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Influence of propyl alcohol addition during processing of molybdenum trioxide powders: Observation of novel dual-wavelength excitation photochromism

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ABSTRACT

The novel dual-wavelength excitation photochromic molybdenum trioxide (MoO_3) powders are fabricated successfully via the hydrothermal method by the addition of propyl alcohol. The samples were fully characterized by means of X-ray diffraction (XRD), scanning electron microscopy (SEM), ultraviolet–visible diffuse reflectance spectroscopy (UV–vis), and colorimetry. The characteristic spectrum of XRD demonstrates that the as-obtained products are highly pure. The results show that the flowerlike MoO_3 sample with the inducer of propyl alcohol has better photochromic properties, which was analyzed by colorimetry. It is shown that both of the samples exhibit a strong adsorption band between 250 and 400 nm in the UV range. Moreover, the UV–vis spectra shows that MoO_3 synthesized with added propyl alcohol absorbs light not only between 250 and 400 nm but also displays an additional band between 500 and 800 nm, demonstrating novel dual-wavelength excitation photochromic properties.

1. Introduction

With distinctive properties of electrochromism, thermochromism and photochromism as a smart material, gas sensor, catalyst and a host material for intercalation, molybdenum trioxide (MoO_3) has been extensively investigated over the past decades [1–5]. As a wide bandgap n-type semiconductor material, molybdenum trioxide has received considerable attention over the last few years because of its many applications [1]. As an example, molybdenum trioxide (MoO_3) is widely used in industry in catalysts, display devices, sensors, smart windows, lubricants, battery electrodes, and nanostructured materials in latest context [6]. Over the last few years, it has been made rapid progress in the development of single-wavelength excitation photochromic MoO_3. In fact, MoO_3 with the general single-wavelength excitation photochromic properties only has one absorption band in the UV–vis range [7–10]. However, as we know, the properties of samples by multi-wavelength excitation can be enhanced compared with materials by single-wavelength excitation [11–13]. Multi-wavelength materials with constant wavelength spacing have attracted extensive interest for many years, because of their wide potential applications in spectroscopy, optical communication [14]. So when the photochromic materials have the multi-wavelength photochromic responses, they can be perfectly used in optical information storage, display device, defense stealth. Actually, how to prepare MoO_3 with polychrome photochromic properties is more and more important. Up to now, the research on the photochromic properties of MoO_3 by multi-wavelength excitation has almost never been reported before. Therefore, the study on dual-wavelength

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excitation photochromic MoO_3 powders is very novel. Advantages of the hydrothermal method include simplicity, low cost, and flexibility with respect to substrate shape and size [15]. Organic inducer in the reaction system not only determined their growth style, but also influenced the crystal structure of samples [16]. Accordingly, the organic inducer is probably to be a good way in preparing MoO_3 with different structures and special properties.

In the present work, we reported a mild hydrothermal approach to synthesis the novel dual-wavelength excitation photochromic MoO_3 powders induced by propyl alcohol. The analysis results showed that when propyl alcohol was assisted, the as-synthesized sample had the novel dual-wavelength excitation photochromic properties.

2. Experimental details

2.1. Hydrothermal synthesis of MoO_3 powders

All chemical reagents were of analytic purity and used directly without further purification. The experimental procedure was designed as follows. In detail, the sodium molybdate powder was dissolved in deionized water to get a transparent solution of 0.20 M and hydrochloric acid was added to the solution adjusting pH to 1.0. After stirring for 4 h, the system was dissolved in variable amounts of propyl alcohol. The amounts of propyl alcohol were 0, 10, and 20 ml labeled S1, S2, and S3 respectively. The resulting solution was transferred into a 100 ml stainless steel autoclave. Hydrothermal reaction was carried out at 120 °C for 48 h. After the hot autoclave was cooled naturally, the product was washed with deionized water and ethanol for several times to remove any possible residue, and finally dried in vacuum.

2.2. Characterization of MoO_3 samples

The crystal phase of as–prepared powders were determined by X–ray diffraction (XRD, Philips Dy 2198X, Holland) with a Cu-Kα radiation of 1.541 Å in wavelength and settings of 40 mA and 40 kV at a scanning rate of 0.02°/s in the 2θ range from 10° to 70°. The surface morphologies of the samples were investigated by scanning electron microscopy (SEM, JSM–5610LV, Japan, 20KV). Furthermore, optical properties were characterized by ultraviolet–visible diffuse reflectance spectroscopy (UV–vis, Shimadzu UV-2550, Japan) with $BaSO_4$ as the baseline correction. And the photochromic properties of the powders were tested by a color difference meter (SC-80C, Beijing Kangguang Instruments Ltd.).

2.3. Test for photochromism

The photochromic properties of the powder samples were tested by color difference meter (SC-80C, Beijing Kangguang Instruments Ltd.), the colors of which were characterized after being irradiated under a mercury lamp (10 cm away from the samples, $\lambda = 365$ nm, 3 W). Then CIE Lab uniform color space for color sheet system was used to figure out the color difference. That is, find out the tristimulus values firstly when color matched. Secondly, work out the three-dimensional rectangular coordinates L^*, a^*, b^*, that any point of the color space represents a color, and the geometric distance between two points represents the color difference of two colors; L^* refers to the transparency index while a^* and b^* refer to the chroma indexes. Finally, calculate the color difference according to the formula below (Eq. (1)):

$$\Delta C = \{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\}^{1/2} \quad (1)$$

ΔC shows the color difference of the corresponding powders before and after being irradiated, that is, the photochromic properties were evaluated by the color difference [17].

3. Results and discussion

The crystalline structure and phase purity of the samples were confirmed by XRD. As shown in Fig. 1, all observed diffraction peaks can be systematically indexed to those of the hexagonal phase of MoO_3, which are in good agreement with the values of standard card (JCPDS card no. 21-0569). No peaks from other impurities are detected in the XRD patterns, indicating that the samples were highly crystalline. At the same time, it can be seen that the relative intensity of peaks of propyl alcohol-induced product is higher than the non-induced powder sample, which reveals that the propyl alcohol-induced sample crystallizes better than the non-induced one.

The surface morphologies of the MoO_3 powders obtained in the presence of different amounts of propyl alcohol are presented in Fig. 2. It is clearly observed that the organic inducer propyl alcohol strongly influenced the morphology of MoO_3 powder. The as-synthesized MoO_3 sample without the inducer of propyl alcohol consists of irregular blocky structures shown in Fig. 2a and b. When the amount of propyl alcohol is chosen as 10 ml (sample S2), the as-prepared MoO_3 powder is composed of some blocky structures and flowerlike structures shown in Fig. 2c and d. As shown in Fig. 2e and f, the MoO_3 powder prepared with 20 ml propyl alcohol has transformed into

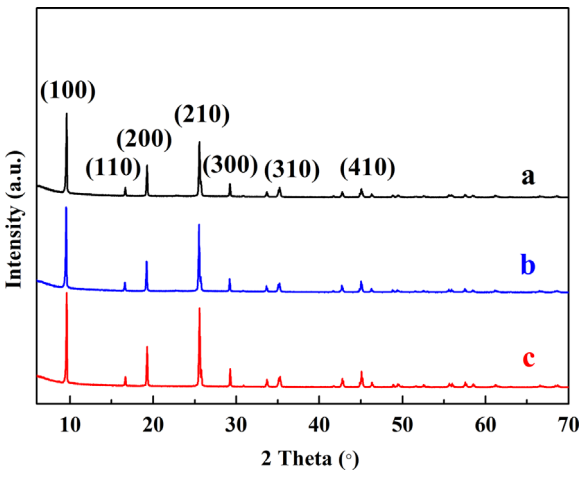

Fig. 1. XRD patterns of MoO_3 samples; (a) without propyl alcohol, (b) with 10 ml propyl alcohol and (c) with 20 ml propyl alcohol.