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Paper 16

Photocatalytic Degradation of Textile Orange 16 Reactive Dye by ZnO Nanoparticles Synthesized via Green Route Using Punica Granatum Leaf Extract

Dr. Edi Syafri, ST, M.Si

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Abstract: Since it does not use any dangerous chemicals and is a simple, low-cost process, the green synthesis approach for nanoparticle creation has several benefits compared to the physical and chemical synthesis routes. The current study describes an environmentally friendly synthesis of zinc oxide (ZnO) nanoparticles (NPs) using an extract of *Punica granatum* plant leaves. Fourier-transform infrared spectroscopy (FTIR), ultraviolet-visible spectrophotometer (UV-Vis), field-emission scanning electron microscopy (FESEM), energy-dispersive X-ray spectroscopy, and X-ray diffraction techniques were used to characterize the morphology, composition, and structural properties of the synthesized zinc oxide nanoparticles. The XRD pattern reveals that the ZnO nanoparticles are crystalline and have a diameter of 20 nm. According to the FESEM studies, the ZnO-NPs have sizes ranging from 50 to 100 nm on average and are almost spherical. When exposed to direct sunlight, the produced ZnO-NPs demonstrate impressive photocatalytic oxidation of textile Orange 16, a re-

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Keywords: green synthesis; zinc oxide nanoparticle; photocatalytic; textile Orange 16 reactive dye

Citation: Al-Zahrani, S.A.; Patil, M.B.; Mathad, S.N.; Patil, A.Y.; Otaibi, A.A.; Masood, N.; Mansour, D.; Khan, A.; Manikandan, A.; Syafri, E. Photocatalytic Degradation of Textile Orange 16 Reactive Dye by ZnO Nanoparticles Synthesized via Green Route Using Punica Granatum Leaf Extract. *Crystals* **2023**, *13*, 172. https://doi.org/10.3390/cryst13020172

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1. Introduction

New nanoscale materials are being produced as a result of the advancements in nanotechnology. These materials have a variety of uses, including in consumer goods, nanomedicine, and nanoelectronics [1,2]. Because of their superior chemical and physical characteristics when compared to their bulk counterparts, research on such materials has increased significantly in recent years. Metal oxide nanostructures have been created, and they have a variety of uses in various industries. A semiconductor with a greater band gap (3.4 eV) is zinc oxide (ZnO). Applications for it include dye degradation, gas sensors, solar cells, and many others [3]. Numerous chemical and physical techniques, such as the sol–gel technique [4], the precipitation method [5], the arc discharge technique [6], the hydrothermal method [7], and the laser ablation method [8], have been adopted for the synthesis of ZnO nanoparticles. Due to its numerous advantages over the physical and chemical approaches—including the fact that it does not use dangerous chemicals and is both environmentally friendly and economically advantageous—synthesis via the green method has been used to prepare ZnO nanoparticles [9,10]. This approach makes use of biological components that are readily available from plants.

Because they are readily available, leaves from the *Punica grantum* plant have been employed. The family of *Punicaceae*, which consists of two different species, is dominated by the pomegranate species (Punica granatum). *Punica grantum* plant leaves contain alkaloids, tannins, triterpenic acids, and flavonoids as phytochemicals [11]. Utilizing chemical reduction, these phytoconstituents produce metal oxide nanoparticles by acting as the stabilizing and reducing agents. As far as we are aware, there has not been any research on the usage of *P. granatum* leaves in the preparation of ZnO-NPs.

Organic substances called dyes are used in the paper, food, leather, and textile industries. Strongly colored, hazardous dyes are present in the effluents from these enterprises. The discharged effluents will enter running water, polluting the surface and groundwater and perhaps endangering the health of aquatic life as well as that of people, fauna, and the environment. Before release, the effluents would be treated to break down the dye into non-toxic species. Different purification methods, including chemical, biological, and physical techniques, have been developed, depending on the type of pollution. ZnO-NPs have been used to degrade dyes with success [12–14]. Through catalytic photooxidation with ZnO-NPs, reactive oxidative hydroxide radicals are created, which destroy the dyes. According to its chemical makeup, the textile Orange 16 reactive dye is an organic substance classified as a disulfonated triphenylmethane dye. Although originally designed for the textile industry, it is now frequently used for protein staining in biochemistry. The catalyst should be stable and reusable in addition to having a high degradation efficiency, as these qualities are crucial for industrial applications. Numerous investigations [15,16] have shown that ZnO-NPs are stable and reusable.

The *P. granatum* leaf extract was used in the current work's green synthesis to create ZnO-NPs. Under exposure to direct sunlight, the green ZnO-NPs that were generated were used to photocatalytically degrade the textile Orange 16 reactive dye.

2. Materials and Methodology

2.1. Materials

We gathered *Punica granatum* leaves from a field in Kaladagi near Bagalkot (India). Zinc nitrate hexahydrate of high purity was procured from Sigma-Aldrich, Mumbai, India. All water used in the experiment was deionized distilled water. We bought textile Orange 16 reactive dye from a local market in Bagalkot, Karnataka, India, through a textile business. None of the chemicals were further purified before use. Glassware washed in a prepared Piranha solution (3:1 volume ratio of H_2SO_4/H_2O_2) was rinsed with deionized (DI) water with a resistivity of 16.4 MΩ·cm (millipore water), dried in an oven, and returned for later use.

2.2. Methodology

2.2.1. Extraction of *Punica granatum* Leaf

Young *P. granatum* leaves were collected from a *Punica granatum* plantation at a Kaladagi village near Bagalkot (India). The leaves' middle rib was cut off. The leaves that remained were cleaned with distilled water to get rid of dust, and they were then dried in the shade to get rid of all the moisture. An aqueous extract of *Punica granatum* leaf was prepared. During the process, the dried leaves were weighed accurately and were finely grounded in a mixture. Then, deionized water was added to the dried fine powder. For about three hours, the mixture was refluxed at 60 °C, using the Soxhlet extractor apparatus. Once the green color turned brownish, the process was stopped. At room temperature, the solution was allowed to cool for the period of ten to fifteen minutes. Furthermore, Whatman No. 41 filter paper was used to filter the solution, and the filtrate was then collected in a dry flask while the residue was thrown away. For future use, the leaf extract was kept at ambient temperature.

2.2.2. Green Synthesis of Zinc Oxide Nanoparticles (ZnO-NPs)

In deionized water, a solution of $Zn(NO_3)_2{\cdot}6H_2O$ (0.1 M) was made. The mixture was made homogeneously by stirring it without being heated. After that, a magnetic stirrer was used to gradually add drops of the aqueous leaf extract to the zinc nitrate solution. After the leaf extract was completely added, the resulting mixture was stirred with heating at 60 °C for 3–4 h on a hot plate until it took the form of slurry. The obtained slurry substance was filtered and placed in a crucible. The sample was calcined at 500 °C in a muffle furnace at a heating rate of 5 °C for 5 h. After calcination, white powder was obtained and stored properly.

2.2.3. Characterization of ZnO-NPs

A Siemens D 5000 (Malvern, UK) powder X-ray diffractometer was used to perform X-ray diffraction on the powdered zinc oxide nanoparticles. The UV-visible spectrum of the zinc oxide nanoparticles was recorded by employing a UV spectrophotometer (Systonic, Bangalore, India). The samples were evaluated between 200 and 800 nm. A Shimadzu (Model: IR Affinity-1, Tokyo, Japan) FTIR spectrometer was used to perform Fourier-transform spectroscopic measurements. A Carl Zeiss (Model: Sigma 300, Bangalore, India) field-emission scanning electron microscope (FESEM) was used to examine the surface morphology and the dimension of the particles of the prepared ZnO nanoparticles. The Oxford instrument (Buckinghamshire, UK), an energy-dispersive X-ray spectroscopy (EDX), was used to obtain the spectrum to check the purity of the synthesized ZnO nanoparticles and the stoichiometry of the samples. The particle size of the synthesized ZnO-NPs was determined using the Zetasizer (Model 3000HS, Malvern, UK). On a cuvette, the zeta average size of ZnO-NPs suspended in DI water was determined. The measurement was repeated three times, and the average value was used for data analysis.

2.2.4. Photocatalytic Activity of ZnO-NPs

A photodegradation study of textile Orange 16 reactive dye in water by exposure to sunlight was used to determine the ZnO nanoparticles as the photocatalysts. For this, an artificial, laboratory-simulated dye was prepared by dissolving the dye in water until the solution's absorbance value was more than one, i.e., 0.4 g in 100 mL of water. To the prepared colored mixture, a known amount of ZnO (i.e., 0.1 g), which would act as a photocatalyst, was added. Later, the aqueous solution was sonicated for 30 min. The solution was then kept in sunlight while being constantly stirred. After 30 min, 5 mL of the solution was withdrawn and centrifuged to settle down the ZnO photocatalyst. The supernatant solution was used to measure the absorbance with a UV-Vis spectrophotometer. From the experiment, it was observed that the value of absorbance reduced after each interval, showing the degradation of the dye by the ZnO-NPs. The detailed stepwise green synthesis of ZnO nanoparticles and their photocatalytic action are shown in Figure 1.

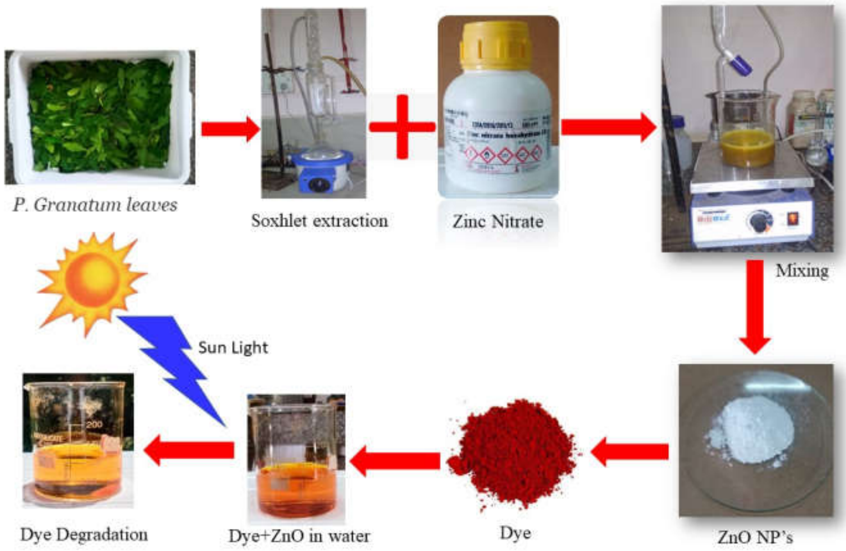

Figure 1. Step-by-step process of ZnO-NP synthesis via green route and the degradation of the dye.

3. Results and Discussion

3.1. X-ray Diffraction Analysis

Figure 2 depicts the X-Ray diffraction graph of the green synthesized ZnO nanoparticles. The sample was examined at various angles, ranging from 0° to 70°. ZnO nanoparticles have significant peaks at 2θ = 31.81°, 34.49°, 36.28°, 47.62°, 56.63°, 62.91°, and 68°, which can be designated as (100), (002), (101), (102), (110), (103), and (200), respectively. The orientation and crystallinity of the ZnO-NPs were revealed by using the X-ray pattern. The JCPDS data sheet/ICDD no. 36-1451 was used to compute the XRD pattern. The obtained X-ray pattern demonstrate that ZnO-NPs were synthesized using the green synthesis route, with the establishment of crystalline and wurtzite hexagonal structures. The particle dimension of ZnO-NPs was computed using the Debye–Scherrer equation from the highest peak (101) in the XRD graph:

$$d = \frac{K\lambda}{\beta cos\theta} \quad (1)$$

where d is the size of the crystallite; λ is the wavelength for diffraction; β is the corrected value of FWHM; θ is the angle of diffraction; and K is the universal and its value is near unity, i.e., 0.94.

The purity of the ZnO-NPs is confirmed by the absence of diffraction peaks from other phases.

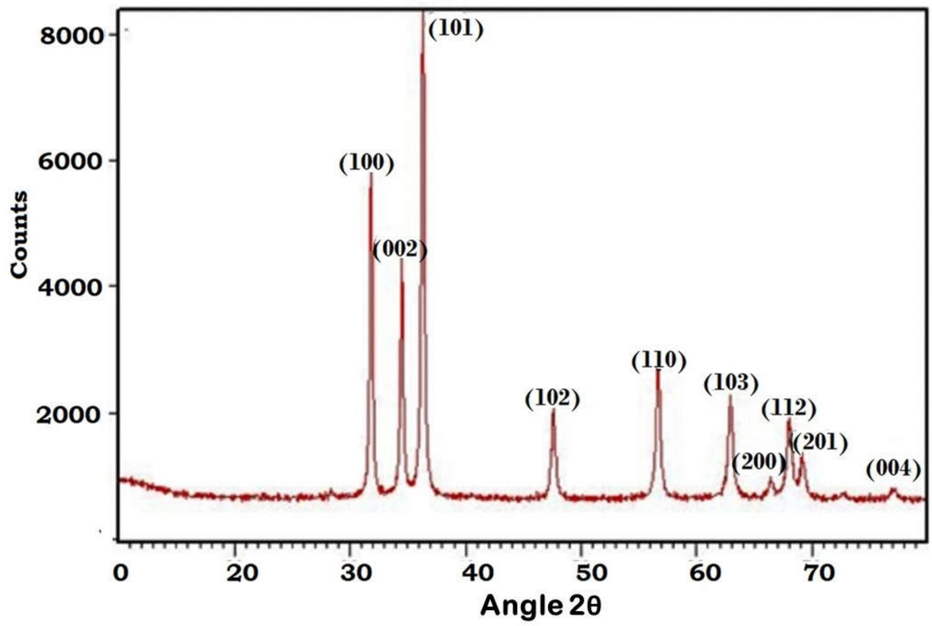

Figure 2. X-ray diffraction curves of green synthesized ZnO nanoparticles.

Bragg's equations 3-5 were used to calculate the inter-planar spacing (d), the lattice parameters (a = b and c) for the hexagonal wurtzite structure, and the volume of the hexagonal system unit cell (V) [17–20]. Table 1 summarizes all of these parameters.

$$n\lambda = 2d \sin\theta \tag{2}$$

$$\frac{1}{d_{hkl}^2} = \frac{4}{3}\left(\frac{h^2 + hk + k^2}{a^2}\right) + \frac{l^2}{c^2} \tag{3}$$

$$V = \frac{\sqrt{3}}{2} a^2 c \tag{4}$$

where d_{hkl} = inter-planar spacing; V = volume of the unit cell; and a, c = lattice parameters for the ZnO-NP.

Table 1. Crystallite size (D), inter-planar spacing (d), lattice parameters ($a = b$ and c), and volume (V) were calculated from the XRD measurements at 50 mmol/kg concentrations of $Zn(NO_3)_2 \cdot 6H_2O$.

[a]m (mmol/kg)	D (nm)	d (nm)	$a = b$ (nm)	c (nm)	V (nm^3)
50	31.158	0.256	0.325	0.527	4.721

[a]m represents the molality of Zn $(NO_3)_2 \cdot 6H_2O$ in water.

3.2. Particle Size Analysis

The particle size of the ZnO-NPs is depicted in Figure 3. The obtained results reveal that the size of ZnO particles ranges between 60 and 100 nm. The average particle size is measured at 80 nm.

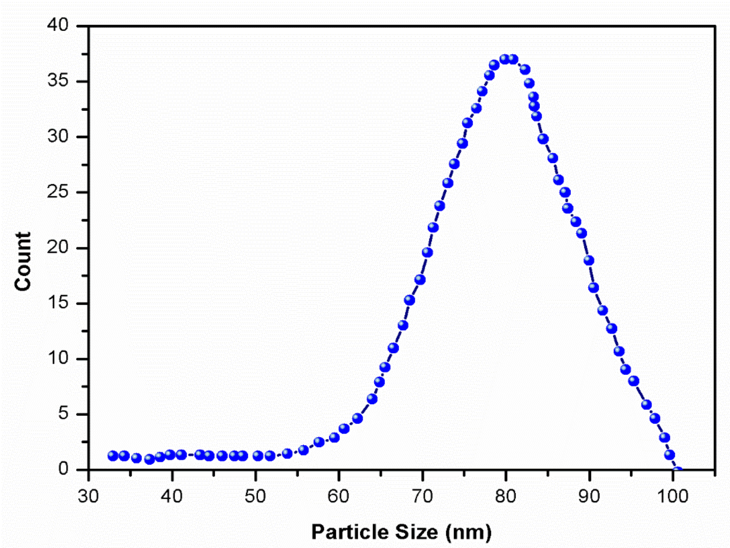

Figure 3. Histogram of ZnO particle size distribution.

3.3. UV-Visible Spectroscopic Analysis

Surface plasmon resonance distinguishes the absorbance pattern of nanoparticles from that of their bulk counterparts. The UV-visible measurement was used to confirm the formation of nanoparticles. Figure 4 depicts the UV-visible absorption spectrum of the ZnO-NPs. Using a systonic UV spectrophotometer, the substance was analyzed between 200 and 800 nm. The color of a solution of zinc nitrate hexahydrate ($Zn(NO_3)_2H_2O$) changed from white to brown when the *P. granatum* leaf extract was added. This resulted from the solution's synthesis of ZnO-NPs. The nanoparticles of zinc oxide are responsible for stimulating the surface plasmon vibrations, which in turn generate color changes. The absorbance peak was found to be centered at 382 nm, indicating that zinc nitrate hexahydrate had been converted to ZnO-NPs [21–23].

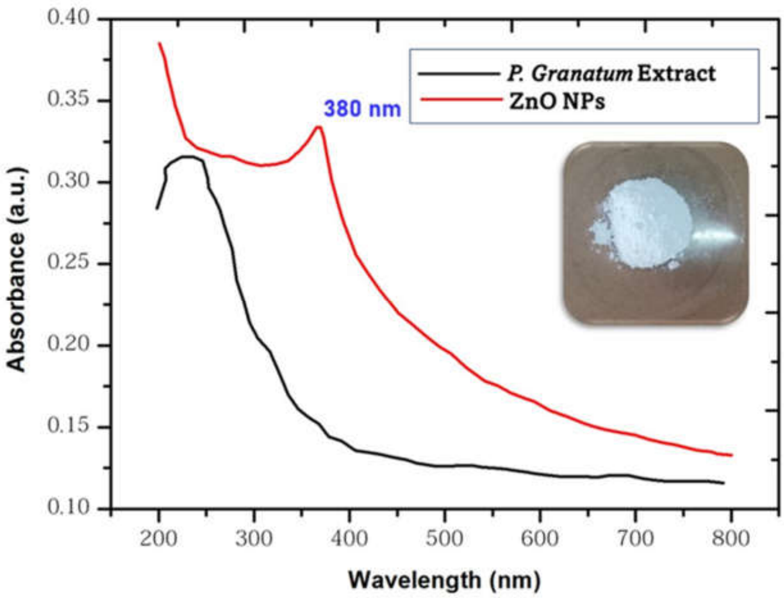

Figure 4. ZnO-NPs and *P. granatum* leave extract spectra measured using an UV-visible spectrophotometer. (Inside: photograph of ZnO-NPs synthesized via green method).

The optical energy band gap (E_g) was calculated using Formula (5), and the results are shown in Table 2.

$$E_g = \frac{hc}{\lambda} \quad (5)$$

From this formula, 'h' represents the Planck's constant (6.626×10^{-34} J s), 'c' corresponds to the velocity of light (the value is 3×10^8 m s^{-1}), and λ corresponds to the wavelength of the peak with the maximum intensity [24].

Table 2. λmax and energy band gap (E_g) results of ZnO-NPs at three different concentrations of zinc nitrate hexahydrate.

[a]m (mmpl/kg)	λ_{max} (nm)	E_g (eV)
5	381	3.436 ± 0.3
10	386	3.369 ± 0.4
50	390	3.361 ± 0.3

[a]m represents the molality of $Zn(NO_3)_2{\cdot}6H_2O$ in water.

The band gap values were calculated, and the results are nearly equivalent to 3.4 eV, which is consistent with previously reported values in the literature. Suresh et al. (3.33 eV) [25] and Hancock et al. (3.39 eV) [26] reported comparable band gap results. The variation in the shape and size of the ZnO-NPs may explain the difference in E_g with Zn $(NO_3)_2{\cdot}6H_2O$ concentration. In addition, a decrease in E_g values is attributed to an increase in ZnO particle size [27].

3.4. FESEM and EDX Analysis

ZnO may be produced into several nanostructures, including nanospheres, nanorods, and others. By looking at and analyzing each minute topographical feature using a field-emission scanning electron microscope (FESEM), the particle size and shape may be determined. The particle size and shape of the synthesized ZnO nanoparticles were analyzed with the aid of a Carl Zeiss FESEM. Figure 5 displays a FESEM picture of the ZnO nanoparticles that were synthesized via the green route. This image demonstrates that the particle is composed of nanostructures, which are even smaller structures. The XRD investigation indicated that the nanoparticles are fully spherical, with an average diameter of 80 nm.

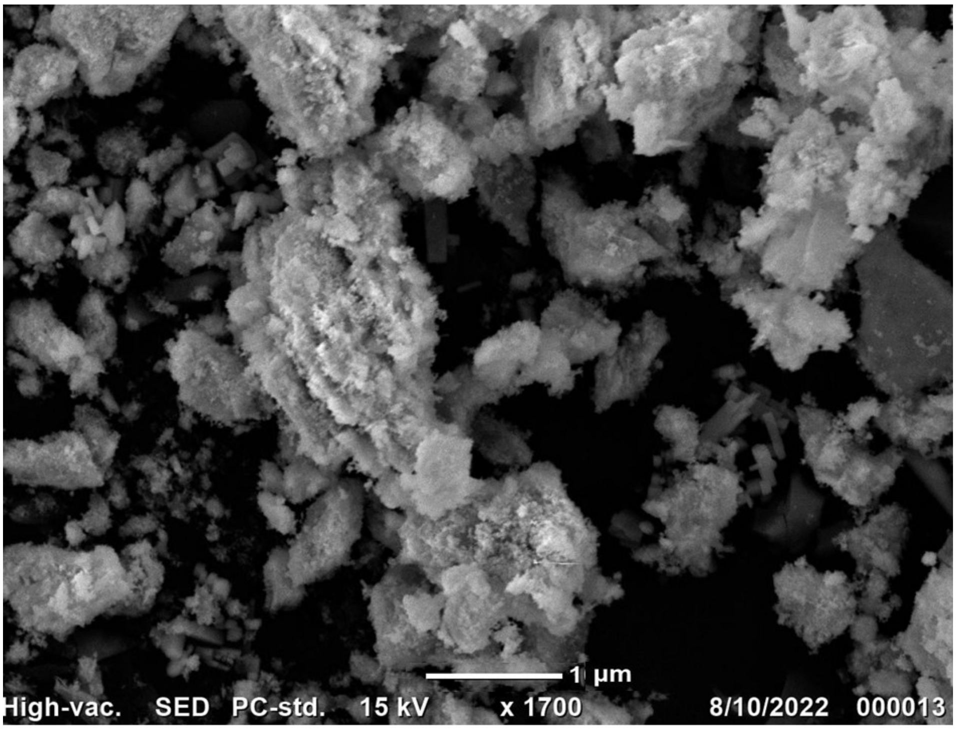

Figure 5. FESEM picture of zinc oxide NPs synthesized via the green route.

EDX was utilized to undertake an elemental investigation or chemical composition on the ZnO nanoparticles made in an environmentally responsible manner. The stoichiometry and chemical purity of the samples were analyzed using an EDX-equipped equipment from Oxford. The EDX spectrum of the ZnO nanoparticles is illustrated here in Figure 6. The EDX spectrum indicates indisputably that ZnO and oxygen (O) ions are present in the ZnO nanoparticles formed by the *P. Granatum* reaction. According to the findings of the elemental analysis, the ZnO powder is made up of 76% zinc and 15% oxygen, which shows that it is quite pure and only comprises a minor number of contaminants. The stoichiometry reveals that the mass percentages of zinc and oxygen should be 80.3% and 19.7%, respectively, if we are to trust the formulae.

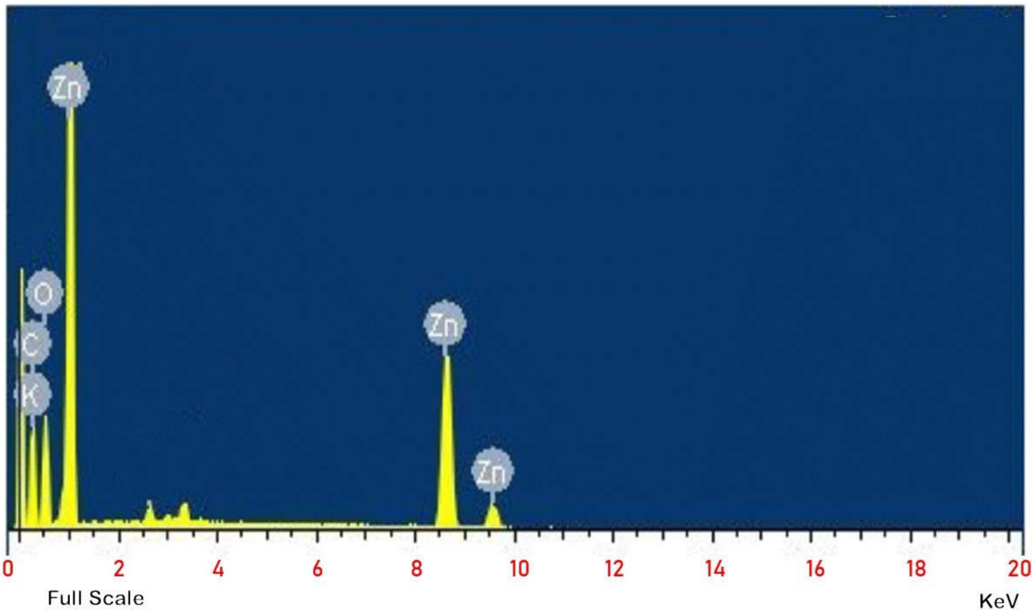

Figure 6. EDAX pattern for the green synthesized ZnO-NPs (Zn—zinc, O—oxygen, and C—carbon).

3.5. FT-IR Analysis

FT-IR spectroscopy was used to establish the presence of the Zn–O bond and its formation mechanism, as well as to detect the photo components that coat the surface of the ZnO-NPs. Fourier-transform infrared spectroscopy was performed using a Bruker Alpha FTIR as the instrument. Figure 7 depicts the FT-IR spectra of the ZnO-NPs that were synthetically generated via the green route. Both the 3610 cm^{-1} and 3822 cm^{-1} spectral peaks are the consequence of O–H stretching. The C–H stretch is responsible for the peak that appears at roughly 2354 cm^{-1}. The peak is induced by C=O stretching and is located at about 1512 cm^{-1}. There is a link between the peaks at 1635 cm^{-1} and ZnO vibrations caused by bending deformation. At 610 cm^{-1}, strong vibrational bands are produced as a result of the stretching modes utilized to form the ZnO nanoparticles. The phytoconstituents of P. granatum prevent the aggregation of ZnO-NPs during their production [22,23,28,29]. This is achieved by stabilizing the nanoparticles' surface.

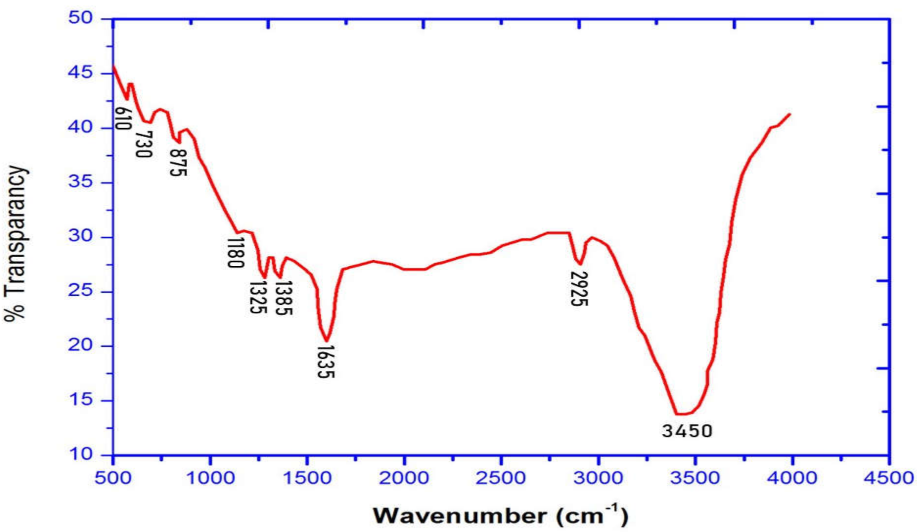

Figure 7. FTIR spectrum of ZnO-NPs.

3.6. Photocatalytic Activity

To evaluate the photodegradation of textile Orange 16 reactive dye in the presence of ZnO nanoparticles, the decrease in the absorbance of the dye was determined. The decrease in the absorbance of the dye solution as a function of exposure length is consistent with a decline in the concentration of textile Orange 16 reactive dye. Over time, the concentration of the blue pigment in the dye solution progressively lost its vibrancy, until it ultimately became light blue. Figure 8 depicts the deterioration of the textile Orange 16 reactive dye stain as a function of time in the samples exposed to sunlight. It can be seen that 600 nm is the wavelength at which textile Orange 16 reactive dye has the highest absorption peak. In addition, it reveals that ZnO-NPs are capable of significantly reducing the pollutant within three hours.

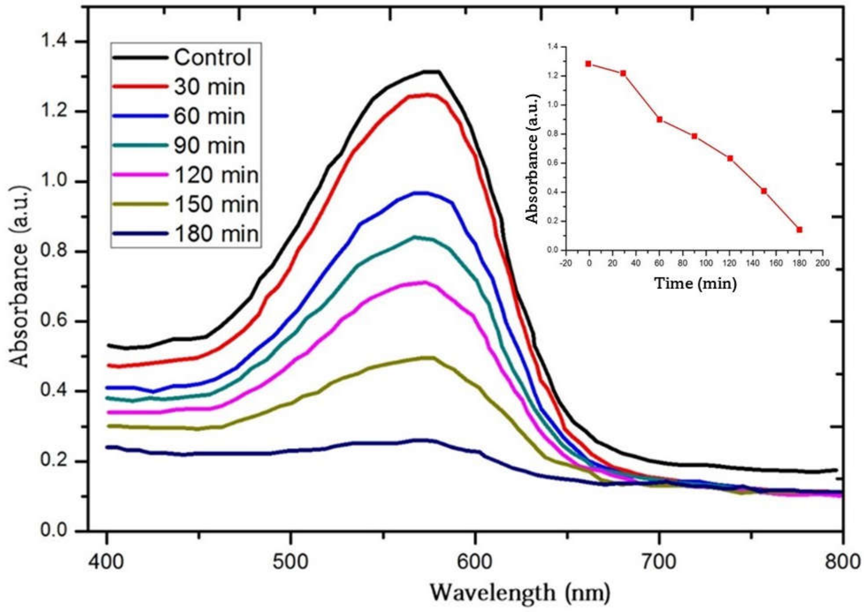

Figure 8. UV-vis absorption spectra showing the degradation of textile Orange 16 reactive dye by photocatalytic action using zinc oxide NPs synthesized via the green route at 30 min time intermissions. Inset: decrease in intensity of textile Orange 16 reactive dye in the presence of ZnO-NPs with time.

3.7. Recyclability and Photostability

Three cycles of photocatalysis were conducted to assess whether or not the photocatalyst could be recycled. The images are depicted in Figure 9. Figure 9a shows a 100% efficiency even after three cycles of photocatalysis. In addition, as seen in Figure 9b, the XRD measurements performed after the photocatalytic phenomenon show that the crystalline structure of ZnO has not altered when compared to before the photocatalytic process was undertaken.

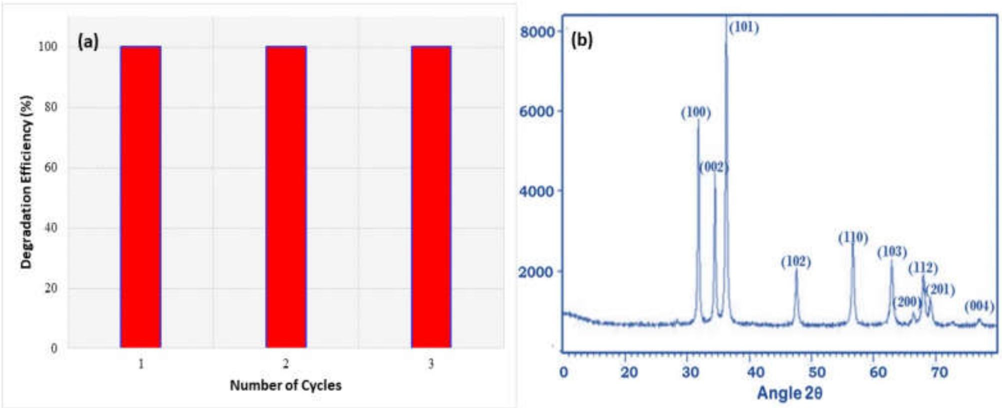

Figure 9. (**a**) The degradation profile of ZnO-NPs for three cycles and (**b**) XRD of ZnO after three cycles.

Figure 10 shows the photodegradation of the Orange 16 dye by using green synthesized ZnO nanoparticles. As observed in Figure 10, the effectiveness of the ZnO-NPs as photocatalysts for the breakdown of organic materials is shown by the decline in the color intensity of the dye with respect to the time of exposure to sunlight.

Figure 10. The color intensity before and after photodegradation using green synthesized ZnO nanoparticles.

3.8. Photolysis

There are three types of photocatalytic dye degradation mechanisms: (1) dye sensitization via charge injection, (2) indirect dye degradation via oxidation/reduction, and (3) direct photolysis of the dye. Photocatalytic substances are generally classified into three generations. One-component (e.g., ZnO and TiO_2) and multi-component semiconductor metal oxide (e.g., ZnO-TiO_2 and Bi_2O_3-ZnO) photocatalysts are classified as first-generation and second-generation, respectively. Third-generation photocatalysts are photocatalysts that are dispersed on an inert solid substrate (e.g., Ag-Al_2O_3 and ZnO-C).

Because some dyes are degraded by direct UV irradiation, it is necessary to investigate the extent to which textile Orange 16 reactive dye is photolyzed in the absence of a photocatalyst. In the absence of the photocatalyst, direct UV radiation exposure photolyzed textile Orange 16 reactive dye by up to 25% in 30 min. When ZnO-NPs were used as a photocatalyst and exposed to UV radiation, it was discovered that the photodegradation of the dye increased by decolorizing the textile Orange 16 reactive dye with an efficiency of 96% in 30 min. The time-dependent photocatalytic degradation and the photolysis of textile Orange 16 reactive dye concentration are shown in Figure 11.

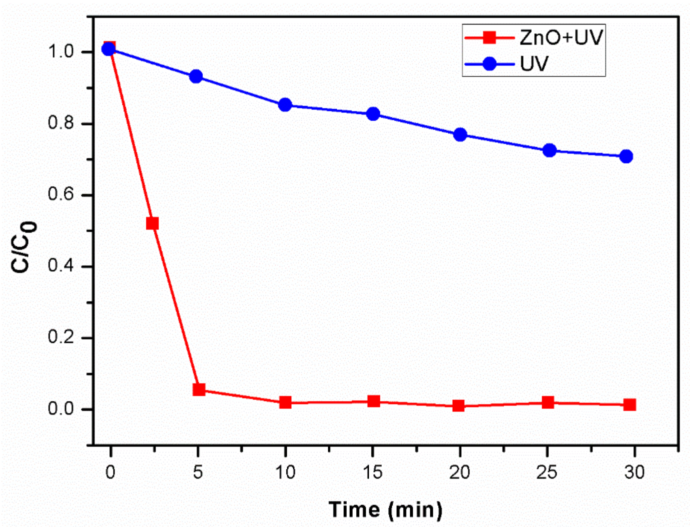

Figure 11. Time-dependent photocatalytic degradation and photolysis of textile Orange 16 reactive dye concentration (C/C_0).

3.9. Comparison

The dye photodegradation in the presence of the synthesized ZnO-NPs was compared to a commercial ZnO and previously reported work. The results are tabulated in Table 3.

Table 3. Comparison of ZnO-NPs as a photocatalyst for dye degradation.

Sl. No	Photocatalyst	Time (min)	% Photocatalytic Degradation of Dye	Reference
1	ZnO	180	94	Present work
2	ZnO	180	95	Commercial ZnO
3	ZnO	160	86.9	[17]

Among the tested ZnO NPs, commercial ZnO performed the best when compared to other ZnO NPs.

4. Conclusions

In the current investigation, an extract of the leaves of *P. granatum* was dissolved in water and utilized in a green synthesis process, which resulted in the successful generation of ZnO-NPs. The preparation of spherical, polydisperse ZnO-NPs with sizes ranging from 50 to 100 nm and an average size of 80 nm was performed. The majority of the nanoparticles are spherical and have an 80 nm diameter. According to the results of the EDX analysis, the ZnO powder has a very high degree of purity and includes nearly no impurities. The powder consists of 76% zinc and 15% oxygen. According to the results of the photocatalytic experiment, the bio-produced ZnO-NPs are able to photodegrade the textile Orange 16 reactive dye with an overall efficiency of 93%. The photolysis of the textile Orange 16 reactive dye concentration without and with ZnO-NPs shows that the ZnO-NPs play the role of photocatalysts effectively. According to the results of this work, the manufacture of ZnO-NPs using the *P. granatum* leaf extract is safe, inexpensive, straightforward, and environmentally friendly, and these nanoparticles have shown efficacy as green photocatalysts in the actual treatment of wastewater.

Author Contributions: Conceptualization, S.A.A.-Z. and M.B.P.; methodology, S.N.M. and A.Y.P.; software, A.A.O; validation, N.M., D.M. and A.K.; formal analysis, A.M.; investigation, E.S.; project administration, S.A.A.-Z. All authors have read and agreed to the published version of the manuscript.

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