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**Investigation of magnesium addition in ZnO matrix using group II heptahydrate**

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**Abstract**

ZnO and MgZnO nanoparticles were prepared by the co-precipitation method utilizing zinc sulfate heptahydrate and magnesium sulfate heptahydrate; structural measurements were also carried out. An x-ray diffraction (XRD) study indicated that no peaks for other possible phases such as MgO or MgZn intermetallic compounds indicating pure wurtzite structure. All nanoparticles crystallized in a hexagonal wurtzite structure with different orientation diffraction peaks; the main peaks were (100), (002), and (101). Grain size (D) increased with increasing Mg concentrations. A scanning electron microscopy (SEM) analysis revealed that nanoparticle size increased by increasing the Mg concentration in a good qualitative with Scherrer equation and not only the size even the grain shape changed. In addition, optical measurements were taken infer that the band gap energy concentration in a good qualitative with Scherrer equation and not only the size even the grain shape.

**1. Introduction**

In international research community zinc oxide (ZnO) in different forms has attracted much attention [1]. Zinc oxide has ~3.3 eV wide bandgap, wurtzite crystal structure, ~60 meV high exciton binding energy, and piezoelectric properties [2] with lattice parameters a = 3.296 Å and c = 5.2065 Å [3]. Zinc oxide is suitable for several applications such as piezoelectric [4], sensors [5], surface acoustic devices [6], transparent electrodes [7], UV absorber [8], light-emitting diodes (LED) [9–11], lasers [12], and solar cells [13]. At Room Temperature (RT), ultraviolet (UV) luminescence has been reported for nanoparticles (NPs) [14] and thin films (TFs) [15]. ZnO absorbs ultraviolet, transparent to visible light and its bandgap changes by doping with elements such as Mn [16] and Cd [17]. Several chemical sources are used to prepare ZnO including zinc chloride [18], zinc acetate [19], and zinc nitrate [20]; however, zinc sulfate is rarely used [21]. Several physical and chemical growth techniques such as radio frequency (RF) magnetron sputtering, chemical vapor deposition (CVD), electron-beam evaporation, spray pyrolysis, ion beam evaporation, and sol-gel process have been utilized for the synthesis of ZnO.

According to Mallika et al. [22], the crystal structure of Mg doped ZnO NPs has maintained its wurtzite arrangement for Mg content less than ~40% as the original structure, therefore the structure of MgZnO appeared without showing MgO rocksalt phase for such concentration. In general, Mg atoms substitute Zn lattice sites in the crystal [23]. Moreover, doping ZnO with Mg has been studied by different ways to various
objectives. The effect of doping has been found to influence optical, electrical, and magnetic properties. Doping ZnO by Mg increases the bandgap [19–21] while on the other hand some groups reported a bandgaps decrease [24–26] due to various reasons. The bandgap of the ZnO changed depending upon many factors such as the concentration of the doping, the molar of the solvent and the temperature degree of synthesis and annealing [24]. Doping ZnO allows to tune the luminescence to cover the ultra-violet and visible light, hence the whole optical properties including bandgap energy and absorption changed. The ZnO luminescence doped with Mg was reported in different wavelengths such as UV [27], blue [28], green [29, 30], orange [19] and red [31]. The ZnO spectrum has a strong absorption in a UV range [32]. The absorption of ZnO NPs has a blueshift with doping by Al [32], and according to Yung et al [33] has a redshift with doping by Sn. While the absorption spectrum of Mg doped ZnO NPs has a redshift [25] and in other hand, has a blueshift [34].

Therefore, this research will prepare ZnO and Mg-doped ZnO NPs with different concentrations (x = 10% and 20%) utilizing zinc sulfate heptahydrate by the co-precipitation method. Then, the research will investigate the effect of high Mg concentration in the structure and optical properties for ZnO and Mg doped ZnO NPs. There is no research work on high Mg concentration with using a co-precipitation method, to the best of our Knowledge. Also, there is no research work on zinc sulfate heptahydrate as a source for preparing ZnO.

2. Experimental method

2.1. Materials

For the preparation of our particles the following materials were used: zinc sulfate heptahydrate [zinc sulfate-7-waterAR] [ZnSO₄,7H₂O] (Avonchem Ltd), extra pure sodium hydroxide [NaOH] (laboratory regent and fine chemical) pellets, cetrimonium bromide [C₁₉H₄₂BrN] (CTAB) (Central Drug House (CDH)), magnesium sulfate heptahydrate [magnesium sulfate-7-waterAR] [MgSO₄,7H₂O], and distilled water.

2.2. Procedure

To prepare ZnO NPs, a 100 ml of 0.2 M NaOH (dissolved in distilled water) was added dropwise into a solution containing 100 ml of 0.1 M Zinc sulfate heptahydrate (dissolved in distilled water) under constant stirring. A capping agent of 10 mg of CTAB, inhibiting the anomalous growth of magnesium hydroxide crystals [21], was added during the precipitation. The resulting solution was then kept at RT for at least 18 h under constant stirring. The precipitates obtained, a white precipitate, was filtered, washed several times with distilled water and then with ethanol. It was then dried at 100 °C in an oven for 4 h and calcined in air at 500 °C for 4 h.

To prepare Mg-doped ZnO NPs, NaOH was added to a mixture of 0.1 (0.01 mol) M zinc sulfate heptahydrate and a 0.01 M (1.11 × 10⁻³ mol) magnesium sulfate heptahydrate solution. The same procedure was repeated to obtain the other sample with different Mg concentrations by following equation (1) to calculate the percentage of Mg in a sample. The obtained samples were calcined in air at 500 °C for 4 h to obtain the Mg-ZnO NPs.

\[
x(\text{content of Mg in a sample}) = \frac{\text{mol of Mg}}{(\text{mol of Mg} + \text{mol of Zn})}
\]

(1)

3. Results

3.1. XRD analysis

XRD peaks are observed in all the sample patterns corresponding to different orientation shown in figure 1 with mainly 100, 002, 101. All XRD has a similar behavior, therefore, the reflection planes indicate that the prepared samples are of a hexagonal wurtzite ZnO structure. No peaks have been seen for other possible phases such as MgO or MgZnO intermetallic compounds indicating pure wurtzite structure. Most of the nanoparticles have their strongest reflection on the 101 plane. This indicates that the preferential orientation is the 101 plane for all samples [1, 35, 36], as seen in figure 1. The peaks positions of the planes (100), (002) and (101) of the samples shows in figure 2. Notably, a small shift moved toward low angle side in sample doped by Mg, the evidence that the structure includes Mg²⁺ ions in the hexagonal wurtzite ZnO structure [37].

3.2. Grain size

The average grain size (D) was calculated using the Debye–Scherrer equation (2) and depending on (100), (002) and (101) planes which is the three highest peaks.
Where $k$ is a constant equal 0.9, $\lambda$ is the wavelength of the x-ray ($\lambda = 1.5405$ Å), $\beta$ is the Full Width at Half Maximum (FWHM), and $\theta$ is the diffraction angle.

The average grain size ($D$) increased with increasing the Mg concentration, in the 10%Mg doped sample and increased again in the 20%Mg doped sample as shown in table 1. The relation between the grain size and Mg concentration illustrated in figure 3. However, the average FWHM ($\beta$) decreased with increasing Mg concentration. This result agrees with the Debye–Scherrer equation, where $\beta$ is inversely proportional to $D$.

Moreover, the annealing temperatures supported the increase in the grain size of the ZnO and Mg-doped ZnO samples; these results agreed with published work [38].

The grain size of Mg-doped ZnO NPs varied as some authors observed increasing grain size [39, 40], while others a decreasing grain size [41–43]. Furthermore, some studies had a non-systematic orientation [22, 38, 44–46]. The reason for the decreasing grain size is the retarding force. The retarding force increased when the number of obstacles per unit volume adhered further to the ZnO surface. For this reason, the grain size of Mg-doped ZnO NPs decreased when the Mg ions increased [42]. On the other hand, the increase in grain size may be due to the accommodation of Mg to the ZnO lattice.

### 3.3. Lattice constant

ZnO has a wurtzite structure with lattice parameters $a = 3.24$ Å and $c = 5.20$ Å [47]. Equation (3) can be used to calculate the hexagonal structure measured by XRD as follows [48]:

$$\sin^2 \theta = \frac{\lambda^2}{4a^2} \left\{ \frac{4}{3} (h^2 + hk + k^2) + \left( \frac{a}{c} \right)^2 l^2 \right\},$$

where $\theta$ is the Bragg diffraction angle; the source of the x-ray wavelength is $\lambda(Cu) = 1.5406$ Å; $a$ and $c$ are the lattice parameters; and $h$, $k$, and $l$ are Miller indices. The lattice parameters were calculated for the (100) and (002) planes. The lattice constant $a$, can be calculated by equation (4) and the lattice constant $c$, can be calculated by equation (5):

For (100): $a = \frac{\lambda}{\sqrt{3}} \sin \theta_{100},$  

For (002): $c = \frac{\lambda}{\sin \theta_{002}}.$

The lattice parameters $a$ and $c$, and the ratio between $c$ and $a$ are shown in table 1. From table 1 the lattice parameters $a$ and $c$ in undoped ZnO NPs were 3.2425 Å and 5.1935 Å, respectively. On increasing Mg content
during the synthesis, the values of lattice constants a and c very slightly increased while the ratio c/a remained almost constant. A similar result was observed in other Mg-doped ZnO [40, 46]. This results of an alteration in its lattice parameter confirm again to that the Mg$^{2+}$ ions set inside the ZnO structure [25]. In addition, the reason behind the slightly increasing in the lattice constant for high Mg concentration (≥8%) is due to the anion-cation Zn–O bond length accordance with Vegard’s law [49].

**Figure 2.** Diffraction peak positions of (a) (100), (b) (002) and (c) (101) planes of undoped and Mg doped ZnO with Mg concentration (x = 0, 10% and 20%) NPs.

| Sample       | Grain size (nm) | Energy gap (eV) | Lattice constant a (Å) | Lattice constant c (Å) | c/a (Å) | The strain ε |
|--------------|----------------|----------------|------------------------|------------------------|---------|--------------|
| ZnO          | 49.45          | 3.255          | 3.2425                 | 5.1936                 | 1.60173 | 0.00244      |
| Mg-ZnO 10%   | 57.09          | 3.165          | 3.24525                | 5.1987                 | 1.60194 | 0.00209      |
| Mg-ZnO 20%   | 73.99          | 3.169          | 3.2459                 | 5.1992                 | 1.60177 | 0.00163      |
3.4. Average strain
The average strain was calculated using the Stokes-Wilson equation (6)

\[
\epsilon = \frac{\beta \cos \theta}{4 \sin \theta}
\]

Where \( \beta \) in radian. The strain for (100), (002) and (101) and the average strain for the ZnO and Mg doped ZnO NPs is mentioned in table 1. As shown in the table that the average strain decreases with increasing Mg concentration. As a result of increasing the grain size, \( [50] \) observed the same result.

3.5. Morphology studies
The SEM images in figure 4 show that the surface morphology of the NPs is strongly dependent on the concentration of the dopant. These particles have different shapes and sizes in the prepared ZnO. In case of undoped ZnO, quasi-spherical grains were found to have regular distribution with some irregular shape grains. After Mg doping, the emergence of increasing grain was noticed followed by the agglomeration of crystallites. Different morphology of the NPs and some hexagonal crystal-shaped were observed due to the defects created by the Mg doping. As the Mg-doping concentration increased, the crystallinity of the NPs significantly increased along with the grain size. The grain size increased as the Mg content increased in accordance with the grain size obtained by the XRD analysis \( [39] \).

3.6. Optical studies
The optical properties of the ZnO and Mg-doped ZnO NPs were determined from the absorption spectra in the wavelength range of 200–800 nm and are presented in figure 5. The bandgap energy was calculated using Tauc’s plot depending on the absorption wavelength in figure 6. As the photoluminescence (PL) properties of the ZnO are very sensitive to oxygen vacancies, the PL spectra of the ZnO and Mg-doped ZnO NPs have been measured in figure 7.

The absorbance of the ZnO NPs changed with the Mg doping percentage. As shown in figure 5, in the visible region, the absorbance value of ZnO and Mg-doped ZnO NPs decreased as the Mg doping concentration increased. This indicates that by increasing Mg doping, the band edge of the ZnO shifts to a higher wavelength side (longer wavelength). The absorption spectra show that the ZnO and Mg-doped ZnO have a UV absorption edge in the region 350–400 nm. This absorption edge was redshifted with the increase of Mg doping concentrations in the ZnO, as in figure 5. The reason of increasing the absorption might be due to defect in the grain sizes, particle size and oxygen deficiency \( [51] \).

The UV absorption edge is directly linked to the optical band gap \( (E_g) \). The optical band gap is an important parameter for Mg-doped ZnO nanoparticles in various optical applications. ZnO is a direct band gap semiconductor. The band gap can be estimated from the absorption edge by applying Tauc’s relationship. Based on the absorption edge in figure 5, the optical \( E_g \) was estimated by assuming a direct transition between the valence and conduction bands from the equation (7):
Figure 4. SEM Images (65 nm width × 56 nm height) with scale bar at 100 nm and ×50,000 magnification of (a) ZnO, (b) 10% Mg and (d) 20% Mg-doped ZnO NPs.
As the transition in the ZnO is direct, \( n \) is set equal to \( 1/2 \):
\[
\alpha h \nu = A (h \nu - E_g)^n
\]  
(7)

where \( A \) is an energy-independent constant, \( h \) is Planck’s constant, \( h \nu \) is the photon energy, \( \alpha \) is the absorption coefficient of the NPs, and \( E_g \) is the band gap energy. The \( E_g \) was obtained by extrapolating the linear portion of the plot \( (\alpha h \nu)^2 \) versus the \( (h \nu) \) plot. Tauc’s plot is illustrated in figure 6. The NPs absorption coefficient was calculated using Beer–Lambert’s law in equation (9):
\[
\alpha = \frac{2.303}{t} \times A
\]  
(9)

where \( t \) is the thickness of the samples in a UV–vis powder holder (1 mm) and \( A \) is the NPs absorbance.

As seen in figure 6, the calculated optical \( E_g \) of the prepared ZnO and Mg-doped ZnO NPs varies between 3.255 and 3.169 eV. We observe a general decrease in the optical \( E_g \) through the Mg doping. First, the band gap energy decreased from 3.255 eV for pure ZnO to 3.165 eV for the 10% and followed by an increase to 3.169 eV for the 20% Mg concentration. The reason behind decreasing bandgap energy with high Mg content is the strong quantum confinements and enhancement the surface area to volume ratio, [52]. The decreasing in \( E_g \) and enhancement of redshift confirm the presence of Mg\(^{2+}\) in some sites in the ZnO structure [52].
Figure 7 shows the PL spectra for ZnO and Mg-doped ZnO NPs upon 345 nm and 500 nm excitations, respectively. The emission spectrum of the NPs indicates a UV region near 380 nm and a broad deep level emission in the visible region at 500 nm. The spectra showed that the annealing temperature increased the PL peak intensity \[53\]. Moreover, figure 7 indicates that the intensity increases by increasing the Mg concentration \[8\]. ZnO PL spectra at RT as usually known are three major peaks: first emission peak at UV near-band-edge around 380 nm, the second one at green emission peak around 520 nm, and the last one in red or orange emission peak around 600 nm \[54, 55\]. Therefore, UV emission in figure 7 is attributed to the radiative recombination of excitons (exciton emission) which is one of the band-edge emissions. Whereas the two broad visible bands are generally attributed to deep-level defects in ZnO crystal, like vacancies and interstitials of zinc and oxygen which is surface anion vacancies. This may be due to the tunneling of surface-bound electrons through preexisting trapped holes. The right shift in the PL peaks (wavelength shift to 500 nm) is due to the addition of Mg, increasing the energy of the vacancy formation and increasing the stability of the oxygen \[25, 54, 56–58\].

4. Conclusion

Mg-doped ZnO NPs with concentrations of 0%, 10% and 20% were prepared by a sol-gel method. The studies and investigations in this work focused on the effects of Mg-doping concentrations on the structural, morphological and optical properties of the NPs. The XRD study indicated that no peaks for other possible phases such as MgO or MgZn intermetallic compounds indicating pure wurtzite structure. All NPs crystallized in a hexagonal wurtzite structure with different orientation diffraction peaks; the main peaks were (100), (002), and (101). Grain size increased with the increasing Mg concentrations. SEM analysis revealed that the size of NPs increased by increasing the Mg concentrations agreed well with the XRD analysis. Optical measurements indicate that the \(E_g\) of the ZnO and Mg-doped ZnO with different Mg concentrations was found to be between 3.255 eV and 3.169 eV. The photoluminescence emission spectra show a peak (ultraviolet band near 380 nm and another peak near 500 nm (green band) for all NPs due to the surface defects in the ZnO such as oxygen vacancies and other impurities.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).
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