J. Water Environ. Nanotechnol., 8(3): 206-214 Summer 2023

ORIGINAL RESEARCH PAPER

An experimental study on photocatalytic degradation to free river water from toxic dye pollutant using Zn doped TiO_2 nanoparticles

Sarathi R.[1, 3*], N. L. Sheeba[1], Selva Esakki E [1, 4], Renuga Devi L[1], Meenakshi Sundar S.[2]

[1] Research Scholar, PG and Research Department of Physics, Sri Paramakalyani College Alwarkurichi, Tamilnadu, India

[2] Principal, Sri Paramakalyani College, Alwarkurichi, Tamilnadu, India

[3] Assistant Professor, Department of Physics, Kamaraj College, Thoothukudi, Tamilnadu, India

[4] Assistant Professor, Department of Physics, Vyasa Arts and Science Women's College, Subramaniyapuram, Tamilnadu, India

Received: 2023-01-28 Accepted: 2023-04-28 Published: 2023-08-5

ABSTRACT

Water pollution by organic pollutants is an ever-increasing problem of global concern. The present study is aimed at synthesizing Titanium dioxide nanoparticles under two different concentrations of Zinc as a dopant material. The synthesized nanoparticles are used as catalysts in degrading malachite green dye an organic pollutant. The morphological studies of zinc-doped Titanium di Oxide nanoparticles were carried out using different spectroscopic and microscopic tools. From the XRD Spectra average crystallite size, lattice parameters, and the unit cell volume are studied. The bandgap of the material was found via using UV-Vis absorbance Spectroscopy. Fourier Transform Infrared Spectroscopy confirms the functional group present in the sample. Under light illumination, metal oxide nanoparticles act as a good photocatalyst in converting a harmful material into a less harmful one. In this aspect, the malachite green dye prepared from river water is degraded under the illumination of visible light. Almost 95% of degradation in 60 min is observed reporting the Zinc doped Titanium dioxide as an eminent photocatalyst.

Keywords: Zinc, Titanium di Oxide, Photocatalysis, Malachite Green Dye, Concentration.

How to cite this article

R. Sarathi, N. L. Sheeba, E. Selva Esakki, L. Renuga Devi, S. Meenakshi Sundar. An experimental study on photocatalytic degradation to free river water from toxic dye pollutant using Zn doped TiO2 nanoparticles. J. Water Environ. Nanotechnol., 2023; 8(3): 206-214. DOI: 10.22090/jwent.2023.03.001

INTRODUCTION

The excess presence of organic dyes from industries like paper and pulp, textiles, food, etc creates great contamination of the environment. According to several reports, 10 – 12% of dyes like Malachite Green, Rhodamine B, Indigo Red, Methylene Blue, Black –T, Carmine, Red 120, and Thymol blue are used in several industries in which the major amount (20%) is discharged into wastewater after synthesis and processing operations [1,2,3]. Living organisms get harmed because of the dye adulterants which are non-recyclable, highly toxic, and contain colored pigments [4]. The aquatic environment gets contaminated due to these hazardous dye molecules even at a very low concentration (< 1 ppm) [5]. Therefore, clearing these dyes from water is more predominant.

In recent years, clearing these dye contaminants from aquatic environments is a risky and challenging task [6]. Various methods like ozonation [7], ion exchange removal [8], adsorption [9], membrane filtration [10], biological/aerobic treatment [11], catalytic reduction [12], and photocatalytic degradation [13] are made use to control the same problem. Due to its simplicity in performance and profitable absorption process, it seems to be a supportive method for wastewater treatment. Due to difficulties such as the removal of pollutants, poor reusability of adsorbent, and low efficiency,

* Corresponding Authors Email: *sarathi.tuty@gmail.com*

this method is not suitable for the removal of pollutants [14]. Recently, the photocatalytic degradation of organic pollutants is highly noticed due to its complete degradation of contaminants in water without producing any secondary toxic material [15].

The heterogeneous photocatalysis seems to be an advantageous one in which the catalyst and reaction mixture are in different phases. The catalyst which is used in this work is easy to recover and can be reused. The catalyst used in this process plays a major role [16]. Titanium dioxide seems to be an effective photocatalyst but its wide bandgap (3.2 eV) restricts its usage in visible light regions. TiO_2 is a commodity chemical with numerous applications in the industry; however, it has recently gained attention in environmental remediation efforts for areas affected by persistent pollution. Although TiO_2's photocatalytic properties have been commercialized through the formulation of materials such as self-cleaning window films and air-purifying roofing tiles, their applications in wastewater treatment processes remain limited [17]. Other semiconductors with similar bandgaps to TiO_2, such as ZnO and CdS, have demonstrated promising photocatalytic properties, but have limitations in terms of catalyst stability and environmental toxicity.

These days, many semiconductor transition metals are doped with titanium dioxide to modify their properties. Zinc material has an equivalent photocatalytic property to titanium dioxide like photocatalytic ability and stability [18]. Since titanium dioxide has a quantum efficiency of less than twenty which confines the separation of electron and hole pair. To enhance the photocatalytic oxidation/ reduction property of TiO_2, Zinc ion is doped with it.

A simple and reasonable microwave-assisted solvothermal method is utilized to synthesize zinc-doped titanium dioxide nanoparticles. The structural and morphological analysis is investigated by XRD and FESEM. The optical analysis of the synthesized zinc-doped titanium dioxide nanoparticles is studied by UV-Vis, FTIR, and PL. The photocatalytic properties of synthesized samples are experimentally tested for malachite green dye degradation using pure river water collected from areas of veeravanallur district which is the novelty of this current work.

EXPERIMENTAL SECTION

Materials Used

In the present work as a precursor of metal ions, Titanium Tetrachloride and Zinc Acetate Dihydrate are used. Ethylene glycol and urea are also used. All the chemicals are purchased from Merck and are of analytical grade.

Synthesis of Zinc doped TiO_2 nanoparticles

Nanoparticles can be synthesized in many ways, though microwave-assisted solvothermal seems to be cost-effective and produces more yield in a very short time [19]. For the synthesis of zinc-doped titanium dioxide microwave-assisted solvothermal method is used. As a first step, titanium tetrachloride/ Zinc acetate dihydrate (0.3 % and 3%) and urea are taken in a ratio of 1:3. Urea is used in this method for the formation of oxide, it also acts as a catalyst to speed up the reaction. Later on, the raw materials are made to dissolve in ethylene glycol using a magnetic stirrer for 60 min. The entire solution is then poured into a ceramic bowl and placed in a microwave oven. The solution is microwave treated for 30 seconds per cycle with an interval of two minutes. Due to this microwave heating, the solvents start to evaporate and the precipitates settle down [20]. The samples are then collected and cleaned well with double distilled water and acetone to remove unwanted impurities present in the sample. The samples are dried under direct sunlight [21]. As a final step, the samples are annealed at 300° C using a muffle furnace.

Photocatalytic Experiment

The samples synthesized are tested for the photocatalytic degradation of malachite green dye by the means of photocatalysis process. The dye solution for this experiment is prepared from river water and raw malachite green dye powder obtained from the reed mat industries of the Veeravanallur district. After illumination with a visible lamp (150 watts), in the photocatalytic condenser setup, the solution is collected, and centrifuged and its corresponding concentration values are recorded using an UV-Vis spectrophotometer. The efficiency of the dye is calculated using equation 1 [22]

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

RESULT AND DISCUSSION

XRD Analysis

The XRD spectra of zinc (0.3% and 3%) doped titanium dioxide nanoparticles are shown in Fig. 1. From the XRD patterns the diffraction peaks of 2θ 25.3°, 37.9°, 48.4°, 54.7°, 62.9° and 71.4° are well matched with their hkl values (101), (004), (200), (105), (204) and (215), respectively (JCPDS Card No: 01-073-1764) [23]. All the peaks represent the crystalline nature of the sample and its phase is purely in the anatase form. The average crystalline size calculated from De-bye Scherrer's equation (D

Table 1: Lattice Parameters and Cell Volume of Zinc (0%, 0.3%, 3%) doped Titanium Oxide.

Sample Code	Lattice Parameters (Å)				The volume of the Cell (Å³)	
	Standard Value (a,b)	Calculated Value (a,b)	Standard Value (c)	Calculated Value (c)	Standard Value	Calculated Value
Zn (0%) doped TiO_2	3.7892	3.7874	9.5370	9.5109	136.93	136.43
Zn (0.3%) doped TiO_2	3.7830	3.7551	9.5100	9.3943	136.10	132.47
Zn (3%) doped TiO_2	3.7760	3.7653	9.4860	9.4677	135.25	134.23

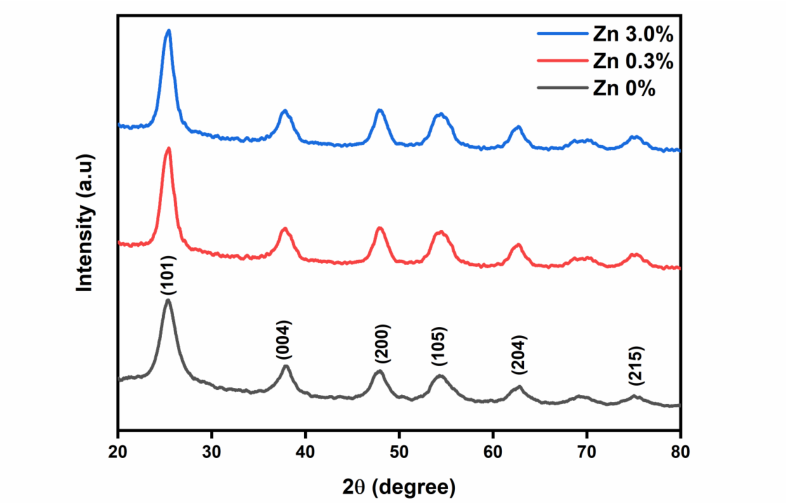

Fig. 1. XRD spectra of TiO_2 doped with (a) 0 % (b) 0.3% Zn and (c) 3% Zn.

= Kλ/βcosθ) [24] is 12.12 nm, 13.33 nm, and 17.16 nm for Zinc concentration of 0%, 0.03%, and 3% in titanium dioxide. An increase in crystalline size is observed when the concentration of Zinc moves from 0.3% to 3% of zinc concentrations. The system in which the synthesized sample is anatase with the space group 141/amd. The lattice parameters a=b and c is calculated using the peaks at (200) and (004) and are compared with their standard a, b, and c values. No characteristic peak of zinc is seen in the XRD pattern. Table 1 provides the characteristics data from the XRD spectra.

FESEM Analysis

The size and shape of the synthesized nanoparticle are characterized by using FESEM analysis. Fig. 2(a) and (b) provides the micrograph image of Zn- 0% and 3% doped TiO_2 nanoparticle. A large number of dispersed nanoparticles [25] with an average particle size of 15.66 nm (Zn 0%) and 18.79 nm (Zn -3%) are observed from the FESEM image. A uniformly sized nanoparticle with a spherical structure is seen for Zn-doped TiO_2 nanoparticles. Figs. 2 (c) and (d) report the EDS graph of pure and Zn 3% doped TiO_2 Nanoparticles. From the EDS spectra the weight percentage of the elements Ti and O for pure TiO_2 is obtained as 67.34% and 23.82% and for Zn, Ti and O for Zn doped TiO_2 is obtained as 49.86%, 47.19%, and 2.96%, respectively.

UV-Vis Analysis

The optical absorption properties are studied by using UV-Vis absorbance spectra in the ranges from 200 nm to 800 nm. Fig. 3(a) points to the absorbance spectra of Zinc (0%, 0.3%, 3%) doped titanium dioxide nanoparticles. An alteration in the TiO_2 bandgap energy and shift in absorbance peak is observed when a transition metal ion gets incorporated into the TiO_2 lattice making the dopant level appear between valance and conduction band [26]. Hence the capacity of TiO_2 to absorb light in

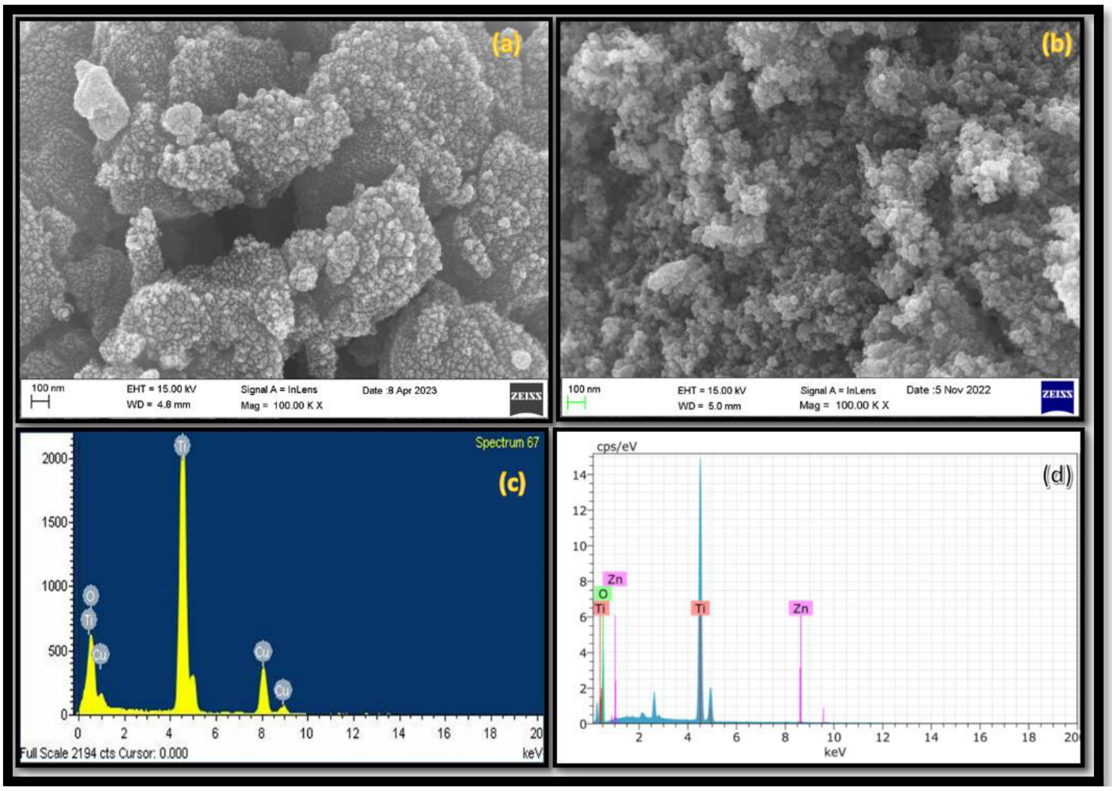

Fig. 2. FESEM Image of (a) Zn 0% (b) Zn 3% doped TiO_2, EDS spectra of (c) Zn 0% (d) Zn 3% doped TiO_2

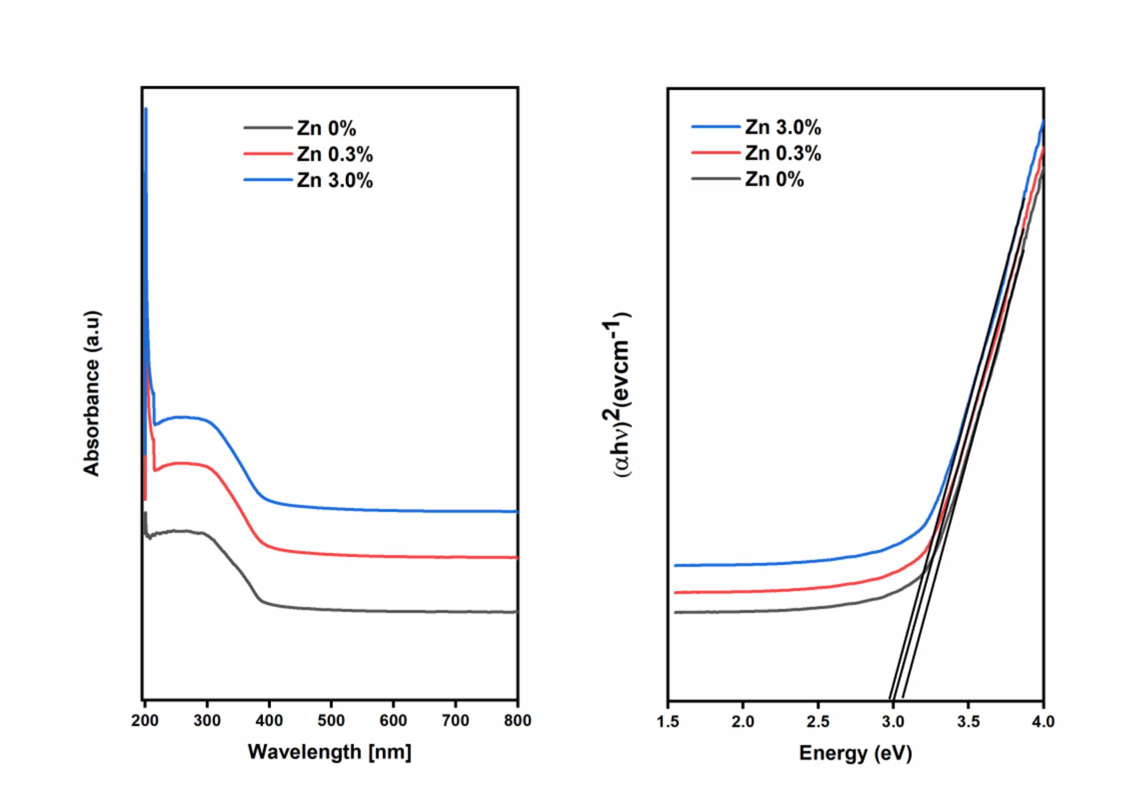

Fig. 3. UV-Vis absorbance spectra of (a) Zn-0.3%, (b) Zn- 3% doped TiO2, Tauc plot of TiO2 doped with (c) Zn – 0.3%, and (d) Zn -3%.

visible regions is enhanced. An increase in shift is noted when the concentration of Zn increases from 0% to 3% [27]. From the Tauc relation, plot between energy and $(\alpha h\upsilon)^2$ [28], the bandgap of the Zn 0%, 0.3%, and 3% doped TiO_2 is calculated as 3.05, eV, 3.00 eV, and 2.96 eV, respectively. The results indicate the incorporation of Zinc ions in the TiO_2 lattice has led to an extended photo response of TiO_2 to a higher wavelength region.

FTIR Analysis

Fig. 4 shows the FTIR spectra of Zn ion-doped TiO_2. The peak at 3217 cm^{-1}, 3178 cm^{-1} 1636 $cm^{-1,}$ and 1633 cm^{-1} might be due to the stretching and bending vibration of the water-absorbed OH group on the surface of the nanoparticle. The broad peak around 600 cm^{-1} – 400 cm^{-1} is assigned to the metal oxide vibrations i.e. to Ti–O stretching and O–Zn–O flexion vibration, respectively [29]. The peak at 1430.67 cm^{-1}, 1437.80 $cm^{-1,}$ and 1063.26 cm^{-1}, 1071.19 cm^{-1} is due to OH bending and CO stretching of alcohol aroused by the usage of acetone during the cleaning process.

PL Analysis

PL spectra have been used to study the efficiency of charge carrier trapping, immigration, transfer, and rate of electron-hole pair recombination in semiconductor nanoparticles. Fig. 5 shows the photoluminescence spectra of Zn ion doped titanium dioxide nanoparticles annealed at 300°C. The excitation PL intensity decreased when the concentration of zinc ion doping increased from 0.3% to 3%. This implies that the amount of zinc ion concentration might slow down the photo-generated electron and hole pair radiative recombination [30]. Hence it may assure that an enhancement in photocatalytic activity might be absorbed for the PL spectra with lower intensity. There might be two reasons for the decrease in PL intensity; One is a decrease in the effective area for absorbing the light of TiO_2 nanoparticles and the other is the decrease in surface oxygen vacancy due to the many chemical bonds of Ti–O–Zn three elements [31].

Photocatalysis Analysis

Dependence of Time

It is well known that the amount of adsorption and the number of active sites on the catalyst surface is crucial for efficient degradation. The percentage of MG dye adsorption on the catalyst surface was calculated by comparing its concentration before and after stirring. Fig. 6 (c) shows the curve for the degradation of malachite green dye using pure and zinc doped Titanium dioxide as a catalyst. The concentration of the dye molecule from the river water starts to decrease when a visible lamp of 150 watts is illuminated. For the successful degradation experimental study of malachite green dye from river water, 0.05g of catalyst is used in 100 mg/l of dye. When the light illumination time increases, the concentration of dye molecules in water decreases resulting in an increase in degradation efficiency [32, 33]. Fig. 6(a) shows the bar graph for the dependence of light illumination time over the degradation efficiency. The degradation efficiency of 95% is observed in a time of 60 min.

Reaction kinetics

The photocatalytic degradation kinetics of malachite green dye is carried out with different conditions ((irradiation time = 60 minutes, initial pH of dye solution = 7, initial dye concentration=100 mg/L, amount of catalyst used = 50 g) and with a continuous atmospheric oxygen supply. Using the pseudo-first-order kinetic relation $\ln (C_0/C) = kt$ where C_0 and C are the initial and final dye concentration at time 0 and t the degradation reaction rate is calculated [34]. The degradation of MG dye is observed as a function of light illumination time and $\ln (C_0/C)$. The linear fit of the curve provides the degradation kinetics rate value following the pseudo-first-order as 0.05895±0.0081 and 0.05666±0.00144 for the catalyst titanium dioxide doped with zinc of 0.3% and 3%. Fig. 6 (d) provides the degradation plot of $\ln (C_0/C)$ vs time of 60 min. Fig. 7 provides the mechanism of photocatalysis.

Effect of initial dye concentration

The effect of initial dye concentration over the photocatalytic degradation using Zn-doped TiO_2 as a catalyst is studied. Four different initial concentrations such as 100 ppm, 200 ppm, 300 ppm, and 400 ppm are chosen for testing the dependence of the initial dye concentration of the synthesized sample. Fig. 6 (b) shows the dependence of initial dye concentration over the degradation efficiency of degrading malachite green dye. When the concentration of dye increases the efficiency of degradation decreases and the time for degradation also increases from 60 min to 90 min. The higher concentration of dye blocks the entry of visible lamp

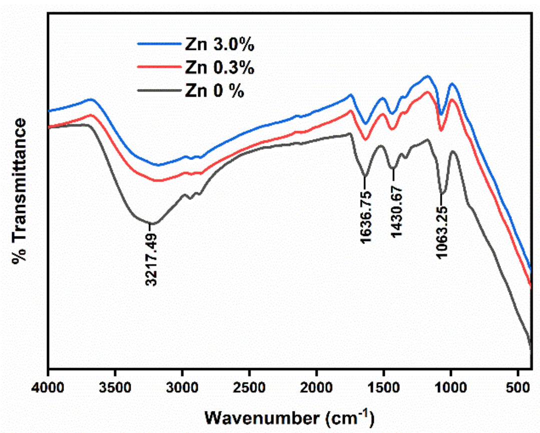

Fig. 4. FTIR spectra of pure and Zn doped TiO_2 nanoparticles.

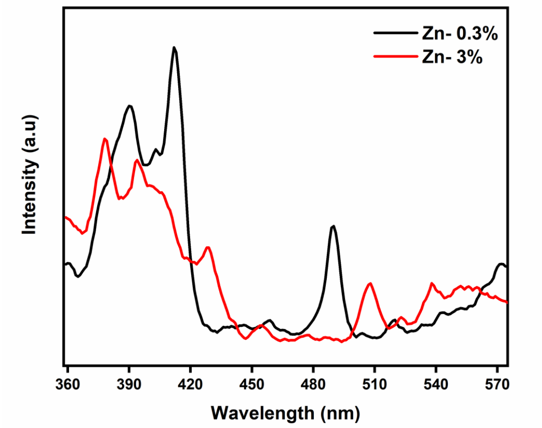

Fig. 5. Photoluminescence spectra of Zn doped TiO_2 nanoparticles.

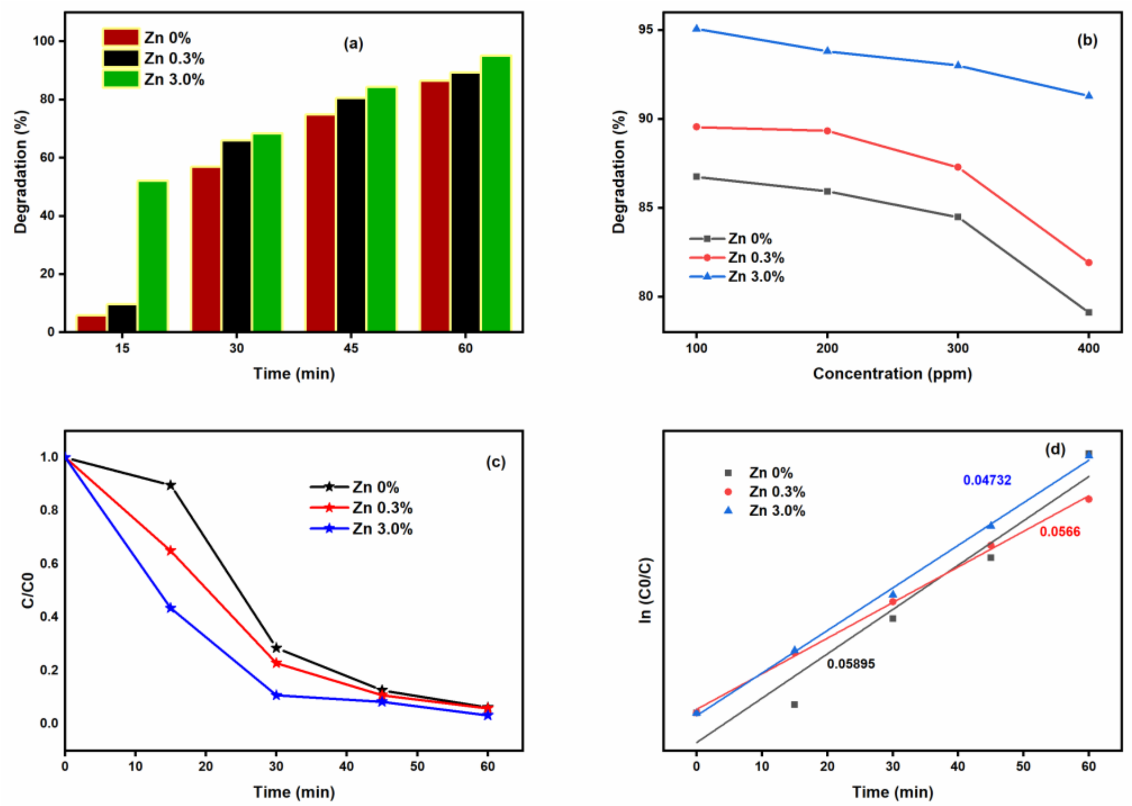

Fig. 6. (a) Degradation Efficiency, (b) Dependence of initial dye concentration, (c) Photocatalysis Degradation Curves (d) plot of $\ln(C_0/C)$ on degradation of MG in 60 min.

and also the electron and hole pair that originate upon the visible lamp illumination has lowered, hence resulting in a decrease of degradation efficiency [35]. The degradation efficiency gets lowered from 95 % to 91 % when the concentration of dye in river water increases from 100 ppm to 400 ppm.

Effect of Initial Dye pH

The initial pH of the dye solution plays a key role in wastewater degradation using photocatalysis. In this work, the initial dye solution pH is adjusted by using Hydrochloric acid and Sodium Hydroxide pellets. The effect of pH level is studied at pH- 1, 3, 5, 7, 9, 11, and 13 for all the synthesized catalysts.

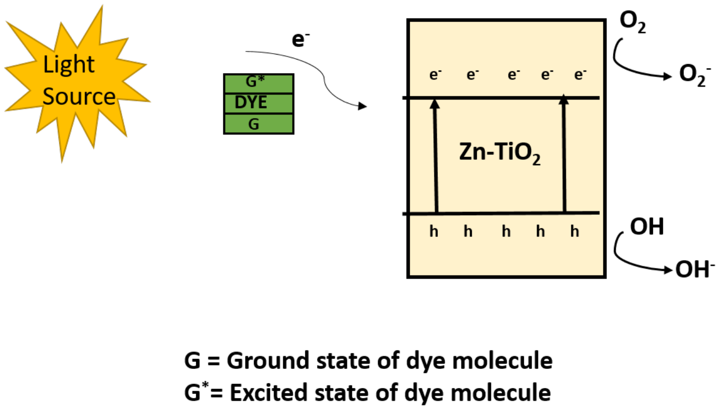

Fig. 7. Mechanism of Photocatalysis

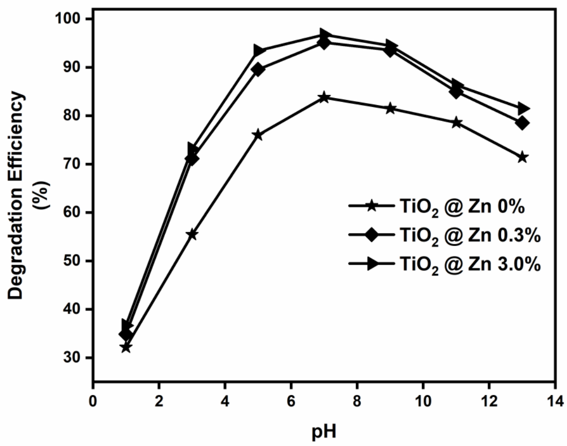

Fig. 8. Dependence of initial dye pH on degradation of MG in 60 min.

As the pH value of the dye solution changes, the surface charge gets altered which gives way for molecules to absorb or repel depending on the charge of a molecule [36]. As malachite green is a cationic dye, the molecules with negative charge trigger adsorption [37]. Fig. 8 shows the photocatalytic performance at various initial pH levels of MG dye solution. All the other parameters like catalyst load (0.05g), dye concentration (100 PPM), and temperature (room temperature) are kept constant throughout the experiment. The efficiency of degradation is observed as 87.7%, 95.12%, and 96.72% at initial pH 7, in other initial dye pH values the degradation efficiency is decreased.

CONCLUSION

Nanosized zinc-doped titanium dioxide nanoparticles are successfully synthesized through the microwave-assisted solvothermal method. The two different concentration of Zinc in titanium dioxide has affected the crystalline size and the bandgap of the synthesized material. The experimental study on the photocatalytic activity of malachite green dye degradation using the synthesized compound as a catalyst has resulted in good photocatalytic efficiency of 95% in a short time. The inclusion of zinc in titanium dioxide has enhanced the photocatalytic efficiency in degrading malachite green dye. The influence of the initial dye concentration study resulted in a decrease in efficiency with an increase in initial dye concentration. To conclude that Zn concentration of 3% on TiO_2 is suggested as an efficient photocatalyst in degrading malachite green dye.

ACKNOWLEDGMENT

The authors would like to express their gratitude to UGC DAE CSR Indore, DST FIST Sponsored Centralized Research Facility, Sri Paramakalyani

College, Alwarkurichi for providing technical support.

CONFLICT OF INTEREST

The authors hereby declare that there is no conflict of interest.

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