International Journal of Nanomaterials and Chemistry

http://dx.doi.org/10.12785/ijnc/010102

# Solvothermal Synthesis, Characterization and Optical Properties of ZnO and ZnO-MgO, Mixed Oxide Nanoparticles

*Roghayyeh Hashemabadi, Ehsan Zamani, Fatemeh Rezaei, Sara Bagheri, Abedin Zebardasti and Alireza Aslani* *

*Department of Chemistry, Faculty of Sciences, Lorsetan University, Lorestan-Khoramabad, Iran.*

Received: 20 Jun. 2014, Revised: 28 Sep. 2014, Accepted: 3 Oct. 2014.
Published online: 1 Jan. 2015.

**Abstract:** ZnO–MgO mixed oxide nanoparticles were successfully produced from a solution containing Zn and Mg Acetate by Solvothermal method. The calcination process of the ZnO-MgO composite nanoparticles brought forth polycrystalline two-phase ZnO-MgO nanoparticles of 40–80 nm in diameters. ZnO and MgO were crystallized into würtzite and rock salt structures, respectively. The optical properties of ZnO-MgO nanoparticles were obtained by solid state UV and florescent. The XRD, SEM and Raman spectroscopies of the particles were analyzed.

**Keywords:** Nanomaterials, Solvothermal; X-ray diffraction; mixed oxide; Electron microscopy.

## 1. Introduction

The metal oxides are extremely important technological materials for use in electronic and photonic devices. In these metal oxides, magnesium oxide (MgO) and zinc oxide (ZnO) is very appropriate for insulation applications due to their low heat capacity and high melting point [1]. Another application for MgO and ZnO are as fundamental materials for chemical heat pump [2], as substrate materials for the epitaxial growth of thin films with desirable magnetic or electronic properties [3]. During the past several years, various methods have been developed for the synthesis of nanomaterials that have included template-assisted [4], vapor liquid solid (VLS) [5], colloidal micelle [6] and electrochemical processes [7]. Because the novel properties of nanomaterials depend on their size and shape, new synthetic strategies and a better understanding of the growth mechanisms by which the size and shape of nanostructures can be easily tailored are key issues in nanomaterials science.

The synthesis and fabrication of nanoscale controlled materials are rapidly developing fields of materials science [8]. Particularly, hollow nanostructures have attracted much attention because their unique shape makes them applicable as delivery vehicles, fillers as well as for catalysis, and could bring about changes in physical and chemical properties [9,10]. Several chemical techniques, such as co-precipitation, sol–gel, hydrothermal method, etc, have been used to prepare nanocrystalline powders[11] Among these chemical techniques, the hydrothermal process has attracted a great deal of attention since particles with the desired characteristics can be prepared with this technique by controlling the solution pH, reaction temperature, reaction time, solute concentration and the types of solvent depending on the particular application.[12] The main advantages of hydrothermal synthesis are related to homogeneous nucleation processes, ascribed to elimination of the calcinations step to produce very low grain sizes and high purity powders.[13]. Zinc oxide (ZnO) is a wide band-gap semiconductor with unique physical properties and a wide range of potentialapplications. It has already been used for applications such as sensors, transparent coating for solar cells and acoustic devices. ZnO has also recently attracted considerable interest for efficient ultraviolet LEDs and laser devices. Indeed, it is an excellent candidate for high-temperature and high-efficiency optoelectronic applications owing to its large exciton binding energy (60 meV), the largest among conventional semiconductors. In addition, an exceptionally important material, magnesium oxide (MgO) is mainly obtained by the decomposition of magnesium hydroxide [14–16], which has been widely used in catalysis [17], toxic waste remediation [18], or as additives in refractory, paint and superconductor products [19]. During the past decades, MgO with nanostructure have received considerable interest due to their potential property modification and is supposed to apply in more areas. The research on the preparation and properties of nanostructured ZnO-MgO in one phase is of great importance. In this paper, our objectives are to investigate the formation of MgO and ZnO nanoparticles in a ZnO-MgO system, and propose a model of the in situ reactions.

* Corresponding author e-mail: a.aslani110@yahoo.com

The insitu reaction model was confirmed in the experiments, and the growth mechanisms for the formation of MgO and ZnO nanoparticles in a ZnO-MgO system are presented.

## 2. Experimental Section

### 2.1 Synthesis of ZnO nano particles

A solution of Zn $(AC)_2$ (2mmol) in EtOH/H2O solvent (30 mL) was added to a solution of NaOH (4 mmol) in EtOH/H2O solvent (10 mL) at room temperature under stirring. After 30 min stirring the mixture transferred into Teflon-lined stainless steel autoclaves, sealed, and maintained at 150 °C for 12 h. Subsequently, the reactor was cooled down to room temperature immediately. The resulting white solid products were centrifuged, washed with distilled water and ethanol to remove the ions possibly remaining in the final products, and finally dried at 60 °C in air. The obtained samples were characterized by X-ray powder diffraction, SEM, solid state UV, solid state florescent and Raman spectroscopies.

### 2.2 Synthesis of ZnO-MgOnano particles

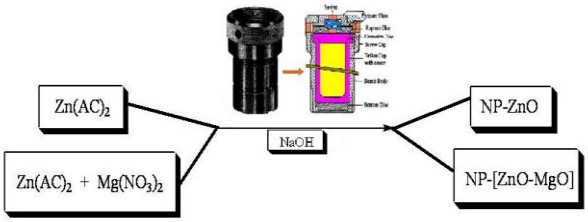
A solution of Zn $(AC)_2$ (2 mmol) in EtOH/H2O solvent (20 mL) was added to a solution of $Mg(NO_3)_2$. $6H_2O$ (2 mmol) in EtOH/$H_2O$ solvent (20 mL). Sodium hydroxide solution (10 M, 10 mL) was added to the above mixture at room temperature under stirring. After 30 min stirring the mixture transferred into Teflon-lined stainless steel autoclaves, sealed, and maintained at 160 °C for 12h. Subsequently, the reactor was cooled down to room temperature immediately. The resulting gray solid products were centrifuged, washed with distilled water and ethanol to remove the ions possibly remaining in the final products, and finally dried at 60 °C in air. The obtained samples were characterized by X-ray powder diffraction, SEM, solid state UV, solid stateflorescent and Raman spectroscopies.

The wave amplitude in each experiment was adjusted as needed. X-ray powder diffraction (XRD) measurements were performed using a Philips diffractometer of X'pert Company with mono chromatizedCukα radiation. The samples were characterized with a scanning electron microscope (SEM) (Philips XL 30) with gold coating. Raman spectra were recorded on a Labram HR 800-Jobin YvonHoribba spectrometer.

## 3. Result and Discussion

### 3.1 Structural study

The reaction between Zn acetate, Mg nitrate and sodium hydroxide to form ZnO and ZnO-MgO nanoparticles has been shown in scheme 1.




**Scheme 1**

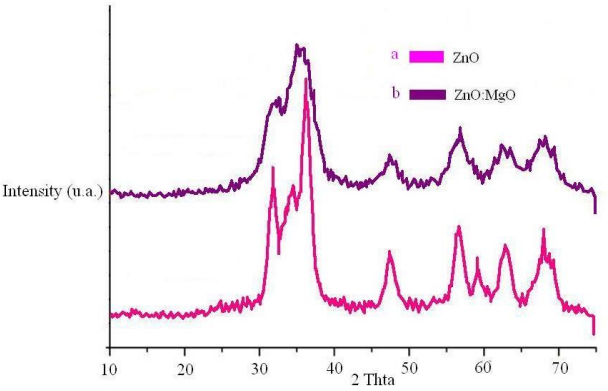



**Fig.1:** X-Ray powder diffraction pattern of (a) ZnO and (b) ZnO/MgO nanoparticles

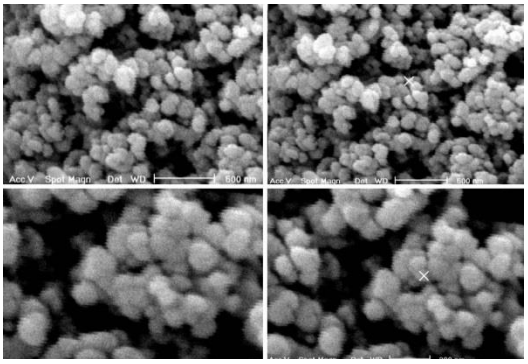


**Fig.2(a)** The SEM images of ZnO nanoparticles.

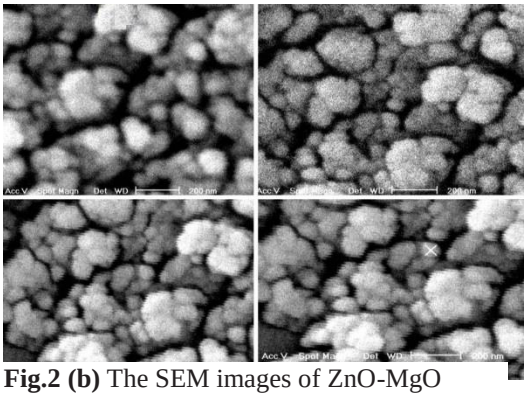


**Fig.2 (b)** The SEM images of ZnO-MgO nanoparticles

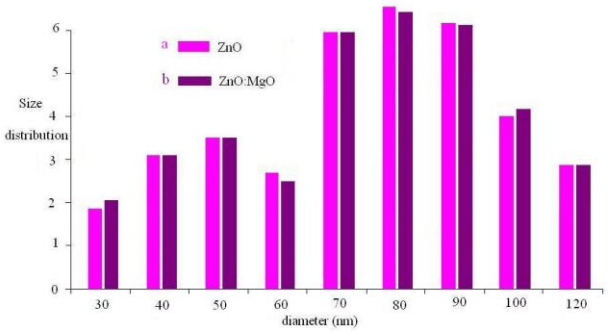



**Fig.3** Particle size histogram of the (a) ZnO and (b) ZnO/MgO nanoparticles.

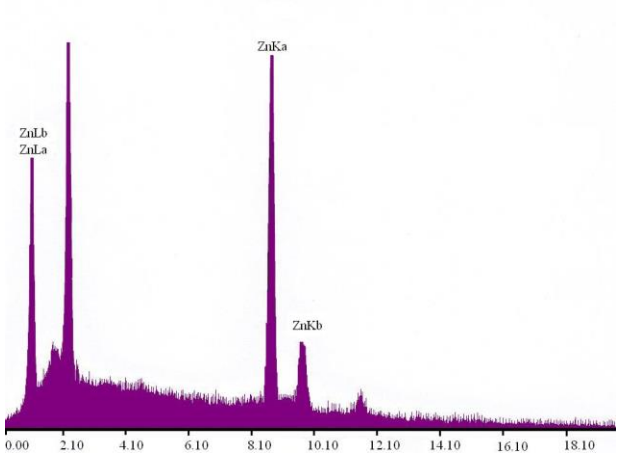



**Fig.4 (a)** EDAX analysis of the ZnO nanoparticles

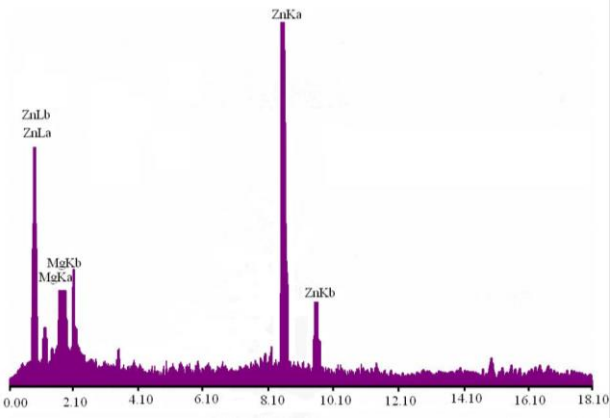



**Fig.4 (b)** EDAX analysis of the ZnO-MgO nanoparticles

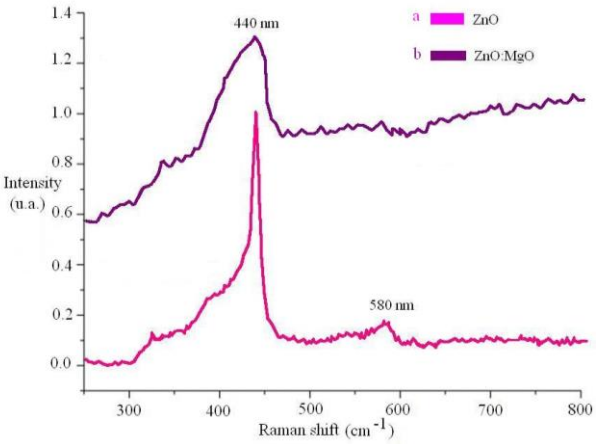



**Fig.5** Raman spectroscopy of (a) ZnO and (b) ZnO-MgO nanoparticles

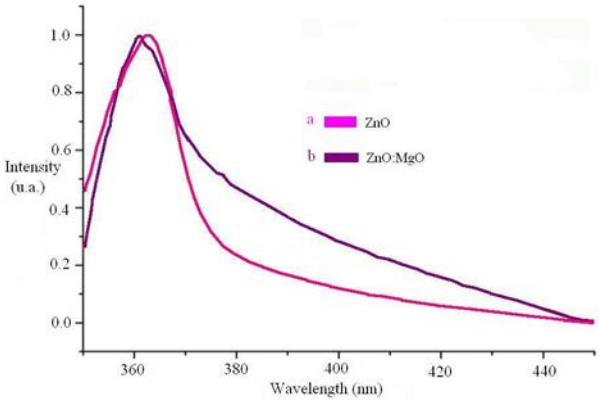



**Fig. 6** Solid state absorption spectrum of (a) ZnO and (b) ZnO-MgO nanoparticles. (λex= 320).

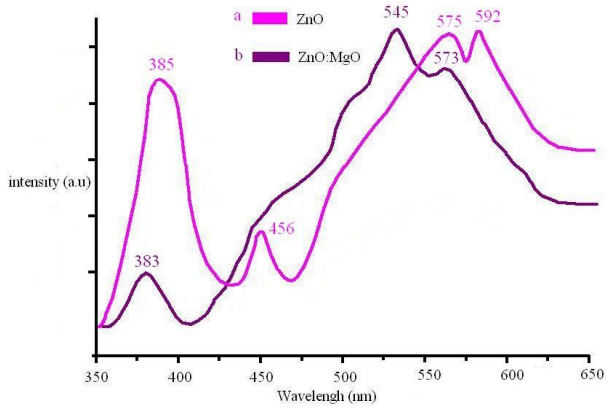



**Fig.7** Solid state PL spectrum of (a) ZnO and (b) ZnO-MgO nanoparticles. (λex= 320).

In the XRD spectra of ZnO and ZnO-MgO nanoparticles we can observe the formation of well crystalline hexagonal structure. On the other hand XRD spectra show that there was no other phase corresponding to MgO. Sharp diffraction peaks shown in Fig. 1 (a and b) indicate good crystalline of ZnO and ZnO-MgO nanoparticles. No characteristic peak related to any impurity was observed. The broadening of the peaks indicated that the particles were of nanometer scale. The morphology, structure and size of the samples are investigated by Scanning Electron Microscopy (SEM). Fig. 2 (a and b), indicates that the original morphology of the ZnO and ZnO-MgO nanoparticles are approximately spherical with the diameter varying between 40 to 80 nm.

To investigate the size distribution of the nanoparticles, a particle size histogram was prepared for ZnO and ZnO-MgO nanoparticles shown in Fig. 3 (a and b). Most of the particles possess sizes in the range from 40 to 80 nm. For further demonstration, the EDAX was performed for the ZnO and ZnO-MgO nanoparticles. The EDAX spectrum given in Fig. 4 (a and b) shows the presence of Zn and Mg as the only elementary components in the ZnO and ZnO-MgOnano particles respectively.Raman spectra of ZnO and ZnO-MgOnano particles are shows in Fig. 5 (a and b). ZnOwurtsit structure shows six Raman active modes. Raman spectra of ZnO nanoparticles show to sharp peak at 580 and 440 cm-1. Raman spectra of ZnO-MgOnano particles shows only one sharp peak at 440 cm-1 correspond to vibration mode of ZnO.Fig. 6 (a and b) shows the solid state UV–vis adsorption spectra of ZnO and ZnO-MgOnano particles. Compare to ZnO-MgO, maximum adsorption wave-lengths of ZnO have 4-6 nm red shift. Fig. 7(a and b) shows the solid state florescent (PL) spectra of ZnO and ZnO-MgO nanoparticles. Compare to ZnO-MgO, maximum excitation wave-lengths of ZnO have 4-6 nm red shift.

## 4. Conclusion

ZnO and ZnO-MgOnanopowders prepared by solvothermal method without any stabilizer or additive. The ZnO and ZnO-MgO particle size was controlled by varying the temperature and aging time of the reaction in solvothermal reactor. The ZnO particle size distributions determined from SEM images and PL spectral data for the ZnO and ZnO-MgO solid state were in good agreement. The PL results for the ZnO and ZnO-MgO nanoparticles showed significant defects in their morphology due to their large surfaces, while the green emission peak in the nanoparticles was due to a decrease in the amount of excitons. Additionally, the PL results showed a blue-shift of the UV peak for the samples that was placed in the low temperature zone, which occurred due to the effect of Mg on the band-gap of the ZnO nanostructures.

## Acknowledgements

Supporting of this investigation by Lorestan University, Khorramabad, IRAN and Giti-Pasand industrial group of Isfahan's gratefully acknowledged.

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