Effect of hydrothermal temperature on structure and photochromic properties of WO₃ powder

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Article history:
Received 29 June 2010
Received in revised form 19 January 2011
Accepted 14 February 2011
Available online 24 February 2011

Keywords:
WO₃ powder
Photochromism
Hydrothermal temperature
Structure

Abstract
Tungsten trioxide (WO₃) powders were prepared via a simple hydrothermal method. The morphology, structure and photochromic activity of the synthesized WO₃ powders were studied by X-ray diffraction, scanning electron microscopy and UV–vis spectrophotometer combined with color difference meter. The results showed the synthesized WO₃ powders with hexagonal phase got much better photochromic properties than the WO₃ powders with cubic phase, the ones not appear until about 160°C. Besides, the WO₃ powder synthesized at 120°C exhibited the best photochromic properties of the samples prepared below 160°C, the particles of which formed a shape of clusters of cactus with uniform size and good dispersion.

1. Introduction
Tungsten trioxide (WO₃) is a hexagonal or cubic symmetrical-structured n-type semiconductor, which has been noted for its outstanding electrochromic, photochromic and photo-catalytic properties [1, 2]. Since its photochromism was discovered by Deb [3] in 1973, the preparation, structure and properties of WO₃ have been extensively studied because of its promising applications in information display devices and high-sensitivity optical storage materials as well as MoO₃ [4–11]. Nowadays the study on WO₃ photochromism has been mainly focused on WO₃ sol and film [12, 13], especially on photochromic WO₃ film’s regulation of solar input to buildings in smart window [14, 15]. However, much less attention has been paid so far to the photochromism of WO₃ powder except for its preparation and structure [16]. In the rare studies on WO₃ powder, strictly-required excitation source (a 350 W high pressure mercury lamp [17]) has greatly restricted its actual applications.

Herein the effect of reaction temperature on the structure and photochromic properties of the WO₃ powder has been analyzed, which was synthesized via hydrothermal method. The results show that the product was transforming from hexagonal phase, the one revealing better photochromic properties, to cubic phase along with the reaction temperature rising. At 120°C, well grown and dispersed WO₃ powder was obtained, whose special morphology endowed it the best photochromic properties of all the samples with hexagonal phase.

2. Experimental

2.1. Synthesis of WO₃ powder
All reagents were analytical grade and used as raw materials without further purification. The sodium tungstate solution of 1.0 mol/L was acidified to a pH of 1.0 using concentrated hydrochloric acid, and then poured into a 100 mL autoclave after being stirred and homogenized for 2 h, in which the hydrothermal reactions went on for 2 days. Then the precipitates were filtered and washed sequentially with distilled water and ethanol, and finally dried in a vacuum oven at 80°C. Each set of experiment was conducted in terms of such procedures, but with different hydrothermal temperatures (80, 100, 120, 140, 160 and 180°C).

2.2. Characterization
The morphology and structure of the samples were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). XRD analysis was conducted using Dy2198 X-ray diffractometer under the measurement conditions of Cu Kα radiation, Ni filter, pipe pressure of 40 kV and electric current of 40 mA; and SEM was carried out using Quanta 200 environmental scanning electron microscope. Furthermore, the UV2550 ultraviolet-visible spectrophotometer (UV–vis) was used, with BaSO₄ as the baseline correction.