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A Simple Method for Preparation of Nano-sized ZnO

Sadraei R*

Islamic Azad University, Saveh, Iran

Research Article

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***For Correspondence**

Sadraei R, Islamic Azad University, Saveh, Iran

E-mail: r.sadraei1989@gmail.com

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ABSTRACT

Nano is an advanced technology which in recent years has had numerous applications in different fields such as industry, biotechnology, energy, environment and etc. In this paper, ZnO nanoparticles were synthesized by simple method of precipitation sounds more plausible. $ZnSO_4$ and NH_4OH were used as precursors to yield ZnO powders. The synthesized ZnO nanoparticles were characterized by X-ray diffraction measurements (XRD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX). According to the results of XRD analysis, the purity of ZnO nanoparticles was obtained; based on the William-Hal method, the mean size of particles was achieved equal to 30 nm. This test has also shown the high crystallinity of the ZnO nanoparticles. As SEM analysis revealed, the morphology of ZnO nanoparticles to be flake like shapes with the mean particle size of 30 nm. Finally, the analysis of the synthesized nano sample by using percentage of EDX has indicated that the purity of ZnO was quite high.

INTRODUCTION

Nanotechnology is the science of production, manipulation and use of materials at subatomic level to produce novel products and processes. At this specific range various optical, biological, magnetic, physical and chemical properties of particles get changed drastically. Nanotechnology has tremendous applications in diagnostic devices, drug delivery, tissue engineering, environmental chemistry, water filtration, producing ecofriendly energy production systems and quantum computers. Nano-sized metal particles have got much attention due to its unique optical, electrical and magnetic properties [1].

Zinc oxide (ZnO) is used as a heterogeneous catalyst, have a high catalytic activity, non-toxic, insoluble, and also a cheap catalyst [2] which is an important n-type [3-6] semiconductor. ZnO is not only a material of particular interest because of its unique optical and electronic properties, but also it has some characteristics including: (i) wide-band gap semiconductor (3.3 eV at room temperature) [2-16], (ii) large exaction binding energy of 60 MeV [3-5,8,10,11,15-18] and (iii) good candidate for numerous possible applications, particularly in the form of thin films, nanowires, nanorods, or nanoparticles [19] in solar cells [7-10,18], gas sensor [3], photo detectors, luminescent oxides [20], photovoltaic [7,9], lasers [7,8,18], optical devices [3,17,18,21,22], sensors [8-10,17,18], electrode [18,23], pharmaceutical [8], and in the production of pigments and cosmetics [5].

The uses of ZnO as a photocatalytic degradation material has been extensively studied [19]. Preparation of nano-size ZnO has been carried out by different methods like hydrothermal method [24], aerosol, micro-emulsion, ultrasonic, sol–gel method, evaporation of solution and suspensions, evaporative decomposition of solution (EDS), solid state reaction, conventional ceramic fabrication, wet chemical synthesis, spray pyrolysis method [3,5,8,18,20,25]. Among these synthetic routes, precipitation approach compared with other traditional methods provides a facile way for low cost and large-scale production, which does not need expensive raw materials and complicated equipment [5,9]. The preparation of zinc oxide nanostructures by the precipitation method from zinc salt solutions, such as $Zn(NO_3)_2$, $Zn(CH_3COO)_2 \cdot 2H_2O$, $ZnSO_4$, etc., with alkali solutions containing LiOH, NH_4OH, NaOH, etc. was investigated [5].

Recently, Li et al. [26] have obtained Nano ZnO flakes by calcinations of the precursor of $Zn(OH)_2$ via the reactive ion exchange method between an ion-exchange resin and zinc sulfate solution at room temperature. Al-Heniti et al. [27] have synthesized Nano crystalline ZnO flakes via solution process using zinc acetate and diethyl amine under refluxing at 85ºC for 4 hours [20]. In the present work, a direct precipitation method was employed to synthesize ZnO nanoparticles by using suitable alternative raw materials, which are attractive for chemical industries [20]. This paper, were utilized zinc sulfate as an initial reagent and NH_4OH as a precipitating agent.

EXPERIMENTAL

Materials and apparatus

All the chemical reagents used in this study were used without further purification which were purchased from Merck chemical company. ZnO crystal structures were characterized by X-ray diffractometer (Made in Italy, GNR, Model: MPQ 300) using at 40 KV accelerating voltage and 1200 W power. Device accuracy reported about 0.01 degree and the particles size were estimated by Williamson–Hall and size–strain plot methods [28]. The morphology of the ZnO particles were determined by scanning electron microscope (Made in USA, Model: KYKY-EM 3200), and the compositional analysis was done by energy dispersive X-ray (Made in UK, Model: Sirus SD).

Preparation of ZnO nanoparticles

Zinc sulfate heptahydrate ($ZnSO_4(H_2O)_7$) and Ammonium hydroxide (NH_4OH) were the two starting materials for the synthesis of ZnO nanoparticles. The synthetic procedure for ZnO nanoparticles by direct precipitations method are briefly summarized in **Figure 1.**

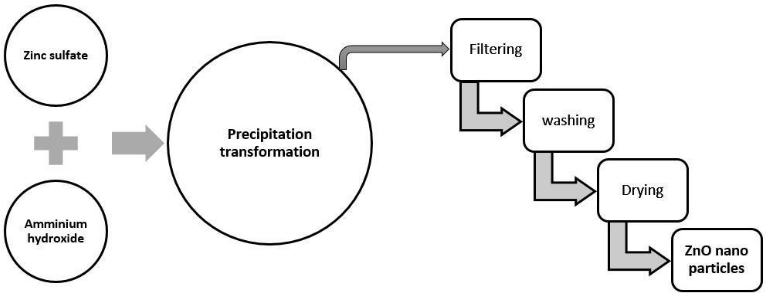

Figure 1. Preparation of ZnO Nanoparticles.

Zinc sulfate heptahydrate (2.9386 mL) were dissolved in 100 mL deionized water. A mother solution prepared from 25 mL of the Zinc sulfate solution (0.2 M) and 50 mL of purified water, and Ammonium hydroxide used as an alkali solution. 25 ml of the alkali solution (NH_4OH, 25%) with an approximate rate of 5 mL/min was slowly dropped into the mother solution [20]. The magnetic stirrer cum heater for vigorous stirring was used at the temperature about 50-60ºC. The precipitates derived from the reaction between the Zinc sulfate and Ammonium hydroxide solution were collected by filtering and were rinsed three times with distilled water by ultrasonic and centrifugation devices. Then, the final precipitates were achieved after drying at 60ºC in oven for 8 hours.

RESULTS AND DISCUSSION

The nano-size ZnO powder was prepared by using Zinc sulfate heptahydrate and Ammonium hydroxide in aqueous solvents; And ZnO powder was investigated by three different analyses. The SEM, EDX, XRD analyses have shown the clear results.

SEM analysis result

The morphology of ZnO nanoparticles was studied by using the SEM which revealed a flake like shape. The SEM image of ZnO flakes powder resulted from drying of Zinc hydroxide at 60ºC for 8 h are shown in **Figures 2 and 3.** It clearly illustrates that the typical SEM images of nano-sized ZnO flakes have large quantity of flake shape with a narrow size distribution. The low-resolution image **(Figure 2)** confirms that the flakes were synthesized in very large quantity; and the high-resolution image **(Figure 3)** of the synthesized flakes reveals that the products have uniform flake shape [20].

EDX analysis result

Chemical purity of the samples was tested by EDX. The corresponding EDX spectrum **(Figure 4)** shows that the ZnO nanoparticles were composed with only Zinc and Oxygen elements, which indicates that the product is high-pure ZnO [20].

XRD analysis result

In order to check the crystallinity and crystal phase of the ZnO nanoparticles flakes XRD analysis using William-Hal method [28] was performed, and result shows that the particle size of samples was approximately 30 nm. According to **Figure 5**, all diffraction peaks in the pattern can be indexed as the pure hexagonal phase of bulk ZnO. The sharp diffraction peaks show that the obtained ZnO flakes have high crystallinity. No characteristic peaks from impurities such as $Zn(OH)_2$ are detected. This indicates that the sample is composed of flake structural ZnO with the lattice constants a=3.249ºA and c=5.206ºA which are consistent with the values in the standard card (JCPDS 36-1451).

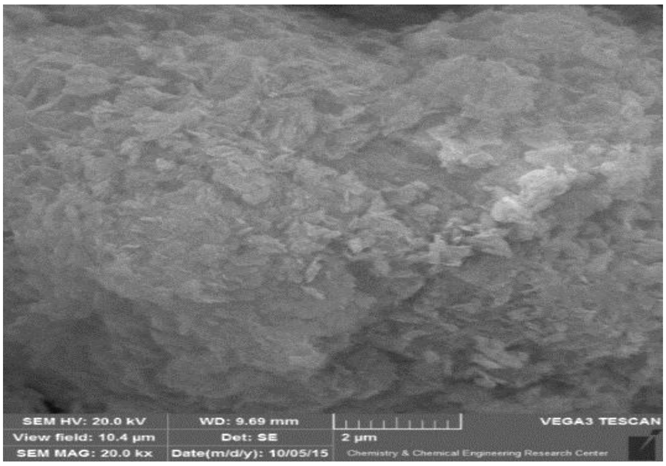

Figure 2. Low Resolution of ZnO Nanoparticles SEM Image

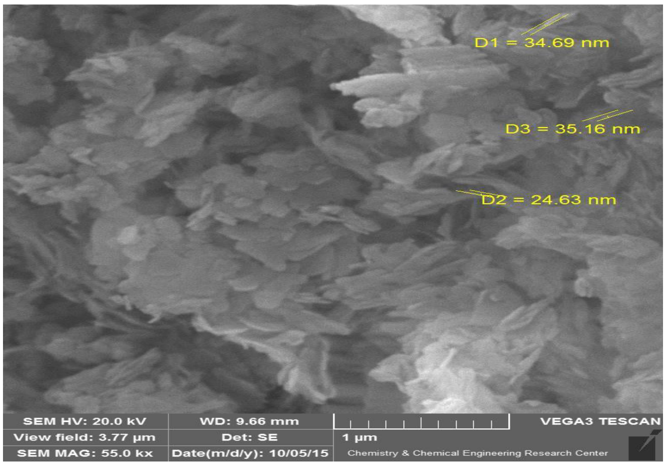

Figure 3. High Resolution of ZnO Nanoparticles SEM Image.

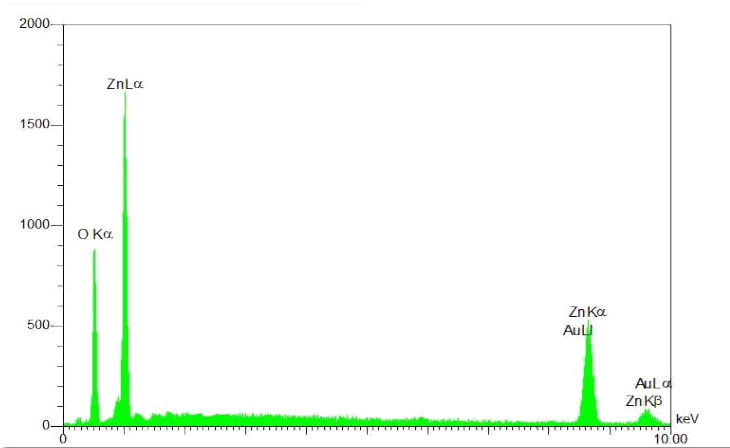

Figure 4. Typical EDX pattern of ZnO Nanoparticles.

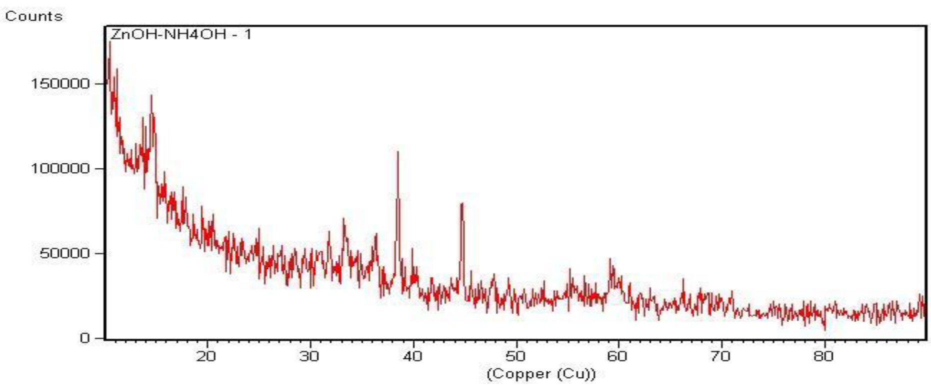

Figure 5. X-ray diffraction pattern of the ZnO Nanoparticle's flake.

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CONCLUSION

In summary, nanoparticles of ZnO crystals were produced by using a simple mixing technique of precipitation. $ZnSO_4$ and NH_4OH have been as precipitating agent in aqueous solutions. A mother solution prepared from 25 mL of the Zinc Sulfate solution (0.2 M) and 50 mL of purified water was controlled within temperature about 50-60ºC. Then, the sample was dried at 60ºC for 8 h in oven. This method employed an inexpensive and reproducible process for the large-scale synthesis of Nano-sized ZnO flakes. Eventually, it was found that ZnO nanoparticles have the average of particle size about 30 nm. SEM images, EDX and XRD pattern indicated that the prepared ZnO nanoparticles have uniform structure and high purity. The leading characteristics of ZnO nanoparticles can be considered in the number of defects, optical properties, stability, electron transport, and other electronic properties. The varied structures allow for numerous applications in different device types. Nano ZnO flakes can be used for new opportunities for anode materials as Zn/Ni batteries and other energy-storage devices.

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