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Microwave Synthesis of Ce/BiVO₄ Nanocomposites Photocatalyst and Their Photocatalytic Properties

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Abstract

Ce/BiVO₄ nanocomposites photocatalyst was synthesized by direct feeding microwave synthesis method, using bismuth nitrate (Bi (NO₃)₃·5H₂O), cerium nitrate hexahydrate (Ce (NO₃)₃·6H₂O) and ammonium metavanadate (NH₄VO₃) as raw material and sodium dodecyl sulfate (SDS) as surfactant. The X-ray diffractometer (XRD) and the scanning electron microscopy (SEM) technology were used to characterize the Ce/BiVO₄ nanocomposites. We investigated the photocatalytic activity of the as-prepared photocatalyst, and methyl orange was used as organic pollutant. The results show that the Ce/BiVO₄ nanocomposite was a good photocatalyst under visible light. In 100 ml of 5 mg/L methylene orange solution, when the catalyst calcined at 673 K was 0.1 g, hydrogen peroxide was 0.5 ml, pH was 2.0, and the degradation ratio of catalyst for methylene orange reached 90.26% within 70 min.

Keywords

 ${\rm Ce/BiVO_4}$ Nanocomposites, Microwave Synthesis, Photocatalytic, Methyl Orange

1. Introduction

In recent years, haze, land pollution, water pollution and other environmental problems are becoming more and more frequent, which have been a serious threat to our survival, so we have to solve these environmental problems.

The treatment of organic wastewater is one of the hot spots. By photocatalytic degradation of organic matter and making it a harmless inorganic small molecule, it is a good green way to discharge. How to find a good photocatalyst, espe-

cially the use of visible light or direct use of sunlight, is the focus of research. The vanadate of nanoscale is a good photocatalyst. Because of its narrow band gap, it can be used for photocatalytic degradation of organic compounds in the visible region. In particular, it can directly use sunlight, the photocatalytic degradation of organic matter [1]-[16].

In this paper, Ce/BiVO₄ nanocomposites photocatalyst is prepared by the method of direct feeding microwave synthesis. The samples were characterized by XRD and SEM technology. The methyl orange was used as the reactant to evaluate the photocatalytic activity of the photocatalyst.

2. The Experiment

2.1. Instruments and Reagents

Microwave oven with 650 W (Sanle General Electric Corp. Nanjing, China) was used to synthesize samples. XRD were collected on a Shimadzu XRD-6100 X-ray diffractometer (Cu K α radiation, λ = 0.15418 nm). The morphology and size were characterized by SEM. he SEM images were obtained on a Quanta 200 FEG field emission scanning electron microscope. Ultraviolet-visible diffuse reflectance spectrascopy was carried out on a ShimazuUV-2600 UV-visible spectrophotometer. Lambda10 UV-vis spectrometer (Perkin-Elme Corp, USA) was used for measuring the absorption spectra on photo-degradation of methyl orange.

All the reagents were analytical purity without further purification. Ultra pure water was used throughout the experiments.

2.2. Direct Feeding Microwave Synthesis of Ce/BiVO₄ Nanocomposites

2.17 g of Ce(NO₃)₃·6H₂O, and 2.42 g of Bi(NO₃)₃·5H₂O were weighed and dissolved in 50 ml of 1 mol/L nitric acid solution, and mixed solution a was obtained. 1.17 g of NH₄VO₃ was weighed and dissolved in 10 ml of a solution containing 1 mol/L NH₃·H₂O, ultra pure water was added to 50 ml, and 1.0 g of SDS was added to form a solution b by ultrasonic dispersion. The A, B solutions were mixed rapidly transferred into 250 mL of flask, with 40% power (total power constant, 30 s work cycle, the work 12 s, stop 18 s) microwave irradiation 20 min, cool to room temperature, high speed centrifugal separation, washed with ultra pure water. The precipitate was transferred to a small beaker with acetone, at 60°C vacuum drying for 4 h, and then placed in a muffle furnace and heat treated 2 h at different temperatures. Products collected for characterization and photocatalytic experiments.

2.3. Photocatalytic Experiment

Firstly, in 100 ml of 5 mg/L methyl orange solution, the pH was adjusted to 2.0 with dilute nitric acid. A certain amount of $Ce/BiVO_4$ nanocomposites and 0.5 ml of 30% hydrogen peroxide were added, and the dispersion was uniform by ultrasonic. It was placed in the dark for 0.5 hours to reach adsorption equili-

brium. Then, it was exposed to visible light for photocatalytic degradation experiments. A certain time interval, centrifuge separation, takes the supernatant to mensurate the absorbance. Finally, according to the change in absorbance to calculate the degradation rate of the solution, the calculation formula is as follows: $D_t\% = (A_0 - A_t)/A_0 \times 100\%$, A_0 and A_t is the absorbance of methyl orange before and after degradation, respectively.

3. Results and Discussion

Figure 1 is a XRD pattern of the sample. It Contains tetragonal zircon crystal phase CeVO₄ (JCPDF NO.12-0757) and tetragonal crystal phase BiVO₄ (JCPDF NO.83-1812). It can be seen that the larger the heat treatment temperature of the sample, the sharper the peak. When the calcination temperature is increased to 573 K, the peak of sample has a very sharp, indicating the crystal growth has been very complete. According to Scherrer equation (D = $0.89 \lambda/B\cos\theta$), we can estimate the size of the crystal. When heat treatment temperature of sample is 573, 673, 773 K, the average size of as-prepared sample is 20, 32.2, 40 nm, respectively.

Figure 2 is SEM image of as-prepared sample. It shows that the majority of the catalyst is a relatively nanoparticle, however, there exists obvious aggregation.

Figure 3 is the sample of UV visible diffuse reflectance spectrum. It can be seen that the reflectivity of 200 - 400 nm is below 5%, and the reflectivity at 400 - 600 nm is near 20% and the reflectivity increases rapidly at 600 nm. Ce/BiVO₄ nanocomposites have strong absorption in the range of visible light, which is a good photocatalyst.

Figure 4 shows the UV-Vis absorption spectra of photo-degradation of methyl orange. The 0.1~g Ce/BiVO₄ nanocomposite calcined at 673 k was added in the methyl orange solution of 100 mL with a concentration of 5 mg/L and degraded in the sunlight. It can be seen within 70 min, the degradation rate of

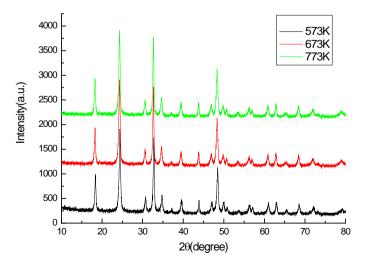


Figure 1. XRD patterns of as-prepared sample.

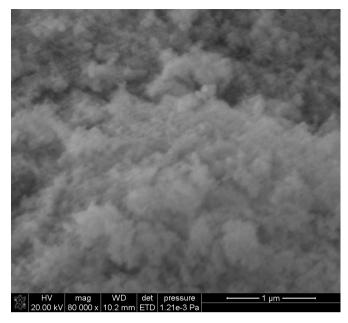


Figure 2. SEM image of as-prepared sample.

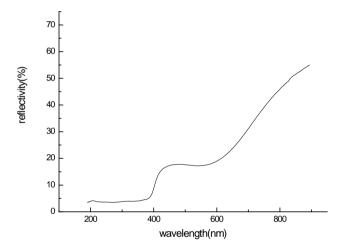


Figure 3. UV-Vis diffuse reflectance spectrum of Ce/BiVO₄ nanocomposite.

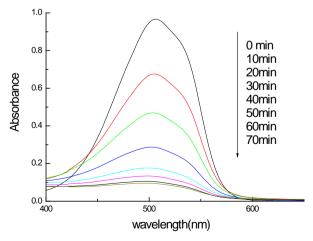


Figure 4. UV-Vis absorption spectra of photo-degradation of methyl orange.

methyl orange solution reach 90.26%. It shows that the as-prepared catalyst has good photocatalytic performance.

We also investigated the influence factors of the photocatalytic performance, such as heat treatment temperature of the catalyst (**Figure 5**), the load of the catalyst (**Figure 6**). The results show that when the heat treatment temperature is 673 K, the load of the catalyst is about 1.5 g/L, and the photocatalytic activity of the catalyst is the best.

4. Conclusion

Using a direct feed microwave synthesis method, Ce/BiVO $_4$ nanocomposites have been synthesized. The results show that the Ce/BiVO $_4$ nanocomposite has excellent photocatalytic performance. In the methyl orange solution of 100 mL with a concentration of 5 mg/L, adding 0.1 g of catalyst, adjusting pH to 2, adding 0.5 mL of 30% $\rm H_2O_2$, the degradation rate could reach 90.26% in 70 min under sunlight.

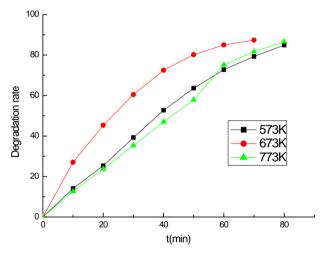


Figure 5. Affect of different heat treatment temperature on photocatalytic activity.

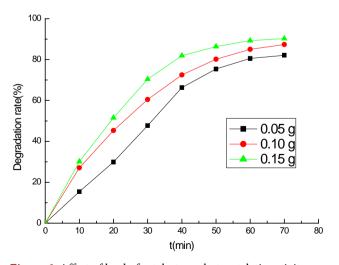


Figure 6. Affect of load of catalyst on photocatalytic activity.

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