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Microwave-assisted the facile synthesis and photocatalytic properties of rhombic ZnO microstructures

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ABSTRACT

ZnO rhombic microstructures were fabricated via microwave assisted approach using $ZnCl_2$ and NaOH as raw materials by simply adjusting the ratios of $[Zn^{2+}]/[OH^-]$ as 1:8 (pH of reaction mixture at 13.9). Controlling $[Zn^{2+}]/[OH^-]$ as 1:1, 1:2, 1:3, 1:4, 1:5, 1:6, and 1:7 respectively (changing the pH of the precursor solution), ZnO microstructures with different morphologies were also synthesized. The characterization results revealed that as-obtained ZnO nanocrystals are of wurtzite structure and of the average grain sizes between 17 and 34 nm. In terms of BET surface areas, the photocatalytic activity of five typical ZnO samples was evaluated by degradations of methyl orange, and rhombic ZnO exhibited the better photocatalytic property, which could be attributed to its larger surface area, high crystallinity, and small particle size. Rhombic ZnO also showed good performance in photocatalytic eliminating Rhodamine-B. The synthesis and application of rhombic ZnO may contribute to development of photocatalyst for environmental issues.

1. Introduction

ZnO is a very important semiconductor material with band gap of 3.37 eV and exciton binding energy of 60 meV [1]. Nano-ZnO has not only excellent piezoelectric and photoelectric properties, but also good stability. ZnO has been applied in nano optoelectronic, piezoelectric devices [2], lasing or photodetector [3,4], hydrogen-storage devices [5], field-effect transistors [6], gas sensors and biosensors [7], and photocatalyst [8–12]. The photocatalytic degradation of water pollutant is a potential wastewater purification technology, and ZnO nano-microstructues can be used as photocatalyst to decompose toxic hard-degradable organics [8], such as organic dyes: azo methyl orange (MO) and triphenylmethane Rhodamine-B (RhB), both are commonly used as the contaminant models of main by-products in the textile industry for the evaluation of photocatalytic degradation activity of organic pollutants. The chemical structures of MO and RhB are shown in Fig. 1. Recently, more and more effort has been made for the controllable synthesis of ZnO with desired morphologies, such as ample nanotubes [13], flower-like particles [8,10,11], nanorods [12], and nanosheet [9,14], since it has been found that the surface morphology, crystallinity, surface area, and porosity of samples can influence the photocatalytic property [8,15,16]. Many methods, such as solid phase [17], gas phase [18] and liquid phase [8–16] methods were used to product various shapes nanoparticles. The newly microwave-assisted (MW) approach exhibits rapid and sustainable process with superlative benefits [19–21]. Therefore, this work describes the synthesis of ZnO rhombic microstructures and other microstructures by MW method, and the photocatalytic activity of typical samples.

2. Experimental

All chemicals were used without further purification. Deionized water was used for all experiments. In a typical procedure, 50.0 mmol $ZnCl_2$ and specific amount NaOH were dissolved in 50 mL water respectively, and $ZnCl_2$ solution was added dropwise into NaOH solution under stirring. After stirring for 30 min, the reaction mixture was placed into microwave reactor (XH-100A: Beijing Xianghu Science and Technology Development Co. Ltd.) and heated to 80 °C (measured by inner set) for 10 min with 900 W. The primary product was left still for 24 h, then the supernatant was removed and precipitate was washed with water for several times. After suction filtration, the precipitate was dried at 150 °C for 3 h to yield a white powder, the as-synthesized sample was denoted as S1-S8 respectively when the molar ratios of Zn^{2+}/OH^- as 1:2, 1:3, 1:4, 1:5, 1:6, 1:7, and 1:8. The product obtained in $[Zn^{2+}]/[OH^-]$ = 1:8 aqueous solution with 5, 15, and 20 min MW reaction time was denoted as S9, S10, and S11 respectively. The XRD

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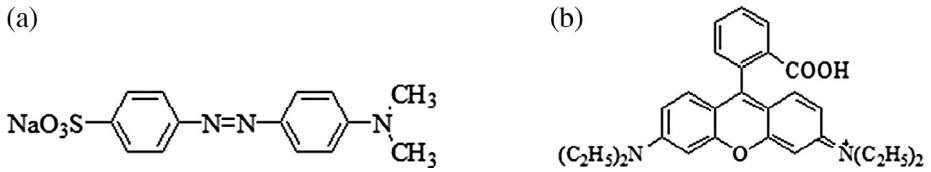

Fig. 1. Chemical structures of methyl orange (MO) (a) and Rhodamine B (RhB) (b).

characterization were performed using a XD-2 (XRD, PURKINJE GENERAL) for S1-S8. SEM images were obtained by a QUANTA-400F (FE-SEM, USA FEI) for S1–S11 samples. N2 adsorption-desorption analysis was performed with a Micromeritics ASAP 2020 apparatus for S1–S8, and the Brunauer-Emmett-Teller (BET) surface area was calculated.

In terms of BET surface areas, five typical samples were used to perform MO photocatalytic degradation experiments. Dispersing each 10 mg catalyst in 60 mL of MO (30 mg·L^{-1}) solution, and the suspensions were kept in dark for 30 min under stirring to reach absorption equilibrium, then exposed to UV light from 300 W mercury lamp set in the photochemical reactor (DGY-1A: Nanjing Duozhu Science and Technology Development Co. Ltd., China). At a given 30 min time interval (180 min total reaction time), each 10 mL solution was centrifuged, the MO concentration was measured using a UV-Vis spectrophotometer (UV2300: Hitachi, Japan). The photocatalytic degradation efficiency was calculated according to degradation (%) = $(A_0 - At)/A_0 \times 100\%$ (where A_0, A_t is the absorbance of dye in solutions at irradiation time (t) of 0 and t, respectively). The superior ZnO sample was also used to photocatalytic degradation of RhB (20 mg·L^{-1}) under the same reaction conditions.

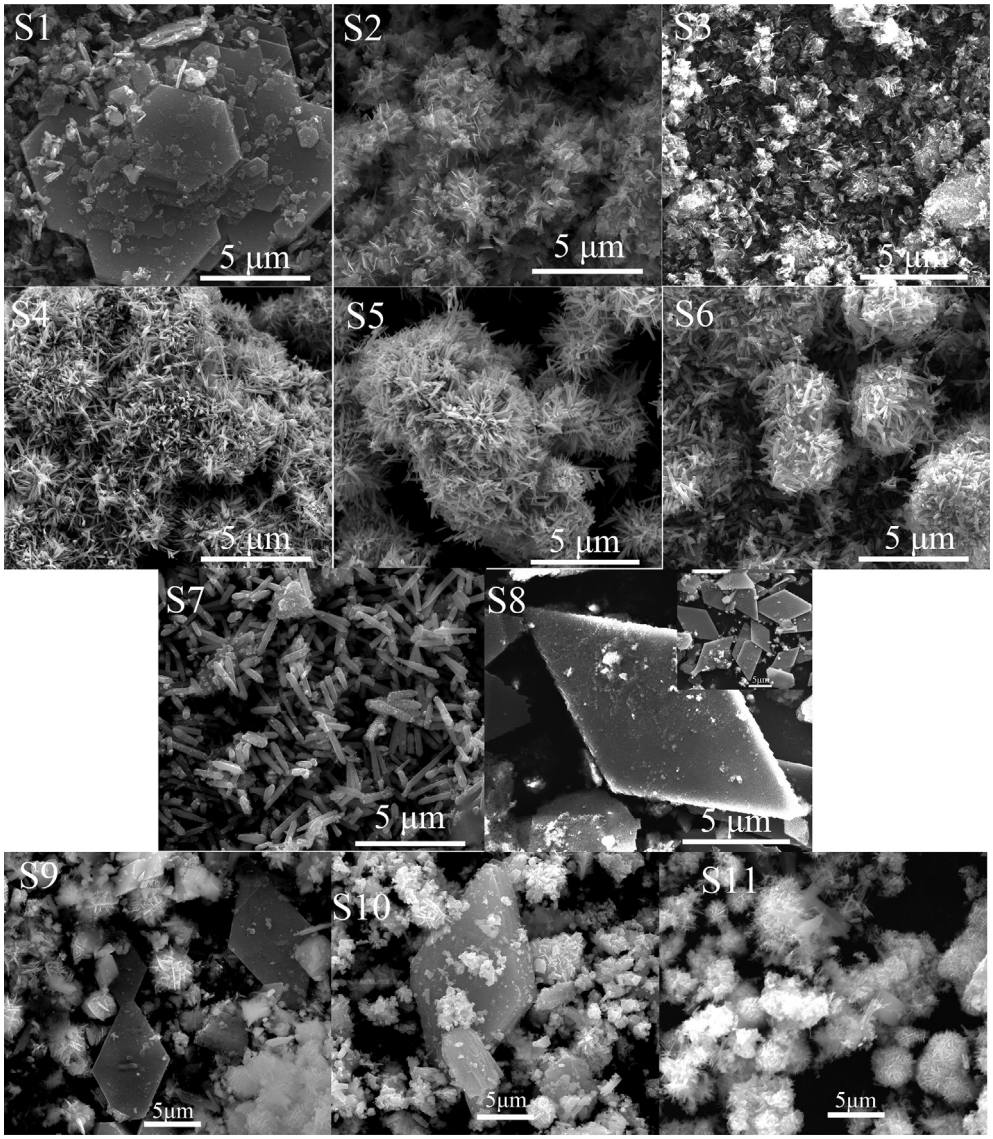

Fig. 2. SEM diagrams of synthesized ZnO microstructures (S1–S11, and the inset of S8).