

Synthesis and characterizataion of magnesium (Mg) doped ZnO nanomaterials with variations in growth time for DSSC applications

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ABSTRACT

This research aims to investigate the effect of growth time variation of magnesium-doped zinc oxide (ZnO:Mg) nanomaterials on the efficiency of dye-sensitized solar cells (DSSC). The synthesis method employed was the seed-mediated hydrothermal process, with a Mg doping concentration of 4% mol and growth times ranging from 9 and 10 hours. The variation of growth time was selected to analyze the relationship between the morphological evolution of ZnO:Mg nanorods and their optical properties as well as crystal structure. The samples were characterized using ultraviolet-visible spectroscopy (UV-Vis) to study optical properties, X-ray diffraction (XRD) to determine crystal structure, field emission scanning electron microscopy (FESEM) to observe morphology, and energy dispersive X-ray (EDX) to identify elemental composition. The results showed that the variation of growth time affected the optical properties, crystal structure, morphology, and DSSC efficiency. The ZnO:Mg sample with an 9-hour growth time exhibited the highest absorbance of 1.45 a.u., the lowest band gap energy of 3.33 eV, and the best crystallinity with an FWHM value of 0.23° and a crystal size of 35.27 nm. FESEM analysis revealed denser and more homogeneous nanorod morphology, while EDX analysis showed the highest Mg content of 0.70% atomic percentage in the 9-hour sample.

Keywords: DSSC; growth time; hydrothermal; nanorod; ZnO:Mg

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INTRODUCTION

Sunlight is the largest source of energy available in nature. It can be converted into electrical energy with the aid of semiconductor devices known as solar cells or photovoltaic cells. Solar cell technology has evolved through three generations [1]. Solar energy has garnered significant attention as an energy source due to its eco-friendly nature, low cost, and virtually inexhaustible supply [2].

The energy provided by the sun manifests as both light and heat. Sunlight can be converted into electrical energy using semiconductor devices called solar cells or photovoltaic cells; this technology has developed through three generations [3]. Solar cells based on metal oxide nanomaterials represent a promising approach for energy conversion, offering a way to reduce the consumption of fossil fuels. Among the various types of solar cells, dye-sensitized solar cells (DSSCs) have emerged as a notable

form of renewable energy technology. Solar energy is highly attractive as an energy source because it is environmentally friendly and essentially inexhaustible [4].

Metal oxide semiconductors are the most widely used materials in this field. Zinc oxide (ZnO) is particularly compelling due to its versatility and broad range of applications; it serves as one of many metal oxide semiconductor materials used in DSSCs [5]. ZnO exhibits high chemical and thermal stability, as well as a large band gap, which allows it to absorb photon energy from sunlight—provided that the photon energy exceeds the band gap. This property enables ZnO to absorb ultraviolet (UV) radiation from the sun [6]. With a band gap of 3.37 eV, ZnO semiconductors are well-suited for use in photovoltaic cells, such as DSSCs, because they can operate within the UV light spectrum [7]. Through synthesis, ZnO nanomaterials can be produced in various mor-

phologies, including nanorods, nanowires, nanotubes, and nanosheets [8].

Doping processes are also employed to expand the utility of ZnO across a wider range of applications. Semiconductors doped with other atoms are crucial for controlling electrical and other physical properties to suit specific applications. Doping ZnO with Mg has been shown to effectively modify its optical and catalytic properties, particularly by tuning its band gap energy [9]. Magnesium (Mg) is a metal utilized in various applications, including refractory materials, optical equipment, and heating elements. Mg-doped ZnO also possesses unique electron-blocking characteristics due to its wide band gap. The structural, surface morphological, optical, thermal, and electrical properties of Mg-doped ZnO have been investigated [10]. The doping process is employed to expand the utility of ZnO across a range of applications. Semiconductors doped with other atoms are essential for tailoring electrical and other physical properties to meet the requirements of specific applications [11].

RESEARCH METHODS

Synthesis of Mg-doped ZnO Nanorods via the Seed-Mediated Hydrothermal Method

The synthesis of Mg-doped ZnO nanorods is carried out in two primary stages: seeding and growth. An FTO substrate (2.0 cm × 1.2 cm) is first cleaned with Decon detergent, rinsed with tap water and deionized (DI) water, and then subjected to sequential sonication in an ultrasonic bath—using DI water, ethanol, and DI water—for 15 minutes each at 60°C. The substrate is subsequently dried in an oven at 90°C for 15 minutes. In the seeding stage, a seed solution is prepared by dissolving zinc acetate dihydrate (ZAD) in 10 mL of ethanol. A 200 μ L aliquot of this solution is applied to the FTO substrate, which is heated on a hot plate at 100°C for 15 minutes. The substrate is then annealed in an oven at 250°C for 1 hour to strengthen the ZnO seed layer. In the growth stage, 0.595 g of zinc nitrate hexahydrate

(ZNH) is dissolved in 10 ml of DI water, and 0.14 g of hexamethylenetetramine (HMT) is dissolved in another 10 ml of DI water. The seeded FTO substrate is placed into a synthesis vial at an angle of approximately 45°. A 4 mol% Mg-doping solution—prepared by dissolving 0.3 g of Mg in 1 ml of acetone—is added to the growth solution. The synthesis vial is then heated in an oven at 90°C for varying durations: 9 hours and 10 hours.

DSSC Fabrication

The counter electrode was prepared by coating an FTO substrate with a plastisol solution, applied dropwise and distributed evenly. This was heated on a hot plate at 100°C for 10 minutes, a process repeated three times. Subsequently, the substrate was immersed in an AgNO₃ solution (dissolved in deionized water) and heated in an oven at 90°C for 8 hours. The natural dye was prepared by crushing 25 grams of dragon fruit peel, adding 80 ml of ethanol and 20 mL of distilled water, sonicating the mixture for 30 minutes, and then allowing it to soak for 3 hours. The electrolyte was prepared by dissolving 0.8 grams of KI in 10 ml of acetonitrile, adding 0.127 grams of iodine, and then incorporating PEG 400 gel and chloroform; the mixture was stirred at 80°C for 60 minutes. A commercial electrolyte, Iodolyte AN-50, was also used for performance testing. The DSSC was fabricated by assembling the components into a sandwich structure. The working electrode (FTO + ZnO:Mg + dye) was placed atop the counter electrode (FTO + plastisol + AgNO₃). The active test area was defined using parafilm, creating a circular surface area of 0.25 cm². The two sides were secured with clamps, followed by the injection of the Iodolyte AN-50 electrolyte.

Characterization

Optical properties were analyzed using UV-Vis spectroscopy over a wavelength range of 200 – 800 nm. The band gap energy was determined via the Tauc plot method, based on the

equation $(\alpha h\nu)^2 = A(h\nu - E_g)$. The crystal structure of the samples was identified using X-ray diffraction (XRD) over a 2θ angular range of $20^\circ - 60^\circ$. Crystallite size was calculated using the Scherrer equation: $D = K\lambda / (b \cos\theta)$. The surface morphology of the samples was analyzed using field emission scanning electron microscopy (FESEM) at magnifications of up to $10,000\times$. The elemental composition of the samples was identified using energy dispersive X-ray (EDX) spectroscopy.

RESULTS AND DISCUSSION

UV-Vis Results

UV-Vis spectroscopy was employed to determine the optical properties and bandgap energy of Mg-doped ZnO (ZnO:Mg) grown over durations of 9 and 10 hours. Figure 1 presents the results of the UV-Vis characterization. The obtained absorbance data reveal variations in light absorption intensity corresponding to the growth duration. Using the Tauc plot method, the bandgap of the ZnO:Mg samples was found to range from 3.34 eV (for a 9-hour growth duration) to 3.36 eV (for a 10-hour growth duration). The increase in the bandgap with growth duration is attributed to a reduction in crystal defects and greater uniformity in nanorod size [1]. A smaller bandgap enhances the stability of excited electrons while remaining within the optimal range for light absorption by a dye.

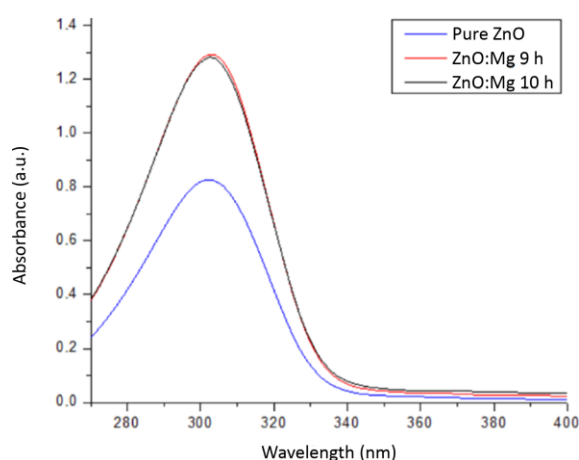


Figure 1. Absorption spectra of Mg-doped ZnO samples with varying growth durations.

The bandgap energy is determined by extrapolating a straight line to the x-axis. This energy value is analyzed to investigate the influence of growth duration on the ZnO bandgap [12]. Table 1 presents the results of the linear extrapolation to the x-axis, providing the bandgap energy values for each sample. The bandgap of undoped ZnO is 3.37 eV, which falls within the typical range for ZnO. Mg-doped ZnO samples grown for 9 to 10 hours exhibit smaller bandgaps compared to pure ZnO. The introduction of Mg dopants into the ZnO structure—at growth durations of 9 and 10 hours—results in a narrower bandgap, a consequence of the difference in ionic radii. This phenomenon arises from the formation of additional energy levels between the valence and conduction bands, resulting from the interaction between Mg electrons and the electronic structure of the ZnO [13].

Table 1. Energy gap of ZnO nanorods doped with varying growth durations.

Sample	Gap energy (eV)
ZnO	3.37
ZnO:Mg (9 hours)	3.34
ZnO:Mg (10 hours)	3.36

X-ray Diffraction Pattern Analysis

The diffraction patterns of the samples are presented in Figure 2, which indicates that the synthesized samples are crystalline, as evidenced by sharp diffraction peaks. The figure displays five diffraction peaks at 2θ angles of 31.75° , 36.23° , and 47.62° . Analysis using "HighScore" software confirms that these diffraction peaks correspond to the (010), (002), (011), (012), and (013) crystal planes. The strongest line (highest peak) occurs at a 2θ angle of 34.66° , corresponding to the (002) crystal plane.

All samples—both undoped ZnO and Mg-doped ZnO (ZnO:Mg)—exhibit diffraction peaks characteristic of a hexagonal wurtzite crystal structure, indicating that the Mg-doping process and variations in growth duration do not alter the primary crystalline phase of the

ZnO. At a growth duration of 9 hours, the peak intensities tend to be lower and the peaks appear broader, suggesting suboptimal crystallinity and relatively small crystallite size. In the Mg-doped ZnO samples, a shift in the diffraction peaks toward larger 2θ angles is observed compared to pure ZnO; this indicates a contraction of the lattice parameters resulting from the substitution of Zn^{2+} ions with Mg^{2+} ions, which have a smaller ionic radius, within the wurtzite ZnO structure [14].

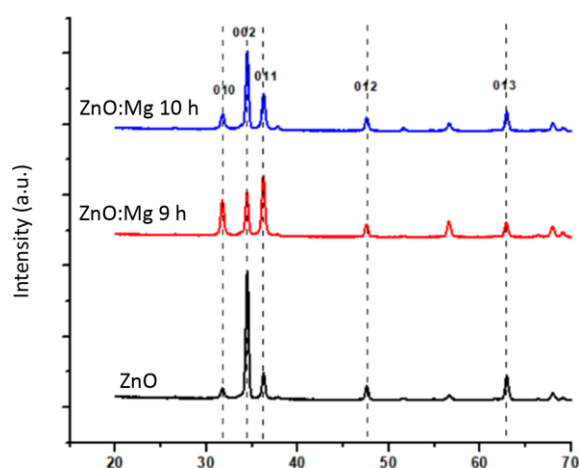


Figure 2. XRD patterns of Mg-doped ZnO samples with varying growth durations

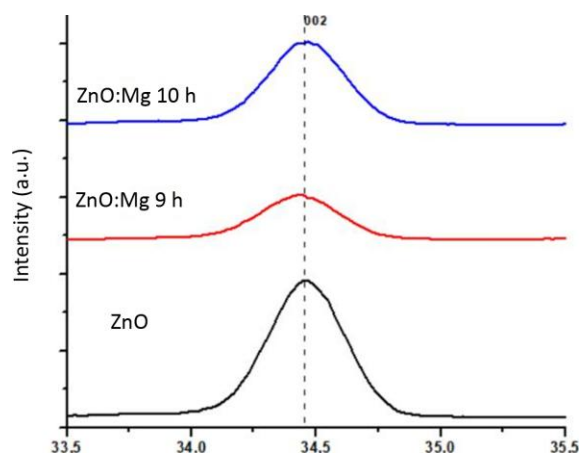


Figure 3. Diffraction peak shift for the (002) plane.

The XRD results, shown in Figure 3, reveal that the primary diffraction peak for ZnO corresponds to the (002) plane. Following Mg-doping, a shift in the position of the (002) peak toward a higher 2θ angle is observed. This shift is caused by the incorporation of Mg^{2+} ions—which have a smaller ionic radius (0.057 nm)

compared to Zn^{2+} ions (0.074 nm)—into the ZnO lattice, resulting in a contraction of the lattice parameters [15]. This is consistent with Bragg's law, where a decrease in the interplanar spacing (d -spacing) leads to an increase in the 2θ value [16]. This phenomenon of diffraction peak shifting also indicates that doping influences the strain and distortion of the ZnO crystal lattice, which can subsequently affect the optical properties and the efficiency of a DSSC.

Table 2. FWHM values and crystallite sizes of Mg-doped ZnO nanorods.

Sample	FWHM (°)	Crystal size (nm)
ZnO	0.37	22.24
ZnO:Mg (9 h)	0.35	23.48
ZnO:Mg (10 h)	0.35	23.46

As shown in Table 2, the FWHM values and crystallite sizes of the Mg-doped ZnO nanorods vary with growth time. The pure ZnO sample exhibits an FWHM of 0.37° with a crystallite size of 22.24 nm, whereas the ZnO:Mg sample grown for 9 hours shows an FWHM of 0.35° and the largest crystallite size, at 23.48 nm. At a growth time of 10 hours, the FWHM remains 0.35° , with a crystallite size of 23.46 nm. These results indicate that Mg doping and variations in growth time significantly influence the crystallinity of ZnO; a smaller FWHM correlates with a larger crystallite size and superior crystal quality, while a larger FWHM suggests the presence of crystal defects, which result in a smaller crystallite size.

Morphological Analysis of Samples via FESEM Images

Figure 4 presents FESEM images of ZnO and Mg-doped ZnO samples, with growth durations ranging from 9 to 10 hours at 1-hour intervals, all captured at $50,000\times$ magnification. The images reveal a hexagonal nanorod morphology for the ZnO, with varying diameters across the samples. Differences in nanorod size and density are observed for each growth duration, indicating that both the growth time and the presence of Mg dopants influence the

nanostructure growth mechanism [7]. Observations indicate that the diameters of the Mg-doped ZnO (ZnO:Mg) nanorods fall within the nanometer scale, as measured using ImageJ software.

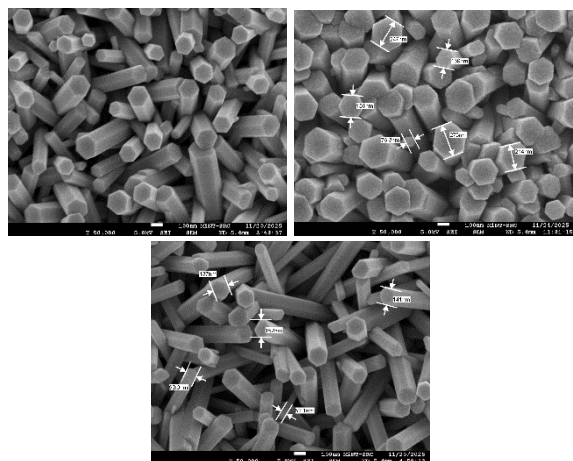


Figure 4. FESEM images of Mg-doped ZnO nanorods with growth durations ranging from 9 to 10 hours, at 50,000x magnification.

Pure ZnO nanorods exhibit non-uniform sizes and irregular morphology. A more regular and uniform morphology begins to emerge in the ZnO:Mg samples at a growth duration of 7 hours, with optimal conditions achieved at 8 hours. The ZnO:Mg nanorods display a homogeneous morphology and a higher density compared to other samples. A high nanorod density can enhance light absorption capacity by increasing the material's active surface area [12].

A growth duration of 10 hours results in less well-defined ZnO:Mg nanorod structures, characterized by rougher surfaces, structural deformations, and signs of the nanorods beginning to break apart. This condition indicates "overgrowth," which leads to an increase in structural defects [2].

Increasing the synthesis time results in larger particle sizes, as atomic diffusion becomes more active during the crystal growth process. At excessively high temperatures, particles may undergo excessive coalescence, leading to agglomeration as the thermal energy exceeds the optimal threshold [6]. FESEM observation results indicate that a growth duration of 9 hours yields the best morphology, characterized by

relatively homogeneous particle sizes and minimal agglomeration.

Elemental Composition Analysis

The results of EDX characterization, showing the elemental composition of pure ZnO samples and magnesium-doped ZnO samples (with growth durations of 9 to 10 hours, at 1-hour intervals), are presented in Figure 5. The EDX spectra for all samples exhibit peaks corresponding to the elements present within the samples [17]. Pure ZnO contains Zn and O, while Mg-doped ZnO (ZnO:Mg) grown for 9–10 hours contains Zn, O, and Mg [8]. The presence of Zn and Mg peaks was detected in each sample; the specific elemental content—expressed in terms of atomic and weight percentages—is detailed in Table 3 and Table 4.

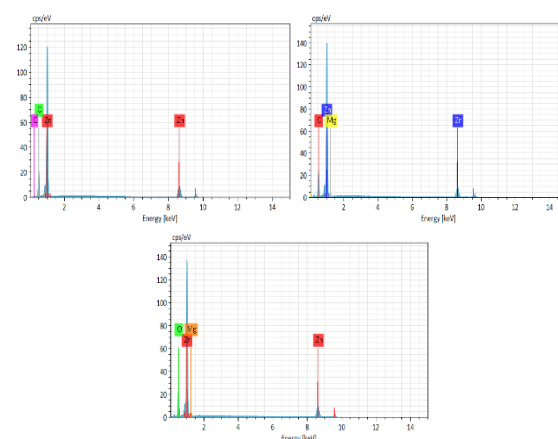


Figure 5. EDX spectra of ZnO and Mg-doped ZnO (ZnO:Mg) nanomaterials with growth durations of 9 – 10 hours.

Table 3. Atomic percentages of ZnO and Mg-doped ZnO (ZnO:Mg) samples (9 – 10 hours growth duration).

Sample	% Atom		
	Zn	O	Mg
ZnO	52.93	47.08	0
ZnO:Mg (9 h)	49.80	49.54	0.66
ZnO:Mg (10 h)	49.31	50.09	0.64

A comparison of the atomic and weight percentages for ZnO and Mg-doped ZnO (ZnO:Mg) with growth durations of 6 – 11 hours is provided in Table 3 and Table 4. The data show that the incorporation of Mg leads to

a decrease in both the atomic and weight percentages of Zn. This indicates that Mg has successfully replaced a portion of the Zn atoms during the synthesis process.

Table 4. Weight percentages of ZnO and Mg-doped ZnO (ZnO:Mg) samples (9–10 hours growth duration).

Sample	% Weight		
	Zn	O	Mg
ZnO	82.12	17.88	0
ZnO:Mg (9 h)	80.18	19.46	0.36
ZnO:Mg (10 h)	79.90	19.76	0.34

The duration of the growth process also correlates with a decrease in the atomic and weight percentages of Mg; this is likely due to the evaporation of Mg during longer growth periods [18]. Therefore, the growth duration influences changes in the elemental composition of the samples.

CONCLUSION

This study demonstrates that varying the growth duration of Mg-doped ZnO nanorods significantly influences their resulting optical properties and crystal structure. UV-Vis characterization reveals an increase in the band gap energy from 3.34 eV at a growth duration of 9 hours to 3.36 eV at 10 hours. This expansion of the band gap is attributed to Mg doping—stemming from the difference in ionic radii between Mg^{2+} and Zn^{2+} —and suggests a reduction in crystal defects, leading to the formation of more uniform nanorods. XRD analysis confirms that all samples are crystalline, with a primary peak corresponding to the (002) plane. A shift in the diffraction peaks toward higher 2θ angles following the addition of Mg indicates the incorporation of Mg^{2+} ions into the ZnO lattice, which causes a contraction of the lattice parameters. Variations in growth duration also affect the full width at half maximum (FWHM) values and crystallite size; the largest crystallite size was observed at 10 hours, while longer durations resulted in fluctuations due to an increase in crystal defects. FESEM analysis reveals a more homogeneous and ordered surface

morphology characterized by hexagonal cross-sections, while EDX analysis shows the highest Mg content in the 9-hour sample, measured at 0.36 wt% (0.66 at%).

AUTHOR CONTRIBUTIONS

Iwantono Iwantono: Conceptualization, methodology, supervision, and writing—review and editing. Fitriatunisa' Al-Fajri: Investigation, data curation, formal analysis, visualization, and writing—original draft. Marlia Morsin: Validation and writing—review and editing. Friska Ziliwu: Investigation, formal analysis, and writing—review and editing. All authors approved the final manuscript.

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DECLARATION OF AI USE

The authors declare that no AI tools were used to prepare this manuscript.

CONFLICT OF INTEREST

The authors declare no conflicts of interest.

DATA AVAILABILITY STATEMENT

All supporting data are included in this article. No additional datasets are available.

ETHICAL APPROVAL

Ethical approval was unnecessary, as no humans or animals were involved.

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