Microwave-assisted synthesis of ZnO nanoflakes: structural, optical and dielectric characterization

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Abstract
In this study, we produced ZnO nanoflakes (ZnO-Nfs) by using microwave-assisted techniques. The structural properties of ZnO-Nfs were analyzed by x-ray diffraction (XRD) technique, Raman scattering spectroscopy and field-emission scanning microscopy (FESEM). The Crystallite size (D) and lattice constants of ZnO-Nfs were calculated. The optical properties of ZnO-Nfs were investigated by using UV-visible diffuse reflectance spectrum and photoluminescence (PL) spectra. Also, dielectric constants of ZnO-Nfs were calculated as related to the refractive index (n) an extinction coefficient (k).

1. Introduction

Recently, metal oxide (ZnO, TiO2, CuO, etc) based nanomaterials have been attracted great attention from the researchers. Among the metal oxide materials, ZnO (zinc oxide) has widely used due to its unique optical, acoustic and electrical properties. Additionally, ZnO is low cost, non-toxic and chemically stable [1, 2]. The development of nano-materials made it possible to fabricate more efficient devices, especially in electronic applications. Recently, ZnO nanoflakes (ZnO-Nfs) have been used in electronic [3], optical [4], and biomedical devices [5] applications. Today, a wide variety of ZnO nanomaterials can be synthesized such as nanoparticles [6], nanoflowers [7], nanoflakes [8], nanowires [9], nanorods [10], nanobelts [11], etc. These ZnO based nanomaterials can be produced by using sol-gel [12], chemical vapor deposition [13], hydrothermal [14], microwave irradiation [15], electrochemical deposition [16], solid-state method [17], sonochemical [18], spray pyrolysis synthesis techniques [19]. In addition, being low-cost and clean, the production method must also allow the control of the size of the material. The microwave-assisted method can be used to control the sizes and shapes of nano-sized materials. Moreover, this method also allows the successful production of large scale nano-sized materials with superior crystal properties.

In recent years, a microwave-assisted method has been used in the production of nanoparticles such as oxide, hydroxide, and sulfur. In addition to being simple, low-cost and clean, this method is unaffected by the disadvantages of temperature changes in the sol-gel and hydrothermal methods. Today, nanomaterials are often produced using hydrothermal and sol-gel methods, but when compared to these methods, the microwave-assisted production technique offers some significant advantages. These advantages include fast reaction time, a simple medium, a short time needed to reach the required temperature for the reaction to take place, and the easy control of the morphology of the particles [15]. In this method, heat is generated with reactants without using an external power supply. Rapid reactions consist of small energy without inside intermediate phases [20]. Therefore, the microwave-assisted technique is a low-cost method to produce ZnO based nano-structure materials. ZnO based nano-structure materials have been produced by using microwave-assisted techniques. Hasanpoor et al [15] produced nanoparticle of ZnO by using microwave-assisted techniques. Laga et al [21] synthesized ZnO rhombic microstructure using microwave radiation. However, high crystalline ZnO-Nfs have never before been produced by using microwave-assisted techniques.

In the present study, the microwave-assisted method was used to produce low-cost and clean nano-size ZnO-Nfs with superior crystal properties, and the structural, optical and dielectric properties of the produced materials were examined. The structural properties of the produced ZnO-Nfs were examined in detail by an
x-ray diffraction analysis (XRD), field emission scanning electron microscopy (FESEM), an energy dispersive x-ray analysis (EDX), Raman spectroscopy and photoluminescence (PL) analysis. Thus, optical analyses were carried out, and the forbidden band gap was calculated using the Kubelka-Munk approach. Dispersion plays an important role in the design of optical devices and studies of the optical characteristics of materials. The single-oscillator model approach is used to examine the behavior of the refractive index by defining the dielectric response for transitions close to the forbidden band gap. The dispersion of the refractive index can be analyzed using the single-oscillator model developed by Wemple and Domenico. Therefore, the dielectric properties of the ZnO-NFs were examined by calculating their dielectric constants.

2. Experimental

2.1. Synthesis of ZnO nanoflakes

ZnO-NFs were synthesized by using the microwave-assisted route. In a typical synthesis, 0.135 M of zinc acetate dihydrate (\(\text{Zn(CH}_3\text{COO)}_2 \cdot 2\text{H}_2\text{O}\)) and 3.23 M NaOH were dissolved separately in deionized water under gently stirring at 30 °C. This solution was obtained after stirring the zinc acetate solution for 15 min, and the prepared NaOH solution was added drop by drop. Then, this mixed solution was stirred to form a milky solution. The prepared solution was transferred into a 500 ml beaker. Then, the beaker was placed in the microwave oven. Microwave irradiation was carried out at 1000 W for 5 min. After microwave processing, the solution was cooled down to room temperature ambient condition. The produced white precipitate was collected by centrifugation, filtration, and washing with water and ethanol and dried for 24 h at 70 °C. Finally, the obtained white powder was dried at 500 °C for 2 h in an oven.

3. Results and discussion

3.1. XRD analysis of ZnO nanoflakes

The structural properties of ZnO-NFs were investigated by using the XRD technique. These studies were performed using the Panalytical Empyrean XRD diffractometer, with Cu K\(_\alpha\) radiation (\(\lambda = 1.54059\) Å). XRD measurements were carried out from 10° to 90° with a 0.01° step size. All obtained diffraction peaks of the nanosized ZnO particles corresponded with the wurtzite structure, hexagonal crystal system, space group \(\text{P}6_3\text{mc}\) reported in the reference JCPDS-98-009-4004 card (\(a = b = 3.247\) Å; \(c = 5.203\) Å). As shown in figure 1, there are no extra peaks relating to any other impurities of ZnO material. As shown in figure 1, the narrow and intense peaks are indicated a good crystallinity and no impurities of ZnO material. The XRD pattern of the ZnO-NFs prepared by de-ionized water had a polycrystalline structure. The highest intensity of the crystal growth orientation was the (101) \(hkl\) plane at \(2\theta = 36.31°\). The average crystalline size of the obtained ZnO powder was calculated along this plane according to the known Debye Scherrer formula [23]:

\[
D = \frac{k\lambda}{\beta_{hl} \cos \theta}
\]  

Figure 1. XRD pattern of ZnO-NFs.
Table 1. The calculated values of crystallite size ($D$), lattice constants, unit cell volume ($V$), X-ray density ($d_{x}$), specific surface area ($S$) and bond length for ZnO-NFs.

| Sample   | Crystallite Size ($D$) (nm) | Lattice Constants | Volume of the unit cell ($V$) ($\text{Å}^3$) | X-Ray Density ($\times 10^4$) ($\text{kg m}^{-3}$) | Specific Surface Area ($S$) ($\text{m}^2 \text{g}^{-1}$) | Positional Parameter ($u$) (Å) | Bond-Length ($L$) (Å) |
|----------|-----------------------------|-------------------|---------------------------------------------|-----------------------------------------------|---------------------------------------------|-------------------------------|------------------|
| ZnO-NFs  | 33.8                        | $a = b$ (Å) = 3.250 | 5.206                                        | 47.62                                         | 0.567                                       | 31.30                         | 0.379            | 1.981            |
The bond length value of ZnO powder was found to be 1.981 Å. The ionic radius of O$^{2-}$ is 1.38 Å and 0.83 Å, respectively. As a result of binding, the length of the Zn-O bond is reported to be 2.21 Å in the literature [23]. The bond length in our study revealed the highly dense and uniform 2D layer structure, a and b were calculated using equation (2) along the (100) plane, and c was calculated using equation (3) along the (002) plane [23, 24].

$$a = \frac{\lambda}{\sqrt{3} \sin \theta_{100}}$$

$$c = \frac{\lambda}{\sin \theta_{002}}$$

The volume of the unit cell ($V$) was calculated as Å$^3$ according to the formula:

$$V = \sqrt{3} \frac{a^2 c}{2}$$

The values of the lattice parameters for the obtained ZnO powder was found as $a = b = 3.250$ Å, $c = 5.206$ Å. The unit cell volume of synthesized ZnO powder was 47.62 Å$^3$. These parameters were also evaluated for the calculation of the positional parameter ($u$) and bond length ($L$).

$$u = \frac{a^2}{3c^2} + 0.25$$

$$L = \sqrt{\frac{a^2}{3} + (0.5 - u)c^2}$$

The bond length value of ZnO powder was found to be 1.981 Å. The ionic radius of O$^{2-}$ and Zn$^{2+}$ formed ZnO material is 1.38 Å and 0.83 Å, respectively. As a result of binding, the length of the Zn-O bond is reported to be 2.21 Å in the literature [25]. Being small from 2.21 Å of our length value is expressed the structural defects such as oxygen vacancies [25]. X-ray density ($d_s$) is obtained by the formula as given below [23].

$$d_s = \frac{nM}{NV}$$

where $n$ is co-ordination number of the unit cell/number of atoms per unit cell, $M$ is the molecular weight of the ZnO-Nfs, $N$ is Avogadro’s number. The x-ray density ($d_s$) was calculated as 0.567 $\times$ 10$^3$ kg/m$^3$. The specific surface area ($S$) of the material was revealed based on the following Sauter equation [23]:

$$S = \frac{6000}{D d_s}$$

The value of the specific surface area ($S$) of our material was 31.30 $m^2$ g$^{-1}$. This value is referred that the specific surface area is an important material property for heterogeneous catalysis, adsorption, and reactions on the surface [26]. This may be contributed to the efficient photocatalytic performance-enhancing the higher degradation ratios in the practical application of devices [23].

### 3.2. Surface morphology and elemental analysis of ZnO nanoflakes

Surface morphological investigation of the prepared ZnO-Nfs powder sample was performed by using scanning electron microscopy (FEI Quanta FEG 650 at 20 kV accelerating voltage). For elemental analysis, weight and atomic percentages of the sample were determined by EDAX. FESEM analysis of the obtained pure ZnO powder in our study revealed the highly dense and uniform flakes-like morphology (figure 2). As seen in figure 2, the particle size of ZnO-Nfs was about 60–200 nm. This size was larger than the crystalline size of ZnO-Nfs calculated by Scherrer’s formula from XRD analysis. Thus, the larger particle size of ZnO-Nfs may be caused to nanoflakes formation [27]. Similarly, Arasu et al showed that the size viewed in FESEM larger than the crystalline size estimated from XRD results. The densely packed surface morphology of ZnO-Nfs is an advantage for the efficient charge carrier separation [28]. EDX spectrum of ZnO-Nfs samples is shown in figure 2(d). The weight percentages of the Zn (88.6%) and O (11.4%) for the ZnO-Nfs sample are shown in figure 2(d). Zn and O are the main constituents of the sample and no trace of impurities could be found within the detection limit of EDX.

### 3.3. Photoluminescence characterization of ZnO nanoflakes

The optic and electronic properties ZnO-Nfs were performed by the photoluminescence (PL) spectroscopy. PL analysis was acquired by using the Cary Eclipse fluorescence spectrophotometer system equipped with a 75 kW Xe pulse lamp as an excitation source at 250 nm excitation wavelength. The excitation and emission slit widths...
For PL measurements were adjusted to 5 and 10 nm, respectively. Figure 3(a) shows the PL analyses of the pattern, and figure 3(b) shows the PL analyses after deconvolution using the Gaussian function. According to PL results, the impurity levels, defects, and electronic band gap, material quality and recombination mechanisms of samples are investigated. The emission bands of the UV and visible regions in PL spectra are revealed in figure 3(a). As shown in figure 3(a), there are two emission bands as the UV and visible regions in PL spectra. For photoluminescence characterization, the crystal quality and structural defects of the sample are analyzed using the emission band in the UV region and the visible region, respectively [23]. The deep level defects such as zinc (Zn$_i$) and oxygen (O$_i$) interstitials, zinc (V$_{Zn}$) and oxygen (V$_{O}$) vacancies in ZnO can be caused PL emission in the visible region [23].
In our PL data, the UV emission band at 361 nm was the result of the recombination of free excitons during the exciton-exciton collision process. This process is known as a near band-edge transition of ZnO [23]. Similar results related to the UV emission band were recorded by Senthilkumar et al (2018) [29]. As can be seen in figure 3(a), the violet emission in the visible region centered at 427 nm. This band has resulted from the electronic transition from zinc interstitial level (Zn_i) to the top of the valance band (VB). Similarly, Das et al [26] observed that the violet emission band at 425 nm. The blue and blue-green emission bands in the visible region were determined at 460 and 488 nm, respectively (figure 3(a)). The transition of electrons from shallow donor levels (Zn_d) to shallow acceptor levels (V_{Zn}) constitutes the blue emission band [26]. The blue-green emission band was originated from surface defects [30]. The PL emission band in the yellow/orange region was observed at 568 nm (figure 3(a)). This band is originated from the oxygen interstitials (O_{i}) [23]. Our green emission band, related to deep-level emission, was observed at 528 nm. This emission band belongs to the singly ionized oxygen vacancy that occurred from the recombination of the photogenerated hole with the single ionized charge state of this defect and/or the electron transition from the bottom of the conduction band to the antisite defect O_{Zn} level [31, 32].

In our result, the high intensity of green emission expresses ZnO nanoflake having a higher number of defect states. This result indicates that the ZnO nanoflake obtained with high defect density is a better material for gas and humidity sensing applications owing to enhanced defect stimulated electrostatic interaction between water and ZnO surfaces [8, 33]. But the presence of these high structural defects is not suitable for optoelectronic devices [8, 33]. XRD results related to our bond length value indicate structural defects. So, this supports photoluminescence analysis data. Thus, figure 3(b) shows the PL analyses after deconvolution using the Gaussian function. Within the plot in figure 3(b), the full width at half maximum (FWHM) values, corresponding to the peak positions, are presented as column graphs. The figure shows a decline in FWHM values as the intensity and sharpness of the PL peaks increase.

### 3.4. Raman characterization of ZnO nanoflakes

The phase, crystalline quality, and defects of the synthesized ZnO-Nfs were investigated by Raman spectra, a nondestructive characterization method. Raman measurements were performed using a Via Qontor Renishaw spectrometer with a 532 nm laser-edge filter (power 10%). Figure 4(a) shows the Raman analyses of the pattern, and figure 4(b) shows the Raman analyses after deconvolution using the Gaussian function. An examination of the Raman results indicated that the optical phonons at the Brillouin zone (Γ) for wurtzite ZnO group were expressed by the following models [34], \( \Gamma_{opt} = A_1 + E_1 + 2E_2 + 2B_1 \), where the \( B_1 \) is silent; the \( E_2 \) modes (\( E_{2(low)} \) and \( E_{2(high)} \)) are non-polar and Raman active only, while the \( A_1 \) and \( E_1 \) are polar modes, both Raman and infrared active. \( E_{2(high)} \) and \( E_{2(low)} \) phonon modes are associated with oxygen atoms and ZnO vibrations, respectively. Additionally, the \( A_1 \) and \( E_1 \) modes are divided into two components (longitudinal (LO) and transverse (TO)) [34].

Figure 4(a) shows, the Raman scattering of ZnO flakes is investigated ranging from 102 cm\(^{-1}\) to 3201 cm\(^{-1}\). In our study, the second-order phonon mode at about 144 cm\(^{-1}\) presented to 2E_{2l}. The peak observed at 332 cm\(^{-1}\) originated the multiphonon (\( E_{2lH} - E_{2l} \)) scattering process [26]. The peak at 438 cm\(^{-1}\), seen to be sharp, narrow and strong, assigned to the nonpolar optical \( E_{2lH} \) mode. This result confirmed to synthesis the ZnO-Nfs having good crystallinity and wurtzite hexagonal structure by supporting XRD results [35]. The small peak centered at 583 cm\(^{-1}\) arisen from structural disorders such as oxygen vacancies and zinc interstitial, was \( A_1(LO) \) phonon mode [26, 36]. This result was associated with the PL emission peak at 460 and 488 nm resulted from
oxygen deficiency [37]. The broad peak at ranging 1050 to 1200 cm\(^{-1}\) indicated the multiphonon process (2LO). This peak belongs to the characteristic properties of II-VI semiconductors. Thus, figure 4(b) shows the Raman analyses after deconvolution using the Gaussian function. Inset the plot in figure 4(b), the full width at half maximum (FWHM) values, corresponding to the peak positions, are presented as column graphs. The figure shows a decline in FWHM values as the intensity and sharpness of the Raman peaks increase.

3.5. Optical properties of ZnO nanoflakes

The reflectance measurements of the produced ZnO-Nfs were performed, and the resulting reflectance spectra are provided in figure 5. The calculated forbidden band gap (\(E_g\)) of the produced semiconductor material is a highly important parameter for materials to be used in optoelectronic devices. In the present study, the forbidden band gap of the ZnO-Nfs was calculated using the Kubelka-Munk approach, which is based on reflectance. The Kubelka-Munk function can be calculated based on reflectance as follows [38]:

\[
\alpha = F(R) = \frac{(1 - R)^2}{2R}
\]

where \(\alpha\) is the absorption coefficient, \(F(R)\) is Kubelka-Munk function and \(R\) is the reflectance. The forbidden band gap of ZnO-Nfs was determined by using Tauc’s equation [38, 39]. Tauc’s equation can be written by the following.

\[
F(R)h\nu = A (h\nu - E_g)^n
\]

where \(h\nu\) represents photon energy, \(A\) is a constant and the value of \(n\) depends on the type of transition. It is 1/2 for direct and 2 for an indirect transition. ZnO-Nfs is an oxide material with a direct forbidden band gap. Using these calculations, the \([F(R), h\nu]^2 - h\nu\) plots of the ZnO-Nfs were drawn, and are provided in figure 6. Using the linear area in which \([F(R), h\nu]^2 = 0\] was in the plot, \(E_g\) was calculated [40, 41]. Through this method, the band gap of the ZnO-Nfs produced in the present study was found to be 3.31 eV. As the crystals were nano-sized (33.8 nm), the quantum size effect within the crystals may have widened the forbidden band gap. This value of the forbidden band gap (\(E_g = 3.31\) eV) is relatively higher than the previously reported values. Su-Yang Han et al [42] calculated the forbidden band gap for ZnO nanoflakes as 3.27 eV by a hydrothermal process. Shinde et al [43] calculated band gap for ZnO nanoflakes 3.09 eV. However, Molefe et al [44] calculated the forbidden band gap for ZnO nanoflakes as 3.91 eV by a chemical bath deposition method. This value is higher than our calculated \(E_g\) value.

3.6. Dielectric properties of ZnO nanoflakes

The refractive index \((n)\) can be calculated from the diffuse reflectance spectrum. The refractive index can be calculated using the Fresnel formula given in equation (11) [45]:

\[
n = \left( \frac{1 + R}{1 - R} \right) + \sqrt{\frac{4R}{(1 - R)^2 - k^2}}
\]

where \(R\) is reflectance, \(n\) is the real part and \(k\) is the imaginary part of the complex refractive index. Besides, \(k\) is known as the extinction coefficient. It is important to know the dielectric characteristics of a solid material. The dielectric constant is related to the state intensity within the forbidden band gap of the material, and so the real
and imaginary parts of the dielectric coefficient should be examined based on the incident photon energy. The real and imaginary parts of the dielectric constant are \( \varepsilon_1 \) and \( \varepsilon_2 \) respectively, and the complex dielectric constant can be written as \( \varepsilon = \varepsilon_1 + i\varepsilon_2 \). The \( \varepsilon_1 \) and \( \varepsilon_2 \) values of complex dielectric constant can be written as follows, based on the refractive index [46]:

\[
\varepsilon_1 = n^2 - k^2
\]  

and

\[
\varepsilon_2 = 2nk
\]

The dispersion of the refractive index can be analyzed using the single-oscillator model developed by Wemple and DiDomenico. The single-oscillator model approach is used to examine the behavior of the refractive index by defining the dielectric response for transitions close to the forbidden band gap. The relationship between the refractive index \( n \) and photon energy \( (E = h\nu) \) according to this model is given in equation (14). Thus, the dielectric response needs to be examined for given photon energy. The real \( \varepsilon_1 \) and imaginary \( \varepsilon_2 \) parts of the dielectric constants versus \( h\nu \) curves are shown in figures 7 and 8, respectively. Figure 7 shows that \( \varepsilon_1 \) increased sharply at values 3.44 and 3.52 eV, but then remained constant. As seen in figure 8, \( \varepsilon_2 \) value keep nearly constant until 3.4 eV and then increases after 3.4 eV. This phenomenon can be described by the existence of some interactions between photons and electrons.

Additionally, the other optical constants of the material can be calculated using the Wemple-Didomenico (WD) single-oscillator model. Using this model, the dispersion behavior of the material can be examined based on its refractive index \( n \) and wavelength \( \lambda \). According to the Wemple-Didomenico model, the connection
between the refractive index, dispersion energy ($E_d$) and single-oscillator energy ($E_o$) can be written as follows [47, 48].

$$n^2 - 1 = \frac{E_o E_d}{E_o^2 - (h\nu)^2}$$

(14)

where $E_o$ is single oscillator energy, $E_d$ is dispersion energy, and $E$ is photon energy. Figure 9 shows the curves of the $n^2 - 1$ versus $E^2$. The $E_d$ and $E_o$ values were calculated from the slope ($E_o/E_d$)$^{-1}$ and intercept ($E_o/E_d$) of figure 9. The dispersion parameters $E_o$ and $E_d$ is calculated using below equation (15).

$$E_d = \sqrt{\frac{1}{\text{Intercept} \cdot \text{Slope}}} \quad E_o = E_d \cdot \text{Intercept}$$

(15)

$E_o$ is corresponding to the distance between the center of valance and conduction band, $E_d$ corresponds to interband optical transitions [49]. Figure 9 $E_o$ and $E_d$ were calculated 5.24 eV and 5.38 eV, respectively. These $E_o$ and $E_d$ values calculated have consisted of previous literature. Caglar et al [50] calculated $E_o$ and $E_d$ for ZnO as 5.28 and 9.52, respectively. Mhamdi et al [51] calculated $E_o$ and $E_d$ for ZnO as 2.09 and 5.43, respectively. Ouni et al [52] calculated $E_o$ and $E_d$ for ZnO as 6.56 and 14.38, respectively.
4. Conclusion

ZnO-NFs have been synthesized by microwave-assisted techniques. The crystallite properties of ZnO-NFs have been characterized by XRD and FESEM analysis. The crystallite size of ZnO-NFs was calculated as 33.8 nm by Debye Scherrer formula. The optical analysis of ZnO-NFs has been investigated via the UV-visible spectrum and photoluminescence spectra. The band gap of ZnO-NFs has been calculated as 3.31 eV. The calculated forbidden band gap (3.31 eV) is thus consistent with the literature. The crystal quality and structural defects of the sample are analyzed using photoluminescence in the UV and the visible region, respectively. Also, the dielectric properties of ZnO-NFs were investigated. The produced ZnO-NFs can be used gas and humidity sensing applications.

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