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Zinc Oxide (ZnO) Nanoparticles: An environmentally friendly synthesis and characterization using pomegranate peel extract

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ABSTRACT

In this work, pomegranate peel extract was used to create zinc oxide nanoparticles (ZnO NPs) in an eco-friendly way. Pomegranate peel extract and aqueous zinc nitrate salt ($Zn(NO_3)2.9H_2O$), a zinc source, were reacted in the presence of 2 M of ammonia hydroxide (NH_4OH) to bring the pH of the solution to eight, and this formed the ZnO nanoparticles. To characterize the nanoparticles, FTIR, XRD, EDX, and SEM were used. In the FTIR spectra, zinc oxide was found as a band at 436 cm^{-1}. The XRD patterns verified that ZnO has a hexagonal phase structure, with an average crystal size of 26.7 nm. The EDX examination demonstrated the exceptional purity of the produced chemical because no impurities were discovered Each element in the sample can be precisely identified using EDX, and the results show zinc and oxygen with weight percentages of 82.80% and 17.20%, respectively. The theoretical percentage value 80.339% and 19.66% zinc and oxygen with respectively, this gives a high compatibility in the weight ratio between the theoretical and real calculations of the prepared compound. SEM investigation shows that the generated particles are spherical and have diameters between 24 and 44 nm.

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1. INTRODUCTION

Plant-based synthesis of nanoparticles (NPs) is a cutting-edge commercial technique that offers numerous financial advantages for industry as well as significant environmental benefits, such as a reduction in chemical pollution [1] [2]. The topic of green synthesis of metals and metal oxides is one of great interest in nanoscience, with plants showing to be the most promising source for large-scale production [3]. In comparison to other biologic al pr esses, the synthesis of nanoparticles using plant extracts is not only more affordable and ecologically benign, but also simpler to scale up, resulting in nano particles with more stability and adaptable forms and sizes. [3] [4]. Zinc oxide (ZnO) nanostructures, one of the most widely used metal oxide nanoparticles (NPs), are driving research efforts because of their unique characteristics and numerous uses. ZnO-NPs can be produced chemically (by deposition, thermal solvent, sol-gel, etc.), physically (by laser ablation, condensation, and evaporation), or biologically using contemporary technologies. Chemical methods are often less preferred for nanoparticle production due to environmental and health risks associated with organic solvents, chemical reactions and process side substances such as vapors, which can leave harmful residues in the environment. Likewise, physical methods is facing obstacles such as high costs and the necessity of using extreme circumstances especially or as an example elevated temperatures and pressure [5], [6]. As a result, the research into green method biosynthesis has increased significantly as a more straightforward, economical, ecologically benign, and antibacterial substitute for conventional chemical and physical activity [3], [7]. Depending on the process, biosynthesis can create pure products and be done in big quantities without requiring expensive equipment or specialized knowledge [8]. Biological materials (such as proteins, enzymes, and extracts) used to decrease and stabilize nanomaterials are playing a crucial role in determining the final properties of the generated nanomaterials, such as their size, shape, morphology, and structure, all of which affect their bioactivity.

The species of the plant, the extraction technique, the solvent used, and the extract concentration all have a significant influence on the final nanomaterials' properties when using plants as a natural product to create them. Notably, the precursor concentration has a significant impact on the morphology of the ZnO-NPs that are produced. Additionally, ZnO-NPs' shape and concentration affect [9].

ZnO nanoparticles made with plant extracts have better optical and biological characteristics than ZnO nanoparticles made with traditional methods. [3][10][11][12]. In addition, metal oxide nanoparticle biosynthesis is particularly advantageous in light of growing concerns about pollution and the growing focus on environmental sustainability [9], [13]. Pomegranates, one of the popular fruits widely distributed throughout the year and known for their sweetness and hard peel, produce a large amount of peel waste, as nearly half of their weight is juice [14], [15]. The peel is rich in biologically active compounds, which contribute to its superior antibacterial properties compared to the leaves and flowers of the plant. Therefore, preparing an extract from pomegranate peels can act as biological agents through which nano oxides, including zinc oxide, can be prepared. Also, very small quantities of pomegranate peels It can give large amounts of extract if treated appropriately [14]-[16]. Verbič et al. provided an environmentally friendly technique for producing zinc oxide nanoparticles (ZnO-NPs) on cotton fabric using wood ash extract as an alkali source and pomegranate peel extract as a reducing agent. They evaluated four different synthesis techniques to find the optimal method for enhancing UV protection. Analytical methods confirmed the successful incorporation of ZnO-NPs on the cotton. The procedure that produced uniformly distributed tiny ZnO crystals with a UV protection factor of 154.0 entailed continuous drying in between immersions, and this approach yielded the best results. This study demonstrates how bio-waste may be used to make ZnO-NPs on cotton at low temperatures and short processing times, resulting in fabrics with superior UV protection. Here's an illustration of a green circular economy approach [17].

This work shows that ZnO-NPs may be produced on cotton using bio-waste at low temperatures and quick processing periods, producing fabrics with better UV protection. This is an example of a circular economy method that is green. Showing significant antifungal, antibacterial, and anticancer properties at low, safe concentrations, they were highly effective against Escherichia coli, Pseudomonas aeruginosa, Bacillus subtilis, Staphylococcus aureus, and Enterococcus faecalis, as well as Candida albicans, Cryptococcus neoformans, Aspergillus fumigatus, and Aspergillus Brasiliense's. With IC_{50} values of 104.9 and 52.4 µg/ml, respectively, the nanoparticles demonstrated anticancer effects on MCF7 and $CaCO_2$ cell lines, while preserving safety for normal cells with an IC_{50} of 155.1 µg/ml. This study highlights the intriguing potential of Ag-ZnO NPs produced using PPE for a range of biomedical uses. [18]. Abu-Dalo et al. synthesized titanium dioxide nanoparticles (TiO_2 NPs) and a nanocomposite (PPP-TiO_2) using pristine pomegranate peel extract (PPP). SEM, DLS, XRD, and ζ-potential were used to characterize the produced nanocomposite. The PPP-TiO2 nanocomposite showed a bimodal distribution with a Z-average size of 1230 nm and better stability (-11.4 mV compared to -6.96 mV for TiO_2 NPs), whereas the DLS analysis showed that TiO2 NPs had a Z-average size of 620 nm with high monodispersed. Biological activity was evaluated through well diffusion, MIC, MBC, and live/dead cell assays, showing that PPP-TiO_2 had 1.5 times the antimicrobial efficacy of PPP and TiO2 NPs, particularly against S. aureus. BOD_5 tests demonstrated that PPP-TiO_2 more effectively reduced microbial communities and organic matter in water samples compared to TiO_2 NPs [19].

Alamdari et al. conducted a study in which they synthesized zinc oxide nanoparticles (ZnO NPs) using leaf extract from *Sambucus ebulus*. The X-ray diffraction (XRD) analysis revealed that these ZnO NPs possess a highly crystalline structure, specifically a wurtzite crystal form, with an average crystallite size of approximately 17 nm. These green-synthesized nanoparticles exhibited strong ultraviolet (UV) absorption properties, along with a prominent yellow-orange emission at room temperature. Additionally, the ZnO NPs demonstrated effective antibacterial activity against various microorganisms and showed a reasonable capability for photocatalytic degradation of methylene blue dye pollutants. These findings suggest that the biosynthesized ZnO NPs hold significant potential for future applications in various fields, including environmental remediation and optoelectronics [20]. Abel, Saka, et al. present an investigation into the biosynthesis of zinc oxide nanoparticles (ZnO NPs) using moringa leaf extract, a method recognized for its cost-effectiveness and eco-friendliness. The paper uniquely emphasizes the selection of bacteria for green synthesis, a topic often overlooked by other researchers. The reduction and stabilization of Zn ions in the ZnO NPs were confirmed using UV-visible spectroscopy, revealing a notable wide bandgap with absorption at 350 nm. Moringa leaf extract proved effective in controlling particle size, ensuring uniformity. The antibacterial efficacy of the synthesized nanoparticles was significant against pathogenic bacteria such as Escherichia coli and Staphylococcus aureus. XRD analysis confirmed the high purity of the ZnO NPs, with no detectable impurities [21].

The purpose of this work is to create zinc oxide nanoparticles (ZnO NPs) by employing a meticulously regulated pomegranate peel extract. Advanced techniques such as (FTIR), (XRD), (EDX), and (SEM) will be used to characterize the produced ZnO NPs.

2. METHOD

2.1. Chemicals:

The experiment included chemicals and solvents from different suppliers, including Sigma-Aldrich, Riedel-de Haën, and SDH. The materials in this study were utilized exactly as received, without undergoing any refining or modification processes. Peels from pomegranates were gathered. The experimental research done in this study, according to a farmer northwest of Diyala Governorate, conforms with pertinent institutional, national, and international norms and legislation for research on plant materials.

2.2. Devices

FTIR, XRD, EDX, and SEM were the techniques used to characterize the iron oxide α-Fe_2O_3 nanoparticles that were synthesized. The FTIR test was conducted using the Perkin Elmer FTIR-65 infrared spectroscopy equipment. To examine the sample composition, an X-ray detector (Philips/PW1730, Cu, Kα = 1.5406 Å) was used. TESCAN, MAIA3, a scanning electron microscope, was used to examine the material's surface properties

.2.3. Procedures:

2.3.1. Preparation of pomegranate peel extract

The plant used in this study is the pomegranate plant, which was obtained from a farm in Diyala Governorate. Pomegranate peels were collected, rinsed with distilled water, and dried in the shade at room temperature and good ventilation conditions from 1-15 /11 / 2023. It was partially ground with an electric grinder to obtain small parts and then stored in a paper bag away from moisture until use. The extraction method was done from a weight of 25 g of pomegranate peels using a sensitive electronic balance and placing it in a conical flask containing 300 ml of deionized water and heating it to a temperature of 60 to 70 ºC for 90 min on a hot plate with moderate stirring to extract the biologically active components. The solid waste is then removed by filtering with cotton several times. Then separation by centrifugation. This process is repeated three times to obtain a filtrate containing the extracted materials without impurities Figure 1.

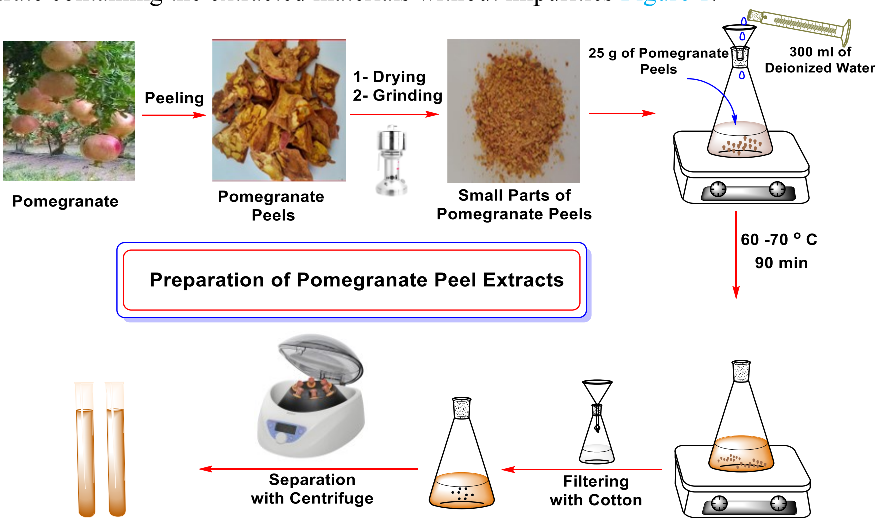

Figure1. Schematic diagram for the preparation of pomegranate peel extracts.

2.3.2. **Preparation of Zinc oxide**

Zinc oxide nanoparticles (ZnO NPs) were synthesized starting with 10 g of zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$), accurately measured on a sensitive balance and dissolved in 200 ml of deionized water in a beaker. This solution was heated on a hot plate at 60 °C with moderate stirring for 1 hr. Subsequently, 100 ml of pomegranate peel extract was gradually added dropwise to a burette. After the addition was completed, the mixture was continuously stirred at the same temperature for another 1 hr. The color was observed to change from yellow to greenish-black indicated the successful synthesis of ZnO NPs. Upon completion of the reaction, the solution was allowed to cool to room temperature for 20 min. The mixture was then filtered, and the resulting precipitate was collected and dried at 150 °C in an oven. The dried product was placed in a ceramic crucible and calcined in a furnace at 500 °C for 4 hr. This process yielded a pure white powder of ZnO nanoparticles, confirming the successful synthesis (as shown in Figure 2.

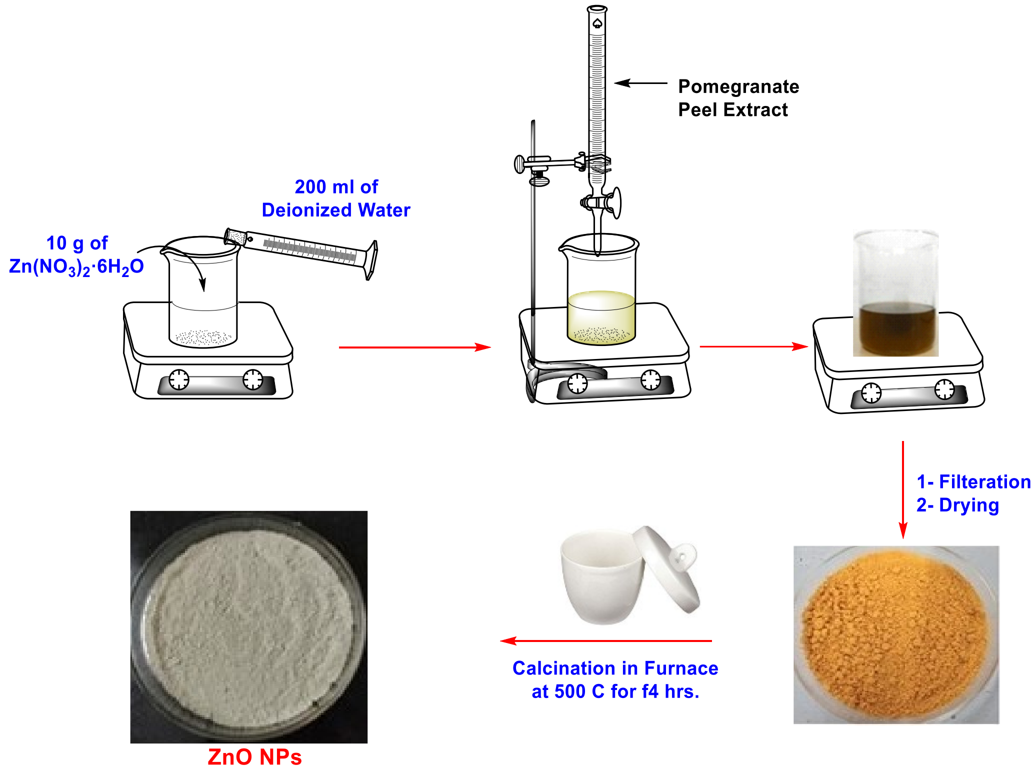

Figure 2. Synthesis of ZnO NPs.

3. RESULTS AND DISCUSSIONS

3.1. Mechanism of Plant-Mediated Synthesis

Figure 3 illustrates the green synthesis technique employed to produce ZnO nanoparticles. Phytochemicals in the plant extract act as reducing agents, transforming metal precursors into metal nanoparticles. These phytochemicals, being non-toxic and rich in antioxidants, serve dual roles as both stabilizing and reducing agents [22].

Key phytochemicals involved in the reduction process include phenolic compounds, aldehydes, alkaloids, flavonoids, and terpenoids. The concentrations of these phytochemical reducing agents vary among different plant extracts, significantly influencing nanoparticle synthesis. Factors such as pH, temperature, contact time, concentration of metal salts, and the phytochemical profile of the plant extract play crucial roles in determining the synthesis, stability, and yield of the nanoparticles [23].

Makarov et al. proposed a three-step mechanism for stabilizing metal ions with an organic coating post-reduction by plant extracts:

1. **Activation Phase**: This involves the reduction of metal ions and their nucleation.
2. **Growth Phase**: This phase enhances the stability of the nanoparticles.
3. **Termination Phase**: This phase determines the final shape of the nanoparticles[24].

Phytochemical activity facilitates the formation of metal oxides from metals such as gold, iron, copper, zinc, titanium, and nickel. Metal ions transition through the growth and stabilization phases due to the action of phytochemicals. Ultimately, the generation of oxygen causes the metal ions to bond, forming nanoparticles with distinct shapes[25].

$$E_v - E = \frac{h}{2.m}\,(k_x^2 + k_y^2) \qquad (1)$$

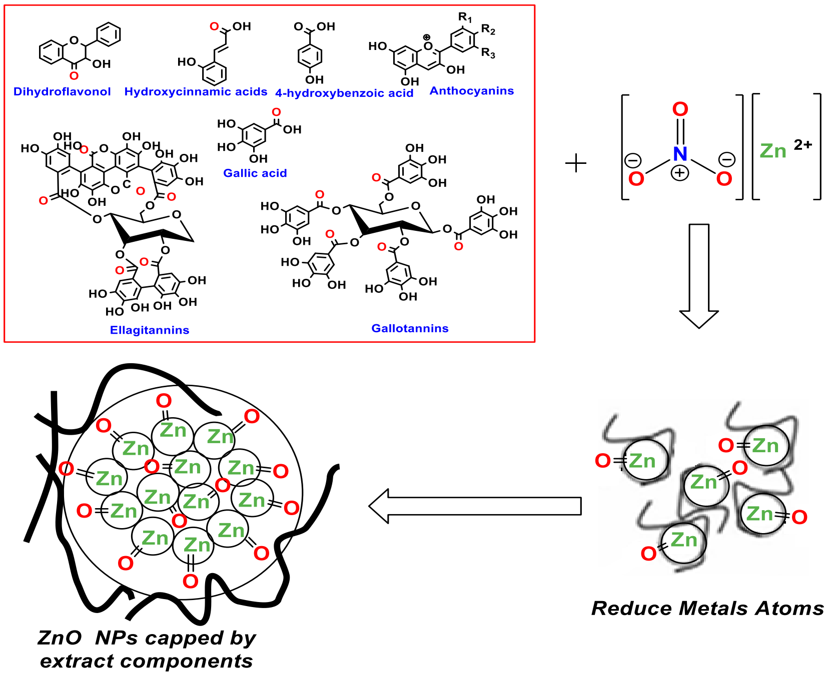

Figure 3. The green synthesis that produces ZnO NPs.

3.2. Fourier-Transform Infrared Spectroscopy (FTIR):
3.2.1. FTIR of Pure Pomegranate peel:

Figure 4 represents the FTIR spectrum of the pure Pomegranate peel before extraction method. The band at 3424 cm^{-1}- indicating stretching vibration of the (OH) groups of alcoholic and carboxylic acids in the pomegranate peel. The weak band centered 2900 cm^{-1} attributed to the stretching vibration of (CH) aliphatic groups. As the bands centered at 1732 and 1619 cm^{-1} corresponded to vibrations of the (C=O) and (C=C) groups, respectively. The peak at 1443 cm^{-1} is due to the C-O groups. Also, the bands at 1352, 1229, 1054 and 618 cm^{-1} corresponded to the vibrations of the (CH_2 bending), (C-N), (C-O-C) and (C-H aromatic) groups, respectively [26], [27], [28].

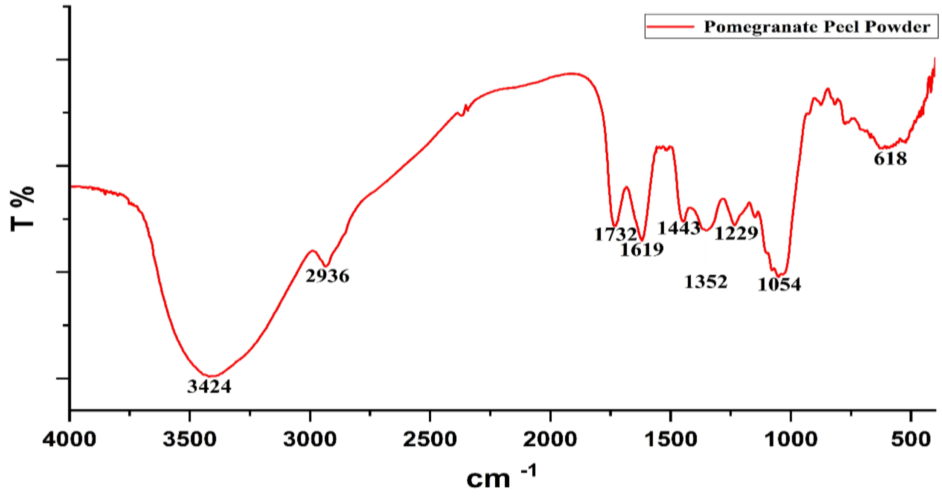

Figure 4. FTIR spectra of Pomegranate peel plant.

3.2.2. FTIR of Zinc Oxide Nanoparticles before Calcination:

Figure 5 represents the FTIR spectrum of ZnO NPs before the calcination using potassium bromide disks within the range 400 - 4000 cm^{-1}. The figure shows two bands centered at 3435 and 1583 cm^{-1}, which belong to the stretching and bending vibrations, respectively, of the O-H groups resulting from the presence of moisture between zinc oxide particles. As for the bands centered at 1364, 1075, and 645 cm^{-1}, they belong to the remains of pomegranate peels on the surfaces of zinc oxide particles. Finally, the band centered at 463 cm^{-1} belongs to the Zn-O group in zinc oxide (ZnO).

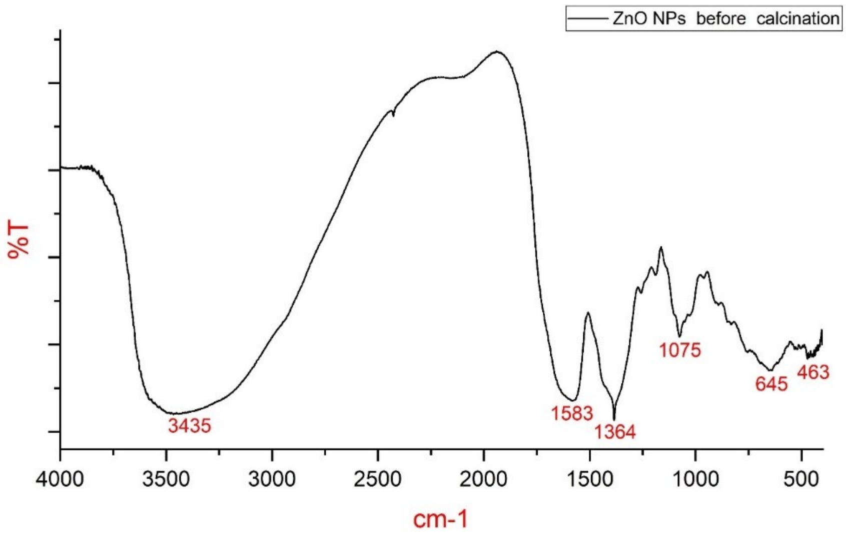

Figure 5. FTIR spectra of ZnO before calcination

3.2.3. FTIR of Zinc Oxide Nanoparticles after Calcination:

Figure 6 represents the FTIR spectrum of ZnO NPs after the calcination process within the range 400 – 4000 cm^{-1}. The figure shows two bands centered at 3455 and 1624 cm^{-1}, which belong to the stretching and bending vibrations, respectively, of the O-H group resulting from the presence of moisture between zinc oxide particles as well as the absorbed OH groups of the plant on the surface of ZnO NPs. The band centered at 436 cm^{-1} belong to the Zn-O group in ZnO, This gives good agreement with a number of previously published studies [29],[30], [31].

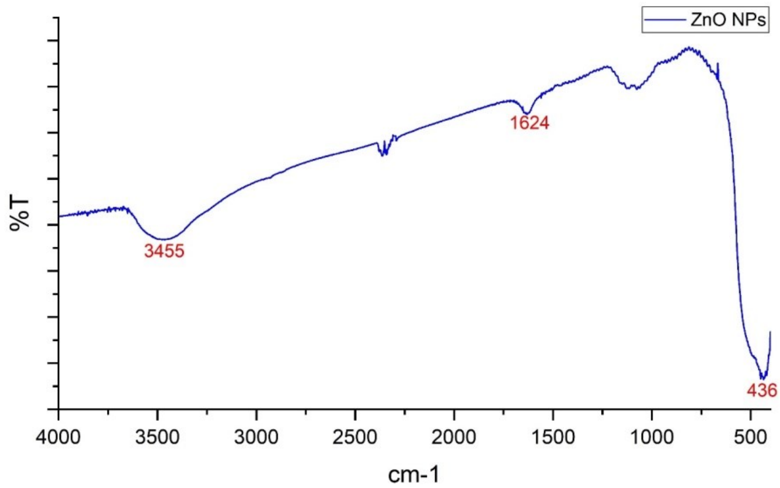

Figure 6. FTIR spectra of ZnO NPs after calcination

3.3. X-ray Diffraction (XRD) of Zinc Oxide Nanoparticles:

The XRD analysis of ZnO NPs, illustrated in Figure 7, reveals several sharp and well-defined peaks, indicating high purity and crystallinity. These peaks are observed in the 2θ region at 31.7189°, 34.3224°, 36.2830°, 47.6227°, 54.1320°, 56.5642°, 59.9838°, 62.9247°, 66.4878°, 68.0870°, 69.1588°, 72.7933°, and 77.1045°, corresponding to the crystallographic planes (100), (002), (101), (102), (110), (103), (200), (112), and (201), respectively. These findings are consistent with the literature (JCPDS Card number 36–1451) and previous studies, confirming the successful synthesis of pure ZnO nanoparticles. The absence of any extraneous peaks within the examined spectrum further underscores the high purity of the synthesized ZnO. The average size of crystals was estimated using the Debye-Scherer equation [32], [33], [34]

$$D = K\lambda / \beta . cos\theta \qquad (2)$$

Where: D is the crystal size (nm), the full width at half maximum (FWHM) of the diffraction peak, the wavelength of the X-ray (1.5406) and θ is the Bragg angle. The average crystal size was found to be in the range of 13.09-47.91 nm. Table 1 show the peak details of the crystal levels of the prepared zinc oxide nanoparticles

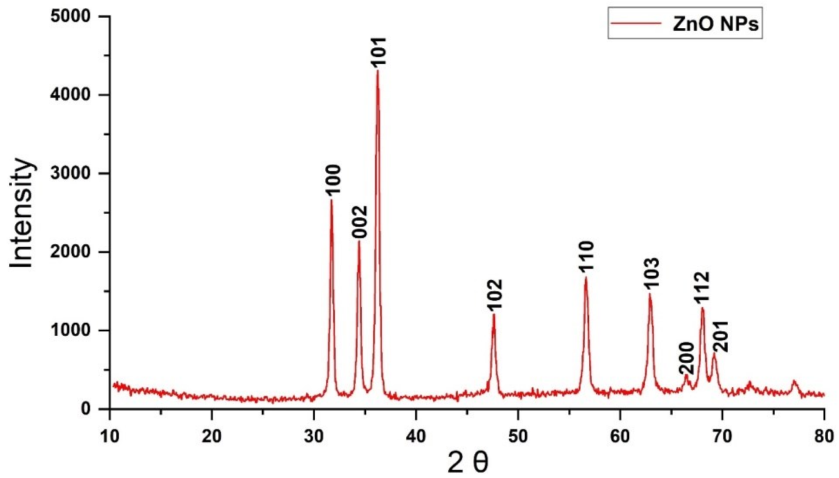

Figure 7. XRD of ZnO NPs

Table 1. XRD Data of the green synthesized ZnO NPs

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [?]	D nm
31.7283	2564.17	0.3444	2.82025	25.06
34.3551	1671.89	0.3444	2.61039	25.23
36.3030	3933.37	0.3444	2.47468	25.36
47.6331	1093.80	0.3444	1.90916	26.35
56.5946	1463.08	0.1968	1.62629	47.91
62.9572	1253.44	0.2952	1.47638	32.97
66.4291	295.32	0.3936	1.40740	25.21
68.0988	1136.72	0.3444	1.37690	29.09
69.2115	558.13	0.3936	1.35746	25.63
72.6827	188.09	0.7872	1.30095	13.09
77.0452	226.35	0.5904	1.23781	17.97

3.4. EDX of the ZnO NPs

The EDX analysis strongly indicates the presence of zinc in its oxide form, with high signals for both zinc and oxygen. Each element in the sample can be precisely identified using EDX, and the results show zinc and oxygen with weight percentages of 82.80% and 17.20%, respectively. The theoretical percentage value 80.339% and 19.66% zinc and oxygen with respectively, this gives a high compatibility in the weight ratio between the theoretical and real calculations of the prepared compound. Two notable zinc peaks at 1 keV and 8.6 keV, as well as a unique oxygen peak at 0.5 keV, are visible in the EDX spectrum. The distinctive peaks of zinc and oxygen validate the elemental makeup of the ZnO nanoparticles that were produced. Furthermore, figure 8 provides additional evidence of the purity and composition of the sample by showing the presence of trace elements, such as gold, alongside zinc and oxygen [35], [36], [37].

3.5. Scanning Electron Microscope (SEM):

In this work, the morphology of the nanoparticles was analyzed using scanning electron microscopy (SEM) to determine the microstructural differences in the samples. The round or irregularly shaped particles detected in the SEM images were zinc oxide nanoparticles. The diameters of the generated ZnO nanoparticles were found to range from 22 to 53 nm, as shown in Figure 9.

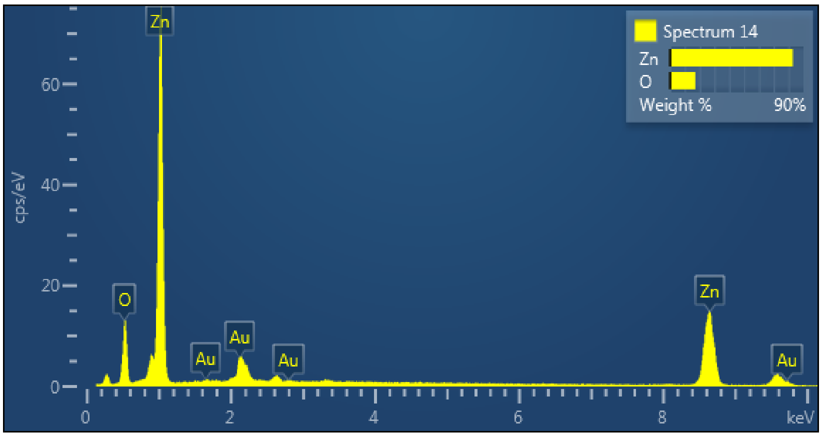

Figure 8. EDX of ZnO NPs

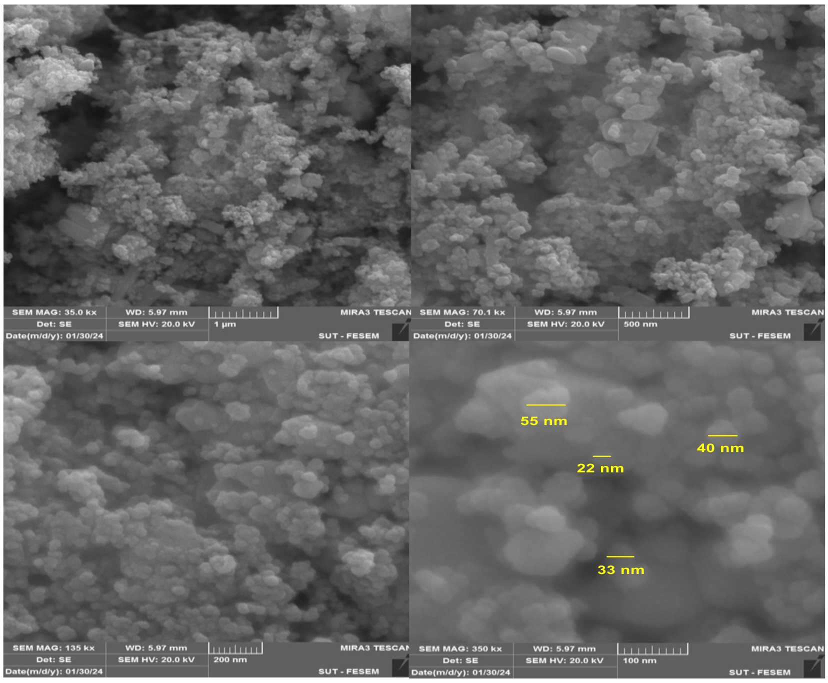

Figure 9. SEM image of ZnO NPs.

It may be inferred from FTIR, XRD, EDX, and SEM analyses that nano-ZnO was effectively synthesized. The FTIR spectra verified the presence of distinctive ZnO bands in the 450–550 cm^{-1} range. XRD analysis verified the zinc oxide's composition and purity, which was in line with previous findings. The average size of the nanoparticles was determined to be 26.715 nm using the Scherrer equation. The great purity of the ZnO was confirmed by the EDX analysis, which found no observable impurities. The surface morphology was obtained using SEM scanning, revealing nanoparticle sizes that ranged from 22 to 53 nm. These sizes nearly matched the expected grain size derived from XRD data. All of these thorough examinations confirm that the produced material is, in fact, zinc oxide.

4. CONCLUSION

The study effectively illustrates the environmentally friendly synthesis of ZnO nanoparticles utilizing an Iraqi pomegranate extract from the Diyala Governorate. This environmentally friendly method adheres to the principles of green chemistry by utilizing plant-based materials and non-toxic, cost-effective chemicals, resulting in a high yield of homogeneous nano-zinc oxide particles suitable for commercial applications. X-ray diffraction analysis confirmed the formation of ZnO NPs, with data consistent with the JCPDS standard file. Additionally, FTIR, XRD, and EDX examinations verified the high purity of the zinc oxide particles, showing no impurities. SEM analysis further validated the nanoscale range of the particles, ensuring their uniformity and suitability for various applications.

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