



Synthesis, Characterization and Photocatalytic Activity of ZrO₂ Nanoparticles using Mint Extract

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Abstract

This work describes a green synthesis route for zirconium oxide nanoparticles (ZrO₂ NPs) by using Zr sulfate and mint extract. This method is distinguished from other processes (physical and chemical) by its simplicity, cost-effectiveness, and low potential risks during preparation. Multiple techniques have been used to characterize and analyze ZrO₂ NPs, including Fourier transform infrared and ultraviolet visible spectra, crystal size calculation by X-ray diffraction, scanning electron and atomic force microscopes, energy-dispersive X-ray, and Zeta potential. Kinetic stability of the ZrO₂ NPs system, as indicated by the Zeta potential of -25.8 mV, suggests a long-term persistence and high surface charge. Finally, photo-catalytic activities of zirconium oxide NPs were examined using methylene blue dye. The results showed that the dye's degradation efficiency under light irradiation was 96.88%, demonstrating its significant photo-catalytic potential. The x-ray diffraction analysis of the crystal size of ZrO₂ NPs, using Debye Scherrer's equation, yielded a value of 18.78 nm. The prepared ZrO₂ NPs were tetragonal.

Keywords: Microscopy, Photo-catalytic activities, Transmission electron, X-ray diffraction, Zirconium oxide nanoparticles.

1. Introduction

Nanotechnology is a multidisciplinary field of interest, attracting researchers from areas such as physics, chemistry, biology, engineering, medicine, and materials science due to its potential to design nanoparticles (NPs) with controlled shapes and dimensions (1,2). While several approaches exist for NPs synthesis, environmentally friendly, cost-effective, and sustainable "green synthesis" techniques are increasingly preferred over conventional physical and chemical methods. Traditional chemical methods often involve high costs and hazardous reagents (3,4), and typically require additional stabilizers or additives to maintain particle stability. These processes may also utilize non-biodegradable solvents and demand extreme conditions such as high temperatures and pressures (5-7). On the contrary, biological or green biosynthesis leverages natural resources—such as bacteria, fungi, algae, and especially plant extracts—to generate NPs without resorting to toxic chemicals. Among these



biological sources, plant extracts stand out for their simplicity, low environmental impact, and cost-effectiveness, as well as for yielding stable NPs (8-11). Plant-derived phytochemicals play a significant role as natural reducing and capping agents during synthesis. Examples include neem (*Azadirachta indica*), hibiscus (*Hibiscus rosa-sinensis*), and Indian gooseberry (*Emblica officinalis*), which have demonstrated effectiveness in producing metal oxide NPs. Spinach, in particular, is a valuable green source due to its rich composition of vitamins (A, C, E, K), folic and oxalic acids, and essential minerals like Mn, Mg, Ca, P, Fe, Zn, Cu, Se, and K. Moreover, it contains important phytochemicals such as carotenoids (β -carotene, lutein, zeaxanthin), chlorophyll, glutathione, α -lipoic acid, betaine, and various phenolic compounds (12-15), all of which contribute to its antioxidant properties and facilitate nanoparticle formation. Previous research has successfully synthesized zirconium oxide (ZrO_2 NPs) using biological agents, including *Euclea natalensis* (16), the fungus *Fusarium solani* (17), various plant leaves (18), *Curcuma longa* (19), pomegranate peel (20), and cinnamon (21). These studies have explored diverse applications ranging from adsorption and thermodynamic analysis to evaluating the biological activity, toxicity, and antioxidant properties of ZrO_2 NPs (22). For instance, synthesis using thyme extract at pH 12 yielded ZrO NPs with an average crystallite size of 18.7 nm, as determined by the Debye–Scherrer equation (23). This study aims to characterize the ZrO_2 NPs prepared from mint plant extract using various methods, such as X-ray diffraction (XRD), field emission scanning electron microscopy and electron density (FESEM-EDS), transmission electron microscope (TEM), and zeta potential. In addition to analyzing the infrared and ultraviolet spectra of the NPs, the photo-catalytic activity (PCA) was utilized as a practical application using methyl blue dye, yielding favorable results.

2. Materials and Methods

The acquired specimens were sourced from a local location and utilized ZrSO_4 (purchased from Merck, Germany), NaOH (obtained from Sigma Aldrich, America), and deionized water (DW) sourced from local markets.

2.1. Preparation of mint extract and ZrO_2 NPs

The same method described in (24) was used. Fresh Mint was gathered and washed by DW to remove any existing impurities, dried, and then blended into a fine, uniform powder. Then, 20 g of this powdered Mint was mixed with 200 mL of DW, and then heated to 70-80 °C for 30 minutes while stirring. The mixture was then filtered and stored in the refrigerator for later use. To synthesize ZrO_2 NPs using the biosynthesis method, 100 mL of Mint extract was carefully added, drop by drop (1 drop per second), to 100 mL of 0.1 M $\text{Zr}(\text{SO}_4)_2$. The solution was stirred for 30 minutes, and then a sodium hydroxide solution (made by dissolving 2 g of NaOH in 50 mL of DW) was added gradually until the pH reached 12. This process led to the formation of a dark green precipitate, which was purified by washing with deionized water using centrifugation and decantation. The purified precipitate was then dried in an oven and calcined at 800°C for 3 hours, ultimately producing a yellowish powder of ZrO_2 NPs.

2.2. The photo-catalytic activity

The PCA of ZrO_2 NPs was evaluated using an organic dye; a 10 mg/L concentration of MB was taken as a model pollutant. The stock solution of MB was diluted with deionized water to prepare the working solution at a final concentration of 5 mg/L. A specific amount of ZrO_2 NPs (0.5 g/L) was weighed and dispersed into the pollutant solution. To ensure homogeneity, sterilize the mixture with ultrasound for 30 minutes, then transfer the suspension to a quartz

photo reactor. The reactor was equipped with a high-efficiency light source, a xenon lamp with a power output of 300 W, to provide the required irradiation. Before illumination, the suspension was stirred in complete darkness for 30 minutes to establish adsorption-desorption equilibrium between the pollutant molecules and the catalyst surface. Following this equilibrium phase, the light source was activated, and the photo-catalytic reaction was initiated, with the suspension exposed to light for 120 minutes. During the reaction, aliquots of the solution were withdrawn at regular time intervals of 20 minutes. Each aliquot was filtered using a syringe filter with a pore size of 0.22 μm to remove residual NPs (24). The residual concentration of the pollutant in the filtered aliquots was measured with a UV spectrophotometer at 664 nm, the characteristic wavelength of MB. **Equation 1** below shows the calculation of the photo-catalytic decomposition efficiency.

$$\text{Degradation Efficiency (\%)} = \frac{C_0 - C_t}{C_0} \times 100 \quad (1)$$

Where C_0 is the initial concentration of the pollutant, and C_t is the concentration at time t . To ensure the accuracy and reproducibility of the results, all experiments were performed in triplicate, and the average values were reported. Control experiments were also conducted to verify the photo-catalytic nature of the degradation process, including tests without the catalyst (light only) and tests in the dark (catalyst only). These control studies confirmed that the observed degradation was solely attributable to the photo-catalytic activity of the ZrO_2 NPs. The results demonstrated that the ZrO_2 NPs exhibit excellent photo-catalytic efficiency, achieving significant pollutant degradation within the reaction time. This highlights their potential as effective photo catalysts for environmental remediation applications (24).

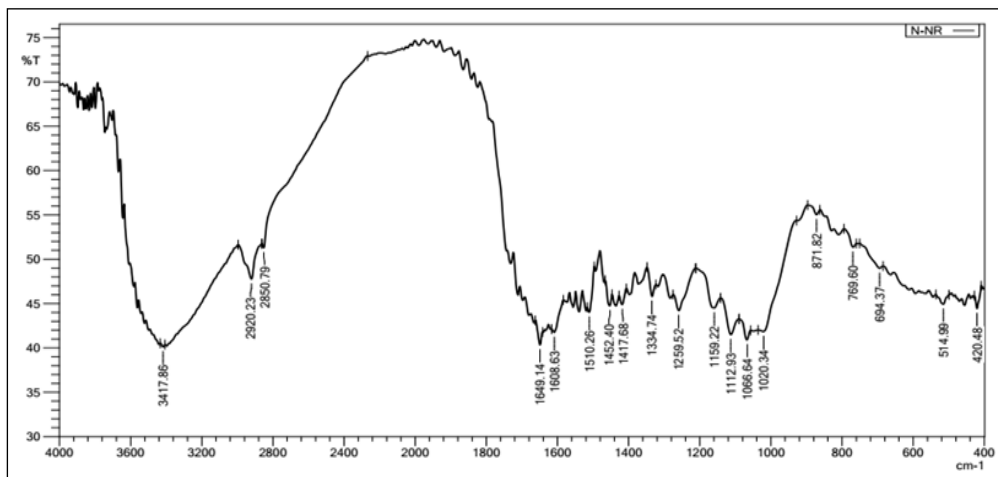
2.3. Product characterisation

The compounds were created and identified using various spectroscopic and microscopic techniques and equipment, including a magnetic stirrer, an electric oven of type PC21-A, Batec, an electric centrifuge of type PLC (80-1), and a sensitive electronic balance (AS 220C1) was used for precise measurements. FT-IR spectrum was measured with a Shimadzu 1800 (Japan) in the range of 400-4000 cm^{-1} . UV-Visible spectroscopy was conducted with a UV-Vis spectrophotometer, and the FTIR and UV-Visible were tested at the University of Baghdad. XRD analysis was performed using a Phillips PW1730 (Netherlands). The structural composition and elemental content were measured by energy-dispersive X-ray spectrometry (EDX) and FESEM-EDS model MIRA II (Czech Republic). Additionally, a TEM model EM10C-100Kv (Zeiss, Germany) was used for further examination. XRD, EDX, TEM, and FESEM-EDS were tested at the University of Kashan/ Islamic Republic of Iran. The Zeta potential was tested at the University of Technology, Baghdad.

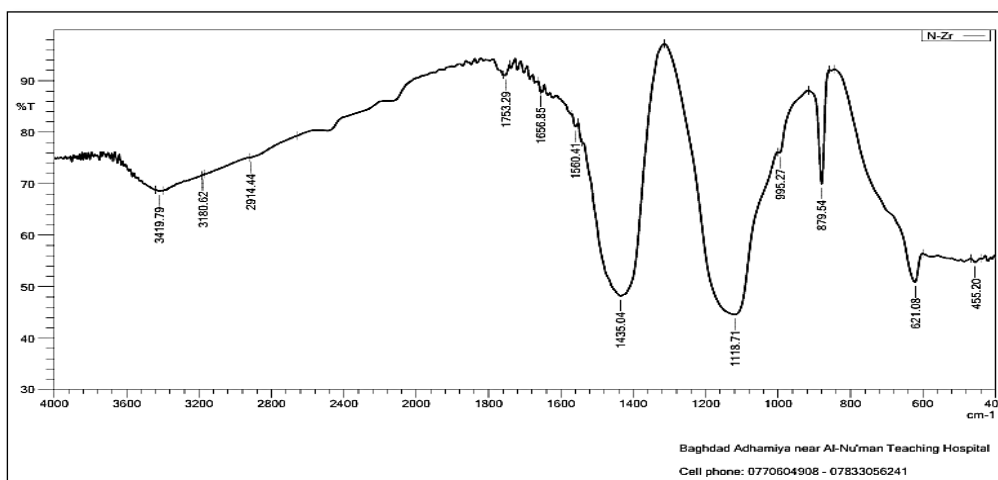
3. Results

3.1. Data analysis of FT-IR

The FTIR of mint leaf extract, **Figure 1A**, shows absorption bands at 3417, 1608, 1417, 1259, and 1020 cm^{-1} . The results of infrared spectroscopy of the obtained ZrO_2 NPs synthesized using Mint extract are shown in **Figure 1B**; many bands are shown at 3419 cm^{-1} .



A



B

Figure 1. Data analysis of the spectrum, **A-** Mint leaves, **B-** ZrO₂ NPs.

3.2. Data analysis of UV-Visible

The UV-vis spectrum of ZrO₂ NPs in 10^{-3} M concentration and DMSO as a solvent in **Figure 2**, displays peaks of absorption at (234 and 370) nm.

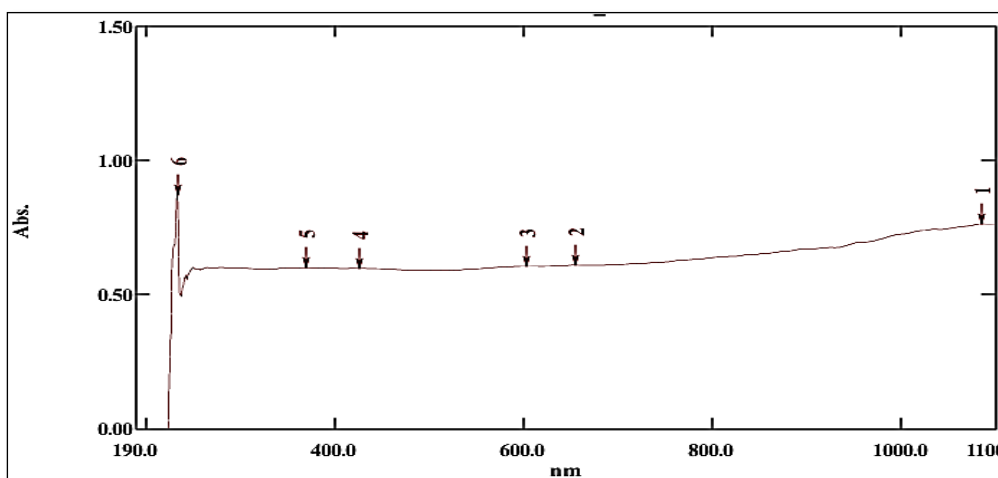


Figure 2. Data analysis of the UV- visible spectrum of ZrO₂ NPs.

3.3. Data analysis of X-RD and EDX

Based on the XRD study presented in **Table 1** and **Figure 3**, it has been determined that the crystal planes of ZrO₂ NPs, which are consistent with miller indices, 101, 103, 202, 321 and

224 have been assigned to several XRD position peaks at ($^{\circ}2\theta$.) values (27.935, 29.618, 31.645, 49.949 and 59.550) respectively, closely match the conventional diffraction peaks of JCPDS card number (No. 24-0734).

Table 1. Data analysis of XRD of ZrO_2 NPs.

Pos. [$^{\circ}2\theta$.]	hkl	FWHM (deg)	2 theta (Rad.)	FWHM (Rad.)	P.s (nm)	Medium size
27.9357	101	0.39	0.255001	0.002	21.74	18.78nm
29.6187	103	0.49	0.285396	0.003	17.46	
31.6458	202	0.30	0.317627	0.004	29.24	
49.9492	321	0.69	0.512779	0.008	13.30	
59.5509	224	0.79	0.525129	0.002	12.15	

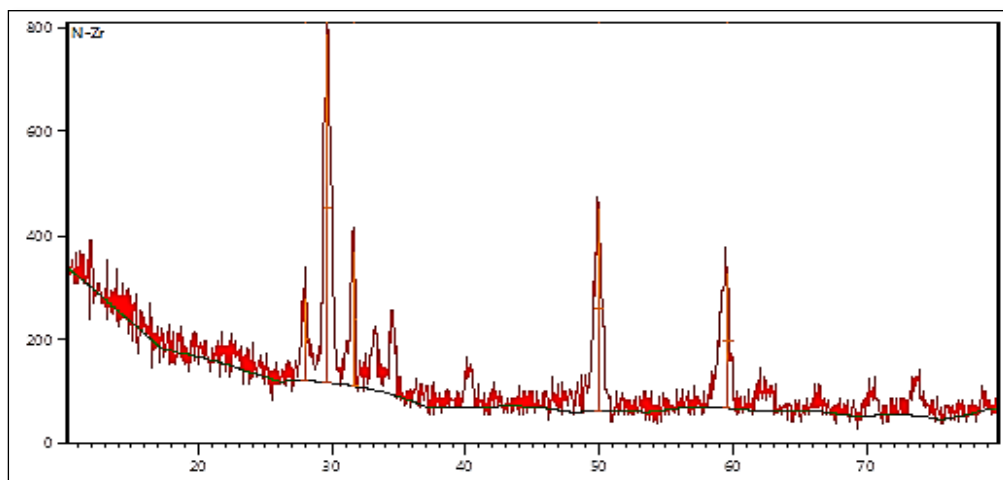


Figure 3. The XRD analysis of ZrO_2 NPs.

The EDX was used to analyze the percentage (%) of elements in the ZrO_2 NPs, as shown in **Figure 4**. The element percentages showed 60% for Zr and 40% for oxygen.

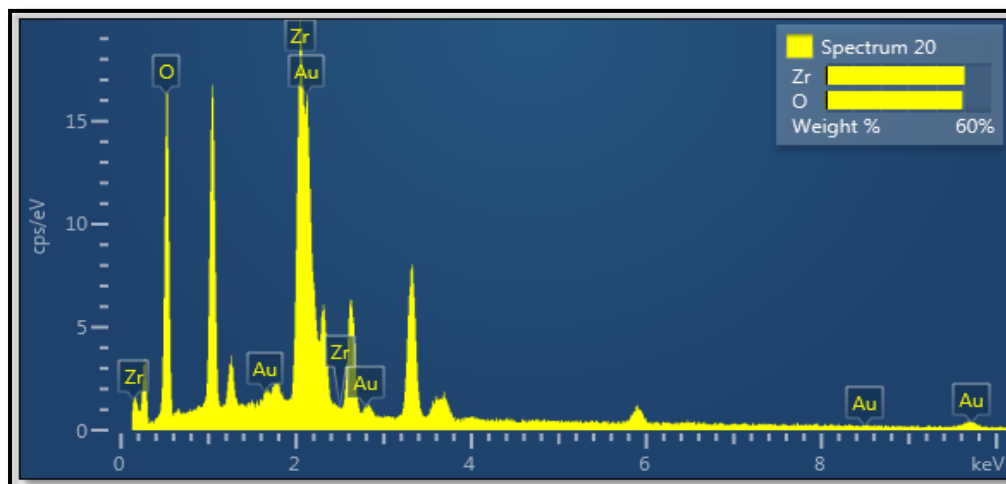


Figure 4. The EDX of ZrO_2 NPs.

3.4. Data analysis of SEM and TEM

The morphology and structure of the nanomaterial were determined by scanning electron microscopy. SEM images in **Figure 5** confirm the precise formation of ZrO_2 NPs in small clusters. The TEM image reveals that the sample consists of spherical and irregular NPs structures within a (15-50) nm range with a zero-dimensional morphology as seen in **Figure 6**.

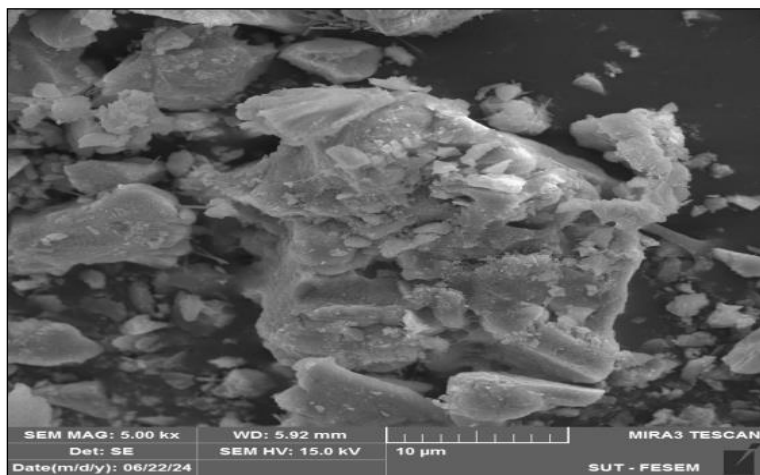


Figure 5. The Images of SME for ZrO_2 NPs.

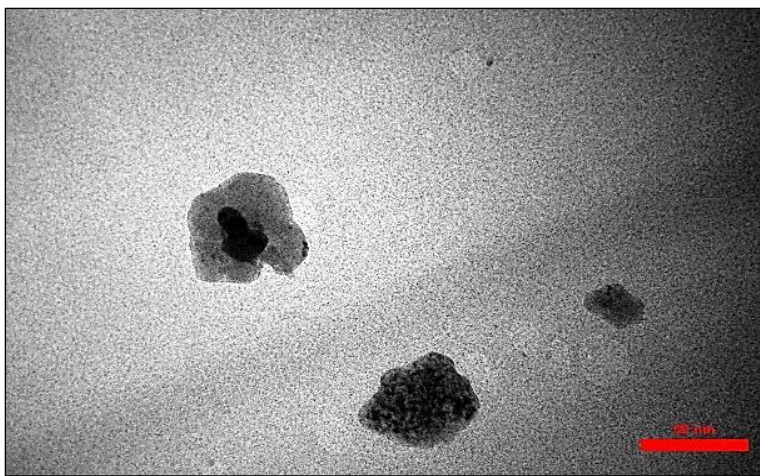


Figure 6. The images of TME for ZrO_2 NPs.

3.5. Zeta potential of ZrO_2 NPs

The zeta potential of ZrO_2 NPs synthesized from Mint plant extract was -25.8 mV, as shown in **Figure 7**.

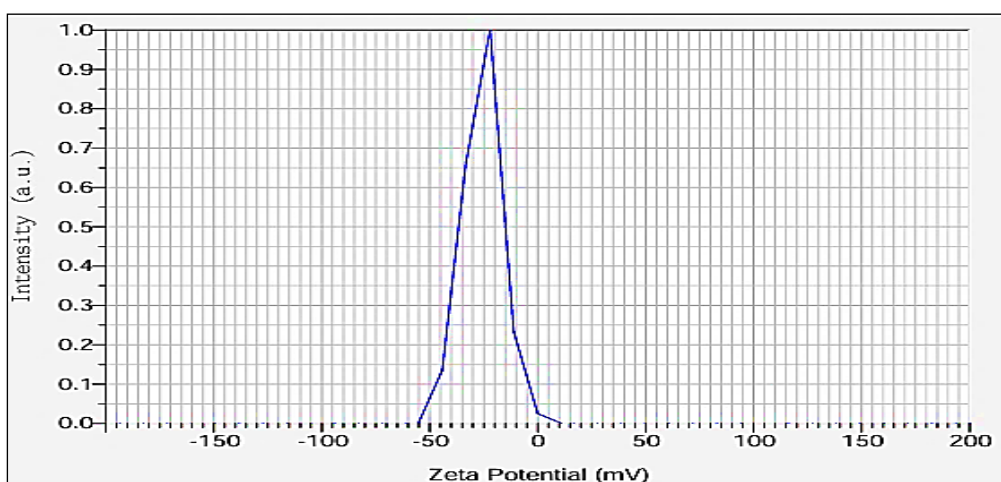


Figure 7. The images of Zeta potential of ZrO_2 NPs.

3.6. Photo-catalytic degradation of MB

The photo-catalytic activity of ZrO_2 NPs was evaluated by light irradiation for the degradation of MB dye. The absorption spectra, measured at different time intervals, exhibited a distinct peak at 664 nm, corresponding to the characteristic absorption of MB. Initially, at time 0 minutes, the absorption was at its maximum (~1.6 absorbance units), reflecting the full concentration of MB in the solution. Over time, a consistent decrease in absorption was observed at intervals of (20, 40, 60, 80, 100, and 120) minutes as shown in **Figure 8**. The gradual degradation of MB molecules, by the end of the experiment (120 minutes), the absorption approached nearly zero, signifying that most of the MB had been degraded.

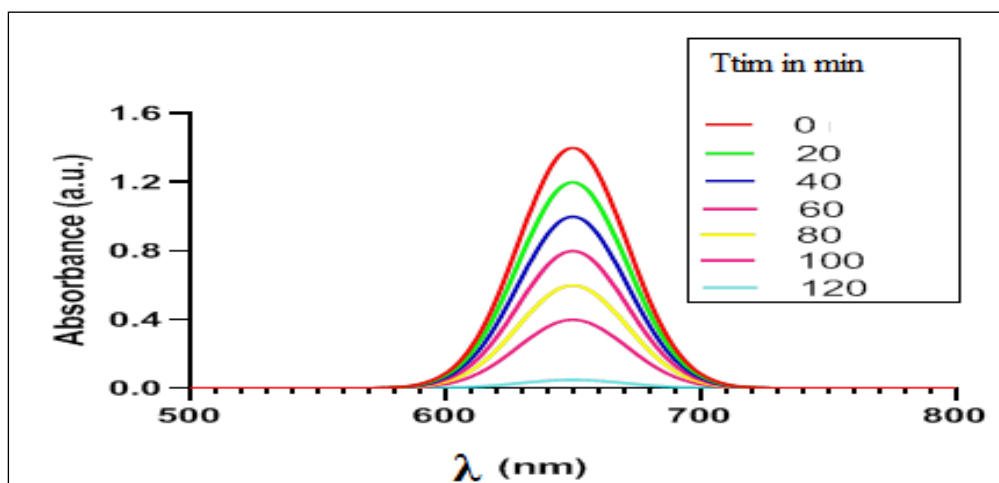


Figure 8. The UV-VIS spectra of MB catalyzed by ZrO_2 NPs.

4. Discussion

4.1. The FTIR spectra

In FTIR, the broad band at 3417 cm^{-1} could be attributed to the presence of the hydroxyl functional groups of alcohols and phenolic compounds that may exist in plant extracts. The sharp peak located at 1597 cm^{-1} is related to the presence of carbonyl-containing compounds, which may be involved in the bio-reduction of the Zr ion. The peaks at $1403\text{--}1279\text{ cm}^{-1}$ arise from the C–N stretching mode of aromatic amine groups. The marked peak at 1055 cm^{-1} can also be related to the stretching vibration of the C–OH bond from proteins in the plant extract.

4.2. The Uv-visible spectrum

The UV-visible spectrum of ZrO_2 NPs shows many peaks at (234 and 370) nm and 400 nm attributed to the transition of holes between Zr and O, indicating a distinctive feature of the biosynthesis process. The absorption peaks above 400 nm are attributed to the d–d transitions of Zr ions in ZrO_2 NPs. The energy gap of ZrO_2 NPs was calculated from the equation: $E_g = 1240/\lambda$, which is to be (5.29 and 3.35) eV (24).

4.3. The XRD pattern and EDX

The observed peaks in **Figure 3** and **Table 1** correspond to the crystallographic planes (hkl) of the prepared ZrO_2 NPs, which were tetragonal crystalline phases as well as monoclinic ones (14). The sizes of the ZrO_2 NPs were determined using the Debye-Scherrer equation, as shown in **Equation 2**, revealing an average size of 18.78 nm.

$$D = \frac{0.9 \lambda}{\beta \cos \theta} \quad (2)$$

Where D: the average crystalline size; λ : X-ray wavelength ($1.5406\text{ \AA}$) of the copper target; β : maximum width at mid-intensity (FWHM) and converted from degrees to radius; K: shape

factor or shape factor, which is about 0.9-0.94; and θ : diffraction angle (24). The XRD pattern shows extra pointless peaks. This may be due to the formation of the crystalline bio-organic compounds and metalloproteins that are present in the extracts (25,26).

EDX was used to analyze the percentage (%) of elements in the ZrO₂ NPs, and the results obtained from this analysis revealed a characteristic peak associated with the % element composition (Zr and O), which corresponds to the exact composition of the sample under study, as shown in **Figure 4**.

4.4. The SEM and TEM analyses

The SEM analysis revealed an approximately spherical shape with a rough surface due to the presence of a limited amount of morphology ZrO₂ NPs with nanostructured morphology. This can be seen from the shapes of the SEM images in **Figure 5**, which confirms the precise formation of ZrO₂ NPs formed in small clusters (27). The TEM image reveals that the sample consists of spherical and irregular NPs structures within a (15-50) nm range with a zero-dimensional morphology. This characteristic is highly desirable in surface chemistry, as it enhances the reactivity and stability of nanomaterials. The spherical morphology (confirmed by TEM) enhances the surface-area-to-volume ratio. The well-defined spherical shape suggests uniform particle distribution, which can improve the material's performance in various applications, as seen in **Figure 6**.

4.5. Zeta potential

The zeta potential of the synthesized ZrO₂ NPs was -25.8 mV, indicating that the kinetic stability of the ZrO₂ NPs system would persist for a long time due to its high surface charge, and thus can be maintained in suspended form in solution (28).

4.6. Photo-catalytic degradation

This degradation is attributed to the photo-catalytic activity of ZrO₂ NPs, which, under light irradiation, produces reactive oxygen species such as (O₂^{-•} and •OH) (28). These reactive species attack MB molecules, leading to the breakdown of their chromophoric structure and conversion into harmless byproducts like (CO₂ and H₂O) (29). The degradation process follows first-order kinetics, where the reaction rate is proportional to the concentration of MB. The degradation efficiency was calculated using the following **Equation 3**:

$$\text{Degradation efficiency \%} = \frac{A_0 - A_t}{A_0} \times 100 \quad (3)$$

Where A₀ (the initial absorbance) and A_t (remaining absorbance) are at the wavelength of 664 nm, respectively, using the values extracted from the spectra (A₀= 1.6 and A_t= 0.05) at 120 minutes (30), the efficiency was determined to be approximately 96.88%. This high degradation efficiency highlights the remarkable photo-catalytic performance of ZrO₂ NPs, making them a promising material for environmental pollution treatment. Their ability to achieve near-complete degradation of MB within 120 minutes demonstrates their potential for use in wastewater treatment systems. Future studies could focus on optimizing experimental parameters, such as catalyst dosage and light intensity, as well as exploring the reusability and long-term stability of ZrO₂ NPs for large-scale applications (31-33).

5. Conclusion

This study describes the eco-friendly synthesis of ZrO₂ NPs using Mint plant extract, which is both efficient and environmentally friendly. FTIR and UV-Vis spectroscopy were used to diagnose the ZrO₂ NPs, which exhibited limited morphology and some deviations due to the presence of large sizes. The SEM technique was used to provide information about the surface morphology and composition of the samples. SEM results showed that the particle size of the ZrO₂ NPs was formed in small clusters. The average crystal size of the ZrO₂ NPs

was 18.78 nm, as confirmed by the XRD and EDX analysis, which showed 60% Zr and 40% O₂. Zeta potential was -25.8 mV, due to the kinetic stability of ZrO₂ NPs. Photo-catalytic tests showed about 96.88% degradation efficiency of MB dye under light irradiation, emphasizing their essential photo-catalytic potential. The particles are small and spherical.

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Conflict of Interest

The authors declare that they have no conflicts of interest.

Funding

None.

Ethical Clearance

This study was approved by the University of Baghdad, College of Education for Pure Science (Ibn Al-Haitham).

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