



Sonochemical synthesis of hierarchical ZnO nanostructures

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ARTICLE INFO

Article history:

Received 7 April 2012

Received in revised form 5 July 2012

Accepted 6 July 2012

Available online 16 July 2012

Keywords:

ZnO

Nanorod

Nanoflower

Sonochemical

Ultrasonication

ABSTRACT

This work is about fabrication of ZnO nanostructures (ZnO-NS) via a simple sonochemical method. The chemicals used for the synthesis of various shaped ZnO are Zn salt, sodium hydroxide and ammonia solution without other structure directing agent or surfactant needed. This method is feasible and green, as it does not require high temperature and/or highly toxic chemicals. The shape of the ZnO-NS can be tuned by adjusting the ultrasound energy dissipated via varying the ultrasonication time from 5 to 60 min. It was found that uniform ZnO nanorods with diameter around 50 nm were formed after 15 min of ultrasonication while flowerlike ZnO-NS was formed after 30 min. This method produces high quality ZnO-NS with controllable shapes, uniformity, and purity.

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1. Introduction

Zinc oxide (ZnO) exhibits several unique properties, such as semiconducting and piezoelectric behavior [1], and it is consequently used in a wide variety of sensors and actuators. ZnO nanostructures (ZnO-NS) have been explored for a wide range of applications in nanoscale and microscale devices, such as generators [2], sensors [3], field-emission transistors [4], ultraviolet photodetectors [5], and in biomedical systems, such as ultrasensitive DNA sequence detectors [6]. In this nanosized range, they are expected to possess interesting physical properties and pronounced coupling quite different from their bulk counterpart [7].

Recently, several new routes have been developed to synthesize simple ZnO-NS, such as oxidation process [8], CVD [9,10], sol–gel synthesis [11], polymerization method [12], precipitation [13,14] solvothermal and hydrothermal [15,16], ultrasonic assisted [17–20], and sol–gel combustion [21]. However, the preparation of hierarchical ZnO nanostructures [22] is a complicated process, and a wide variety of different variables may affect the properties of the final product. Some important variables have distinct effects on the properties of the final product, while others may have only minor effects. In the case of preparation of hierarchical ZnO-NS, it is very important to obtain a narrow size distribution of the final product and to be able to control the morphology.

Sonochemical method is a green and feasible technique. In this work, a new, fast, and simple sonochemical route was designed to prepare hierarchical ZnO-NS in which zinc acetate was used as the starting material. The size, morphology, and crystallization of the resulting ZnO-NS were investigated by several tools.

2. Experimental

To prepare the Zn²⁺ solution, 11 g zinc acetate [Zn(CH₃COO)₂·2H₂O] was dissolved in 100 mL NH₃·H₂O 25%. Then 4 g sodium hydroxide [NaOH] was added to the solution. All these processes were carried out at room temperature without any specific conditions. After that, deionized water was added till the final concentration became 1 M Zn²⁺ solution. Each of the four 30 mL glass bottles was filled with 25 mL of the solution, followed by ultrasonication for 5, 15, 30, and 60 min, respectively. The temperatures of the samples were measured during the ultrasonication and they were found 31, 40, 57, and 70 °C for samples prepared at 5, 15, 30, and 60 min, respectively. The resulting white color product was centrifuged (4 min, 4000 rpm) and then washed with distilled water for three times. The obtained white precipitate was dried in an oven at 60 °C for 24 h. The weights of the obtained products were measured for each dried sample. The results were presented in Table 1 and compared to the theoretical measurements.

To check the effect of ultrasound energy on synthesis process, two separated glasses of the solution was chosen named as sample A and B. Sample A was placed on stirrer and the solution stirred for

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Table 1

The theoretical and experimental weight measurements of the prepared samples at 5, 15, 30, and 60 min using ultrasonication.

Ultrasonication time (min)	Theoretical (g) ± 0.01	Experimental (g) ± 0.01
5 (Zinc hydroxide)	2.98	2.32
15 (Zinc oxide rods)	2.44	1.96
30 (Zinc oxide flowers)	2.44	2.21
60 (Zinc oxide flowers)	2.44	2.28

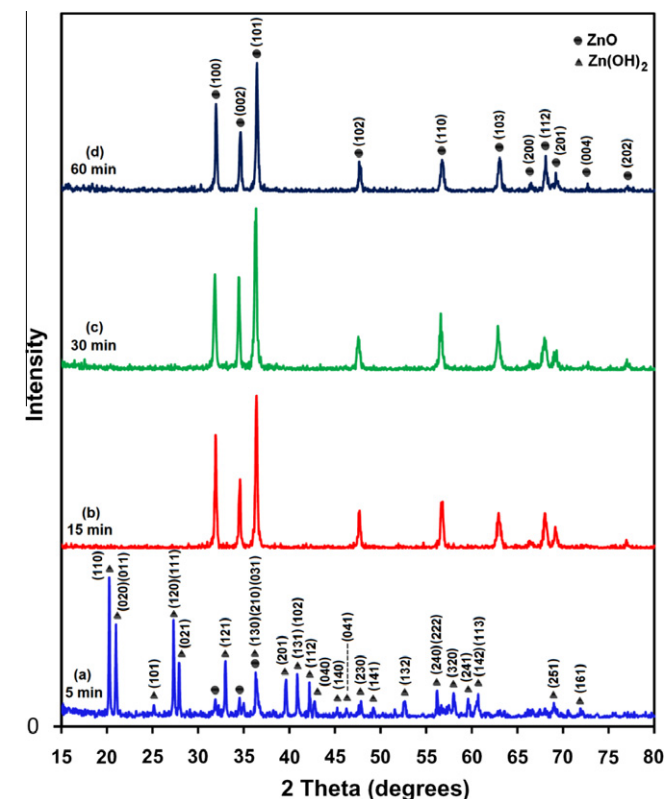


Fig. 1. XRD patterns for samples prepared after various ultrasonication time (a) 5 min, (b) 15 min, (c) 30 min, and (d) 60 min.

60 min. No change in color was observed for the solution. It means that there is no material formed during the stirring process. On other hand, the temperature of the solution was measured during the ultrasonication and it was found to be about 70 °C. The same process as sample A was applied to sample B but at 70 °C. There was no change in color for this sample as well as sample A, which means that the sound energy plays a very important role in the ZnO-NS formation process. To insure that there is no material in the solution, the UV–Vis absorbance of the solutions (pri-

mary sol, the sample A and B) were obtained (Fig. 5). A maximum absorbance is observed for each solution that is corresponding to Zn⁺² [28] and there is no absorbance peak related to the other materials like Zn(OH)₂ or ZnO. It shows that the stirring does not have any effect on the formation of ZnO from zinc acetate.

The structure of the as-prepared ZnO-NS was characterized by X-ray diffractometer (XRD, Philips, X'pert, Cu K α), field emission scanning electron (FESEM, FEi Quanta 200 at 20 kV), and field emission transmission electron microscopes (TEM, JEOL, 2010F). The UV–Vis spectra were recorded in the range of 200–1000 nm by a Thermo Scientific Evolution system (300 UV–Vis spectrophotometer, USA).

3. Results and discussions

3.1. Structural studies

The XRD patterns of the Zn(OH)₂ and the prepared ZnO-NS with ultrasonication times at 5, 15, 30, and 60 min are shown in Fig. 1a–d, respectively. With 5 min of ultrasonication, XRD pattern in Fig. 1a shows that the sample is mainly composed of Zn(OH)₂ phase (PDF card No: 01-076-1778) plus a small fraction of ZnO phase (PDF card No: 00-036-1451). After 15 min ultrasonication, however, Fig. 1b–d clearly show that the samples are fully composed of ZnO phase since no extra peaks observed, which indicates that all the Zn(OH)₂ has completely transformed into ZnO phase. The crystallite sizes of the ZnO-NS could be estimated by Scherrer equation, $D = (k\lambda/\beta_{hkl}\cos\theta)$ [23], where D is the thickness of (hkl) crystal plane, λ is the wavelength of the incident X-ray (1.5406 Å for Cu K α), k is a constant equal to 0.93 [24], β_{hkl} is the peak width at half-maximum intensity, and θ is the peak position. The (101) plane (the strongest) was selected to calculate the crystallite size. Corresponding to the ultrasonication time of 15, 30, and 60 min, the estimated crystallite sizes were 30 ± 2 , 38 ± 2 , and 36 ± 2 nm, respectively.

3.2. Morphological characterizations

Fig. 2 shows the morphological evolution from the newly formed Zn(OH)₂ crystals at formation time of 5 min and ZnO-NS prepared at the following times of 15, 30, and 60 min, respectively. By a comparison of the XRD patterns in Fig. 1 and the SEM observation in Fig. 2, it is noted that the Zn(OH)₂ crystals were not stable under ultrasonication and a phase transformation from Zn(OH)₂ to ZnO (nanorods in Fig. 2b, diameter ~ 50 nm, length ~ 5 – 8 μ m) occurred after 15 min ultrasonication. The significant morphological evolution (from Fig. 2a to b) matches with the corresponding XRD patterns in Fig. 1a and b, owing to the phase transformation. From nanorods in Fig. 2b to nanoflowers in Fig. 2c and d, however, it is interesting to note that the morphological evolutions lead to no change on crystal structure of ZnO-NS (See Fig. 1b–d). In addition, with increasing the ultrasonication time from 30 to 60 min,

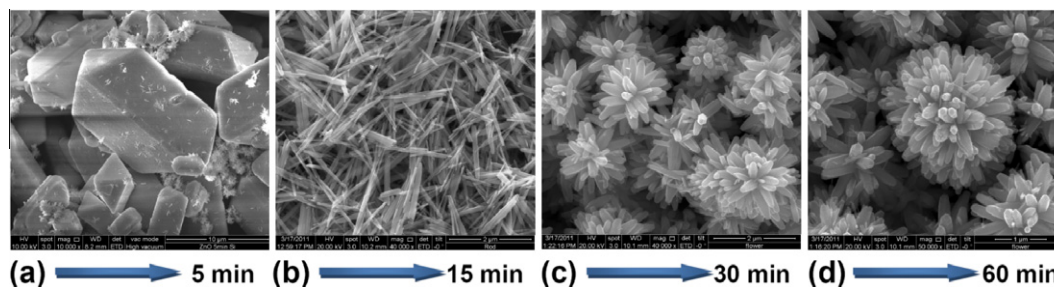


Fig. 2. SEM micrographs for (a) Zn(OH)₂ crystals, (b) ZnO nanorods, and (c, d) ZnO flowers.

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