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Short communication

Preparation and photocatalytic properties of zinc oxide nanoparticles by microwave-assisted ball milling

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Abstract

Hexagonal wurtzite ZnO nanoparticles with the average size of about 15 nm were successfully prepared by microwave-assisted ball milling. The as-prepared ZnO nanocrystals were characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), UV–visible spectrophotometer, fluorescence measurements, and electroconductivity detections. The results showed that single-phase hexagonal wurtzite ZnO nanoparticles were obtained after 20 h by microwave-assisted ball milling. The UV spectrophotometer analysis confirms that photocatalytic activity of as-synthesized ZnO nanoparticles under ultraviolet light is demonstrated via methyl orange degradations of 95%. In addition, the measurement results of the relative content of OH radicals in the aqueous solution and the relative conductivity of the solutions verify that the coupling of microwave and ball milling significantly enhanced the chemical reaction.

Keywords: D. ZnO; Nanoparticles; Microwave; Ball milling; Photocatalysis

1. Introduction

Zinc oxide (ZnO) is an important semiconductor material because it possesses piezoelectric properties, wide band gap (3.37 eV), and large exciton binding energy (60 meV) [1]. It can act as efficient photocatalyst, which can induce redox-processes owing to the electronic transitions between filled valence band and an empty conduction band [2]. In the last decade, ZnO nanoparticle has been the subject of interest for its unique and fascinating potential applications in transparent electrodes of solar cells, gas sensing, light emitting diodes (LED), ultraviolet (UV) photodetectors and photocatalysts [3–8].

Various preparation methods of nanocrystalline ZnO such as the precipitation method [9], hydrothermal method [10], sol–gel method [11], ball milling, [12,13] and microwave assisted synthesis [14] have been developed. However, there are still limited reports about obtaining high-quality single phase ZnO nanoparticles through a low-cost one-step synthesis.

Recently, a new synthesis method, microwave assisted ball milling, was developed by our group. And several ferrite nanoparticles were synthesized [15–18]. In this present paper, ZnO nanoparticles were directly synthesized by this ball milling approach and its photocatalytic properties were investigated. Furthermore, the coupling effect of microwave irradiation and mechanical force in this process was discussed.

2. Experimental details

The experiments were carried out in an ultrasonic wave-assisted ball milling device designed by the authors [19]. Zn$(CH_3COO)_2 \cdot 2H_2O$ of analytical grade were used as the raw materials. The brief process is as follows: Mass ratio of raw materials to milling balls (stainless steel balls of 2 mm in diameter) was 1:100, the power and frequency of the microwave were 0.8 kW and 2450 MHz respectively. For confirming the coupling effect of microwave-assisted ball milling experiment, ball milling processing without microwave and

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microwave treatment without ball milling were carried out separately on the same device. After washing and drying, the synthesized particles were characterized by following equipment.

Structural and morphology characterization were performed by X-ray diffraction (XRD,D-5000, Siemens, Cu Ka) and transmission electron microscopy (TEM, JEOL-1230). The relative fluorescence intensities of the aqueous solutions after different processing times were tested using a FL-2500 fluorescent analyzer. In addition, electrochemical methods were used to test the relative conductivity of the solutions at different times. The electrical conductivity was determined using a conductivity meter (DDS-11A).Photocatalytic decomposition of methyl orange (MO) is used to evaluate the photocatalytic activities of the prepared ZnO nanoparticles. The photocatalytic activity of ZnO (100 mg) prepared was evaluated by decolorization of 400 mL MO solution (the concentration is 5 mg/L) under the identical conditions. After being centrifuged at 5000 rpm for 5 min, samples were analyzed for their absorption by using UV–visible spectrophotometer (UV-6000PC) at 464 nm. Finally, the degradation efficiency of the MO by catalyst was calculated using Eq. (1) as follows:

$$\% \text{ degradation} = \frac{C_o - C}{C_o} \times 100\% \quad (1)$$

where C_o and C are absorbance of the dye solution before and after UV irradiation, separately.

3. Results and discussions

3.1. XRD and TEM characterization

The change of mass percentage of ZnO in the as-milled products with milling time is shown in Fig. 1(a), which clearly reveals the evolution of raw materials $Zn(CH_3COO)_2{\cdot}2H_2O$

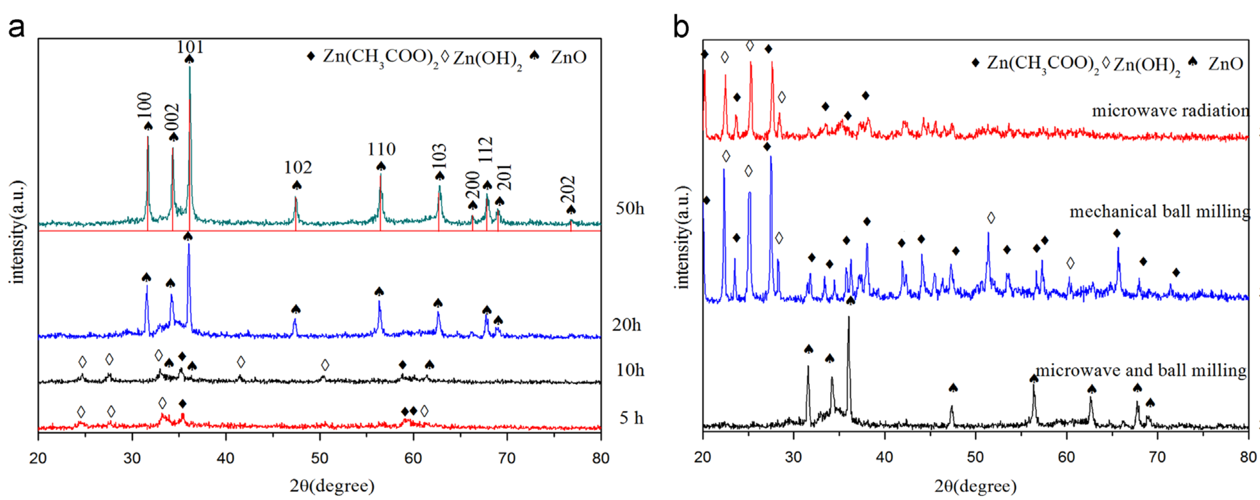

Fig. 1. (a) XRD patterns of reaction products processed by microwave-assisted ball milling after different time and XRD patterns of nanosize ZnO. (b) The X-ray diffraction patterns of reaction products prepared by three different approaches including microwave radiation, mechanical ball milling, and microwave-assisted ball milling, with processing time of 50 h.

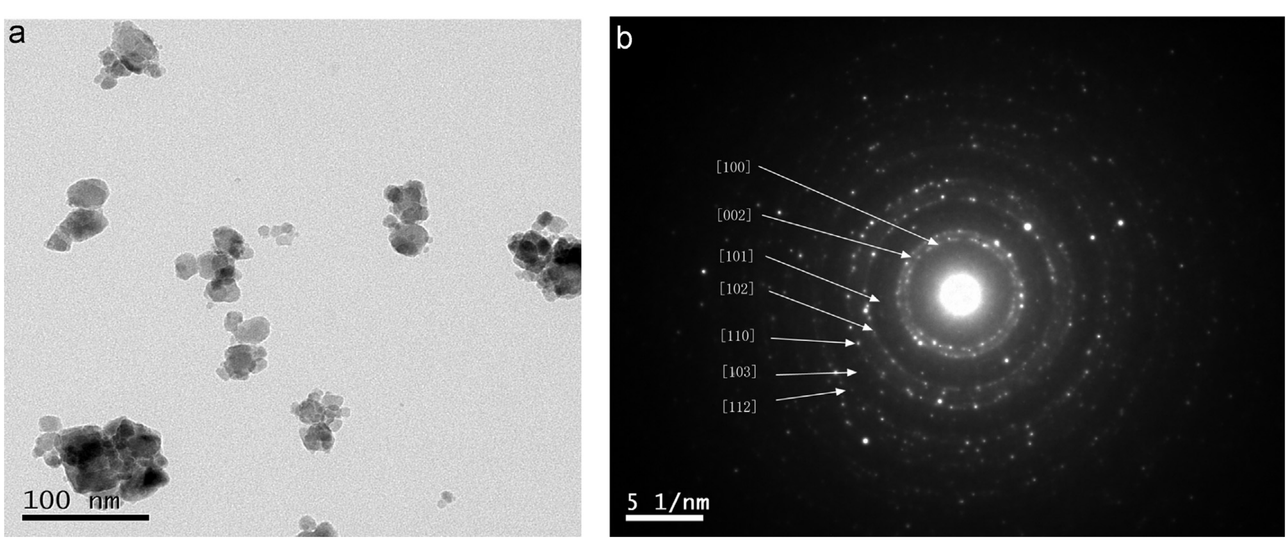

Fig. 2. TEM image (a) and SAED patterns (b) of as-synthesized ZnO nanoparticles by microwave-assisted ball milling for 50 h.