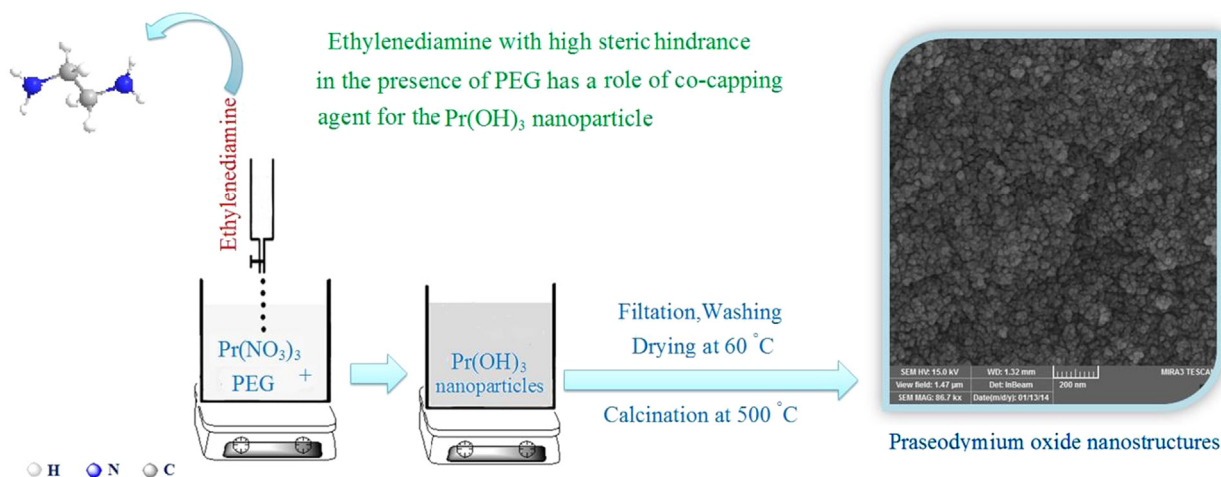
and 3 mmol of PEG under magnetic stirring for 10 min. The green gel-like product was filtered and washed with distilled water for three times. The final product was dried at 60 °C and calcined at 500 °C for 3 h (sample 1). Schematic diagram of formation of Pr_6O_{11} nanostructures is depicted in Scheme 1. The experiment was carried out by using 2, 3, 4, and 5 mmol of en at the same conditions. To investigate the effect of en, a blank test was performed by NH_4OH instead of en. In the blank test, 8 mmol of ammonia dissolved in 20 ml distilled water and was added into a solution including 1 mmol of PN and 3 mmol of PEG dissolved in 50 ml of distilled water. To study the effect of PEG, one experiment was carried out without PEG (blank test 1). The as-synthesized products were characterized by FT-IR, SEM, TEM, XRD, EDS, PL and UV–vis techniques.

2.3. Photocatalytic measurements

The photocatalytic activity of praseodymium oxide nanostructures obtained from sample no. 4 was tested by using 2-naphthol solution. The degradation reaction was performed in a quartz photocatalytic reactor. The photocatalytic degradation was carried out with 0.0012 g of 2-naphthol solution containing 0.05 g of Pr_6O_{11} . This mixture was aerated for 30 min to reach adsorption equilibrium. Then, the mixture was placed inside the photoreactor in which the vessel was 40 cm away from the UV source of 400 W mercury lamps. The quartz vessel and light source were placed inside a black box equipped with a fan to prevent UV leakage. The experiment was performed at room temperature. Aliquots of the mixture were taken at periodic intervals during the irradiation, and after centrifugation they were analyzed with the UV–vis spectrometer. The 2-naphthol degradation percentage was calculated by Eq. (1) as follows:

$$D.P. (t) = \frac{A_0 - A_t}{A_0} \times 100 \quad (1)$$

where A_0 and A_t are the absorbance value of solution at 0 and t min, respectively.



Scheme 1. Schematic diagram of the synthesis of praseodymium oxide nanostructures.

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