

## Structural and Optical Characterization of ZnO and Glucose Capped ZnO Nanoparticles

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**Abstract:** Zinc Oxide and Glucose capped Zinc Oxide nanoparticles have been prepared using modified chemical reaction method. X-ray diffraction analysis showed that the prepared samples possess polycrystalline nature with hexagonal structure. Surface morphology has been analyzed using scanning electron microscopy. The estimated value of band gap was found to be 3.41 and 3.87 eV for Zinc Oxide and Glucose capped ZnO respectively. Fourier Transform Infrared spectroscopic analysis has been carried out to find the chemical bond and elemental composition present in Zinc Oxide and Glucose capped Zinc Oxide.

**Keywords:** Band gap; SEM; X-ray diffraction; ZnO

### 1. INTRODUCTION

Transparent conducting oxides (TCO's) found to exhibit some specific characteristics which make them interesting for various applications in Photovoltaic devices, Light Emitting Diodes and Solar cells [1, 2]. Besides, Zinc Oxide (ZnO) is a semiconductor with an energy gap of 3.3 eV, large exciton binding energy (60 meV) thus leads to various application viz., optoelectronic devices, piezoelectric devices and chemical sensors [3-5]. Numerous techniques have been used to prepare ZnO nanoparticles such as mechano-chemical synthesis [6], metal alkoxide hydrolysis [7], hydrothermal [8] and sol-gel reaction process and so on [9, 10]. Synthesis of metal oxide nanoparticles is currently interesting and its has been limited due to usage of organic compounds to obtain metal alkoxides using metallic salts as source of metals. When metal alkoxides are used as precursors, the rate of hydrolysis and poly-

condensation can be easily controlled by adjusting conditions of the reaction [11]. The process of capping has been obtained by the addition of organic molecule as capping agent with metal oxide nanoparticles thus leads to passivate the surface of the particles without agglomeration.

In the present work, ZnO nanoparticles have been obtained in which the base material NaOH is used in presence of inorganic metal salts. Also, Glucose capped ZnO nanoparticles have been obtained by using NaOH with inorganic metal salts in addition to organic molecule (Glucose) as capping agent. X-ray diffraction analysis has been carried out to determine crystalline nature and phases of the prepared samples. Surface morphology has been analyzed using scanning electron microscopy. Optical absorption analysis has been carried out to find the band gap value of the prepared samples. Fourier Transform Infrared (FTIR) spectroscopic analysis has been used to find out the presence of chemical bonds and elemental composition in the prepared samples. The effects of capping agent (Glucose) with ZnO on structural, mor-

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phological, compositional and optical properties of the prepared samples were investigated.

## 2. EXPERIMENTAL DETAILS

The chemicals used in the present work were of AR grade reagents (99.99% purity, purchased from Sigma Aldrich). Zinc acetate dihydrate  $Zn(CH_3COO)_2$  and Sodium hydroxide (NaOH) were used as source materials, Glucose molecule as a capping agent for the preparation of ZnO and Glucose capped ZnO nanoparticles. The molarity of Zinc acetate was fixed as 0.3 M by carrying out the experiments at various molarity values in the range between 0.1 and 0.3 M. The preparation of 0.3 M Zinc acetate solution was obtained by dissolving suitable amount of  $Zn(CH_3COO)_2$  in double distilled water in 100 ml glass beaker. By the addition of an adjustable amount of 0.1 M NaOH the pH value of the reaction bath reaches  $9.0 \pm 0.1$  leading to produce a white precipitate at the bottom of the beaker. The resulting white precipitate was washed thoroughly with double distilled water followed by ethanol to remove the unwanted impurities present in it. Thereafter, the resultant product was centrifuged at 10,000 rpm for 20 minutes. The powder sample was separated from the reaction bath and kept in vacuum desiccators. Thereafter the resulting white powder samples were dried in hot air oven at  $150^\circ C$  for the time period of 3 hours. Similar procedure was carried out by the usage of commercially purchased Glucose as capping agent for the preparation of Glucose capped ZnO nanoparticles. Similar experimental procedure was carried out for the preparation of ZnO and Glucose capped ZnO nanoparticles, which has been reported earlier [12].

### 2.1. Characterization

The crystalline nature of the prepared powder samples was identified with the help of X-ray diffractometer (XPERT PRO PANalytical, Netherland) using  $CuK_\alpha$  radiation ( $\lambda=1.5406 \text{ \AA}$ ) and with step increment of  $0.02^\circ$  within the  $2\theta$  value in the range between 10 and  $80^\circ$ . Surface morphology of the prepared samples was analyzed using a Scanning electron microscope (JEOL JSM 840). Optical absorption and transmittance measurements of the prepared samples were analyzed using an UV-Visible-NIR (Shimadzu Model 200, Singapore) spectrophotometer within the wavelength range between 300 and 1100 nm. Fourier Infra Red spectroscopic analysis was carried out using a FTIR (Shimadzu 8400S) spectrometer within the value of wavenumber range between 400 and  $4000 \text{ cm}^{-1}$ .

## 3. RESULTS AND DISCUSSION

### 3.1. Structural Analysis

X-ray diffraction analysis has been carried out to find out the crystalline nature of the prepared powder samples. Figure 1 shows the XRD pattern recorded for ZnO and Glucose capped ZnO nanoparticles obtained at optimized condition. It is observed that the prepared samples are found to be polycrystalline in nature with hexagonal structure. The diffraction peaks of ZnO are found at  $2\theta$  values of angles  $31.8, 35.01, 36.32, 47.53, 56.60, 62.86, 68.01$  and  $76.20$  degree corresponding to the lattice planes (100), (002), (101), (102), (110), (103), (112) and (202), respectively. The "d" values observed in the present work are found to be in close agreement with JCPDS ICDD file for hexagonal ZnO [13]. All the identified peaks are from ZnO and hence there is no observation of peaks

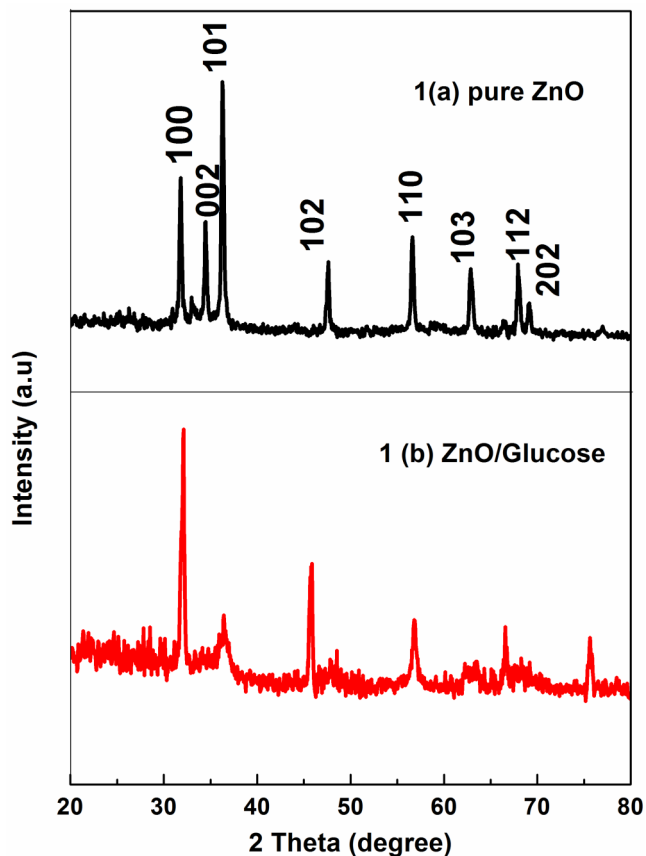


Figure 1. X-ray diffraction pattern of (a) ZnO (b) Glucose capped ZnO nanoparticles

corresponding to Zn and O, which are denoted in Figure 1a. It is observed that the intensity of (100) plane is found to be higher than all other peaks in the diffractogram indicating that the crystallites are preferentially oriented along (100) plane. On the other hand, there is change in preferential orientation of the plane from (100) to (101) and slight shift in the peak position for Glucose capped ZnO, which is denoted in Figure 1b. The observation of the broad peak at  $2\theta$  value of  $36.32$  degree corresponding to a decrease in the value of crystallite size of the prepared samples may be due to the addition of Glucose molecule as capping agent [14]. The crystallite size of the prepared samples was determined using FWHM data and Debye Scherrer formula [15,16].

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

where,  $\lambda$  is the wavelength of  $CuK_\alpha$  target used ( $\lambda=1.5418 \text{ \AA}$ ),  $\beta$  is the full width at half maximum of the peak position in radian and  $\theta$  is the Bragg's diffraction angle at peak position in degree. The value of lattice constants and stress for the prepared powder samples are determined using the following equations [17,18].

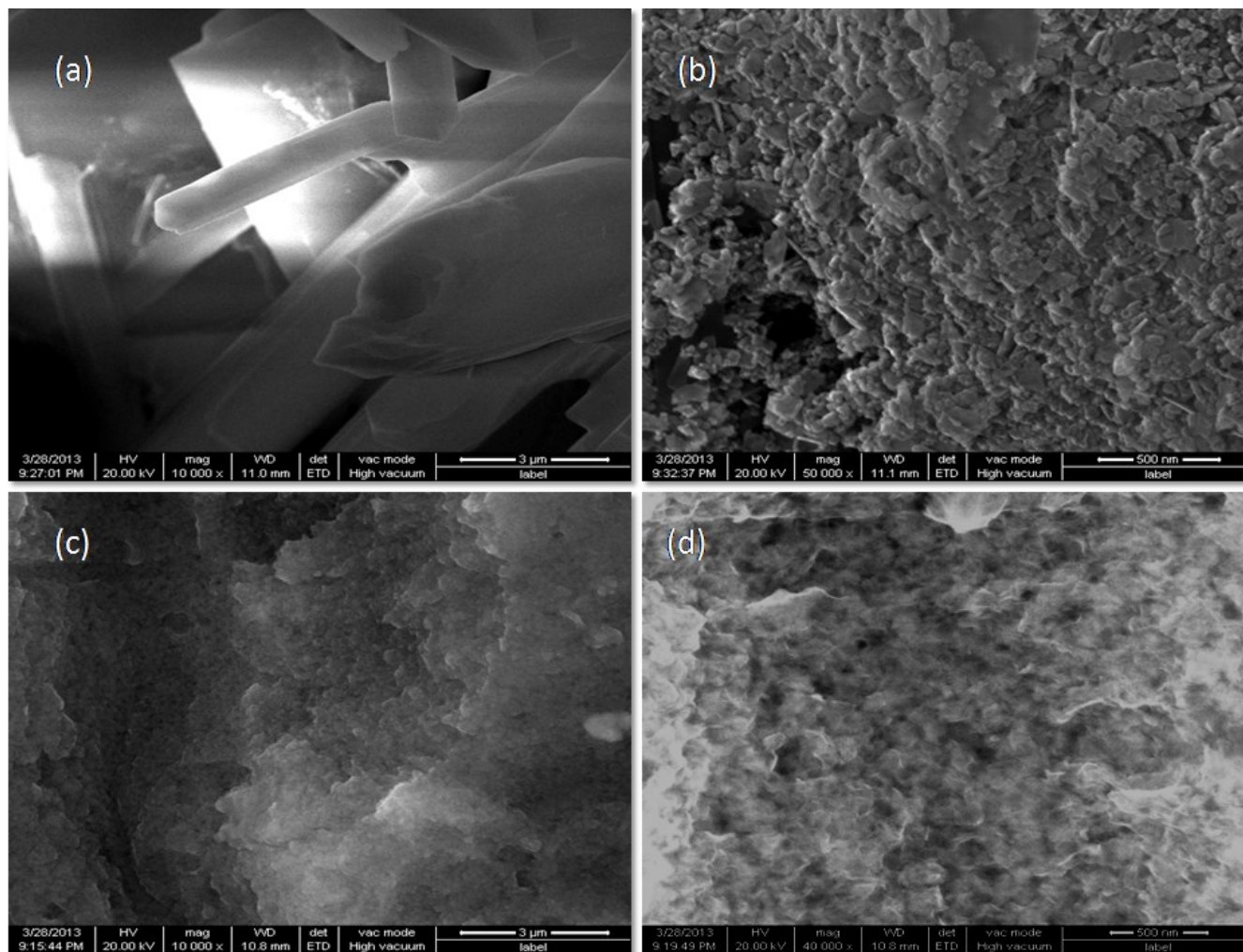


Figure 2. SEM images of (a) ZnO (b) Glucose capped ZnO nanoparticles

$$\frac{1}{d^2} = \frac{4}{3} \left[ \frac{h^2 + hk + l^2}{a^2} \right] + \frac{l^2}{c^2} \quad (2)$$

$$\sigma = -233 \frac{c - c_0}{c_0} \quad (3)$$

The size of the crystallites for ZnO and Glucose capped ZnO samples are found to be in the range between 41 and 22 nm. The

Table 1. Structural parameters of ZnO and Glucose capped ZnO nanoparticles

Sample	Lattice constants (Å)			Crystallite size (D) nm	d spacing (Å)	Stress (σ) 10 <sup>-9</sup>
	a	c	c/a			
ZnO	3.2451	5.1901	1.5994	41	2.8136	-37.334
Glucose capped ZnO	3.2594	5.2073	1.5976	22	2.7929	-33.409

estimated value of structural parameters such as lattice spacing, lattice constants, stress and crystallite size are given in Table 1.

### 3.2. Morphological Analysis

The surface morphology of ZnO and Glucose capped ZnO samples was analyzed using scanning electron microscopy. Figure 2 shows the scanning electron microscopic images for ZnO and Glucose capped ZnO nanoparticles. The process of agglomeration produces small rod shaped grains for ZnO nanoparticles, which is seen in Figure 2a. The addition of Glucose molecule as capping agent can produce changes in surface morphology, but also prevent the process of agglomeration for Glucose capped ZnO, which is seen in Figure 2b. During the process of capping, Glucose molecule combined with OH groups of already existed Glucose molecules attach with ZnO nanoparticles. Thus, the capping agent provides protection of organic shell structure to prevent the nanoparticles from the agglomerated solution. Therefore, by changing the value of ratio for precursor and capping agent produces change in surface morphology of the system, since the ratio of variation show the way to stabilize the plane of orientation [19].

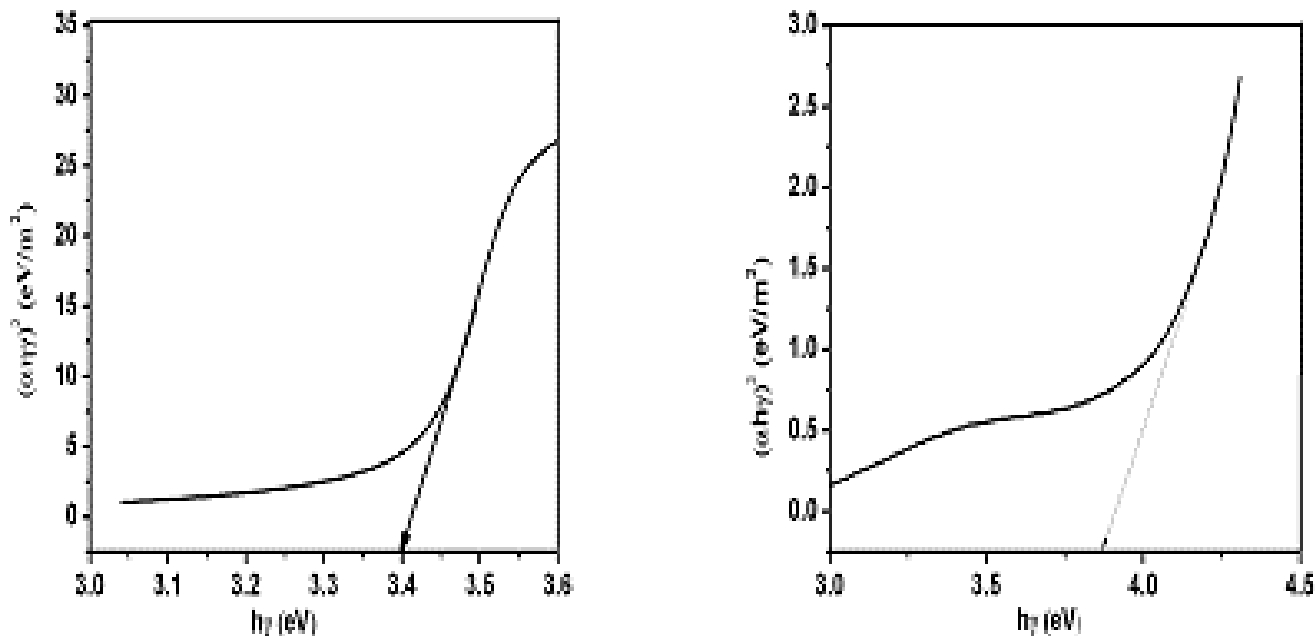


Figure 4. Plot of  $(h\nu)$  versus  $(\alpha h\nu)^2$  for (a) ZnO (b) Glucose capped ZnO nanoparticles

### 3.3. Optical Absorption Analysis

Optical absorption analysis was carried out to determine the optical properties of ZnO and Glucose capped ZnO. Figures 3a and 3b show the optical absorption spectra recorded for ZnO and Glucose capped ZnO nanoparticles within the wavelength range between 300 and 1100 nm. It is noted that the observation of wavelength shift from 307 to 362 nm correspond to shift in value of band gap from 3.41 and 3.87 eV for ZnO to Glucose capped ZnO. The observed value of band gap in the present work must be quite closer to the value of ZnO. The observation of red shift in absorption wavelength for Glucose capped ZnO may be due to the process of capping Glucose molecule with ZnO. The value of band gap is defined as the energy in which the value of absorption coefficient is higher than  $10^4 \text{ cm}^{-1}$ . A graph was plotted between  $(h\nu)$  versus  $(\alpha h\nu)^2$  for ZnO and Glucose capped ZnO nanoparticles as shown in Figure 4. The linear portion of the graph is extrapolated to energy (x-axis) axis, which gives the band gap value of the material. The estimated band gap value for ZnO and Glucose capped ZnO is found to be 3.41 and 3.87 eV respectively, which is quite closer to the value reported earlier.

### 3.4. Fourier Transform Infrared Spectroscopy Analysis

Fourier Transform Infrared Spectroscopy is one of the important analytical techniques, which is used for investigating vibrational properties of synthesized materials. The absorption peak and band positions not only depend on composition, but also the coordination of surfactant on the surface of the prepared samples. The organic molecules present in synthesis have high surface energy to stabilize their thermodynamically unfavourable state. The stabilizing agents control the process of nucleation, growth and its ability to bind on the surface of the sample and stabilize them against ag-

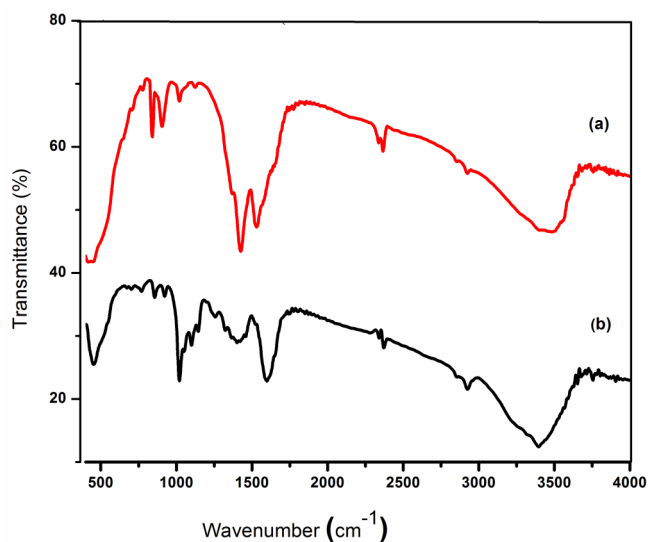


Figure 5. FTIR spectra of (a) ZnO (b) Glucose capped ZnO nanoparticles

glomeration [23]. FTIR spectra recorded for ZnO and Glucose capped ZnO nanoparticles is shown in Figure 5. The appearance of the broad peak at  $3595 \text{ cm}^{-1}$  may correspond to the vibration mode of O-H bond indicating the formation of  $\text{Zn}(\text{OH})_2$ . The observation of peaks at  $1430 \text{ cm}^{-1}$  corresponds to asymmetric-symmetric stretching mode of vibration for C = O bond due to the association of residual organic impurities in the prepared powder samples. FTIR spectra recorded for Glucose capped ZnO molecule is shown in Figure 5b. It is noted that the presence of peaks at 1430 and 1017

$\text{cm}^{-1}$  corresponding to C-H bond due to the process of coupling of Glucose molecule which acts as capping agent for ZnO. It is also noted that the observation of peak intensity at 1607, 840  $\text{cm}^{-1}$  is attributed to C-O, Zn-O bond indicating the interaction (capping) of Glucose with ZnO.

#### 4. CONCLUSIONS

ZnO and Glucose capped ZnO nanoparticles were successfully prepared using modified chemical reaction method. X-ray diffraction analysis showed that the prepared samples are found to be in nanocrystalline nature with hexagonal structure. There is change in preferential orientation of the plane from (100) to (101) corresponding to ZnO to Glucose capped ZnO. The observation of change in surface morphology without agglomeration of grains is observed for ZnO and Glucose capped ZnO. The shift in the value of band gap from 3.41 to 3.87 eV is noted for pure and Glucose capped ZnO. The appearance of peaks at 1607, 840  $\text{cm}^{-1}$  characterized the C-O and Zn-O bands indicating the interaction process of Glucose molecule with ZnO. The process of capping Glucose with ZnO nanoparticles can produce changes in structure of ZnO nanoparticles with Carbohydrate as a biotemplate addressing biomedical applications, food packaging, water purification and water preservative processing technologies.

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