

Preparation and Characterization of ZnO NPs using the Capparis Spinosa Plant

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Abstract

This study examines two distinct methods for preparing zinc oxide nanoparticles (ZnO NPs) the chemical method and the green method. In the chemical method, zinc acetate and deionized water were used, whereas in the green method, the plant extract from Capparis spinosa fruit was used. The ZnO nanoparticles were analyzed using X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDS), field-emission scanning electron microscopy (FESEM), and Fourier transform infrared spectroscopy (FTIR). ZnO nanoparticles prepared by chemical precipitation and biological methods were characterized using UV-Vis spectroscopy to analyze the absorption spectra. The FESEM images revealed the spherical shape of the nanoparticles. The sizes of the nanoparticles were experimentally calculated using XRD and FESEM. The particle size measured by the XRD pattern was 26 nm for the chemically synthesized ZnO NPs sample and 6 nm for the ZnO NPs sample prepared by green synthesis. FTIR spectra showed that the stabilizing agent made the particle surface inert. The results indicate that the ZnO NPs sample synthesized using the green method is better than the chemically synthesized samples. This comparative study highlights the influence of the capping agent and the resulting morphologies on surface area and recombination kinetics, while also noting that it can be used in medical applications due to its environmentally friendly method.

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Keywords:

Capparis Spinosa Fruit, Zinc Oxide Nanoparticles, XRD, Green Synthesis, Chemical Method.

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1. Introduction

Nanoscience is a modern field of scientific research that studies the structures of materials whose dimensions fall within the nanometer range 1-100 nm [1]. Nanoscience has received widespread attention from scientists and researchers due to its properties, as its physical, chemical, and biological characteristics may differ from their bulk counterparts [2]. In addition to the small size of nanomaterials, they also have a large surface area compared to their volume [3]. Larger particles are tied to atomic or due to these unique properties of nanomaterials, they have been used in many fields, including medicine, drug delivery, wound treatment, and cosmetics [4]. There are many methods for preparing materials, including physical, chemical and biological methods [5, 6]. Nanomaterial preparation methods are generally classified into two basic approaches. The first is the top-down approach, where large materials are converted into nanoparticles using physical and mechanical techniques such as photoetching and pulsed laser erosion. The second approach is the bottom-up approach, where nanoparticles are built from small components, such as atoms and molecules, through chemical and biological reactions, such as chemical precipitation, thermal decomposition, and green methods [7, 8].

Manufacturing nanomaterials by physical or chemical methods requires high energy, safe laboratories, and the use of more hazardous materials. Therefore, alternative methods for preparing nanomaterials, namely biological methods, have been used. These methods are safer, less toxic, and environmentally friendly [9, 10]. Therefore, inexpensive agricultural waste plant extracts containing secondary metabolites can act as stabilizing, reducing, and capping agents during the biosynthesis of metal nanoparticles in a simple

and economical synthetic manner. These include stems, leaves, fruits, and roots, which contain reducing agents, such as amino acids, citric acid, aldehydes, and flavonoids [11].

zinc oxide (ZnO) is a well-known electrical and optical semiconductor has a wide direct band gap, an extensive range of 3.37 eV, different from other semiconducting materials, and a high energy of 60 meV at ambient temperature [12, 13]. ZnO is an inherently n-type semiconductor [14]. Since ZnO has a broadband gap of 3.37 eV and strong chemical stability, it can be used in transistors, light-emitting diodes, gas-sensing devices, and solar cell transistors [15]. ZnO NPs were chosen for this investigation because of their high stability. They last longer than organic-based antimicrobials and disinfectants [16, 17]. There are several methods for creating differentiated ZnO particles, both chemically and physically. Along the way, numerous techniques have been used to synthesize diverse zinc particles from a variety of sources, including bacteria, fungi, algae, and other plants, due to the increasing availability of diverse green materials [18]. As a result, a green approach for synthesizing ZnO nanoparticles is urgently needed. Biological methods, which use either plant extracts or microorganisms, have offered a dependable and environmentally acceptable substitute for chemical and physical methods [19]. The use of microorganisms to reduce metal ions in the treatment of toxic metals is promising. Green synthesis methods are more advantageous than traditional physical and chemical methods due to their simplicity, cost-effectiveness, and lack of toxic and environmentally harmful chemicals. Consequently, they have gained significant importance in recent years in the production of nanomaterials, including nanoscale zinc oxide [20]. Recently, an increasing interest has been noticed in scientists' desire to manufacture nanomaterial oxides, including ZnO, using plant extracts. This is due to ensuring their non-toxicity and their use in medical journals. Nanozinc oxide has been manufactured using reducing agents found in plant extracts [21-23].

In this research, ZnO NPs were synthesized using two different methods: the chemical method and the environmentally friendly green method using the extract of the Caparison fruit plant, which is a substance that has been used very little. It is considered a reducing agent for nanoparticles. The effect of organic matter on the properties of the nanomaterial was studied for the possibility of using it in various medical applications, such as using it as a layer to protect the skin from ultraviolet rays.

2. Experimental Work

2.1. Preparation of ZnO Nanoparticles by Chemical Method

To prepare ZnO NPs by chemical precipitation, 2.1 g of zinc acetate was taken as raw material and dissolved in 100 mL of deionized water at a concentration of 0.1 M. Then, sodium hydroxide was added drop by drop at a concentration of 1 M to reach a pH of 8.5. A white suspension was formed with continuous stirring using a magnetic stirrer (Model: VS-130Sh, made in Korea) at a temperature of 60°C. ZnO NPs were obtained after filtering the material using filter paper and washing the material twice with deionized water and once with alcohol, and drying it at a temperature of 50°C, then calcining it at a temperature of 400°C for two hours. Fig. 1 illustrates the preparation steps of pure ZnO NPs by the chemical method.

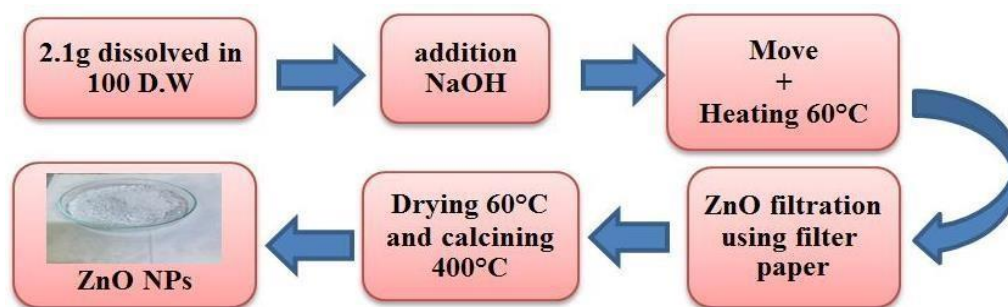


Figure 1: Preparation steps of pure ZnO NPs by the chemical method.

2.2. Preparation of ZnO NPs by Biological Method

2.2.1 Preparation of Capparis Spinosa Extract

Samples of the Capparis spinosa plant were collected from the northern region of Diyala Governorate, Iraq, during April and June 2024. The plant fruit extract was prepared with minor modifications to known protocols for preparing plant extracts. Fresh plant fruits were collected and washed thoroughly using tap water to eliminate surface impurities, and then they were rewashed with deionized water to ensure sample purity. The fruits were allowed to air dry at room temperature. The dried fruits were ground using a clean electric blender until they turned into a fine powder. Then, 10 g of the dried powder was weighed and mixed with 100 mL of deionized water. The mix was boiled for 3 hours at 90°C with continuous magnetic stirring (Model: VS130Sh, made in Korea) to ensure homogeneity. After boiling was completed, the solution was cooled to room temperature. It was then subjected to centrifugation (Model 1710 made in Germany) at 5000 rpm for 20 minutes to separate impurities from the extract. The extract was filtered using sterile filter paper (Whatman No. 1) and stored at 4°C for further analysis [24].

2.2.2 Preparation of ZnO NPs using Capparis Spinosa Extract

To prepare ZnO NPs using the green method, 30 mL of the Capparis plant fruit extract was first taken and added dropwise to 0.1 M zinc acetate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$) solution and 70 mL of deionized water, and placed on a magnetic stirrer for two hours at a temperature of 60°C. After the formation of a light brown precipitate, it was filtered using a centrifuge, a separator device, and the material was washed twice with deionized water and alcohol using a magnetic stirrer for half an hour. After that, the material was filtered using filter paper and dried at a temperature of 60°C, ground with a hand pestle, and calcined at a temperature of 400°C for two hours. Fig. 2 illustrates the preparation steps of pure ZnO NPs using Capparis spinosa fruit.

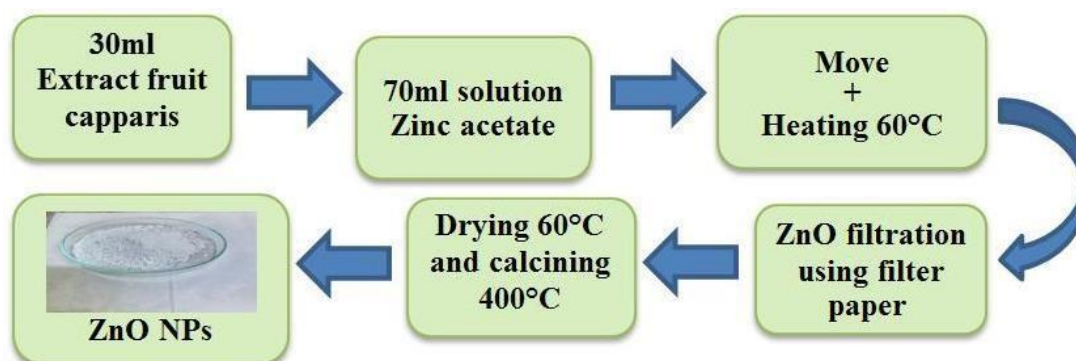


Figure 2: Preparation steps of pure ZnO NPs using Capparis spinosa fruit.

3. Results and Discussion

3.1. X-Ray Diffraction (XRD)

To study and characterize the crystal structure of ZnO NPs, XRD analysis was used. Fig. 3 shows the XRD pattern of ZnO NPs prepared by the chemical method. It was clearly identified where the peaks obtained were at 2θ values of 32.0657°, 34.719°, 36.5368°, 47.822°, 56.8622°, 63.1154° and 68.2175°. The results are consistent with the ICDD card (no. 01-075-1526). In Fig. 4, it is noticed that the ZnO NPs prepared by the green method have 2θ peaks of 31.800°, 34.4729°, 36.2787°, 47.5611°, 56.6308°, 62.9021°, and 67.9740°. The results are consistent with the ICDD card (no. 96-900-4182) [25, 26]. There are distinct peaks seen in the figures of ZnO NPs. This indicates that the ZnO NPs from both techniques are very crystalline [27]. As shown in Tables 1 and 2, the nanoscale structure created by the binding nature of biomolecules in organic matter has the most orientation peak diffraction line at (101) [28, 29]. Additionally, the crystalline size was assessed using Debye-Scherrer's equation, Eq. (1) [30].

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

Where D is the size of the crystal, the constant K has a fixed value of 0.94, λ and β are the wavelength and the Full Width Half Maximum (FWHM), respectively, and θ is the Bragg diffraction angle [31]. The average particle size of ZnO NPs synthesized by the chemical method was calculated to be in the range 27 to 21 nm, and that of the green method was in the range 17 to 14 nm. The reason for the smaller particle size of ZnO NPs prepared by the green method compared to the chemical method is due to the use of the plant extract of the fruit of the Capparis plant in the preparation, which in turn acts as a reducing agent and prevents clumping, thus reducing the ZnO particle size [32].

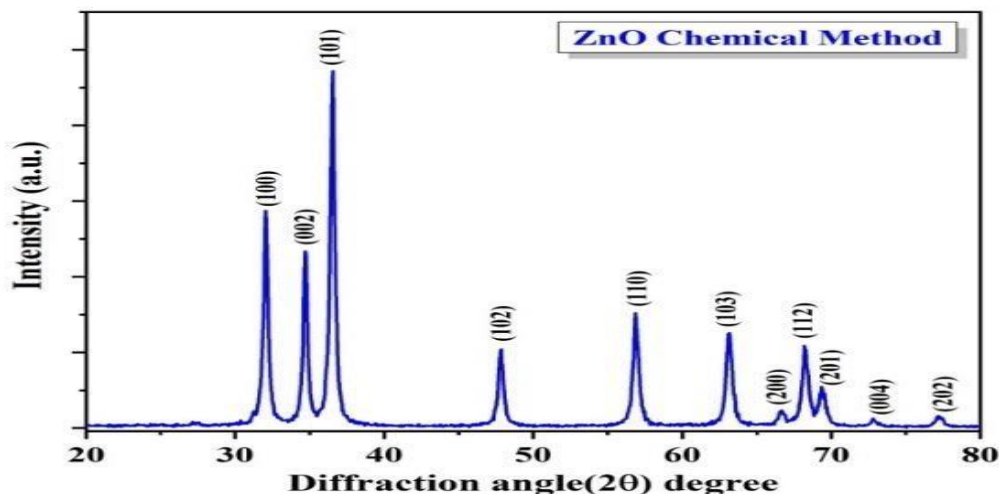


Figure 3: XRD diffraction pattern of pure ZnO nanoparticles prepared by the chemical method.

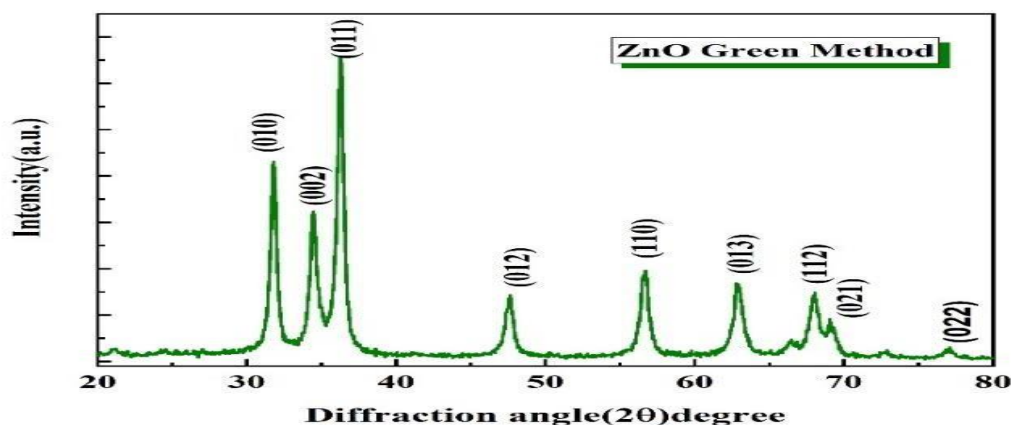


Figure 4: XRD diffraction pattern of pure ZnO nanoparticles prepared by the green method.

Table 1: Crystallin size and structural parameters of ZnO NPs prepared by the chemical method.

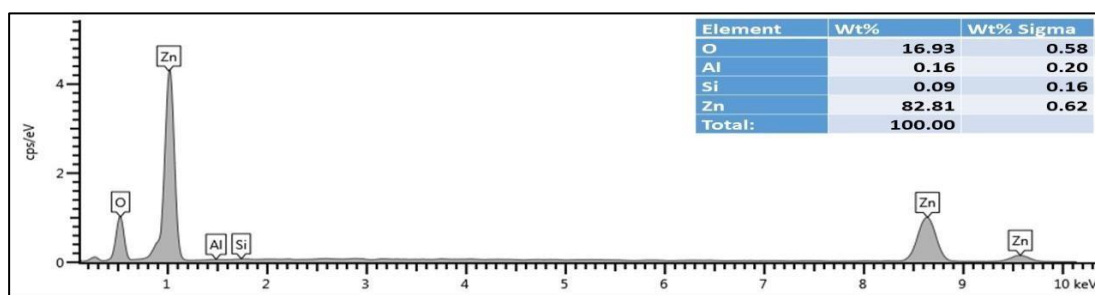
2θ (Degree)	FWHM	Size of the Crystal D(nm)	hkl
32.0657	0.3349	24.67178402	(100)
34.719	0.298	27.92002145	(002)
36.5368	0.3352	24.94835007	(101)
47.822	0.3527	24.62921177	(102)
56.8622	0.3389	26.64603751	(110)
63.1154	0.4269	21.83042737	(103)
68.2175	0.36170	26.51674939	(112)

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

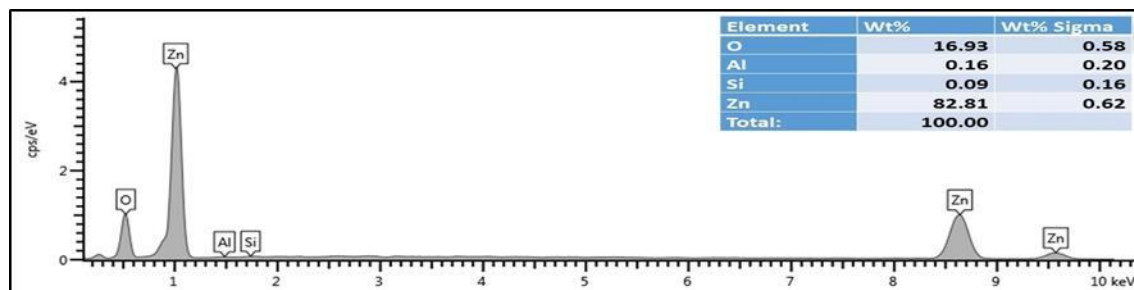
2 θ (Degree)	FWHM	Size of the Crystal D(nm)	hkl
31.8000	0.48090	17.17	(010)
34.4729	0.53780	15.46	(002)
36.2787	0.50670	16.49	(011)
47.5611	0.60790	14.27	(012)
56.6308	0.60750	14.84	(110)
62.9021	0.62160	14.97	(013)
67.9740	0.66060	14.49	(112)

3.2. Energy Dispersive X-Ray Spectroscopy (EDX)

EDS is a method used for the analysis of the chemical composition of materials by analyzing the radiation that results from the interaction of the sample with an electron beam or the radiation beam of an X-ray. The EDX spectrum of ZnO nanoparticles by the chemical method is shown in Fig. 5, showing that the sample consists of Zn and O. The large peaks correspond to Zn and O. The XRD measurements and this analysis agreed well. For various reasons, including chemical interactions, silicon, aluminum, or other materials may appear in an EDX examination. The different components in the sample and the electron beam or X-ray employed for the evaluation may undergo chemical reactions, causing silicon and aluminum signals to emerge. This implies that everything depends on how the samples are prepared for examining ZnO.

**Figure 5: EDX patterns of ZnO NPs prepared by the chemical method.**

The EDX spectrum of ZnO NPs prepared using a biological process, as shown in Fig. 6, indicates that Zn and O are present in the sample. Besides, the samples contained additional elements, such as potassium, silicon, phosphorus, and sulfur. The presence of these elements is due to the use of plant extracts. This result is consistent with the findings of another research [33, 34].

**Figure 6: EDX patterns of ZnO NPs prepared by the biological method.**

3.3. Field Emission Scanning Electron Microscopy (FE-SEM)

The surface morphology of ZnO NPs was detected with a FE-SEM, showing nanoparticles prepared by the chemical precipitation and biological methods, as shown in Fig. 7 A, B, C and D.

Image analysis indicates that the synthetic ZnO NPs prepared by both methods possess a spherical form granule with an even spread [35]. The ZnO NPs produced by the biological method employing Capparis spinosa fruit extract have a size ranging from 38 to 36 nm, and the particles exhibit good crystallization and are dispersed with little agglomeration, as illustrated in Figs. 7(A and B). The ZnO sample synthesized by the chemical technique has a range in size of 38-48 nm, as shown in Figs. 7(C and D). Consequently, the observed effect was a growth in the size of the nanoparticles. The difference in size seen between the XRD and SEM measurements is attributed to the difference in measuring techniques.

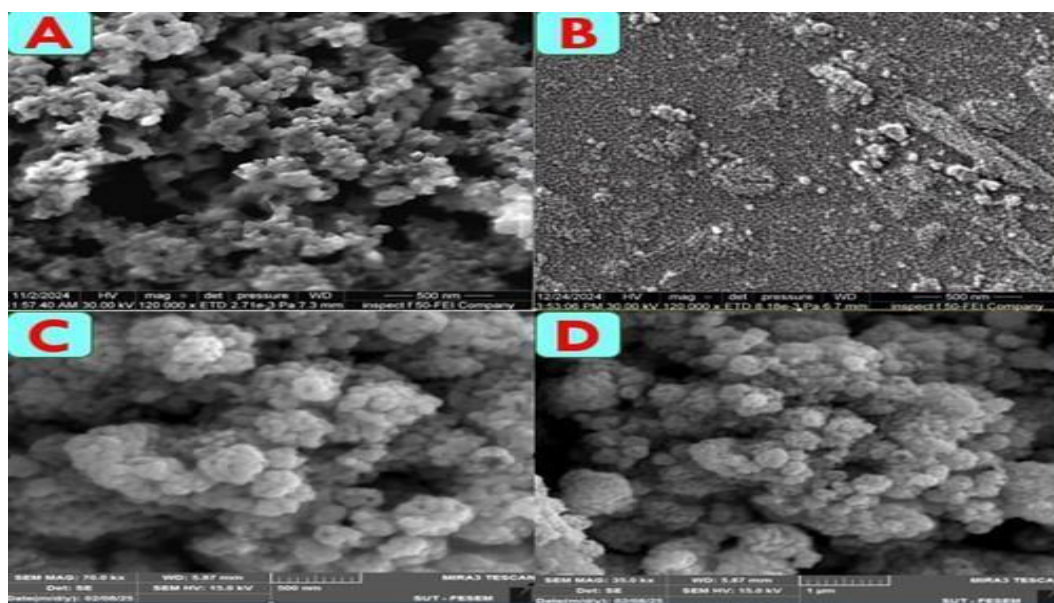


Figure 7: The FE-SEM images at two different magnifications for (A) and (B) ZnO NPs prepared by the green method and (C) and (D) ZnO NPs prepared by the chemical method.

3.4. UV-Vis. Analysis

The optical properties of the ZnO NPs prepared by chemical precipitation and biological methods were examined utilizing the UV-Vis. absorption spectrum. Fig. 8 displays the ultraviolet-visible absorption spectra of ZnO NPs at standard ambient temperature. The spectra revealed a distinct peak in UV absorption at 356 nm for ZnO NPs prepared by the chemical precipitation method and at 290 nm for those prepared by the biological 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 with the primary absorption edges in the sample and may be used to determine the energy band gap of the nanomaterial [36]. The optical energy band gap of ZnO nanoparticles was calculated using the Planck equation [29]:

$$E_g = \frac{hv}{\lambda_{\max}} \quad (2)$$

The energy band gap value for the sample prepared by the chemical method was 3.84 eV, and its value for that prepared by the green method was 4.2 eV. The reason for this is that the material prepared by the green method had a small particle size due to the effect of the plant extract, which is a reducing agent. Thus, due to the phenomenon of quantum confinement, a shift occurred in the energy band gap. The relationship is inverse; the smaller the particle size, the larger the energy band gap, and this is consistent with Shnawa et al. [37].

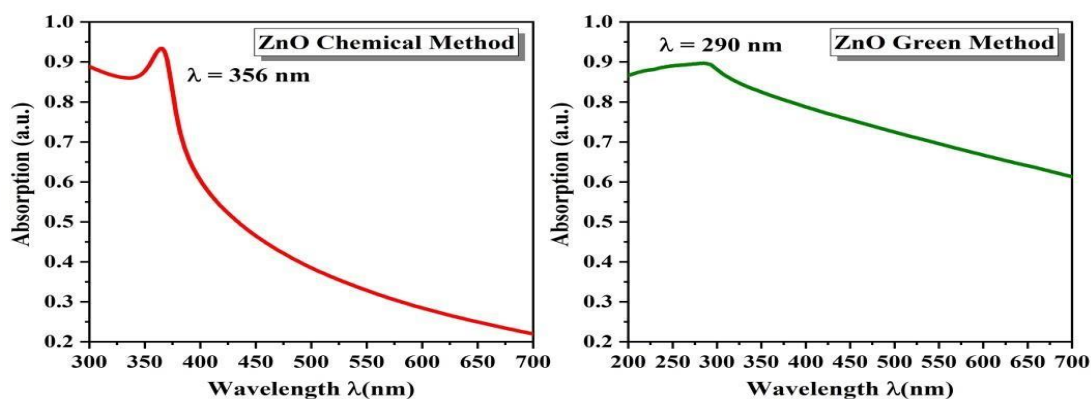


Figure 8: UV-Vis absorption spectra for ZnO NPs prepared by the chemical method and biological method.

3.5. Fourier transform infrared spectroscopy (FTIR) Measurements

ZnO was studied spectroscopically in several ratios between 4000 and 500 cm^{-1} . The assay was carried out to find possible biomolecules in the extract that were in charge of ZnO stabilization and reduction, as shown in Fig. 9. In addition, the figure shows the spectrum of zinc oxide prepared by the participant method, revealing a band at 3429 cm^{-1} that represents O-H, a primary site for metal ion adsorption, and a band at 2348 cm^{-1} that represents the O=C=O stretch. It was believed that the C-H stretching mode was responsible for the band observed at 2946 cm^{-1} , the O=C=O stretch was responsible for the band at 2348 cm^{-1} , the C=O stretching mode in the amine group, which is frequently found in proteins, was responsible for the band at 1624 cm^{-1} , and the C-O stretch was responsible for the band at 1119 cm^{-1} . Stretches of zinc oxide are represented by the peak between 400 and 500 cm^{-1} [38].

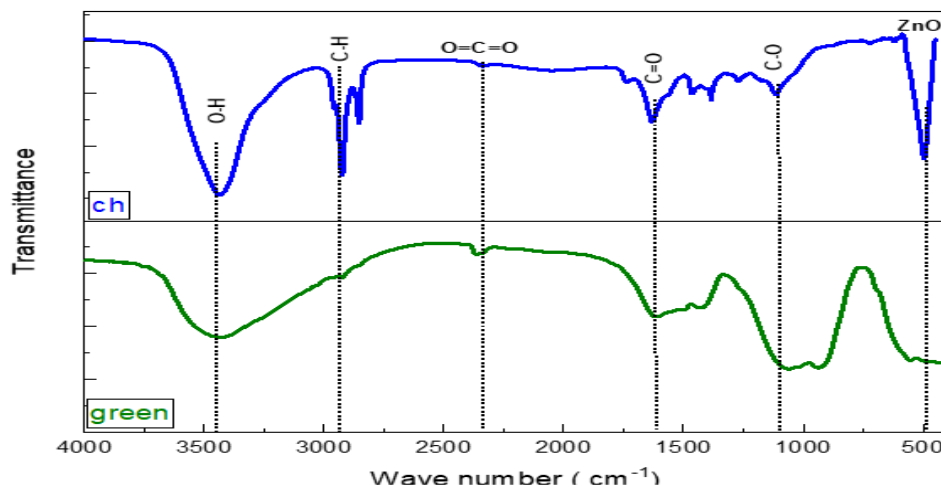


Figure 9: FTIR spectra of ZnO NPs prepared by the chemical method and the biological method

4. Conclusions

ZnO NPs were effectively created using a straightforward chemical reaction with an aqueous medium as the capping agent and through environmentally friendly synthesis using the extract of Capparis spinosa fruit. The XRD pattern showed the hexagonal phase development of the ZnO nanoparticles. For chemically made ZnO, the crystallite size measured in the XRD pattern was 26 nm, while the ZnO samples created through green synthesis were 6 nm. FESEM images revealed an intriguing aggregation of chemically synthesized nanoparticles with a spherical-like shape. Additionally, the FESEM image demonstrates that the particle sizes of chemically generated materials are larger. The stabilizing substance passivated the particle surface, according to the

FTIR spectra. Results revealed that the ZnO nanoparticles synthesized by the green method showed enhanced structural and optical properties compared to the chemically synthesized ZnO nanoparticles.

The results obtained showed that the material prepared using Capparis spinosa fruit extract is more stable, has smaller nano-sized particles and less toxicity, and as a result, it can be used in various medical applications, such as using it as a layer to protect the skin from ultraviolet rays.

Conflict of Interest

The authors declare that they have no conflict of interest.

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تحضير وتوصيف جسيمات أكسيد الزنك النانوية باستخدام نبات الكاباريس سبينوزا

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الخلاصة

تدرس هذه الدراسة طريقتين متميزتين لإعداد جسيمات أكسيد الزنك النانوية (ZnO NPs) الطريقة الكيميائية والطريقة الخضراء. في الطريقة الكيميائية، تم استخدام أسيتات الزنك والماء منزوع الأيونات، بينما في الطريقة الخضراء، تم استخدام المستخلص النباتي من ثمرة نبات *Capparis spinosa*. تم تحليل جسيمات أكسيد الزنك النانوية باستخدام حيود الأشعة السينية (XRD)، ومطيافية الأشعة السينية المشتتة للطاقة (EDS)، ومجهر مسح الإلكترون بالانبعاث الميداني (FESEM)، ومطيافية الأشعة تحت الحمراء بتحويل فورييه (FTIR). تم تمييز جسيمات أكسيد الزنك النانوية المحضرة بالترسيب الكيميائي والطرق البيولوجية باستخدام مطيافية الأشعة فوق البنفسجية المرئية لتحليل أطراف الامتصاص. كشفت صور FESEM عن الشكل الكروي للجسيمات النانوية. تم حساب أحجام الجسيمات النانوية تجريبياً باستخدام XRD و FESEM. بلغ حجم الجسيمات المقاس بنمط حيود الأشعة السينية 26 نانومتراً لعينة جسيمات أكسيد الزنك النانوية المصنعة كيميائياً، و 6 نانومتراً لعينة جسيمات أكسيد الزنك النانوية المحضرة بطريقة التخليق الأخضر. أظهرت أطراف الأشعة تحت الحمراء بتقنية فورييه أن عامل التثبيت جعل سطح الجسيم خاملاً. تشير النتائج إلى أن عينة جسيمات أكسيد الزنك النانوية المصنعة بطريقة التخليق الأخضر أفضل من العينات المصنعة كيميائياً. تُسلط هذه الدراسة المقارنة الضوء على تأثير عامل التغطية والشكل الناتج عنه على مساحة السطح وحركية إعادة التركيب، مع الإشارة أيضاً إلى إمكانية استخدامه في التطبيقات الطبية نظراً لطريقته الصديقة للبيئة.

الكلمات المفتاحية: فاكهة القبار، جسيمات أكسيد الزنك النانوية، حيود الأشعة السينية، التخليق الأخضر، الطريقة الكيميائية.