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Dependence of Structural and Optical Properties on Annealing Time of ZnO Nano-Structure

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A B S T R A C T

ZnO nano-structure was processed by the coprecipitation method using hexahydrate zinc chloride ($ZnCl_2.6H_2O$) as a starting chemical and then adding sodium hydroxide (NaOH) dropwise. The annealing process for ZnO nano-structure was carried out at a temperature of 400 C° at different intervals, varying from 0 to 8 hours. The effects of annealing time in the structural and optical properties are detailed and the results are compared among the experimental techniques. The structural and optical characteristics of ZnO powders were synthesized by X-Ray Diffraction (XRD), Transmission Electron Microscope (TEM), Fourier Transform Infrared (FTIR), and Ultraviolet-Visible spectroscopy (UV-Vis). The XRD patterns reveal that the fabricated samples have a crystal structure, and an improvement in crystallinity occurs by increasing the annealing time. TEM images exhibit the formation of a "hexagonal wurtzite" structure of "ZnO" nano-structure that agglomerates slightly by increasing the annealing time. The "FTIR" Spectra in a range of 400 to 4000 cm^{-1} exhibit peaks that correspond to "Zn-O" stretching modes and a redshift occurs upon increasing the annealing time. From the "UV-VIS" absorption spectra, the optical band gap is found to decrease, and Urbach energy increases by increasing the annealing time.

1. Introduction

Recently, ZnO nano-structure have had a great importance due to their novel properties such as having a wide bandgap, which is considered a natural n-type diluted semiconductor material **[2]**. ZnO nano-structure also have a great excitation binding energy of (60 mV) at room temperature, and this energy is greater than many other materials [4, 11, 22, 34, 40]. Furthermore, ZnO nano-structure have a heat resistance that exceeds that of organic and inorganic materials [37]. Even though all these critical properties have been discussed, there has not been a focus on the annealing time, which is considered one of the most important factors in controlling such structural and optical properties. Current literature shows that the coprecipitation method with the annealing time plays a massive role in the altering of physical properties in ZnO nano-structure. ZnO nano-structure could be synthesized by many methods like "Sol-gel processing" [39], "hydrothermal synthesis" [20], "thermal decomposition" [36], "spray pyrolysis" [17], "vapor transport process" [5, 38] and "direct precipitation or coprecipitation" [16, 28]. Among the methods of preparation are wet chemical processes at low temperatures, such as hydrothermal process, hydrolysis, and coprecipitation; are low-cost, and they are used in preparing various types of ZnO nanostructures [6]. Otherwise, "chemical vapor deposition (CVD) and physical vapor deposition (PVD)" have been developed

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to synthesize ZnO nano-structure into more complex structures applying multiple steps and high temperatures. ZnO has three main crystallization forms: hexagonal wurtzite structure, cubic zinc blend, and rock salt. The "hexagonal wurtzite structure" is considered the most stable phase, and it is characterized by inter-connecting O^{2-} and Zn^{2+} tetrahedrally. A hexagonal unit cell has lattice parameters of "a=0.3296 nm" and "c=0.52065 nm" with a ratio of "c/a = 1.633" [30, 31]. ZnO nano-structure have a variety of applications, such as photocatalyst [3], solar cell transducers [27], medical and dental materials, rubber, UV shielding, pigments, ceramic, concrete, antibacterial materials, and semiconductors. [7, 18, 19, 23, 24, 33, 35]. As the effect of annealing temperatures has an essential role in altering the structural and physical properties of prepared ZnO nano-structure [14, 21] therefore ZnO nano-structure were prepared using coprecipitation method. The aim of this work is the study of the annealing time effect on structural and optical properties of ZnO nano-structure.

2. Experimental procedures

2.1 Preparation of ZnO nano-structure

Hexahydrate zinc chloride "$ZnCl_2.6H_2O$, 99 % Sigma-Aldrich" and sodium hydroxide "NaOH, 99 % Sigma-Aldrich" were used as starting chemicals.

ZnO nano-structure were prepared using a simple coprecipitation technique. $ZnCl_2.6H_2O$ was dissolved into 100 ml of distilled H_2O at room temperature. A suitable volume of NaOH liquid solution was added dropwise into the Zinc Chloride Hexahydrate solution. The clear solution was transferred to milky white slurry suspension. NaOH is very important because it is the source of the hydroxide group (−OH), which tends to make a bond with Zinc Ion; i.e. Zinc hydroxide and sodium chloride are formed. The white colloidal solution was stirred for a suitable time at room temperature. White precipitates were carefully collected by a filter paper and washed many times with distilled water to get rid of salts. The precipitate was dried at 100 ºC for 6 hours. Finally, the product was crushed using pestle and mortar to produce fine powder before structural and physical characterization. The prepared samples of powder were annealed at 400 C^o for different annealing periods (1, 4, and 8 hours); the samples were kept to reach room temperature and were collected the next day.

2.2. Characterization

The physical ZnO nano-structure properties were characterized by "Philips' X-Ray diffractometer", "X'PERT-MPDVC PW 3040" with CuKα radiation (λ = 1.5406 Aº) in the 2Θ range of "10-80°" with "2º/min" scanning rate. The morphological feature of the ZnO nano-structure was observed by a transmission electron microscope "JEOL: 1200 EX II, Japan". TEM measurements were made by dropping ZnO nanostructures dilute suspension onto copper grids. Fourier Transform Infrared (FT-IR) spectra were measured by scientific "NICOLET 6700 FT-IR", with a frequency range from 400 to 4000 cm^{-1}, using KBr disks. The optical properties of prepared ZnO nano-structure were analyzed via "Jasco V-560 UV-VIS spectrometer". Transmission was measured in the range between (500 and 4000 nm) using JASCO V-560 UV/VIS/NIR spectrophotometer. The instrument specified by resolution 0.1 nm and wavelength accuracy ±0.3 nm (at a spectral bandwidth of 0.5 nm). The measurements were made on glass, immediately after glass preparation and all spectra were measured at room temperature.

3. Results and Discussion

3.1 Structural Analysis

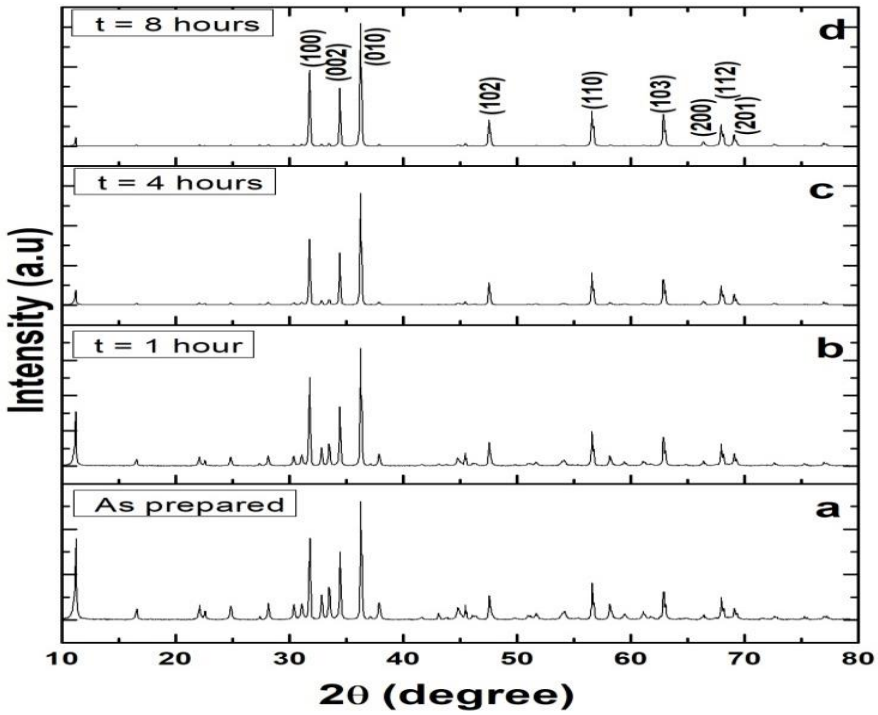

Figure 1 "XRD patterns of ZnO nano-structure samples for (a) as-prepared and annealed for (b) 1 hour (c) 4 hours (d) 8 hours".

Figure 1 (a-d) shows the ZnO nano-structure X-ray diffraction patterns at constant temperature 400ºC and different annealing times: as-prepared, 1, 4, and 8 hours. All the XRD peaks in the 2θº range were characterized, and they correspond to the hexagonal structure of ZnO with lattice constants "a = 3.253Aº" and "c = 5.214Aº". The standard data card "JCPDS card No. 36-1451" shows an agreement with the output data. The good crystallization of samples is shown by the sharpness of the diffraction peaks.

The crystallite size (D) of ZnO is calculated and found to be in the range of 30-40 nm for different annealing times using Scherrer's equation.

$$\boldsymbol{D = \frac{K\lambda}{\beta Cos\theta}} \quad (1)$$

"Where; K=constant (0.89 < K < 1), λ = wavelength of the X-ray, β= Full Width at Half Maximum (FWHM), θ = diffraction angle".

By increasing the annealing time, crystallite sizes were found to increase due to the agglomeration of smaller crystallite into larger ones "[8, 13, 15, 29]". Furthermore, the crystallinity enhancement of the prepared samples increases upon increasing the annealing time [12, 32]. This improvement may contribute to the migration of atoms to the correct positions in the crystal lattice, which leads to the recrystallization in ZnO lattice; so, in turn, defects get reduced. [1, 9, 10].

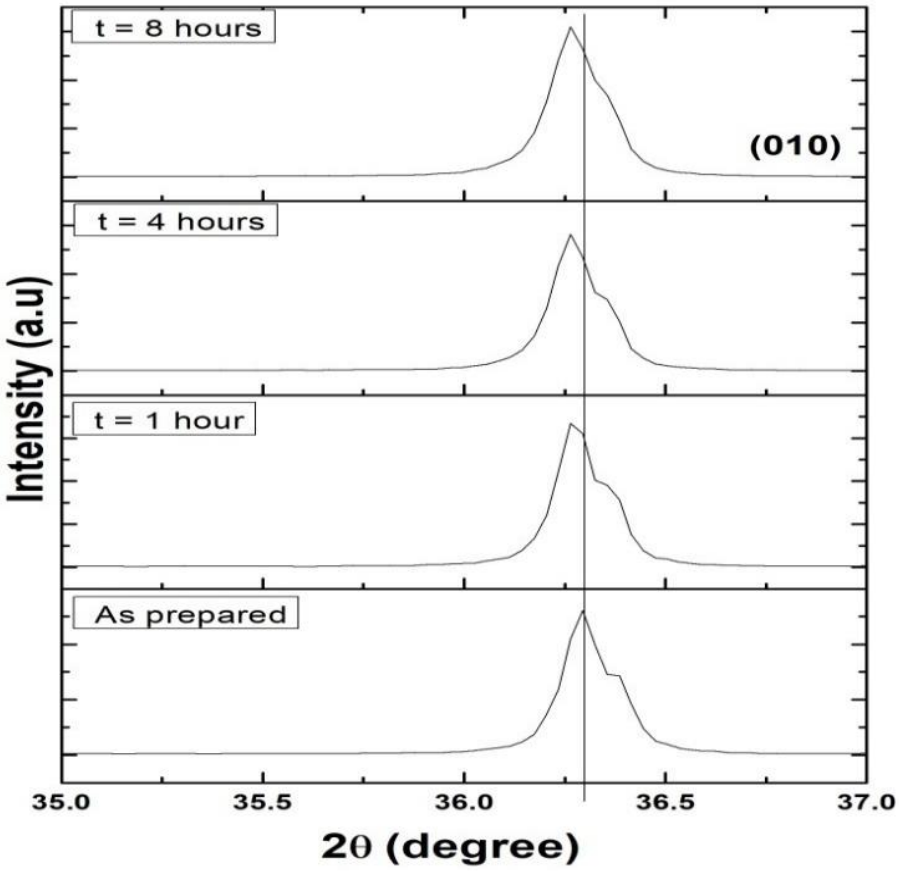

Figure 2 "shift of (101) crystal plane towards lower diffraction angle (2θ°) with the increase of the annealing time for as-prepared and annealed ZnO nano-structure".

Figure 2 shows the shift of (101) plane to a lower diffraction angle (2θ°) with the increase of annealing period; this shows an inter-planer distance increment of the ZnO crystal, which, as a result, led to a decrease after annealing in micro-strain of ZnO lattice.

Table 1. "Measurements of 2θ°, FWHM, crystallite sizes at different annealing time from XRD profiles"

"Annealing period" (hours)	"2θ°" (degree)	"FWHM for (101) peak"	"Calculated crystallite size (D) nm by Debye Scherrer's formula"
"as-prepared"	36.295	0.49	31.6
1	36.272	0.46	33.1
4	36.27	0.45	33.8
8	36.263	0.41	36.9

3.2 Transmission Electron Microscopy (TEM) Analysis

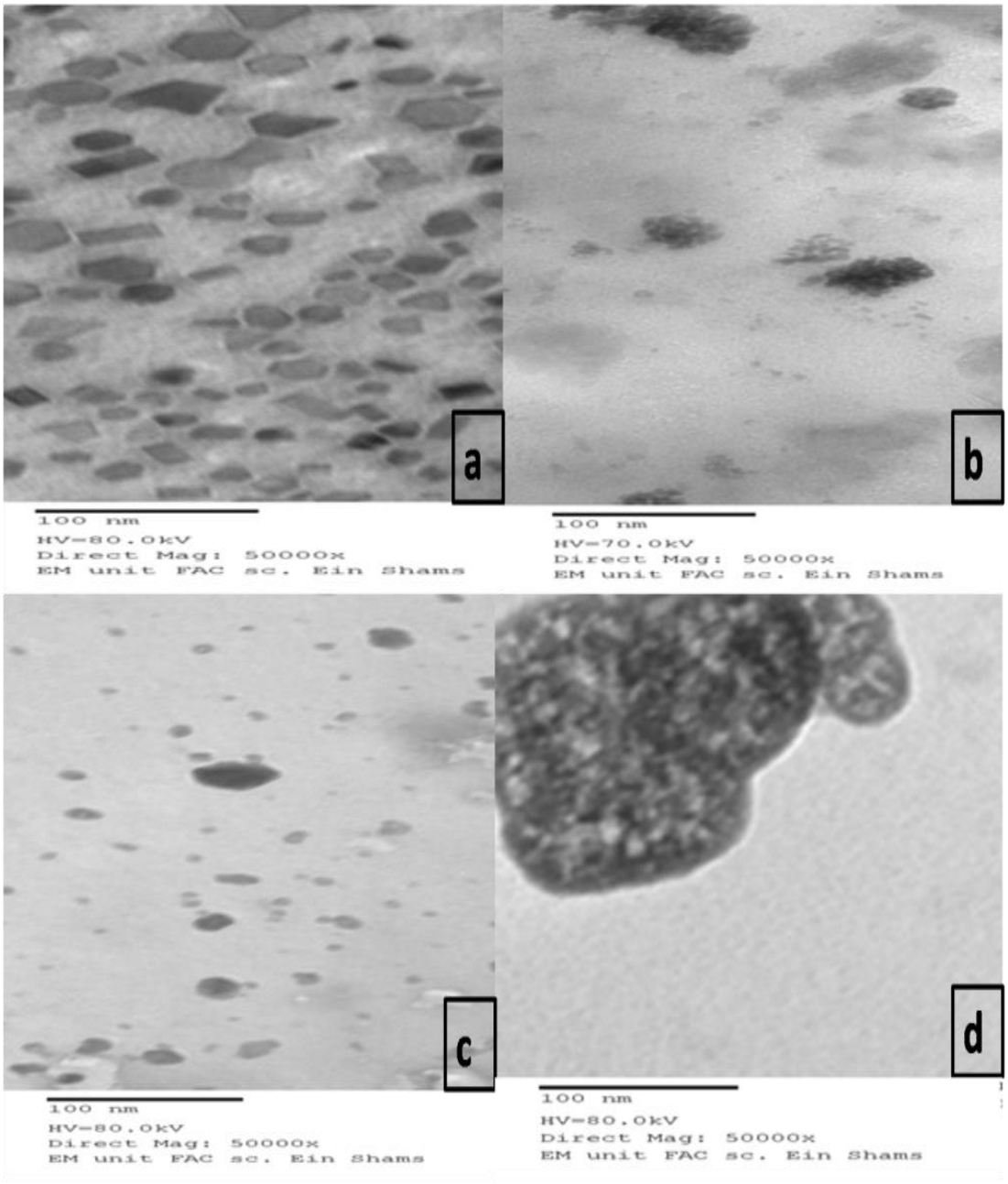

Figure 3 "TEM images of ZnO nano-structure samples for (a) as-prepared and annealed for (b) 1 hour (c) 4 hours (d) 8 hours".

TEM images show that the ZnO nano-structure have many shapes. By increasing annealing periods, an increment in the size of the nanoparticles occurs. For the as-prepared sample (**Figure 3a**), the average nano-structure diameter ranges from 30-40 nm, which is in good agreement with the value obtained from Scherrer's formula. For the annealed samples (**Figure 3 b, c**), an aggregation occurs in ZnO crystals; the nano-structure, in turn, convert into clusters. These clusters may reach several hundred nanometers (**Figure 3d**). TEM results differ from the XRD results according to the duration of the annealing time: the longer the annealing time, the more variation occurs. The fact that the applicability of Scherrer's formula is

restricted to small particles (<100 nm) has been reported; large ZnO crystallites show differences that ensure the inapplicability of Scherrer's equation to Larger particles [18].

3.3 Fourier Transform Infrared spectroscopic analysis

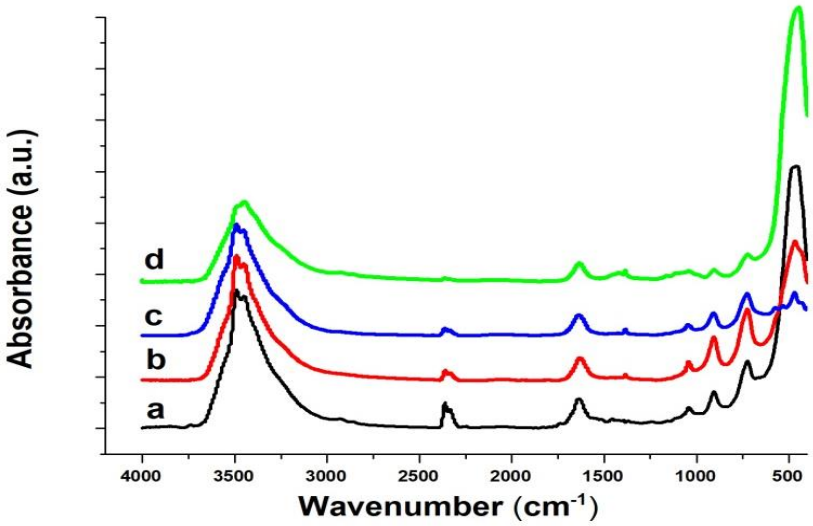

Figure 4 "FTIR spectra of ZnO nano-structure for (a) as-prepared and annealed samples for (b) 1 hour (c) 4 hours (d) 8 hours".

Figure 4 reveals the FTIR spectrum of the ZnO nano-structure prepared by the "chemical precipitation method"; the FTIR spectrum was measured in the range of "500-4000 cm^{-1}". The modes within 3150-3620 cm^{-1} are correlated to absorb symmetric H_2O. From **Figure 4** we can also observe that by increasing the annealing time, the sharpness of the characteristic peaks for the crystalline nature of ZnO increases, indicating the increase in particle size. The peak of 2850 cm^{-1} corresponds to OH stretching vibration. The mode of 2350 cm^{-1} corresponds to the CO_2 asymmetric bond. The adsorbed band at 1650 cm^{-1} corresponds to OH bending vibration. The absorption peak at 1350 cm^{-1} is a result of the formation of C-OH bending vibrations. The mode at 500 cm^{-1} indicates the stretching vibration of ZnO nano-structure [25].

3.4 (UV-VIS) absorption spectroscopy

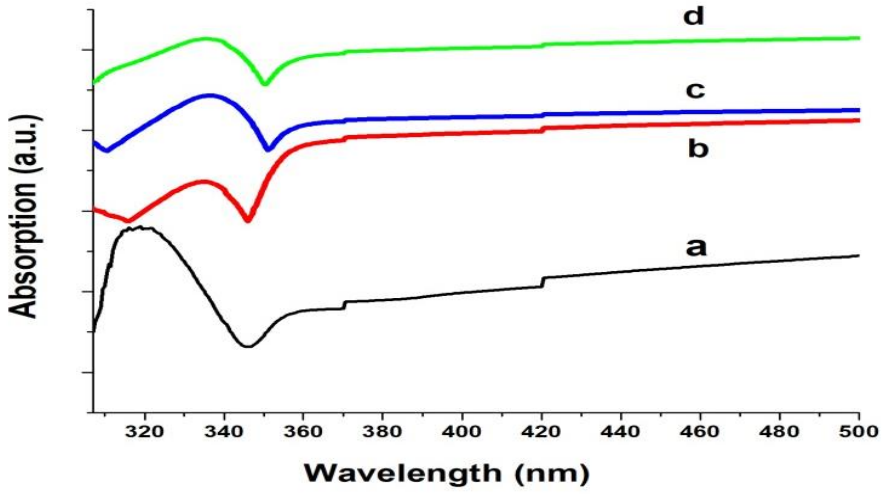

Figure 5 "UV-VIS absorption spectra at room temperature of (a) as-prepared and annealed ZnO samples for (b) 1 hour (c) 4 hours (d) 8 hours".

Figure 5 shows the effect of varying annealing periods on (UV-VIS) absorption spectra of annealed and as-prepared ZnO samples. The absorption peak for ZnO as-prepared sample is found to be around 335 nm. Otherwise, a small redshift in the absorption peak is observed for the annealed ZnO samples; this may be due to the difference in crystallite size. By plotting $(\alpha h\upsilon)^2$ versus $(h\upsilon)$, the optical bandgap of the prepared samples was estimated using the equation:

$$\boldsymbol{\alpha h\nu = (h\nu - E_g)^c} \quad \textbf{(2)}$$

"Where α is the absorption coefficient, hυ is the incident photon energy, E_g is the bandgap energy. The value of c depends on transitions nature: for indirect and direct transitions n = 2 and 1/2 respectively".

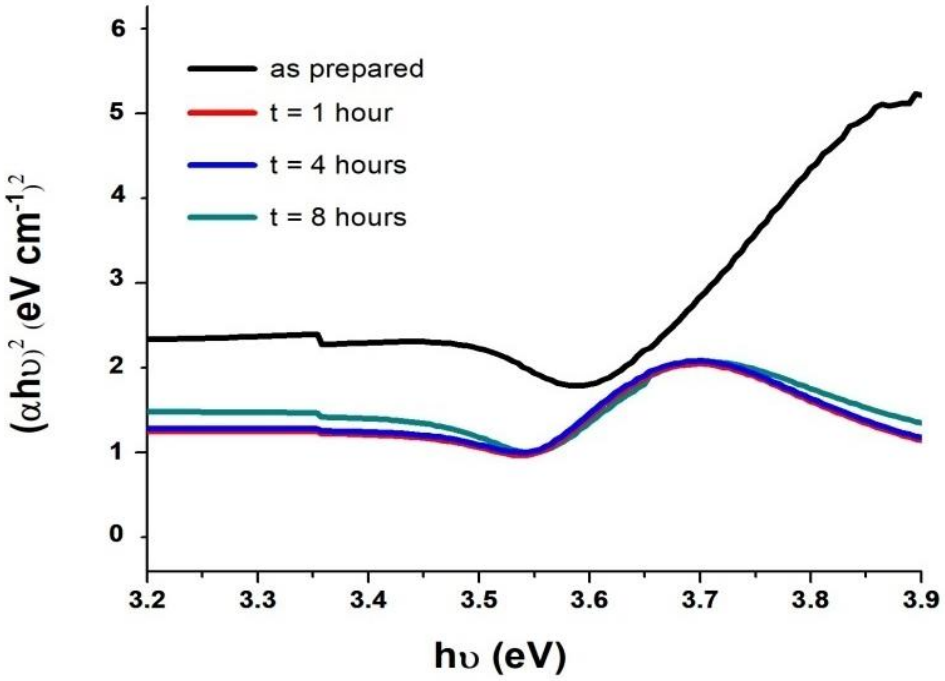

Figure 6 "plot of $(\alpha h\upsilon)^2$ versus $(h\upsilon)$ for (a) as-prepared and annealed ZnO nano-structure for (b) 1 hour (c) 4 hours (d) 8 hours"

By plotting the curves and introducing the linear part of it as shown in **Figure 6**, the bandgap energy is determined by the intersection on the x-axis. These values are tabulated in **Table 2**.

Table 2 "the bandgap energy for as-prepared and annealed ZnO nano-structure".

Annealing time	Bandgap energy (eV)
as-prepared	3.52
1 hour	3.45
4 hours	3.442
8 hours	3.431

From [**Table 2]**, it is obvious that the increasing trend in crystallite size is accompanied by a decrease in bandgap, upon the increase of the annealing time [33, 36].

4. Conclusion

The ZnO nano-structure prepared via coprecipitation method were synthesized after annealing for different periods, ranging from 0-8 hours. XRD, TEM, FTIR, and UV-Vis spectroscopy revealed the optical and structural properties as well as the particle size of the annealed samples. The Wurtzite structure of synthesized ZnO nano-structure was confirmed by XRD analysis. The average particle size of ZnO nano-structure (as-prepared) was shown by TEM figures ranging within 30-40 nm. Besides, XRD and TEM

analysis confirmed that by increasing annealing time, the average particle size of ZnO nano-structure increases. A wide absorption band shown by FTIR is related to Zn-O vibrational mode. Besides, UV-Vis absorption was estimated by the bandgap energy of ZnO nano-structure; it was observed that with increasing annealing time, a slight redshift in the absorption edge occurs. It could be concluded that annealing time is considered an important tool in modifying the optical properties and in improving the crystal quality of prepared ZnO samples.

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