Microwave hydrothermal synthesis of AgInS₂ with high visible light photocatalytic activity

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Introduction

In recent years, $AgInS_2$ has aroused extensive attention because of the photovoltaic and optoelectronic applications of I–III– VI_2 ternary compounds [1]. And ternary $AgInS_2$ has absorption of visible-light, which is expected to have photocatalytic activity. $AgInS_2$ has been synthesized through a lot of methods. However, these methods always involve high processing temperature, long reaction period [2], complicated pretreatment processes [3], and special surfactant or solvent. Microwave synthesis, well known to be efficient and time saving, represents a great breakthrough in synthetic chemistry methodology. Herein, we have successfully synthesized pure phase $AgInS_2$ by microwave hydrothermal method, which is simple pretreatment and only costs minutes to obtain final sample. Besides, in this paper, $AgInS_2$ is firstly used for visible light photocatalytic degradation of environmental pollutants, and presents good activity toward Congo Red.

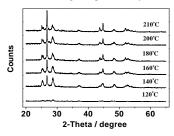
Materials and Methods

All chemicals were analytical grade and used as received without further purification. 0.5 mmol Ag_2SO_4 , 0.5 mmol $InCl_3\cdot 4H_2O$, and an excess of thioacetamide were dissolved in 15 ml distilled water. Then 0.1 mol/L HCl was used for adjusting the solution pH 4. This solution was added into a glass vessel of 35 ml capacity and maintained at different temperatures for total 35 min using a microwave system (Explorer48, CEM Co.). The sample was characterized by X-ray diffraction (XRD, Bruker D8 Advance), UV-vis diffuse reflectance spectroscopy (DRS, Varian Cary 500), and high resolution transmission electron microscopy (HRTEM, JEOL model JEM 2010 EX).

Results and Discussion

As showed in Fig. 1, the XRD patterns of the samples prepared at 140, 160, 180, and 200 °C had similar shapes and were all in agreement with the standard (JCPDS No. 025-1328). For the sample synthesized at 210 °C, a small peak at about 30.5 degree appeared which demonstrated the sample was impure. The peaks of the sample synthesized at 120 °C were so weak that they could not be recognized compared with other samples. Fig. 2 showed DRS of AgInS₂ synthesized at different temperatures. The band gaps varied from 1.44 (140 °C) to 1.63 (200 °C) eV as the temperature rising. Fig. 3 was the HRTEM image of the AgInS₂ sample synthesized at 200 °C. The lattice interplanar spacing was measured to be 0.333 and 0.315 nm, corresponding to the (002) and (121) plane of AgInS₂, respectively. We investigated the photocatalytic activities of pure AgInS₂ samples toward Congo Red (CR). When the CR solution (10 ppm, 80 ml) was irradiated without catalyst by the light (420 nm < λ < 800 nm), the concentration of characteristic absorption features of an aqueous CR did not decline with the time increasing, indicating no degradation occurred. In the dark experiment, after the CR solution with catalyst was stirring for 1 hour, adsorption-desorption equilibrium was

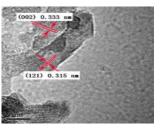
established, and the concentration of CR solution also didn't decrease with the subsequent illumination. While illumination for the dispersions of the samples (0.04 g) in a 6 hours period all led to a continued diminution of CR concentration in the solution bulk. This confirmed the photodegradation of CR. The results also showed the sample prepared at 160 °C had the best photocatalytic activity, while that of 140 °C displayed the worst. Further work for the mechanism of AgInS₂ photocatalytic activity is now being explored in our group.



E 140°C 180°C 200°C 200°

Figure 1. The XRD patterns of AgInS₂ prepared at different temperatures.

Figure 2. DRS of AgInS₂ prepared at different temperatures. The insert showed the plots of $(ahv)^2$ vs. hv.



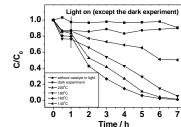


Figure 3. High-resolution TEM images of the synthesized AgInS₂ prepared at 200 $^{\circ}$ C.

Figure 4. Visible light photocatalytic activities of AgInS₂ prepared at different temperatures for degradation Congo red.

Significance

Exploiting efficient visible light active and easy synthesized photocatalysts for degradation of environment pollutants is the top goal at present. The easy and rapid synthesized AgInS₂ showed high photocatalytic activity under visible light by degradation of CR.

Reference

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