



## Enhanced The Properties of ZnO Thin Film by Graphene Oxide for Dye Sensitized Solar Cell Applications

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### ABSTRACT

In this present work, the effects of coating of graphene oxide (GO) at different concentrations (0, 0.2, 0.3, 0.4, 0.5, and 0.6 mg/ml) onto zinc oxide (ZnO) nanostructured were investigated. ZnO and ZnO coated with GO (ZnO/GO) were prepared using immersion method. The structural, morphology and optical properties of all samples have been studied using x-ray diffraction (XRD), field emission scanning microscopy (FESEM) and UV Vis spectroscopy. The peak obtained from the XRD pattern shows that all samples are in the hexagonal-wurtzite structure. The (002) peak shows the strongest intensity for all samples with the highest (002) peak obtained for the ZnO/GO sample coated at a GO concentration of 0.5 mg/ml. The diameter of ZnO/GO nanostructured samples decreased after coating with GO at concentrations of 0.2 to 0.5 mg/ml and the diameter increased again when ZnO nanostructures were coated with GO at above 0.5 mg/ml. The highest transmission spectrum was obtained for the ZnO/GO sample coated with GO at a concentration of 0.5 mg/ml. In conclusion, the effect of GO coating on ZnO nanostructured can be changed at different concentrations of GO. The optimal properties of ZnO/GO may be suitable as a photoanode in DSSC applications.

## 1. Introduction

The world's energy consumption is increasing in tandem with population growth. On the other hand, conventional energy resources are rapidly depleting, raising serious concerns about energy crises. To overcome the current issue, attempts are being explored to use renewable energy resources [1]. The dye-sensitized solar cells (DSSC) are the most popular third-generation solar cells that were developed by Grätzel and co-workers in 1991 [2-4]. Nowadays, DSSC has received a lot of attention because of its potential for low-cost solar energy conversion applications.

The DSSC is also known as photovoltaic and exhibits the capability to convert sunlight to electricity. This DSSC has emerged as a promising candidate to harvest energy due to its low-cost

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production, environmentally friendly, high-power conversion efficiency, good performance in diverse light conditions, higher indoor efficiency than silicon solar cells, long-term stability, and stable performance with light intensity [5,6].

The DSSC consists of a semiconductor photoanode, a light-absorbing sensitizer attached to the photoanode's surface, the redox electrolyte, and the counter electrode (CE). Tin oxide ( $\text{SnO}_2$ ), zinc oxide (ZnO), and titanium oxide ( $\text{TiO}_2$ ) are common metal oxides used in the formation of DSSC due to their physical and chemical properties [7]. Commonly, ZnO metal oxide was chosen as a photoanode material over all other metal oxide materials because it has a wide bandgap energy (3.37eV), strong electron mobility ( $115\text{-}155\text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ ), high excitation binding energy of 60 meV at ambient temperature, better stability against photo corrosion, and huge surface area [8,9]. The properties of ZnO such as structure, particle size, porosity, and pore size distribution will be affected the electronic device applications. Other than that, ZnO offers several intrinsic advantages, such as a greater electron migration rate, a lower process temperature, and structure can be easily created by varying the parameters process.

However, slow electron injection at the ZnO/dye interface has been reported to be the major limitation of overall photogenerated current in ZnO-based solar cells [10]. Researchers are currently looking at a variety of ways to improve electron transportation in photoelectrodes and reduce charge recombination to enhance the cell performance of ZnO-based DSSC, such as the introduction of materials with high electronic transport and charge carrier mobility. Carbon nanotubes and graphene both have high electrical conductivity and have been introduced into the photoanode of DSSC to improve cell performance by lowering charge recombination [11,12]. Recently, graphene has been identified as a potential candidate for improving dye absorption and carrier transport properties of DSSC [13]. According to Song and Wang [10], the photoelectric conversion efficiency (PCE) of the device could be enhanced by introducing reduced graphene oxide (rGO) into ZnO. The use of graphene increases the porosity of anode films and provides a huge surface area for more dye adsorption sites. Thus, it can increase dye loading and light harvesting efficiency.

Adding graphene oxide (GO) which is one of the graphene derivatives into ZnO will increase the efficiency of DSSC [14-16]. GO is a well-known carbon material that has been the subject of numerous research studies in recent years due to its multifunctional character based on its large surface area, amphiphilicity, and tuneable electronic properties through surface modification [16]. GO is made by oxidizing and exfoliating graphite and consists of a single graphene sheet with conjugated  $\text{sp}^2$  domains and  $\text{sp}^3$ -like regions. GO contains oxygen functional groups covalently bonded to carbon atoms in the basal plane and on the edges. The chemically active surface of GO allows other organic molecules or metal oxides to attach to it, changing its electrical characteristics. Víctor-Román *et al.*, [17] developed the ZnO-GO hybrids for the degradation of methylene blue under UV-light irradiation. The set of ZnO-GO hybrids has been synthesized in an ultrasonication process involving ZnO nanoparticles [17]. Boukhoubza *et al.*, [18] used the simple hydrothermal process to improve the ZnO NR/GO nanocomposites with different GO amounts. Sha *et al.*, [19] improved the PCE of DSSC by constructing ZnO/rGO photoanode by using one step electrodeposition method.

ZnO nanostructures are commonly synthesized via solvothermal, hydrothermal and sol-gel immersion methods [20]. In the fabrication of nanostructured material, a low-temperature method of preparation is gaining popularity to allow deposition on thermally adjustable substrates. ZnO nanostructured produced by immersion method are well known to be dependent on preparation parameters such as material concentration, deposition temperature, and time. The morphologies and sizes of a ZnO nanostructure are mostly controlled by concentration [21]. The immersion method is one of the processes that operates at low temperature, simple production process, and can produced in large-scale nanostructured on substrates with low energy process.

In our recent work, we used a new approach to prepare ZnO/GO nanostructured using a double solution immersion method. At first, ZnO nanostructured were grown on a glass substrate coated with ZnO seeds layer and followed by GO coating on top of ZnO nanostructured. The structural, morphological and optical properties of ZnO/GO nanostructured were investigated as photoanode in DSSC applications.

## 2. Methodology

### 2.1 Growth Process

The preparation of the ZnO doped seeded layer was explained in detail by Mohamed *et al.*, [22]. ZnO/GO nanorod arrays were grown on ZnO seeded layer-coated substrate using the aqueous solution immersion method. Zinc nitrate hexahydrate ( $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , 98.5%, Riendemann Schmidt) and hexamethylenetetramine ( $\text{C}_6\text{H}_{12}\text{N}_4$ , 99.5%, HMTA) was used as a precursor and stabilizer, respectively [22]. A measured amount of  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  was dissolved in deionized water (DI) to form aqueous solution, and then HMTA was added to the solution. The mixed solution was sonicated under an ultrasonic water bath for 30 min at 50°C. Then, the solution was continuously stirred for 3 hours at room temperature and then poured into a container with an Al: ZnO seed coated glass substrate inserted into the container. The immersion process was done by immersing sealed vessels inside the water bath for 60 minutes at 95°C for nanorod growth [23]. For the second immersion process, GO solution with different concentrations (0, 0.2, 0.3, 0.4, 0.5, and 0.6mg/ml GO) was poured into each vessel onto the ZnO nanorods. The vessels were then immersed again for 20 minutes at 95 °C. DI water was used to rinse the ZnO nanorods and finally, the thin films were annealed at 500°C for 30 minutes in ambient conditions. The as-synthesized ZnO/GO with 0, 0.2, 0.3, 0.4, 0.5 and 0.6mg/ml of GO were labeled as ZnO, ZnO/GO-1, ZnO/GO-2, ZnO/GO-3, ZnO/GO-4, and ZnO/GO-5 respectively. The ZnO/GO nanostructured structure properties were then characterized using x-ray diffraction (XRD) UV-VIS spectroscopy, field emission scanning electron microscope (FESEM), and fourier-transform infrared (FTIR) spectroscopy.

## 3. Result

### 3.1 X-ray Diffraction Analysis

The XRD analysis was used to determine the structure and diffraction peaks of samples. XRD patterns of ZnO and ZnO/GO nanostructured at different GO concentrations were presented in Figure 1. The obtained peaks confirm the hexagonal-wurtzite structure for all samples is in good agreement with JCPDS-ICDD card no. 36-1451 [18]. Based on Figure 1, there are three peaks observed for all samples which correspond to (100), (002), and (101) diffraction peaks in the range of 5°–90°. These thin films show a polycrystalline structure that displayed the highest peak intensity at the (002) plane, suggesting the structure was primarily grown along the c-axis or in the direction perpendicular to the substrate on the seed layer [24,25]. The relative peak intensity of the nanostructured was calculated using Eq. (1) [26].

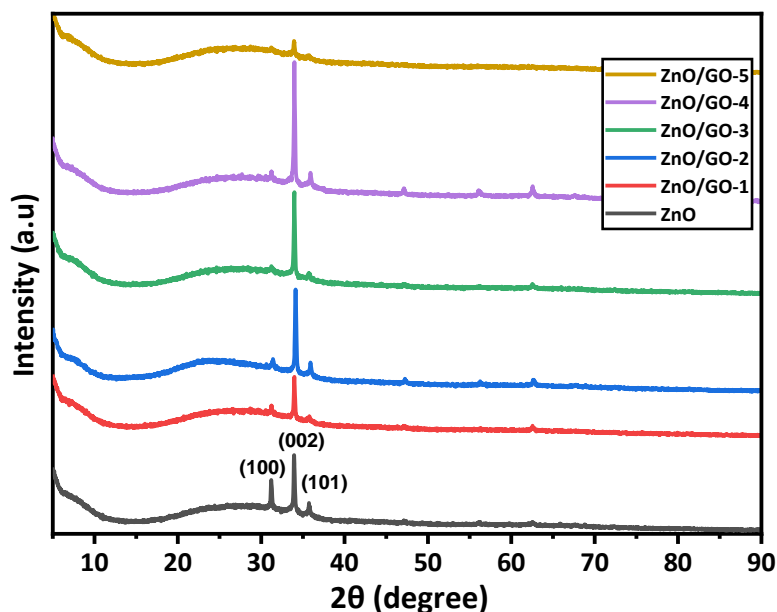


Fig. 1. XRD pattern for ZnO and ZnO/GO nanostructured at different GO concentrations

$$P_{(hkl)} = \frac{I_{(hkl)}}{\sum I_{(hkl)}} \tag{1}$$

where  $I(hkl)$  is the measured relative intensity for (002) plane and  $\sum I(hkl)$  is the intensity of all the diffraction peaks in all plane. The relative peak intensities of the (002) planes are shown in Table 1.

**Table 1**  
 The 2θ peak, FWHM, crystallite size and peak intensity related to the samples

Samples	FWHM (degree)	Crystallite size (nm)	Peak Intensity	Dislocation density, $\delta$ ( $\frac{Lines}{m^2}$ )	Interplanar distance, $d$ (Å)
ZnO	0.19	46.00	0.48	$4.77 \times 10^{14}$	2.63
ZnO/GO-1	0.19	44.00	0.51	$5.21 \times 10^{14}$	2.63
ZnO/GO-2	0.21	42.00	0.60	$5.80 \times 10^{14}$	2.63
ZnO/GO-3	0.23	38.00	0.64	$6.96 \times 10^{14}$	2.64
ZnO/GO-4	0.24	36.00	0.67	$7.59 \times 10^{14}$	2.64
ZnO/GO-5	0.18	47.00	0.41	$4.52 \times 10^{14}$	2.64

According to the estimated values, the relative peak intensity of the (002) peak increases as the concentration of GO coated on ZnO nanorods increases. The relative peak intensity of ZnO/GO nanostructured improves from 0.2- 0.5 mg/ml of GO concentration. This implies that the ZnO/GO-4 nanostructured sample has the highest relative peak intensity along the c-axis and better crystallinity than other samples. Thus, it is this sample nanostructured might be suitable for ZnO-based photoanode. The higher relative peak intensity along the c-axis presents good crystallographic planes and nanostructured formation at minimum surface energy. However, the intensity of the peak ZnO/GO nanostructured decreased with further addition of at 0.6 mg/ml of GO concentration. This reduction behavior in the crystallinity of ZnO/GO structure might be due to the uniformity structure and increase of grain boundary of nanostructured when coated with GO over 0.5 mg/ml concentration. The full-width half-maximum (FWHM) values of the nanostructured are around 0.24 and 0.18 respectively. Diffraction peaks of GO were not found due to its small amount. The absence

of typical diffraction peaks of GO stacking layers could be attributed to the fact that introducing ZnO into the sheets of GO destroys the regular stacking of GO layers [27].

The average crystallite size of nanostructured samples can be estimated from Debye-Scherrer's Eq. (2).

$$D = \frac{0.94\lambda}{\beta \cos\theta} \quad (2)$$

The crystallite size of ZnO/GO nanostructured decreases when concentration of GO coated on ZnO nanorods increases. The smallest crystallite size was shown by ZnO/GO-4. Chava and Kang [28] reported that, as the crystal size decreases, smaller particle sizes can be obtained. Therefore, in electronic applications such as DSSC, a smaller particle size of samples is good to be used for the absorption of dyes due to a large surface area [28]. It obviously indicates that ZnO/GO-4 has a large internal surface area for more dye loading. In general, a more dye-loaded photoanode harvests more photons from incident light, resulting in a higher Jsc [29]. Dislocation density,  $\delta$  of the nanostructured is estimated from crystalline size using Eq. (3).

$$\delta = \frac{1}{D^2} \quad (3)$$

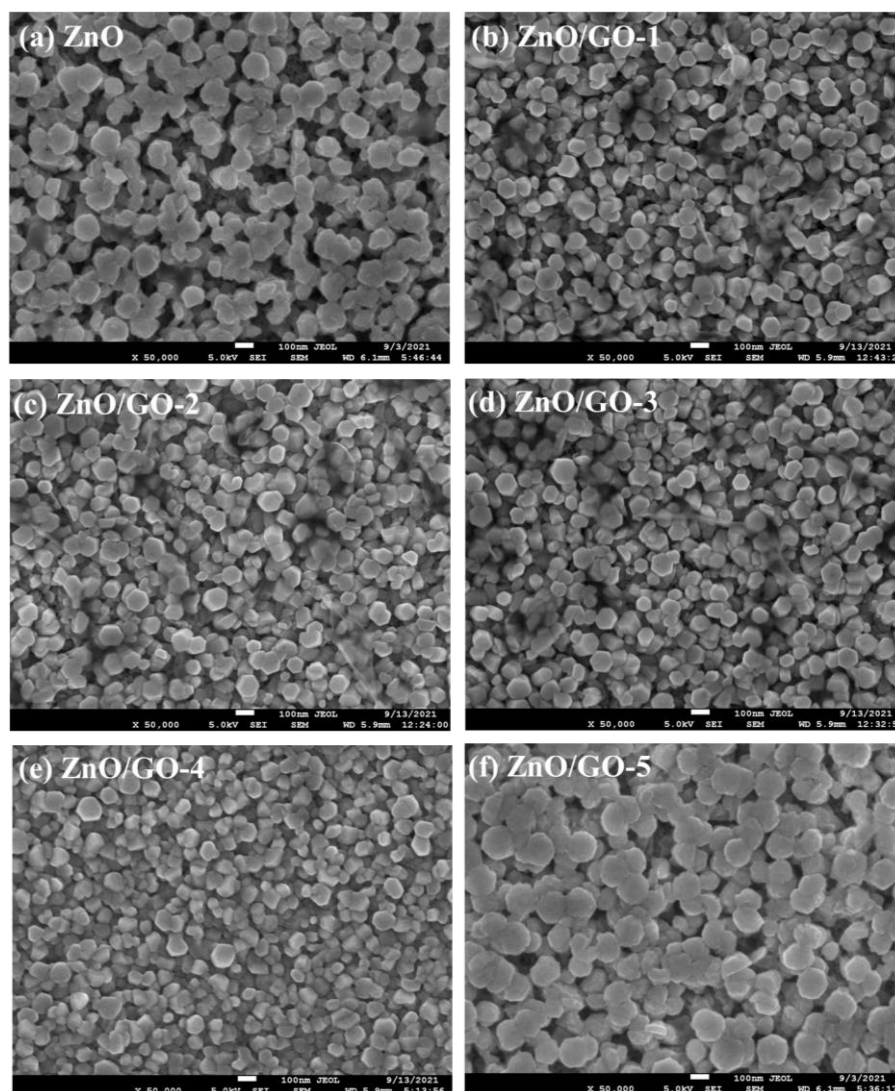
where D(002) is the crystallite size. The dislocation density of ZnO and ZnO/GO nanostructured increase by GO coating concentration from 0 to 0.5 mg/ml. Because of the introduction of defects such as oxygen vacancies and zinc interstitials, stress and strain have a significant impact on the film properties. According to Bragg's equation, the inter-planar distances of the diffracting planes  $d$  and the lattice constants  $a$  and  $c$  of wurtzite structure films were determined using Eq. (4) [26]:

$$2d_{hkl} \sin \theta = n\lambda \quad (4)$$

where  $d_{hkl}$  is the distance between lattice planes of Miller indices ( $h$ ,  $k$ , and  $l$ ),  $n$  is the order of diffraction (usually  $n=1$ ),  $\lambda$  is the X-ray wavelength of  $\text{CuK}\alpha$  radiation ( $1.54 \text{ \AA}$ ) and  $\theta$  is Bragg's angle (half of the peak position angle).

### 3.2 Field Emission Scanning Electron Microscope (FESEM)

The FESEM images in Figure 2 show that all the samples were grown vertically aligned hexagonal structures on the glass substrate ZnO seed layer. The coating of GO at different concentration from 0 - 0.5 mg/ml indeed influences the ZnO nanorods growth by reducing the size of nanorods from 104 to 56 nm, respectively (Table 2). The size of ZnO nanorods decrease when concentration of GO coating increase over 0.5 mg/ml. It was found that ZnO/GO-4 sample has the smallest size with better distribution of nanostructured. Atanacio-Sánchez *et al.*, [7] discovered that a sample with greater distribution of particles has the highest efficiency of DSSC. The decrease in the diameter of the nanorods could be attributed to the cleavage of some larger nanorods and the surface area of the thin film can increase [18]. A larger surface area will cause more dye molecules to be absorbed onto the surface of the nanostructure. Thus, it will produce more current and might be suitable to increase power conversion efficiency for DSSC [18,30].



**Fig. 2.** FESEM micrograph of (a) ZnO, (b) ZnO/GO-1, (c) ZnO/GO-2, (d) ZnO/GO-3, (e) ZnO/GO-4 and (f) ZnO/GO-5 thin films

**Table 2**

The average diameter of all the thin films

Samples	Average Diameter (nm)
ZnO	104
ZnO/GO-1	74
ZnO/GO-2	69
ZnO/GO-3	67
ZnO/GO-4	56
ZnO/GO-5	113

### 3.3 Fourier-Transform Infrared (FTIR) Spectroscopy

The surface functional groups in ZnO and ZnO/GO nanostructured were studied using FTIR spectroscopy as shown in Figure 3. The oxygen functional group peaks in the ZnO/GO sample are found at 1071, 1178, and 1730  $\text{cm}^{-1}$  which correspond to C–O stretching vibrations, C–OH stretching, and  $\text{sp}^2$ -hybridized C=C groups, respectively. The appearance of new peaks between 600 and 1180  $\text{cm}^{-1}$  can be attributed to Zn-C stretching bonding. Any shift or change in the position and intensity

of peaks in the FTIR spectra of samples indicates the contribution of functional groups of GO with ZnO nanorods [31]. Broadband in  $1080\text{ cm}^{-1}$  is observed as part of the nature of the C–O–C stretching vibration of some residual chemical agent in the ZnO and ZnO–GO, which is why the stretching vibration is more pronounced in the ZnO–OH. Furthermore, as can be seen, the ZnO/GO samples exhibit a stronger downward disturbance than the ZnO. This disturbance is caused by more energy being absorbed, which may be due to particle size [7,32].

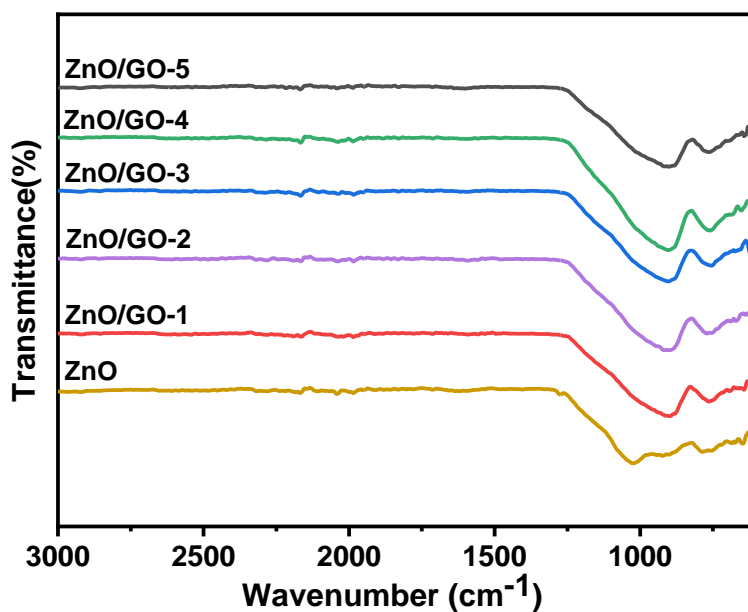


Fig. 3. FTIR spectra of ZnO, and ZnO/GO thin films

### 3.4 UV-visible Analysis

#### 3.4.1 Transmittance

The transmittance spectrum of ZnO and ZnO/GO samples are presented in Figure 4. The transmittance spectrum increases with increasing of GO coating concentration from 0 to 0.5 mg/ml that presented by ZnO to ZnO/GO-4, respectively. Decrease of transmittance for ZnO/GO-5 nanostructured occurred at higher GO coating concentration which is over than 0.5 mg/ml. The increase in transmission spectrum may be related to the increase in carrier concentration in ZnO/GO nanostructured due to the high degree of vertical alignment, low surface roughness, and uniformity of ZnO/GO nanostructured [22]. The transmission dropped dramatically near the visible region, which corresponded to the intrinsic bandgap energy of ZnO [33]. The reduction of transmittance is attributed to the structural properties and film thickness [34]. Previous research suggests that ZnO film transmittance decreases as the nanostructured size decreases [35]. The porosity of the ZnO thin films can be calculated using the following Eq. (5) [28]:

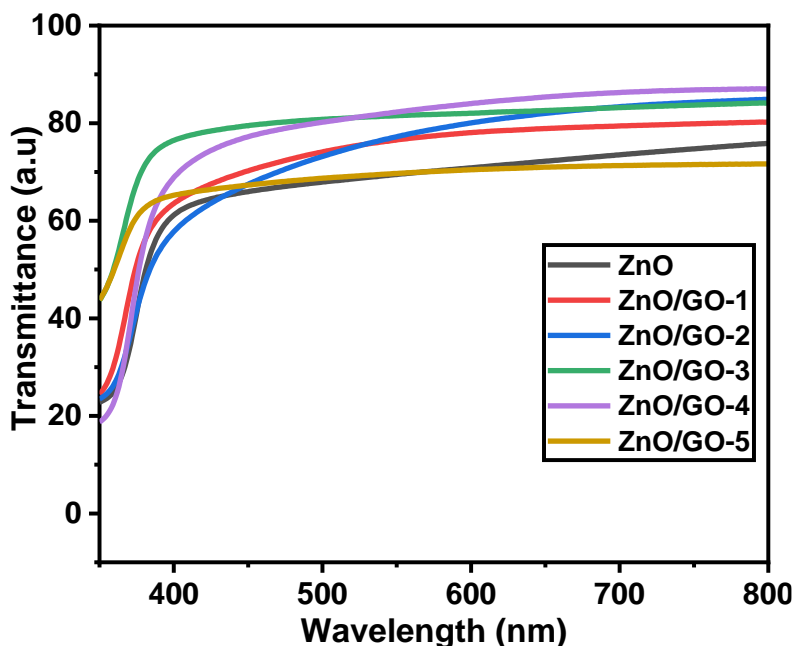


Fig. 4. Comparison of the optical transmittance of ZnO thin films for various GO concentrations

$$\text{Porosity} = 1 - \frac{\left(\frac{n_f^2 - 1}{n_f^2 + 2}\right)}{\left(\frac{n_s^2 - 1}{n_s^2 + 2}\right)} \quad (5)$$

where  $n_f$  is the refractive index of the porous ZnO films and  $n_s$  is the widely accepted refractive index of the ZnO skeleton, which is 2. The following equations are used to calculate the refractive index,  $n_f$ , in the transmittance region where the absorption coefficient, 0.

$$n_f = [N + (N^2 + S^2)^{1/2}]^{1/2} \quad (6)$$

$$N = \frac{2S}{T_m} - \frac{(S^2 + 1)}{2} \quad (7)$$

$T_m$  is the envelope function of the maximum and minimum transmittance values, and  $S$  is the substrate's refractive index, which in this case is 1.52 for the transparent glass substrate. The  $T_m$  value can be calculated by averaging the transmittance data from the transparent region between 400 and 800nm wavelength or where the value is close to 0. ZnO/GO-4 shows the highest porosity state that may be attributed to good transmissibility for dye adsorption and can improve electrolyte ion exchange rates, reducing recombination losses and increasing conversion efficiencies (Table 3) [36]. As mentioned by Ghann *et al.*, [37], the porous surface of the material of photoanode suggests an enhancement in the adsorption of dye into the ZnO structure.



**Table 3**

The average transmittance and porosity of ZnO thin films with different GO concentrations

Samples	Average Transmittance	Porosity (%)
ZnO	70.4921	1.3045
ZnO/GO-1	76.2186	1.3054
ZnO/GO-2	77.4929	1.3055
ZnO/GO-3	81.7557	1.3058
ZnO/GO-4	82.6607	1.3060
ZnO/GO-5	68.4822	1.3042

#### 4. Conclusions

In summary, we have successfully prepared the ZnO/GO nanostructured thin films via double step solution immersion method. The XRD pattern indicates that ZnO and ZnO/GO thin films present a hexagonal wurtzite structure, which preferentially grows along the (002) orientation. The ZnO/GO-4 thin film gives the smallest crystallite size which is around and diameter. The FESEM images indicated the hexagonal shape of all samples with the smallest diameter (56 nm) shown by ZnO/GO-4 sample. The highest transmittance spectrum is shown by ZnO/GO-4 which might be due to the increase in carrier concentration in ZnO/GO nanostructured due to the high degree of vertical alignment, low surface roughness, and uniformity of ZnO/GO nanostructured. These findings demonstrate that the coating ZnO with GO improves the properties of ZnO nanostructured and the optimal sample might be suitable to be used as a photoanode in DSSC.

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