

Influence of Using Different Preparation Methods on the Properties of ZnO Nanoparticles

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Abstract

Zinc oxide nanoparticles (ZnO NPs) were created using two different preparation methods: chemical precipitation and the green method using the leaves of *Ficus carica* extract. The nanoparticles were examined using X-ray diffraction (XRD), field-emission scanning electron microscopy (FE-SEM) and energy dispersive X-ray Spectroscopy (EDX). Analytical techniques such as XRD were applied to verify the crystallinity of ZnO NPs as well as used to calculate the crystal size for the prepared samples. The XRD pattern exhibited a hexagonal structure, and the mean diameter of the crystal size for ZnO NPs prepared by chemical precipitation was 27.44 and 33nm for ZnO NPs prepared by the biological method. Nanoparticles of ZnO have a spherical shape, as examined by FE-SEM. The EDX test indicated the existence of peaks corresponding to zinc and oxygen. The surface properties, such as root mean square roughness (Rq) and average roughness (Ra) were examined by atomic force microscopy (AFM), where Rq and Ra were 35.8 and 29.3 nm, respectively. The ZnO NPs made using chemical precipitation and biological methods were studied with UV-visible spectroscopy (UV-Vis) to look at the absorption spectra, and it was found that the absorption spectrum increased with the green method.

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1. Introduction

The field of nanotechnology deals with creating and manipulating materials on a scale ranging from the size of a single molecule or atom to that of a submicron, as well as incorporating these smaller entities into larger systems [1]. Depending on their size and form, nanomaterials exhibit physicochemical characteristics that vary from those of the bulk material. By manipulating its size and structure on a microscopic scale, the nanomaterial unexpectedly takes on a new nature with enhanced powers [2]. Extensive attention from researchers in materials chemistry, medicine, agriculture, IT, biological processes, optics, electronics, catalysis, environment, energy, and senses is drawn to metal oxide nanoparticles due to their crucial significance in these domains [3-5]. Nanoparticles, which are the basic components of nanotechnology, typically range in size from 1 to 100 nm [6]. Metal nanoparticles, such as gold, zinc, silver, selenium, and copper, have lately gained significant interest due to their basic and technological importance [7-12]. Many scientists have lately been interested in zinc oxide (ZnO) due to its role in the biogenesis of nanoparticles (NPs). ZnO is a metal oxide semiconductor with a broad band gap of 3.36 eV and a high excitation binding energy. It is used in drug delivery, solar cells, photo catalytic degradation, and personal care goods such as sunscreens and cosmetics [13]. ZnO has excellent chemical and physical stability [14]. Iron oxide is the most abundant, with zinc oxide being the second most prevalent. It is cost-effective, secure, and simple to prepare. There are some physical and chemical techniques for synthesizing nanoparticles [15, 16]. Nevertheless, it is not always possible to avoid using harmful substances in the synthesis process [17]. As a result, finding a



more sustainable way to create nanoparticles is a top priority. Green method that uses either fungus or plant extracts are a dependable and environmentally conscious substitute for traditional physical and chemical processes [18, 19]. Green synthesis utilizes a method of fabricating nanomaterials that is both clean and safe while also being cost-effective and ecologically beneficial. One of the most well-regarded ways is the creation of nanoparticles utilizing organisms [20]. Within this group of organisms, plants and extracts derived from plants seem to be the best option due to their nature as “chemical factories” because they may include many bioactive compounds, which vary depending on the plant or plant material they are derived from. Plant extracts typically include flavonoids, terpenoids, and phenols [21, 22].

Within this study, zinc oxide NPs were synthesized utilizing two different techniques: the chemical approach and green approach, through an extract derived from the *Ficus carica* plant, which acts as a reducing and capping agent for nanoparticles. This study also investigated the impact of the preparation procedure on the characteristics of the material being prepared.

2. Experimental Part

2.1. Preparation of ZnO Nanoparticles by Chemical Method

The direct precipitation process was used to produce ZnO NPs, using zinc nitrate hex hydrate and NaOH as precursors. 0.2 M zinc nitrate hex ahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and 1 M NaOH were used. Both solutions were prepared using deionized water. A white suspension was formed by adding drops of NaOH solution to zinc nitrate solution at 60 °C with continuous stirring for 1 h. The white substance was centrifuged at 4000 rpm for 20 min. Then, it was cleaned three times with distilled water, and finally it was cleaned with absolute ethanol. The resulting product was subjected to calcination at a temperature of 400°C in the presence of air for 3 hrs [23], as shown in Fig.1. The weight was determined using an Eq. (1) [24]:

$$M = \frac{Wt}{M. wt} \times \frac{1000}{V} \quad (1)$$

M represents the molecular concentration, Wt represents the weight, V represents the volume of distilled water (DI), and M.wt represents the molecular weight of the precursor.

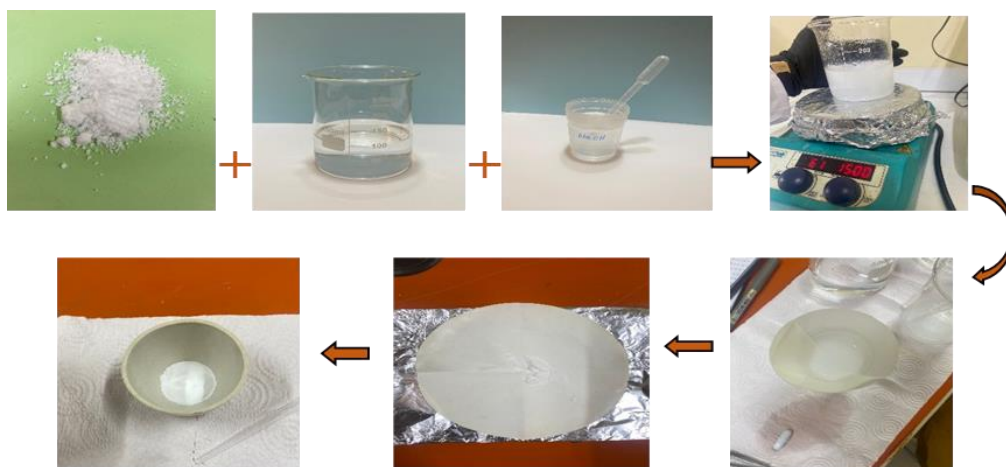


Figure 1: The preparation steps of pure ZnO NPs by chemical precipitation method.

2. 2. Preparation of ZnO NPs by Green Method

2. 2. 1. Preparation of Aqueous Leaf Extract

Ficus carica (fig) leaves were collected from the home garden, washed well with distilled water to clean them from dust, dried in the shade for two days at room temperature, and ground with an electric grinder. 5 g of leaf powder were combined with 400 mL of deionized water in a 500 mL beaker and heated to boiling for one hour. The solution was left to cool at ambient temperature, as shown in Fig.2. The solution was filtered using Whitman's filter paper to obtain the aqueous leaf extract, which was kept at 4°C for further use.

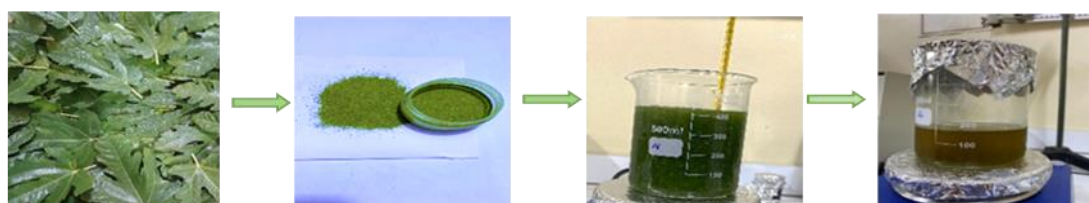


Figure 2: steps of the aqueous leaf extract preparation.

2. 2. 2. Preparation of ZnO nanoparticles using *Ficus carica* leaves extract

80 mL of *Ficus carica* leaf extract was heated to 60°C with stirring. After that, the extract was mixed with 5 g of zinc nitrate at a temperature of 80°C for 1 h with continuously stirring at speed of 400 revolutions per minute. The paste was made by heating the ingredients to 80°C for 1 hour. The paste was annealed at 400°C for 2 h. The substance was ground into a powder using a mortar and pestle after being heated. Next, the powder was left to dry naturally to get the pure ZnO NPs. Fig. 3 illustrates the preparation steps of pure ZnO NPs using *Ficus carica* (fig) leaves.



Figure 3: preparing pure ZnO NPs using *Ficus carica* (fig) leaves.

3. Results and Dissection

3. 1. XRD Measurement

To investigate the characteristics of the structure of pure ZnO NPs, X-ray diffraction analysis was performed within an arrangement of 2θ from 20° to 80° . Fig.4(a, b) shows the XRD patterns of the pure ZnO NPs prepared by the chemical and green methods. The presence of the hexagonal wurtzite phase is confirmed for all the observed peaks. The results are consistent with [ICDD card no. 36-1451] [25, 26]. The figure indicates the presence of sharp peaks in the XRD patterns of ZnO NPs prepared by both methods. This shows that the zinc oxide nanoparticles are highly crystalline. ZnO nanoparticles were grown along the planes, as shown in Table 1 and Table 2. The nanoscale structure, created by the binding nature of biomolecules in organic matter, has the most orientation peak diffraction line at (101) [27].

The crystalline size (D) was evaluated by the following Debye Scherrer's formula [28]:

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

The constant K has a fixed value of 0.94. The variables λ and β indicate wavelength and the full width at half maximum (FWHM), respectively, while θ represents the Bragg's diffraction angle [29]. It was found that the average crystalline size for pure ZnO NPs was 27.4433 nm. These results are in good agreement with that of Soto-Robles et al. [30]. The crystalline size values and structural parameters of ZnO NPs prepared by the chemical precipitation method are shown in Table 1. Table 2 illustrates the crystalline size values and structural parameters of ZnO NPs prepared by the green method.

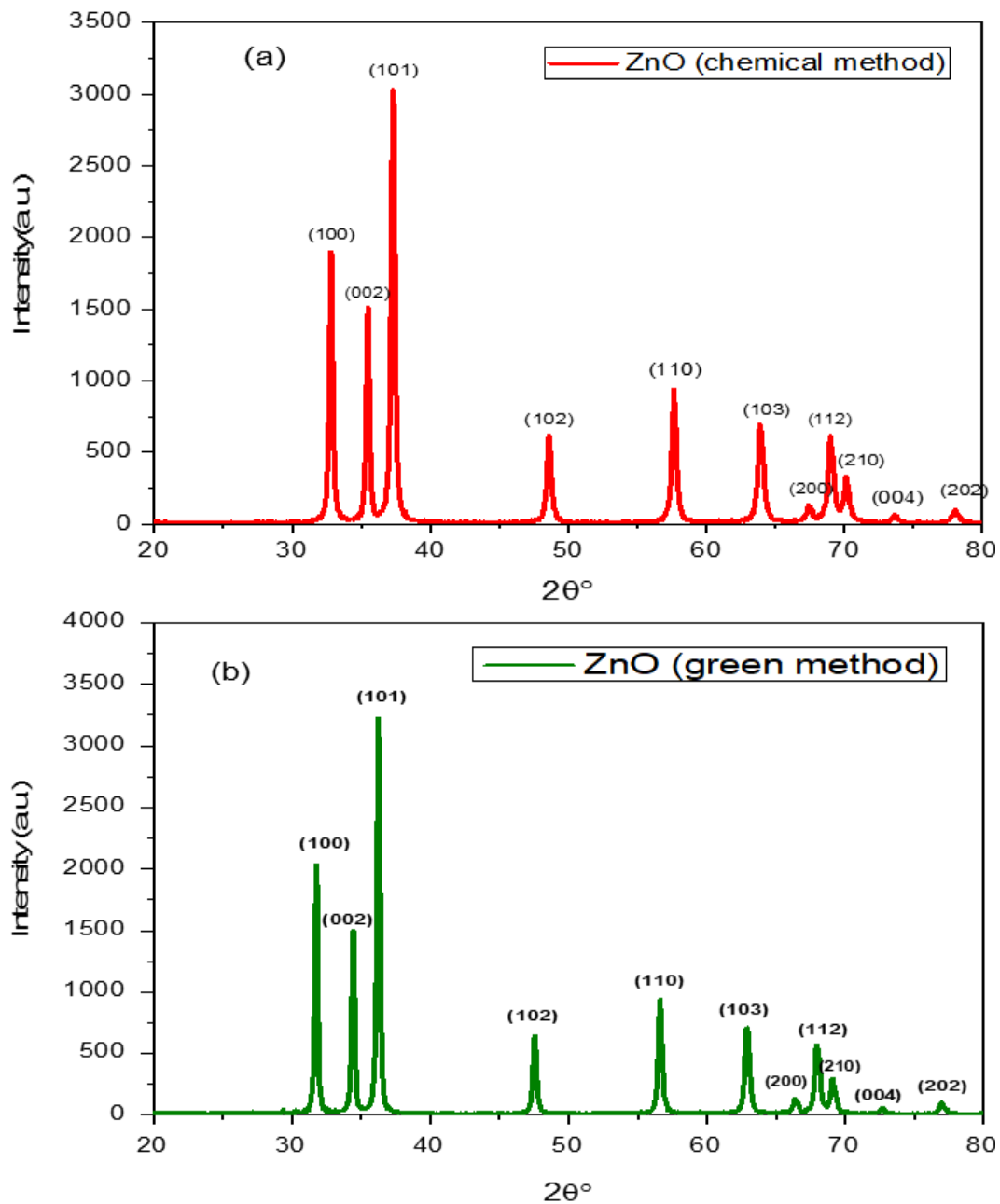


Figure 4: X-ray diffraction pattern of pure ZnO nanoparticle prepared by (a) chemical method (b) green method.

Table 1: Crystalline size and structural parameters of ZnO NPs prepared by chemical method

2 θ (Deg)	FWHM (rad.)	Crystalline size D(nm)	(hkl)
32.8425	0.2854	29.00805	(100)
35.5226	0.2598	32.09643	(002)
37.3305	0.3029	27.67267	(101)
48.6224	0.3387	25.72755	(102)
57.6501	0.3362	26.96103	(110)
63.9406	0.3951	23.69287	(103)
67.4258	0.301	31.7165	(200)
69.0244	0.4255	22.64937	(210)

Table 2: Crystalline size and structural parameters of ZnO NPs prepared by green method

2 θ (Deg)	FWHM (rad.)	Crystalline size D(nm)	(hkl)
34.4379	0.22570	36.83569	(002)
36.2585	0.24130	34.62914	(101)
47.5447	0.25900	33.50362	(102)
56.5864	0.25770	34.99657	(110)
62.8690	0.31990	29.0939	(103)
66.3499	0.28470	33.32507	(200)
67.9417	0.30020	31.89716	(112)
69.0651	0.29590	32.57743	(201)
76.9237	0.32860	30.86342	(202)

3. 2. Energy Dispersive X-Ray Spectroscopy (EDX)

Energy dispersive spectroscopy (EDX) is a method used to analyze the chemical composition of materials by studying the radiation resulting from the interaction of the sample with an electron beam or an X-ray beam. The EDX spectrum of ZnO nanoparticles prepared by the chemical method is shown in Fig.5 (a), revealing that the sample includes Zn, O and C. The prominent peaks correspond to Zn and O. This analysis showed good agreement with the XRD data. Carbon, aluminium, silicon, or other materials may appear in an EDX examination for several reasons, including chemical reactions. Chemical reactions may occur between the various components in the sample and the electron beam or X-ray used in the examination, leading to the appearance of signals of nickel, aluminum, or carbon. This means that it all comes down to the method of preparing the samples for examination. The EDX spectrum of ZnO nanoparticles prepared by the green method, as shown in Fig. 5(b), revealed that the sample includes just Zn and O. The prominent peak, which corresponds to Si, is due to sample preparation on a glass substrate. This analysis showed good agreement with the XRD data [31]. Tables 3 and 4 display the EDX patterns for ZnO nanoparticles.

3. 3. Field Emission Scanning Electron microscopy (FE-SEM)

A Field-Emission Scanning Electron Microscope (FE-SEM) was used to detect the surface morphology of zinc oxide nanoparticles prepared by the chemical precipitation and green methods, as seen in Fig. 6 (a, b). Image analysis revealed that the ZnO NPs prepared by both methods showed that the grains have a spherical shape with a uniform distribution. The ZnO NPs synthesized by the chemical method has a size range of 28.32-51.00 nm, as shown in Fig.6(a). In contrast, the ZnO NPs produced through the green method employing *Ficus carica* leaves extract has a size range of 50-78 nm, as illustrated in Fig.6(b). The particles exhibited excellent crystallization and were evenly dispersed with little agglomeration. Consequently, an observed effect was a growth in the size of the nanoparticles. The disparity in size seen between the XRD and SEM measurements

suggests that the ZnO nanoparticles have formed aggregates as a consequence of the annealing process [32].

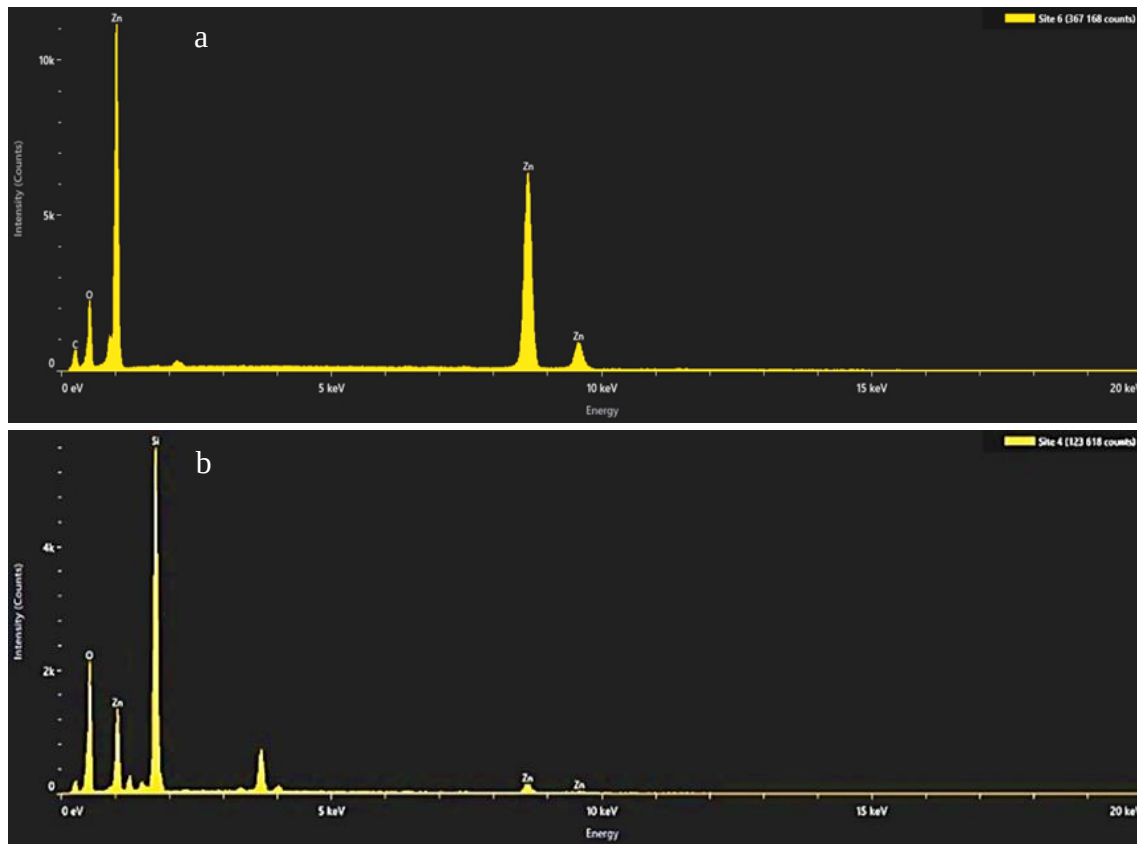


Figure 5: EDX patterns of ZnO NPs prepared by the (a) chemical method (b) green method.

Table 3: EDX patterns for ZnO NPs prepared by the chemical method.

Element	Weight %	Atomic % Error	Atomic %
Zn	78.7	0.5	52.5
O	21.3	0.3	47.5

Table 4: EDX patterns for ZnO NPs prepared by biological method.

Element	Weight %	Atomic % Error	Atomic %
Zn	18.3	0.3	5.2
O	81.7	0.8	94.8

3. 4. Atomic Force Microscopy (AFM)

The surface's structure and elevation may be determined by analysing the plot topographies with an atomic force microscope. This method allows for the analysis of digital images from various perspectives, including 3D modelling, and provides for the measurement of quantitative surface properties such as RMS roughness (Rq) and average roughness (Ra). The three-dimensional AFM images and the granularity distribution of ZnO NPS produced using chemical and green methods are shown in Fig.7 (a, b). All samples obtained exhibited a polycrystalline nature. The root mean square roughness (Rq) for ZnO NPs prepared by the chemical precipitation was 35.8 nm, and the average roughness (Ra) was 29.3 nm. The root mean square roughness (Rq) for ZnO NPs prepared by the green method was 51.43 nm, and the average roughness (Ra) was 66.72 nm. Surface roughness increases the surface topography and increases the ability to improve cell attachment, consequently increasing cell adhesion. Fig.7 illustrates the AFM images

for zinc oxide nanoparticles (ZnO NPs) prepared by the chemical precipitation and the green methods [33, 34].

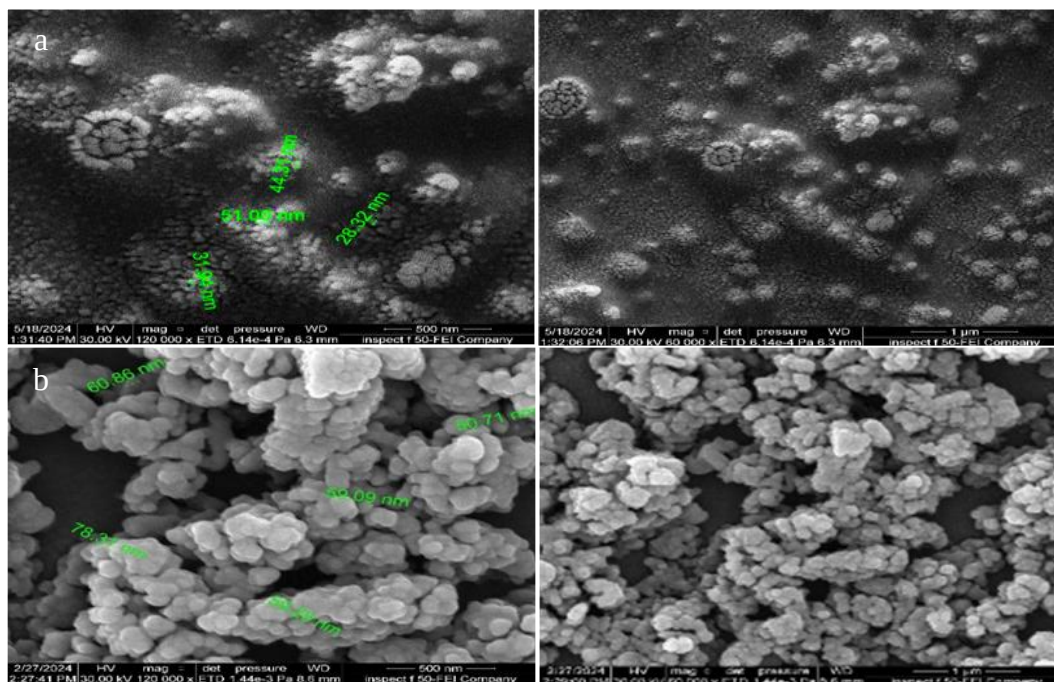


Figure 6: FE-SEM images of ZnO NPs prepared by (a) chemical method, (b) green method.

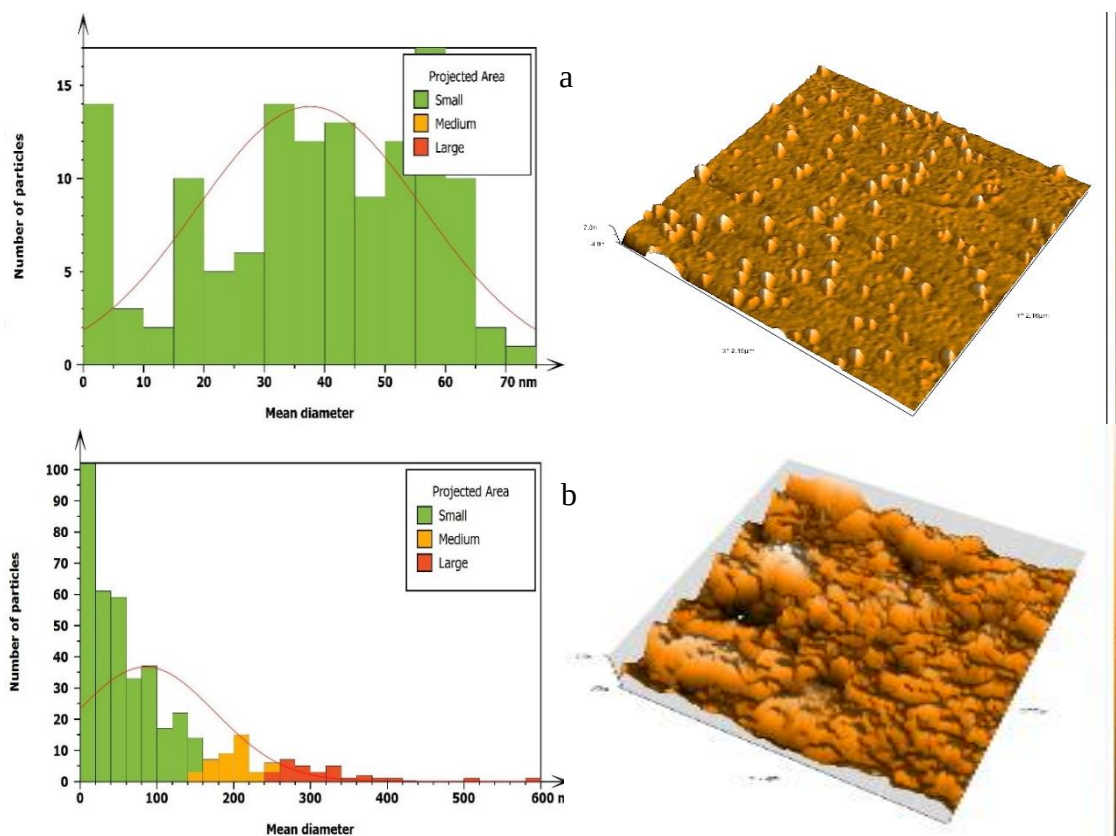


Figure 7: AFM images of ZnO NPs prepared by (a) chemical method, (b) green method.

3.5. UV–Vis Analysis

The optical characteristics of ZnO nanoparticles prepared by chemical precipitation and green methods were investigated using UV-Vis spectroscopy. The investigation of the nanoparticles included studying their absorbance spectra over the wavelength range of 290-800 nm, as seen in Fig. 8, which shows the ultraviolet-visible absorption spectra of ZnO at standard ambient temperature. The spectra showed a distinct peak in UV absorption at 291 nm for ZnO NPs prepared by the chemical precipitation and at 383 for ZnO NPs prepared by the green method. Additionally, the presence of a linear component indicates that the transition process in these powders occurred directly. The peak with the greatest intensity in the spectra correlates to the primary absorption edges in the sample and may be used to determine the band gap of the nanomaterial. The Optical energy band gap of ZnO nanoparticles was calculated using the Planck equation [35, 36]

$$E(\text{eV}) = \frac{1240}{\lambda(\text{nm})} \quad (3)$$

The energy gap value for the sample produced by the chemical technique was 4.26 eV. The observed behavior may be identified to the volume quantization effect in the sample [29]. The optical energy gap value of samples obtained using Ficus carica leaf extract through the green approach was 3.24 eV. This result is in good agreement with that of Haque et al. [37]. The reason for the decrease in the energy gap in the green method is possibly due to the increase in the aggregation of ZnO NPs. This occurs because of defects that accompany the preparation of ZnO NPs.

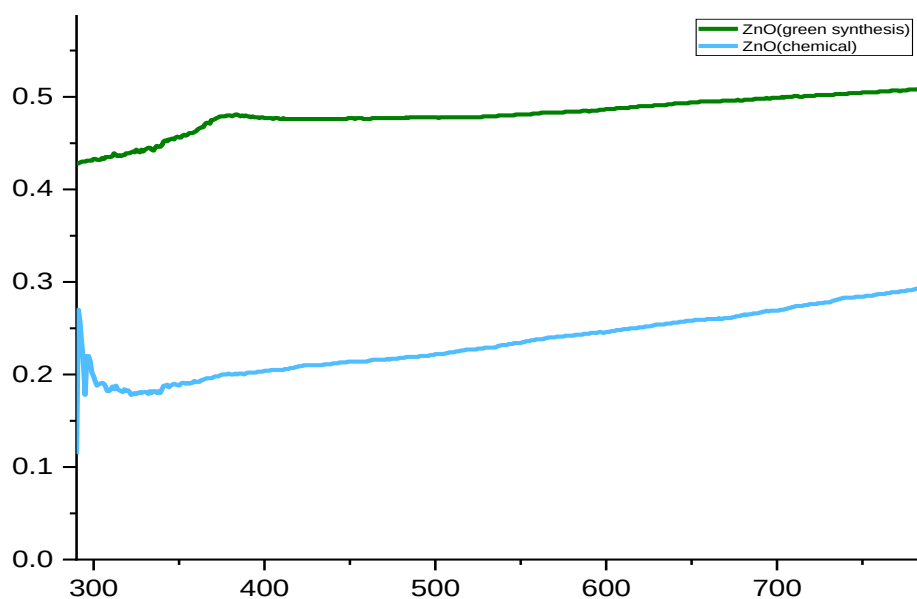


Figure 8: Absorbance spectra for ZnO NPs prepared by the green method and ZnO NPs prepared by chemical method.

4. Conclusions

This study is based on the synthesis of ZnO NPs using two different methods, chemical and green. XRD patterns confirmed that the ZnO nanoparticles formed were polycrystalline with a crystal size of 27.44 and 33nm for the chemical and green methods, respectively. The study of the FE-SEM images revealed that the nanoparticles had a spherical form and varied in size. Based on the collected findings, the average grain size was almost similar, with slight differences in the energy gap value. Still, there was a significant difference in the average roughness value. The average roughness (Ra) for

ZnO NPs prepared by chemical method was 29.3 nm, so it is possible to manufacture sensors and solar cells. The average roughness (Ra) for ZnO NPs prepared by the biological method was 66.72 nm so that it can be used in medical applications such as antibacterial and antifungal agents and treatment of cancer cells. In solar cells, a rough surface is needed to increase the absorbance and decrease the reflectivity; as it was observed that the transmittance absorbance spectrum was very high, meaning that ZnO NPs prepared by the green method can be used as a window for solar cells and not as a solar cell.

Acknowledgment

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Conflict of interest

Authors declare that they have no conflict of interest

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تأثير استخدام طرق تحضير مختلفة على خواص جزيئات أكسيد الزنك النانوية

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الخلاصة

تم تحضير جزيئات أكسيد الزنك النانوية (ZnO NPs) باستخدام طريقتين مختلفتين للتحضير: الترسيب الكيميائي والطريقة البيولوجية من خلال استخدام أوراق مستخلص نبات اللبخ كإريك. تم تحليل الجسيمات النانوية باستخدام حيود الأشعة السينية (XRD)، والمجهر الإلكتروني الماسح الانبعاث المجال (FE-SEM)، والتحليل الطيفي للأشعة السينية المشتتة من الطاقة (EDX). تم تطبيق التقنيات التحليلية مثل حيود الأشعة السينية (XRD) للتحقق من تخليق جسيمات أكسيد الزنك النانوية البلورية. أظهر نموذج XRD أشكالاً سداسية، وكان متوسط قطر حجم البلورة لكلا التقنيتين المستخدمتين (33، 27.44) نانومتر، على التوالي. الجسيمات النانوية لأكسيد الزنك لها شكل كروي، كما هو موضح بواسطة (FE-SEM). أشار اختبار (EDX) إلى وجود قيم تتوافق مع الزنك والأكسجين. يعد الفحص المجهرى للقوة الذرية (AFM) وطريقة تستخدم لتوفير صور رقمية عالية الدقة تسمح بإجراء تقييمات دقيقة لخصائص السطح، مثل جذر متوسط خشونة الترتيب (Rq) ومتوسط الخشونة (Ra). تم توصيف ZnO NPs المحضرة بواسطة الترسيب الكيميائي والطريقة البيولوجية باستخدام التحليل الطيفي للأشعة فوق البنفسجية (UV-Vis) لتحليل أنماط الامتصاص.

الكلمات المفتاحية: AFM، جسيمات أكسيد الزنك النانوية، طريقة الترسيب، SEM، مطيافية UV-VIS.