

Studying the effect of irradiation time in preparing zinc oxide nanoparticles prepared by microwave method

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Zinc oxide nanoparticles were prepared by microwave method by dissolving zinc nitrate salt ($Zn(NO_3)_2 \cdot 6H_2O$) in deionized water. The prepared nanoparticles were characterized by X-ray diffraction method (XRD), Field Effect-scanning electron microscopy (FE-SEM), infrared spectroscopy (FTIR), and UV-visible spectroscopy (UV-Vis.). The results of the structural tests showed that the prepared nanomaterial was of a hexagonal wurtzite structure with an ideal growth along the C-axis with an average particle size of about 24 nm. While the images of the (FESEM) showed that the nanoparticles were arranged in the form of nanorods and they had an irregular distribution. The purity of the samples was confirmed (ZnO) by EDAX data. And the results of FTIR showed distinct locations at the band (464 to 430 cm^{-1}), which indicates the characteristic stretching mode of the Zn-O bond. The results UV-Vis. showed that the prepared materials have a band gap between 3.23 and 2.21 eV.

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1. Introduction

ZnO is a semiconductor with many possible uses due to its n-type direct band gap (3.37 eV), high exciton binding energy (60 MeV), and high dielectric constant. Because of its high optical efficiency [1], ZnO can be used for a wide range of tasks requiring operation in the visible and near-ultraviolet spectrums. [2]. Many exciting properties and technological applications exist for nanoscale zinc oxide [3]. Photocatalysis, quantum dots chemical cleanup, photoinitiation of polymerization reactions, solar energy conversion, biochemical sensors, chemical electrodes, cosmetics, and pigments are some of the applications.

Several distinct nanostructures, including nanoneedles, nanobelts, nanorods, nanowires, nanobows, and nanonails, have been observed in ZnO [4]. Zinc oxide nanostructures can be made in a number of ways, such as by thermal decomposition, spray pyrolysis, chemical vapor deposition, sol-gel hydrothermal precipitation, and others. [5, 6].

Using a conventional thermostatic system, convection or conduction heats up the walls of the reactor. However, it takes more time for the center of the sample to reach the desired temperature, resulting in inhomogeneous temperature profiles. Microwave heating has emerged as a promising synthesis method in both organic [7, 8] and inorganic [9] chemistry as a potential solution to this problem. Using this method, the reaction mixture can be heated quickly and uniformly to the desired temperature while saving both time and energy. The two mechanisms by which microwave heating converts electromagnetic radiation into heat energy are dipolar rotation and ionic conduction, and both processes are inextricably linked to the chemistry of the reaction mixture being heated. The microwave absorption properties of various compounds can be used to selectively heat a reaction mixture.

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This method has several advantages over more traditional methods, including high heating rates that result in an increase in the rate of chemical reactions; the ability to use a wide variety of reaction conditions (from mild to autoclave temperatures and pressures); high yields; and reaction selectivity as a result of differences in microwave absorbing properties; and precise regulation of reaction conditions; ease of handling; and quick and easy optimization. It is simple, inexpensive, safe for the environment, and doesn't require the use of a template

2. Experimental part

2.1. Synthesis of zinc oxide (ZnO)

ZnO nanoparticles were prepared by a typical synthesis process, in which (1.487g), of the precursor salt ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) was diluted in 50 mL deionized water to obtain a Zn + 2 solution and add of base (NaOH) (0.108 gm) was drip (2 min) into the above described solution with magnetic stirring at room temperature to obtain a colloidal system, which was kept while stirring for 10 min, then the reaction mixture was transferred to microwave and processed at Specific temperature (85 °C) for a specified period (5, 10) min under temperature control mode in a microwave accelerated reaction system operating at different power (400 W). Upon completion of the reaction and cooling to room temperature, the white precipitate was collected by filtration and washed with deionized water times. Finally, the product was dried at 70 °C in a vacuum oven for 1 hour.

2.2. Characterization

The synthesis of zinc oxide (ZnO) nanoparticles was studied using powder X-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), Utilizing energy dispersive X-ray spectroscopy, we were able to determine the elemental make-up of as-prepared ZnO. (EDX, Inca Oxford, attached to the FESEM), Fourier transform infrared spectroscopy (FTIR), and ultraviolet-visible spectroscopy (UV-vis). Scherer's formula was used to determine the average crystallite sizes (D) [9] (Table 1) of the ZnO nanostructures, and X-ray diffraction Cu-K radiations (Model: PW1730) were used to characterize the synthesized ZnO powders in the 2θ range of 10° to 80° .

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

where λ is the X-ray source's wavelength, β is the diffraction peak's full width at half maximum intensity at 2θ and θ is the Bragg angle. A carbon label was used to transport a sample of solid state material for FE-SEM analysis (Model: Mira3). A secondary electron detector was used to examine the samples. The ZnO nanoparticles' infrared spectra were recorded using a spectrophotometer model Equinox 55, Tensor 27, in transmittance mode. The UV-Vis spectra of colloidal systems are then modeled using Lambda850.

3. Results and discussion

To see how a variable that can be changed, like irradiation time, affected the shape of the ZnO that was made, a number of experiments were done with the same experimental conditions but different irradiation times: 5 minutes and 10 minutes after the desired temperature was reached. Figure 1 shows the X-ray diffraction pattern of ZnO powder as it was made. The diffraction lines, which match the values reported in the ZnO database, are clear proof that hexagonal wurtzite was formed (JCPDS card no. 036-1451). Pure ZnO nanoparticles were made during this process. All of the diffraction peaks are very sharp, which shows that the ZnO sample is very crystalline. Using the Debye-Scherrer equation and the XRD results, the size of the crystallites in the ZnO particles that had already been made was found. (Table 1).

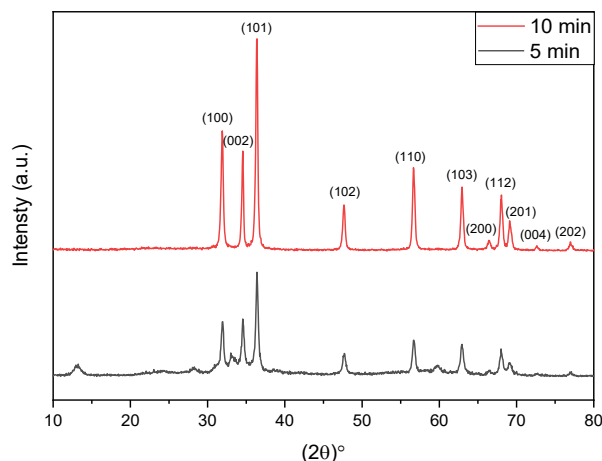


Fig. 1. The ZnO nanoparticles' X-ray diffraction (XRD) pattern as prepared.

In Figure 2, In this picture, we see how the morphology of ZnO nanoparticles changes as a function of irradiation time, with more time under irradiation leading to more clearly defined structures and a more uniform particle size and morphology distribution in the final system As shown in Figure 2 (b), it is represented by obtaining nanoneedles or nanowires.

Table 1. Structural parameters of ZnO nanoparticle.

Synthesis code	Experimental conditions (irradiation time)	Zn precursor	Base	Average crystallite size, nm	Energy gab (eV)
MW-1	5 min , 400 W	Zn(NO ₃) ₂ .6H ₂ O	NaOH	24.32	3.23
MW-2	10 min ,400 W			28.11	2.21

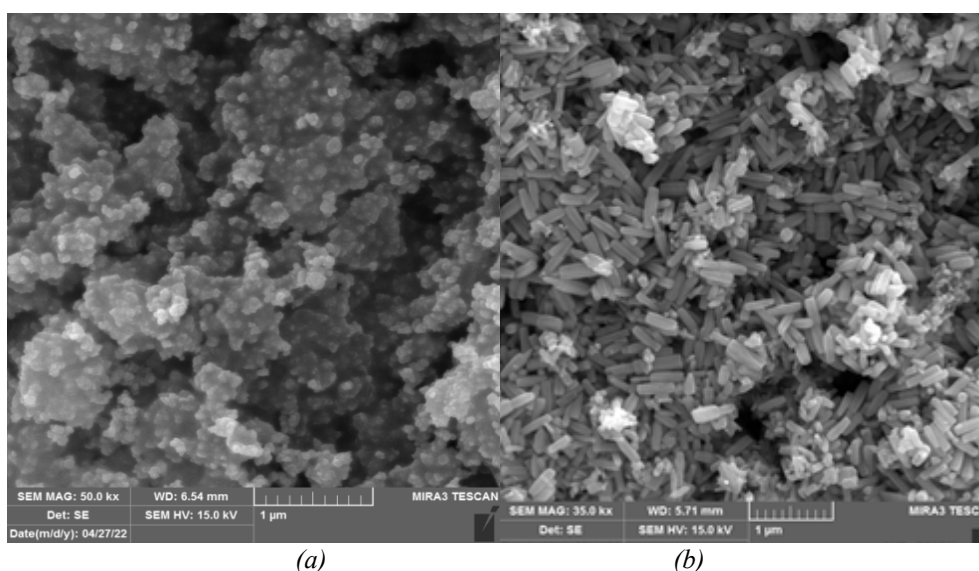


Fig. 2. ZnO images from FE-SEM taken at various irradiation times a: 5 min; b: 10 min.

In Figure 3 The FTIR spectrum of ZnO There is a broad band with very weak intensity. peaking at $\nu = 3435 \text{ cm}^{-1}$, which can indicate the presence of moisture or the presence of O–H. Three distinct peaks at 464.43 cm^{-1} and 430.04 cm^{-1} were seen in the FTIR spectrum, consistent

with the Zn–O stretching vibration mode and other reports [11]. Because the number of atoms making up the surface may change under these circumstances, a different stretching vibrational mode of ZnO was observed. Additional bands are seen between(800 - 900) cm^{-1} , which are attributed to the C-H bend [12]. The bands between(1600- 1300) cm^{-1} are associated with the C=O bonds and the O–C–O bonds. C-O bond bending modes, both symmetric and asymmetric, were detected between (1525- 1580) cm^{-1} [10]. The results show that ZnO nanoparticles were successfully made by using a simple, cheap, and quick method of combustion that was helped by microwave energy .

An EDX analysis was performed on the material after it was synthesized to learn more about the ZnO nanorods' chemical makeup. In Fig. 3, we see an example of an EDX spectrum obtained from ZnO nanorods. EDX analysis revealed that only Zn and O signals were present in the prepared nanorods, proving their composition to be solely of those two elements. There was no sign of a secondary phase or impurity, which means that the ZnO nanorods were very pure.

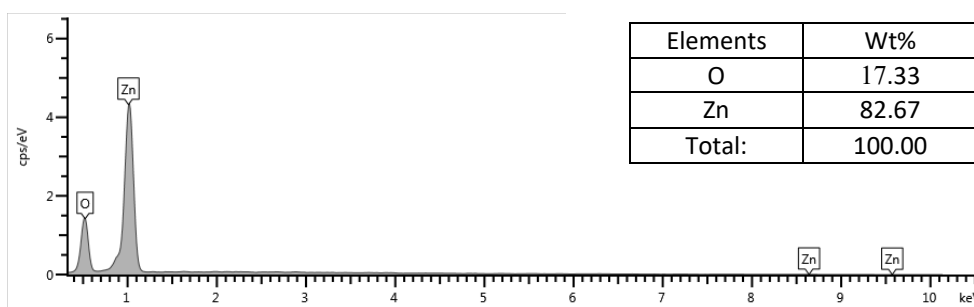


Fig. 3. EDX spectrum of the synthesized ZnO nanorods by microwave irradiation (10 min).

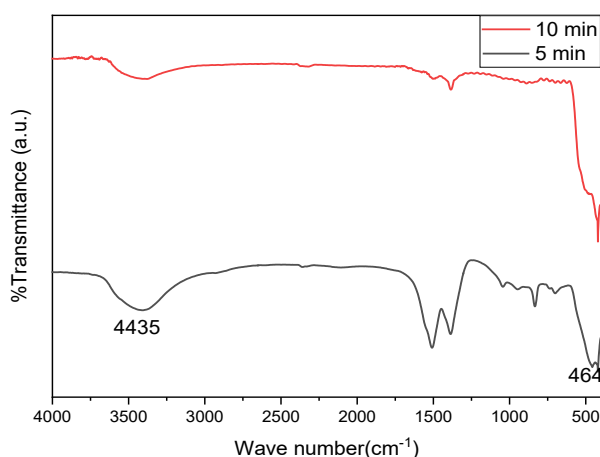


Fig. 3. FTIR of the ZnO nanoparticles.

With the understanding that particle size and shape play a role in colloidal absorption, the band gap energy was calculated from their absorption curves using Tauc plots in order to gain a better understanding of the optoelectronic properties of NP [13]. In this experiment, we measured the absorption spectra of synthesized ZnO nanoparticles. Figure 4 shows that the band gap (E_g) was calculated by extending the linear portion of the graph to the horizontal axis [14]. Table 1 summarizes the observed band gap values and the corresponding UV absorption maxima (374 nm and 338 nm) for this material. When exposed to UV light, electrons move from the valence band to the conduction band, resulting in UV light absorption.

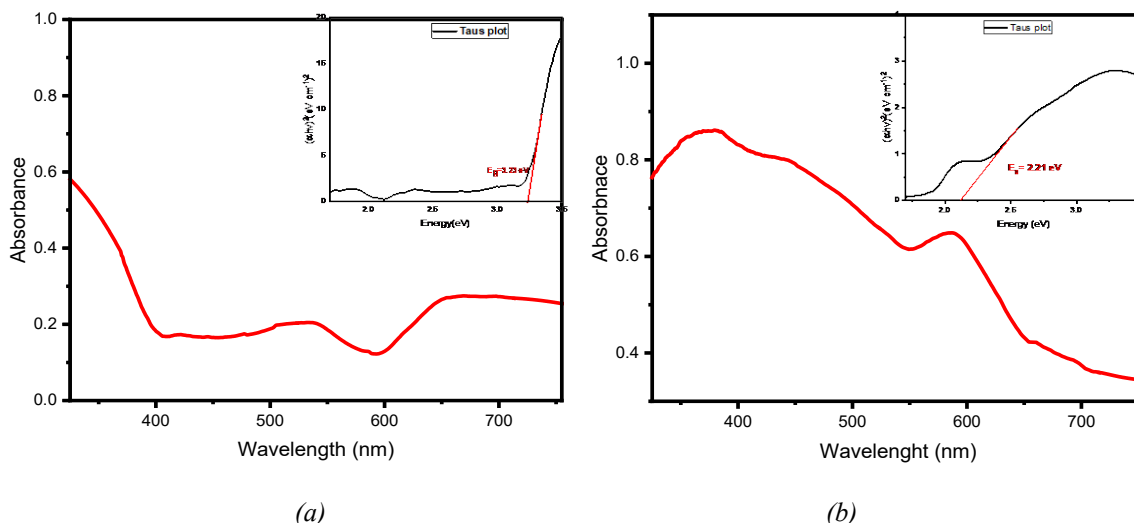


Fig. 4. The UV spectrums of synthesized ZnO nanoparticles: The Taucs The plot of band gap calculation for samples is given in onset (a) 5 min (b) 10 min.

4. Conclusion

As new methods for their synthesis are developed and explored, ZnO nanomaterials' many applications expand, which in turn affects their properties when used to create cutting-edge devices. Improvements in microwave technology have facilitated the rapid and pure synthesis of nanomaterials with a variety of desirable morphologies. $\text{Zn}(\text{NO}_3)_2$ is used as a precursor reagent in an. The method is straightforward and efficient with microwave-assisted aqueous solution. ZnO with high crystallinity and purity was successfully synthesized. Furthermore, the effects of a number of parameters on the final ZnO nanoparticles have been analyzed. Extending the reaction time from 5 minutes to 10 minutes produced a system that was both highly pure and dimensionally uniform. More time under the radiation causes the c direction to develop, resulting in a more uniform distribution of particle size and morphology and a clearer system. and well-defined structures. ZnO NPs' absorption edge and band gap were relatively unaffected by their size and shape. ZnO nanoparticles had a band gap of between 3.23 and 2.21 eV

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