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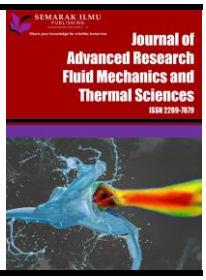

Effect of GO Solution Temperature on the Structural, Optical and Electrical Properties of ZnO Nanostructured for Photoanode Function

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ABSTRACT

The purpose of this study is to determine the effect of different graphene oxide (GO) solution immersion temperature on the structural, morphology, optical, and electrical properties of zinc oxide/graphene oxide (ZnO-GO) nanostructures. The ZnO/GO nanostructures prepared at various GO solution immersion temperature from 75-95°C using solution immersion method. The structural properties of the samples were investigated using X-ray diffraction (XRD), and the recorded patterns revealed that all the samples had a preferred orientation along the (002) plane. The crystallinity of ZnO/GO nanostructures were enhanced with increasing GO solution immersion temperature. The morphology of ZnO/GO nanostructures was determined using field emission scanning electron microscopy (FESEM). Fourier transformation infrared spectroscopy (FTIR) was used to determine the molecular compounds of ZnO/GO nanostructures. The peak intensity of GO with ZnO nanoparticles is shifted at 744 to 1243 cm-1 when the temperature of GO solutions increases. The UV–visible spectrophotometer was used to examine the optical properties of ZnO/GO nanostructures. It is found that the highest transmittance ZnO/GO nanostructures was obtained at the highest GO solution immersion temperature which is 95℃. Based on the current-voltage(I–V) measurement, the electrical properties of ZnO/GO nanostructures increase when the GO solution immersion temperature increases. Thus, by variation of GO solution immersion temperature the structural, morphology, optical, and electrical behaviour were improved.

1. Introduction

Zinc Oxide (ZnO) is widely used in several areas of technology due to its unique properties, including a large excitation binding energy of 60meV and has a direct wide band gap [1]. ZnO nanostructured can be grown at low temperatures which are extensively used for blue-ultraviolet and white light emitting devices [2].

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The properties of ZnO nanostructured materials are highly dependent on their microstructure, including crystal size, specific surface and on their morphology. Most research is mainly focused on one-dimensional ZnO nanostructures, such as nanowires, nanorods and nanotubes because of their unique features such as less grain boundaries, surface defects and dislocations. ZnO can be synthesized through various techniques. Spray pyrolysis [3], thermal decomposition method [4], solution immersion method [5], atomic layer deposition[6] and radio frequency (RF) sputtering [7] are among the techniques used for the synthesis of ZnO nanostructured. One of other techniques is the solution immersion method which is the most simple and effective technique in terms of cost and less power consumption than the other techniques[8]. ZnO nanostructured and its physical properties produced through the immersion method can be controlled by preparation parameters such as material concentration, growth temperature and time [9].

The purpose of this study is to analyze the effect of GO solution immersion temperature on ZnO/GO nanostructures by using the immersion method. The growth layer on ZnO/GO nanostructures process was carried out at various GO solution immersion temperature ranging from 75°C to 95°C. The structural, morphology, optical and electrical of ZnO/GO nanostructures were studied.

2. Methodology

2.1 Seeded Layer Preparation

Glass substrate was used to deposit ZnO as seeded layer using spin coating technique. Detailed explanation process can be obtained in previous study [10].

2.2 Preparation of ZnO Solution

The ZnO solution was prepared by mixing 0.1M zinc nitrate hexahydrate, and 0.1M hexamethylenetetramine that act as a precursor and stabilizer, respectively. These two materials were mixed and dissolved in deionized (DI) water. The solution was sonicated using an ultrasonic water bath for 30 min at 50°C. Then, it was stirred for 3 hours at room temperature.

2.3 Preparation of GO Solution

The solution was prepared by mixing 50 mg of GO powder and 100 ml of DI water. The solution then undergoes the sonicating process for 30 min at 50°C and continues by stirring for an hour.

2.3 Growth Process

ZnO/GO nanostructures were grown on ZnO seeded layer-coated glass substrate by using the aqueous solution immersion method. The ZnO solution was poured into the Scotty bottle that contained ZnO seeded-layer-coated glass substrate at the bottom of the bottle. The schott bottles were left in a hot water bath for an hour at 95°C to grow ZnO nanostructures. After that, remove the ZnO solution and pour the GO solution into schott bottle, the schott bottle was immersed 40 min inside the water bath with various temperature (75, 80, 85, 90 and 95 °C) to grow the GO onto the ZnO nanostructures. DI water was used to rinse the ZnO/GO nanostructures and were annealed at 500°C for 30 minutes in ambient conditions. The ZnO/GO nanostructures were characterized using x-ray diffraction (XRD), field emission scanning electron microscope (FESEM), fourier transform infrared spectroscopy (FTIR), UV-visible spectrometer, and current-voltage (IV) measurement.

3. Results

3.1 Structural Result

XRD is used to characterize the crystallinity of ZnO/GO nanostructures. Figure 1 shows the XRD pattern of ZnO/GO nanostructures, and all the samples well indexed with the standard JCPDS card no. 36-1451. The XRD diffraction peaks of ZnO/GO nanostructures are indexed to (100), (002) and (101) crystal planes that correspond to (2θ) of 31.8˚, 34.4˚ and 36.3º, respectively. The diffraction peaks along (002) plane has the highest peak intensity of ZnO/GO nanostructures which typically refer the formation of structures along the c-axis or perpendicular to the substrate[11]. As the GO solution immersion temperature is increased from 75℃ to 95℃, the intensity of the (002) plane is remarkably increased. The highest and lowest peak intensity along (002) plane for ZnO/GO nanostructures was at 95℃ and 75℃ of GO solution immersion temperature. Other study reported that the crystalline quality of nanostructures increases as they develop at higher temperatures which is good agreement with pattern in this study [12]. The high intensity of the diffraction peaks was produced due to the good of nanostructures alignment on the substrate [13]. Previous research also mentioned that the diffraction peak intensity is high because of the reduction in defects structure and has higher quality of film [14]. A relatively high intensity (002) plane for the ZnO/GO nanostructures suggests that the preferred orientation along the c-axis is perpendicular to the substrate [15]. As reported by Liang *et al.,* the active materials that has better crystallinity structure would give better dye absorption as well as faster electron injection in DSSC [16].

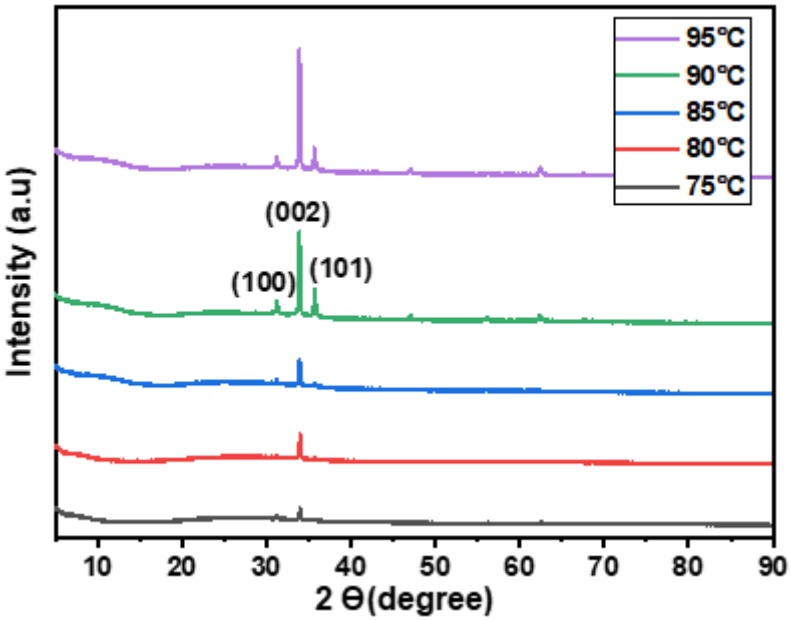

Fig. 1. X-ray diffraction pattern of ZnO/GO nanostructures at different GO solution immersion temperature

The full width half maximum (FWHM) value was used to calculate the average crystallite size using the Scherrer equation 1 [17].

$$D = \frac{0.94\lambda}{\beta cos\theta} \quad (1)$$

where λ represents the incident X-ray wavelength of Cu Kα radiation, which is 1.54 Å, β represents the maximum full-width half value, and θ is the angle of the diffraction peak. The relative peak intensity,*P* was calculated using equation 2 [18].

$$P_{(hkl)} = \frac{I_{(hkl)}}{\Sigma I_{(hkl)}} \quad (2)$$

where I(hkl) is the (002) peak intensity, and ΣI(hkl) is the sum of the intensities of all the diffraction peaks at (2θ) for the ZnO thin film deposited on a glass substrate. Figure 2 shows the crystallite size and relative peak intensity value of ZnO/GO nanostructures samples. The crystallite size that measure using Scherrer equation were 48, 46, 43, 39, and 37 nm for the ZnO/GO nanostructures that prepared at GO solution immersion temperature 75, 80, 85,90 and 95℃, respectively. The crystallite size of ZnO/GO nanostructures of all samples decrease as temperature of GO solution increase. ZnO/GO nanostructures that prepared at 95℃ of GO solution immersion temperature gives the smallest crystallite size might be ideal to be used as photoanode in DSSC. Previous study reported that small crystallite size would provide a large surface area that can absorb more dyes and improve the efficiency of photoanode [19].

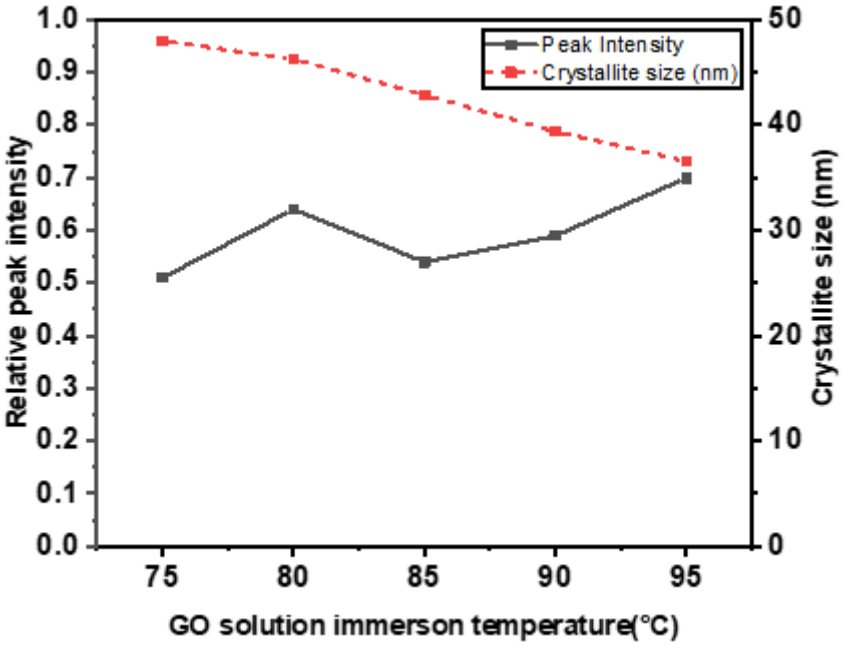

Fig. 2. The graph of crystallite size and peak intensity related to the samples

3.2 Morphology Result

Morphology of ZnO/GO nanostructures at 50k magnification is shown in Figure 3. The obtained ZnO/GO nanostructures are in hexagonal structure. It was observed that there is a significant change in the growth of ZnO/GO nanostructures on the seed layer when the GO solution immersion temperature is varied. The average diameter of ZnO/GO nanostructures were 147, 133, 127, 120, and 113 nm for the sample prepared at GO solution temperature 75, 80, 85, 90 and 95℃, respectively. The average diameter of the ZnO/GO nanostructures grown at 95˚C GO solution immersion temperature has smallest value compared to other samples. The patterns of this results are similar

with previous research and as reported that the increasing temperature of the solution produced adequate heat energy for the atom nucleation which improve the surface mobility to be more active [17]. This demonstrates that the GO solution immersion temperature has a significant influence on the size of ZnO/GO nanostructures. The smallest diameter of nanostructures will result in larger surface area. Thus, more dye molecule could be absorbed onto the surface of nanostructures, it will produce more current and thus it is suitable for DSSCs since it will enhance its power conversion efficiency [20,21].

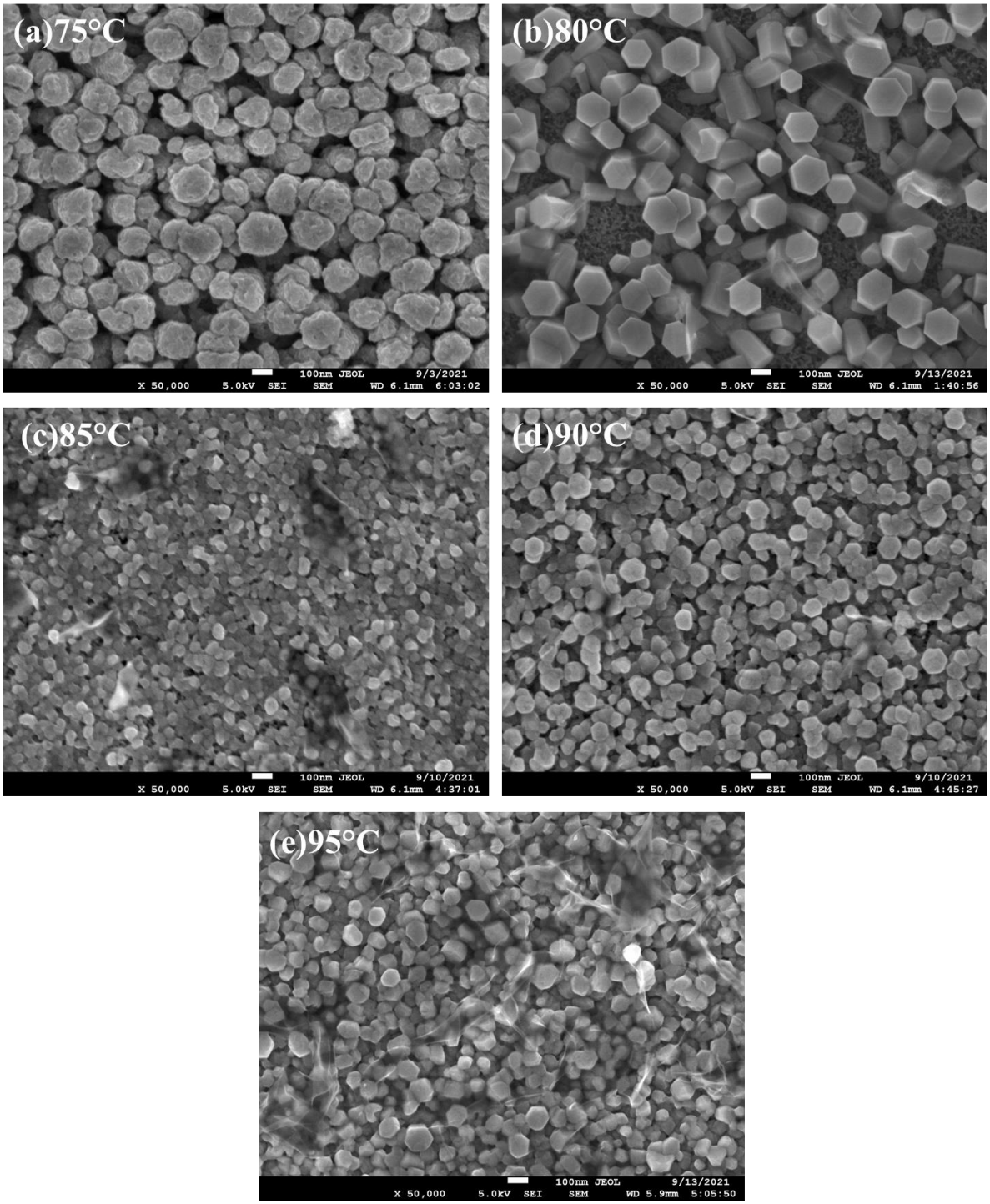

Fig. 3. FESEM images of the ZnO/GO nanostructures at 50k magnification

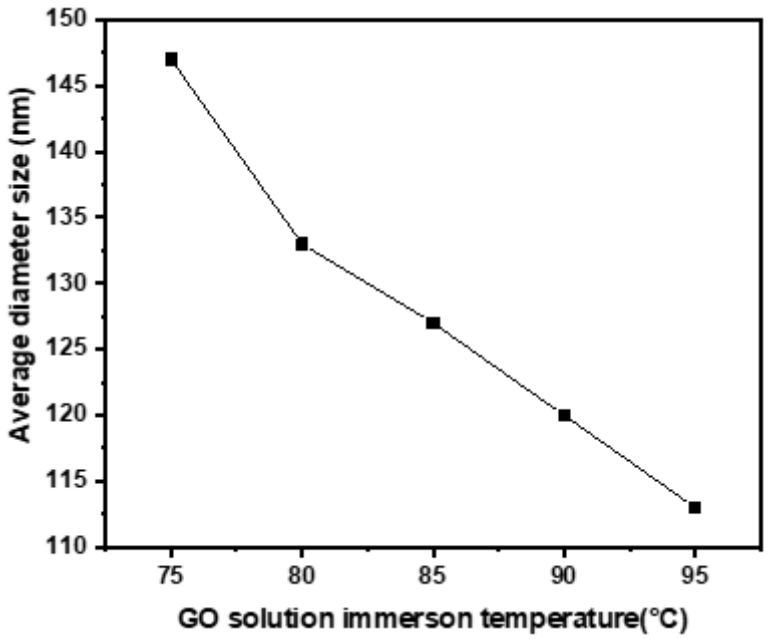

Fig. 4. The average diameter of ZnO/GO nanostructures

3.3 Fourier-Transform Infrared (FTIR) Spectroscopy

The surface functional groups in ZnO/GO nanostructures were studied using FTIR spectroscopy. Figure 5 shows a few absorption bands in the FTIR spectrum of ZnO/GO nanostructures. The oxygen functional group peaks in the ZnO/GO nanostructures are found at 1071, 1178, and 1730 cm^{-1}, which correspond to C–O stretching vibrations, C–OH stretching, and sp^2-hybridized C=C groups. The appearance of peaks between 600 and 1180 cm^{-1} can be attributed to Zn-C stretching bonding. We observed a few peaks shift for ZnO/GO nanostructures at 744 to 1243 cm^{-1}. As mentioned by Alamdari *et al.,* 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 nanoparticles or the partial reduction of GO [22]. A broad band in 1080 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 nanostructures exhibits a stronger downward disturbance than the ZnO, this disturbance is caused by more energy being absorbed, which may be due to particle size [23,24].

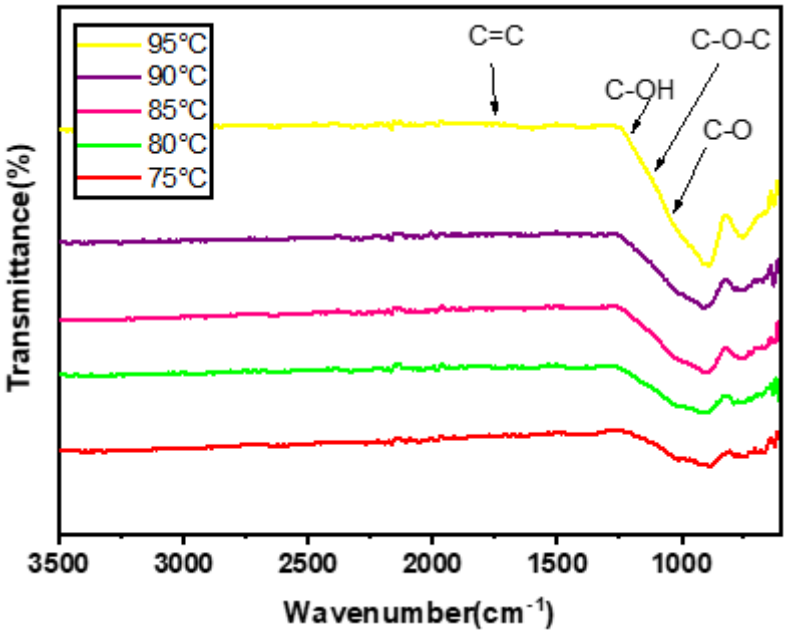

Fig. 5. The FTIR spectra of ZnO/GO nanostructures

3.4 Optical Properties

The optical properties of ZnO/GO nanostructures are shown in Figure 6. In this study, the optical transmittance of selected ZnO/GO nanostructures is measured using a UV–vis spectrophotometer with an integrating sphere at wavelengths ranging from 350 nm to 800 nm. The average transmittance value of the ZnO/GO nanostructures grown at 95℃ of GO solution immersion temperature is clearly higher compared to the other samples at the visible wavelength. This indicates that the light transmittance of the film may be improved by the higher temperature. The porosity of ZnO/GO nanostructures can be calculated using the equations below.

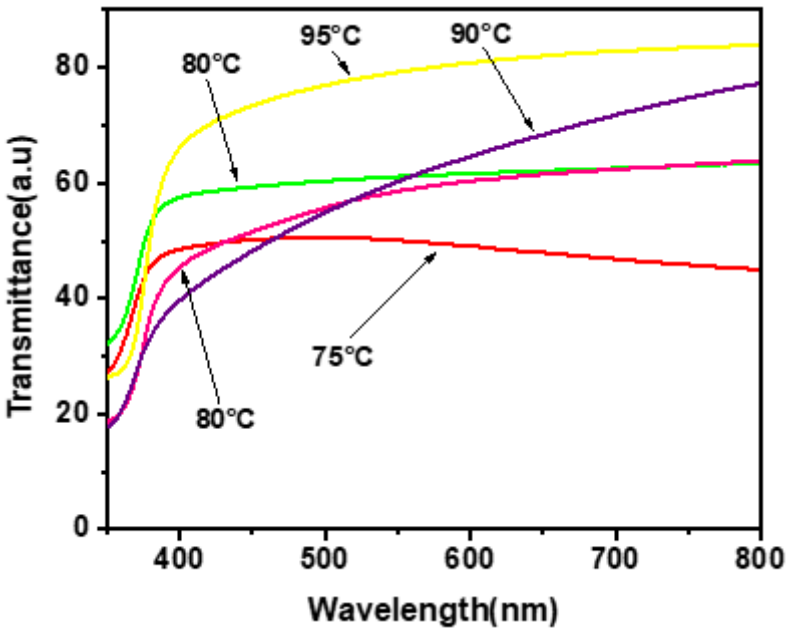

Fig. 6. Transmittance spectra of all the samples

$$\text{Porosity}= 1 - \frac{\left(\frac{n_f^2-1}{n_f^2+2}\right)}{\left(\frac{n_s^2-1}{n_s^2+2}\right)} \quad (3)$$

where nf denotes the refractive index of porous ZnO/GO nanostructures and ns denotes the widely accepted refractive index of the ZnO skeleton, which is 2. The following equations are used to calculate the refractive index, nf, in the transmittance region where the absorption coefficient, 0.

$$n_f = [N + (N^2 + S^2)^{1/2}]^{1/2} \quad (4)$$

$$N = \frac{2S}{T_m} - \frac{(S^2+1)}{2} \quad (5)$$

Tm is the envelope function of the maximum and minimum transmittance values, and S is the refractive index of the substrate, which is 1.52 in this case for the transparent glass substrate. The Tm value is calculated by averaging the transmittance data from the transparent region between 400 and 800nm wavelengths, or where the value is close to 0. The porosity increases as the GO solution immersion temperature increase meaning that the porosity did influence by the GO solution immersion temperature. From Figure 7, the highest porosity is shown by the ZnO/GO nanostructures that grown at 95℃ of GO solution temperature. The ideal photoanode should have a high porosity for efficient mass transport and be transparent to reduce the loss of incident photons in visible light. The ideal photoanode should have a high porosity for efficient mass transport and be transparent to reduce the loss of incident photons in visible light [25].

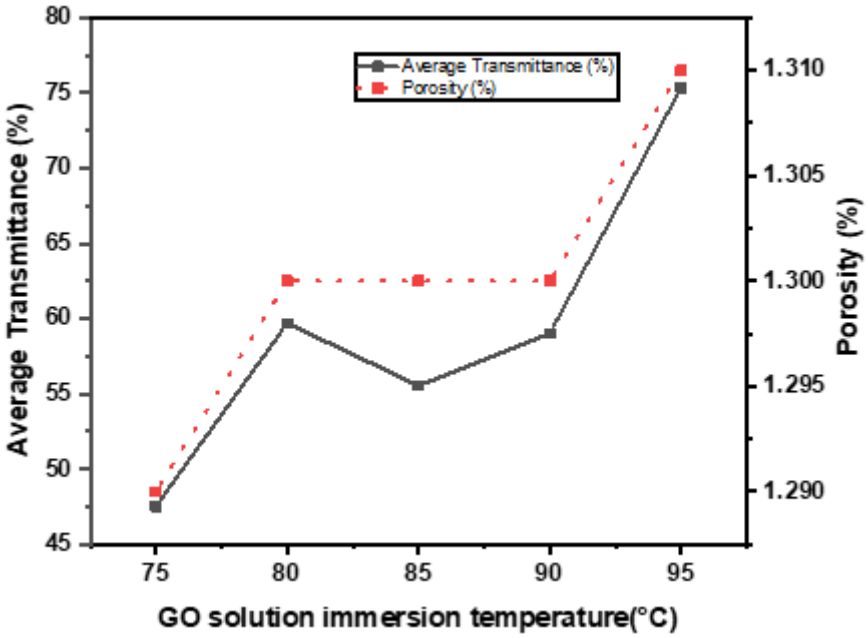

Fig. 7. Average transmittance and percent of porosity in all samples

3.5 Electrical Result

Figure 8 shows the current- voltage (I-V) characteristics for sample ZnO/GO nanostructures. The resistivity and conductivity were calculated from the slopes of the linear I-V plot using the following formulas

$$\rho = \left(\frac{V}{I}\right)\frac{wt}{l} \tag{6}$$

$$\sigma = \frac{1}{\rho} \tag{7}$$

where V is the supplied voltage, I is the measured current, w is the electrode width, t is the film thickness, l is the length between the electrodes and σ is the film conductivity.

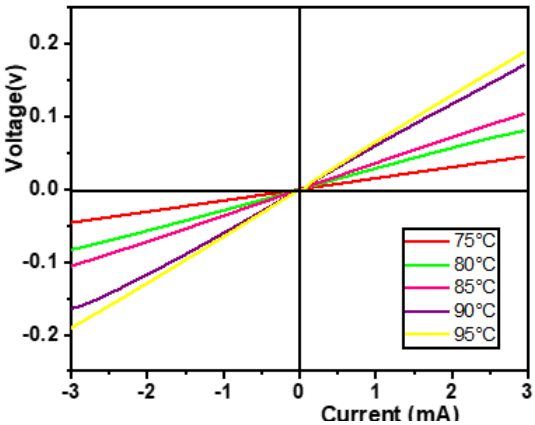

Fig. 8. I-V curve of ZnO/GO nanostructures at different temperature

Figure 9 shows the variation in resistivity with respect to the GO solution immersion temperature. The I-V measurement was done at room temperature. The results show that the electrical resistivity of the ZnO/GO nanostructures decreases when GO solution immersion temperature increase up to 95℃. The ZnO/GO nanostructures at 95℃ of GO solution temperature shows highest conductivity which is 2.033 ×10-3 S.cm-1. At this condition, this sample may be suitable to be used as a photoanode in DSSC application.

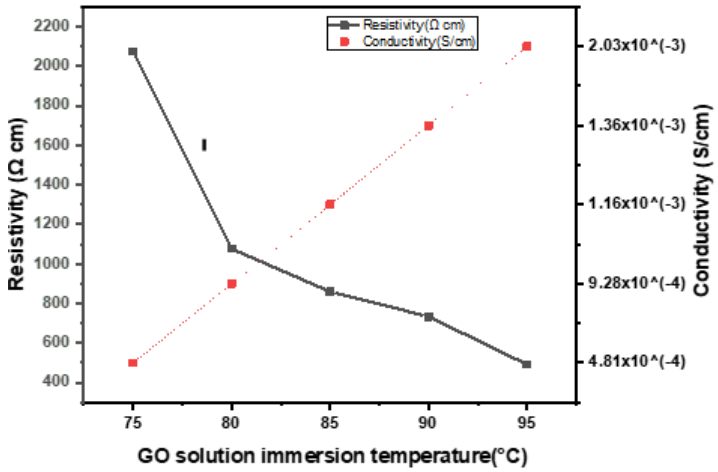

Fig. 9. The resistivity and conductivity of samples

4. Conclusions

In summary, we have successfully prepared the ZnO/GO nanostructures via solution immersion method at different GO solution immersion temperature. From XRD result, it was observed that the crystallite size decreases as the GO solution immersion temperature increases indicates that the increase in GO solution immersion temperature improved the crystallinity of ZnO/GO nanostructures. The ZnO/GO nanostructures growth at 95℃ of Go solution temperatures shows that smallest crystallite size which is 37 nm. FESEM characterization showed the smallest diameter and uniform grain size for the ZnO/GO nanostructures grown at 95℃ of GO solution immersion temperature. This sample also showed the highest porosity and average transmission values compared to other samples. From I-V measurement, the conductivity of ZnO/GO nanostructures increases with increasing GO solution immersion temperature. It can be concluded that ZnO/GO nanostructures grown at a temperature of 95℃ of GO solution show optimal conditions. Based on the results obtained, this sample may meet the characteristics as a photoanode in DSSC applications.

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