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ISSN: 2455-0191



Green Synthesis of MoO₃ Nanoparticles by Reflux Method for Efficient Photocatalytic Degradation of Methylene Blue Dye

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ARTICLE DETAILS

Article history:

Received 20 July 2026

Accepted 19 August 2026

Available online 23 August 2026

Keywords:

MoO₃

Photocatalysis

Degradation

Green Synthesis

ABSTRACT

The present study reports a green approach for the synthesis of MoO₃ nanomaterial and its application as a photocatalyst for the degradation of methylene blue (MB) under natural solar irradiation. The structural, morphological, compositional, vibrational, and optical properties of the synthesized material were investigated using X-ray diffraction (XRD), scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDS), Raman spectroscopy, and UV-Vis spectroscopy. XRD analysis confirmed the formation of crystalline orthorhombic MoO₃ with a crystallite size of approximately 24 nm, along with a minor contribution from structural disorder. SEM observations revealed a sheet-like morphology, while EDS confirmed the presence of molybdenum and oxygen in the synthesized material. Raman analysis indicated the presence of oxygen-vacancy-related defects in the MoO₃ structure. The UV-Vis study showed an optical band gap of 2.93 eV, with the observed red shift attributed to defect states associated with oxygen vacancies. These defects can provide additional active sites and facilitate the photocatalytic process by improving the interaction of the material with the dye molecules and incident solar radiation. The photocatalytic performance was evaluated using MB as a model organic pollutant in aqueous medium under natural solar irradiation at an ambient temperature of approximately 25–30 °C. The synthesized MoO₃ exhibited appreciable photocatalytic activity, achieving about 94% degradation of MB within 80 min. The degradation followed apparent first-order kinetics, with a rate constant of 0.03565 min⁻¹. The results indicate that the synthesized MoO₃ nanomaterial possesses suitable structural and optical characteristics for solar-assisted photocatalytic degradation and can be considered a promising material for the treatment of dye-containing wastewater.

1. Introduction

To solve numerous problems, the exploration of various nanomaterials is under the vision of scientists. As the circumstances vary, so do the techniques to handle them [1,2]. Metal oxide nanomaterial proves to be a great resource for many applications such as photocatalysis, solar cell applications, sensors, display material, batteries, biological applications, etc. [3-5].

The common threat to life is the synthetic dyes used in cosmetics, textile, paper, and food industries, containing toxic elements like heavy metals with atomic weights between 63 and 200, making their way to the water bodies having a great impact on the ecosystem [6,7]. The textile industry uses some azo dyes that decompose into toxic aromatic amines, leading to fatal health problems, and the cosmetic field dyes cause skin issues like irritation, redness, allergies, etc. [8], hence, many such dyes have been restricted by the government in order to reduce such fatal pollutants. Still, a large number of dyes is used, which needs immediate and effective solution [9].

In the past decades, extensive research has been performed looking for the answers. Such a study is done by taking a transition metal oxide, i.e., molybdenum oxide (MoO₃), for the purpose. Up till now, diverse oxides have been explored of titanium, tin, manganese, iron, silver, zinc, tungsten, bismuth, cerium, and molybdenum [7,10].

Among these, MoO₃ has attracted a lot of attention following layered structure, multiple oxidation states, high thermal chemical stability and low toxicity [11-13]. Further, the TMO viz. MoO₃ comprises tetrahedral and octahedral gaps in its lattice, offering an easy channel for

photocatalytic transitions [14]. The multiple oxidation states help to maintain oxygen vacancies and increase interlayer distances [12]. It has an impact on lattice distortion and finally on photocatalytic activity [12,15,16]. Moreover, the synthesis techniques used to synthesize the material are physical (laser ablation, ball milling, thermal decomposition, sputtering, etc.) [17-20] or surfactant-based chemical methods (as solvothermal, hydrothermal, sol-gel method, chemical precipitation, etc.) [21,22] which exhausts harmful byproducts to the environment, just a handful of modalities follow the green way and one such method is reflux method by which the nanomaterial is synthesized here [23].

The reflux method allows to follow the route that least exhausts harmful gases to synthesize the prescribed nanoparticles [24,25]. The synthesized material is crystalline in nature and aligns well with photocatalysis application with constructive results without any addition of external agents like CO, etc.

The present study demonstrates the green synthesis of sheet like and orthorhombic molybdenum trioxide nanostructures to overcome the challenges of catalysts loading and artificial radiation in the existing photocatalysis applications.

2. Experimental Methods

2.1 Materials

Ammonium heptamolybdate tetrahydrate (NH₄)₆Mo₇O₂₄·4H₂O purchased from Fisher Scientific, HNO₃ solution 25% from Loba Chemie, and double distilled water. Methylene blue dye was purchased from Rankem. Without any supplementary purification, all of the chemicals and reagents utilized in this investigation were used just as supplied.

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2.2 Synthesis of MoO₃ Nanoparticles

The nanoparticles were prepared by modifying the procedure reported by Wongkrua et al. [26]. For the synthesis, 30 mM of ammonium molybdate tetrahydrate was dissolved in 40 mL of distilled water at room temperature. The solution was stirred continuously for 10 min to obtain a homogeneous precursor solution. Subsequently, 2 M HNO₃ was added gradually with constant stirring until the solution became colorless. The addition of nitric acid was used to establish the required acidic conditions for the subsequent chemical transformation and precipitation of the precursor.

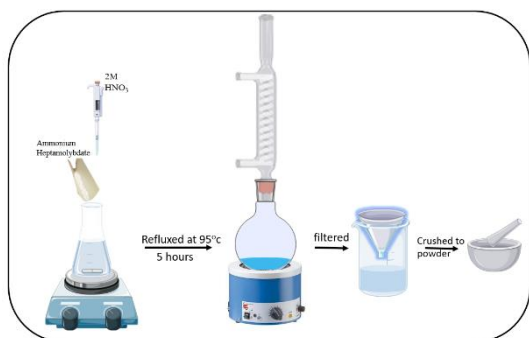


Fig. 1 Schematic of synthesis for MoO₃ by Reflux method

The resulting solution was refluxed at 95 °C for 5 h. During refluxing, the elevated temperature and acidic reaction conditions facilitate the conversion of the dissolved precursor species and promote nucleation of the solid phase. After completion of the reflux treatment, a white precipitate was obtained. The precipitate was separated by filtration and dried to remove the remaining moisture. The dried material was collected in powder form and annealed at 250 °C before being used for further studies. Schematic representation of the synthesis is shown in Fig. 1.

The formation of nanoparticles can be associated with the nucleation and growth processes occurring during the reflux treatment. Under the controlled acidic and thermal conditions, the precursor species undergo chemical transformation, followed by the formation of numerous nuclei. The subsequent growth of these nuclei is limited by the reaction conditions, favoring the development of particles in the nanoscale range. Annealing at 250 °C was carried out to improve the structural stability and crystallinity of the resulting material and to remove residual volatile species or moisture.

2.3 Photocatalytic Dye Degradation Method

The experimental work was done in the summer season at noon at an elevation of 1915 feet at 19.902 latitude and 75.310 longitude, and the solar power incident was around 1.03–1.09 kWh m⁻² day⁻¹. Methylene blue dye was used for the photocatalytic measurements of MoO₃ under solar irradiation. For the process, 10 ppm of the dye was taken in 100 mL distilled water with solution pH = 7. A quantity of sample weighing 25 mg was introduced into the solution, followed by a period of 10 minutes in darkness to achieve adsorption-desorption equilibrium. For the readings, 3 mL samples were taken every quarter of an hour. Absorption at 666 nm was measured using a UV-Vis spectrophotometer. Certain parameters, such as degradation efficiency and rate order, were calculated.

2.4 Characterizations

By the application of Cu-Kα radiation with wavelength 1.5406 Å at room temperature, the morphological characteristics are studied by Bruker D8 Advanced Diffractometer with 0.02° as step size. The photocatalytic activity was carried out by using methylene blue as a dye. The absorption coefficient and band gap were investigated using a Perkin Elmer Lambda 25 UV-Vis spectrophotometer. To study the chemical bonding of the material, Raman spectra were recorded for the range 200–1000 cm⁻¹. The characterizations have been performed at room temperature. The structural and elemental properties are analyzed with the help of SEM-EDS analysis.

3. Results and Discussion

3.1 X-Ray Diffraction Analysis (XRD)

The annealed sample shows an orthorhombic structure with a high purity of 79% crystallinity, and the peaks align well with the standard JCPDS card 05-0508 [27] as shown in Fig. 2. The morphological parameters such as lattice constant, dislocation density, crystallite size, <https://doi.org/10.30799/jnst.375.26110503>

strain, interplanar spacing, volume, and stacking fault were calculated. The crystallite size was calculated using the Debye-Scherrer formula,

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

where, D represents grain/ crystallite size, λ is the wavelength of the X-ray used, and β is FWHM.

The orthorhombic structure has characteristic equations for lattice parameters as,

$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \quad (2)$$

where, a, b, and c are lattice parameters, and (hkl) are Miller indices.

Other parameters, such as dislocation density (δ) and microstrain (ε), of the prepared samples are determined by the following Eqs.(3) and (4), respectively, and summarized in Table 1.

$$\delta = \frac{1}{D^2} \quad (3)$$

$$\epsilon = \frac{\beta \cos \theta}{4} \quad (4)$$

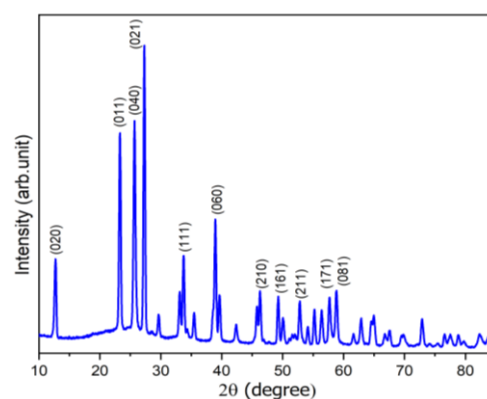


Fig. 2 X-ray diffraction pattern of synthesised MoO₃ nanoparticles

Table 1 Structural parameters of MoO₃ nanoparticles

Lattice Parameters (Å)			Volume	D	δ	ε*10 ⁻³	Crystal-	FWHM
a	b	c	(Å) ³	(nm)	(nm) ⁻²		linity (%)	
3.9636	13.86	3.6796	202.14	24	1.736	1.248	79	0.3344

The dislocations and strains in the structure were introduced in the synthesis process, and while annealing, forming oxygen defects held up the performance of the MoO₃ nanomaterial as a dye-degrading agent. Slightly amorphous nature proves the absence of oxygen atoms from some of the sites. The dislocation density and microstrain specify that oxygen impurities can be added within the space and can affect the photocatalytic activity, too. Hence, having an insight into these parameters is of great importance. The results are compared with previously reported results as it was found to be a better alternative for photocatalysis [22,26].

3.2 UV-Visible Spectral Analysis

The optical properties of the prepared MoO₃ nanoparticles were studied using UV-visible absorption spectroscopy at room temperature. The absorption spectrum shows a strong response in the ultraviolet region, which is characteristic of the electronic structure of MoO₃. The spectrum obtained for the present sample shows the main absorption maximum at approximately 287.6 nm, with an absorbance of about 0.589.

The absorption observed in the UV region is mainly related to electronic transitions associated with the Mo–O network. To estimate the optical band gap, the absorption data were further analysed using the Tauc approach. The relation used for the analysis is [16,22,26].

$$\alpha h\nu = A(h\nu - E_g)^{0.5} \quad (5)$$

where, E_g is the bandgap, hν shows the photon energy, and α represents the absorption coefficient.

Fig. 3 presents the Tauc plot obtained for the synthesized MoO₃ sample. From the extrapolation, the optical band gap was estimated to be approximately 2.93 eV. This value confirms the semiconducting nature of the prepared material and indicates that the electronic structure of the synthesized sample differs from that of highly crystalline bulk MoO₃.

The relatively lower band-gap value observed in the present sample may be associated with structural imperfections introduced during the

synthesis process. In particular, oxygen vacancies and other defect states can modify the electronic structure by introducing localized energy levels within the forbidden gap. These defect-related states may contribute to the observed shift in the absorption edge and consequently result in a lower estimated optical band gap. The additional features observed in the Raman spectrum provide further evidence for modifications in the local structural environment of the synthesized MoO_3 [14,15,24,25].

The band gap obtained in the present investigation is lower than values commonly reported for bulk or highly crystalline MoO_3 . This difference may arise from the combined influence of defects, oxygen deficiency, crystallite size and morphological characteristics of the prepared sample. Therefore, the reduced band gap should not be attributed to a single structural factor alone [8,12,17]. From the photocatalytic point of view, the observed band gap of 2.93 eV is of particular interest. A reduction in the optical band gap can increase the range of radiation that can participate in photoexcitation and may assist in the generation of electron-hole pairs. These features can be beneficial for photocatalytic reactions. However, the photocatalytic performance of MoO_3 is also governed by other factors, including surface area, defect concentration, charge-carrier recombination and the efficiency of charge separation [25,26].

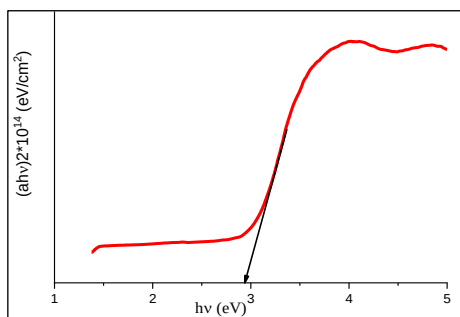


Fig. 3 Tauc plot of synthesised MoO_3 nanoparticles

3.3 Raman Analysis

Raman spectra (Fig. 4) comprise three segments depicting structural modes of vibration as follows: 600 - 1000 cm^{-1} is the stretching mode, followed by 200 - 400 cm^{-1} showing the deformation mode, and lastly, the lattice modes below 200 cm^{-1} [27]. Intense peaks were obtained at 274.33 cm^{-1} , 656.88 cm^{-1} , 810.65 cm^{-1} , and 987.14 cm^{-1} . Three peaks were visible viz. 656.88 cm^{-1} B3g-m(OMo_3), 810.65 cm^{-1} B1g-m(OMo_2) and 987.14 cm^{-1} Ag, B1g-m($\text{O}=\text{Mo}$) in the stretching mode pointing towards the stretching of $\text{Mo}=\text{O}$, Mo_2-O and Mo_3-O bonds respectively representing the orthorhombic phase. The other peaks at 233.479 represents $\text{Ag}-\delta$ (OMo_2), 274.33 $\text{B}_{2g}-\delta$ ($\text{O}=\text{Mo}$), 327.46 $\text{Ag}-\delta$ (OMo_3), 367.84 $\text{Ag}-\delta$ ($\text{O}=\text{Mo}$), 463.33 $\text{B}_{1g}-\nu$ (OMo_3) [26-31] represents bands assigned to respective wavenumber. The peaks are slightly shifted and can be associated with variation in the force constants of the bonds, i.e. the positive peak shift shows larger force constants and vice-versa [32] corresponding to stacking fault and dislocations, oxygen vacancies in the structure and oxygen defects introduced in the lattice leading to redshift [13,14].

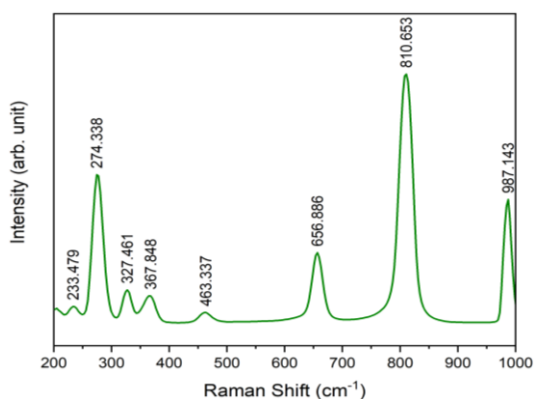


Fig. 4 Raman spectral response of synthesised MoO_3 nanoparticles

3.4 SEM-EDS Analysis

The SEM images in Fig. 5 show the sheet-like structure synthesized from green synthesis. These sheets have a large surface area, which helps degrade the dye more effectively [26]. The EDS data exhibits that pure MoO_3 has been synthesized successfully. Furthermore, the atomic percentage of Mo and O is 23.27% and 76.73%, respectively, revealing the formation of MoO_3 as they are present in the atomic ratio of nearly 1:3. <https://doi.org/10.30799/jnst.375.26110503>

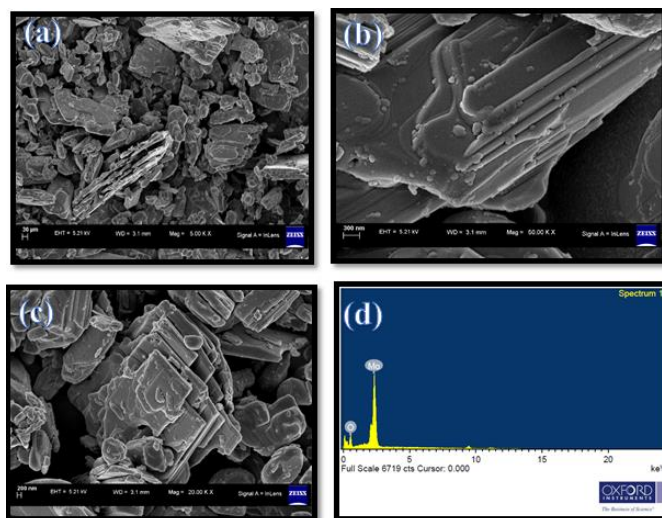


Fig. 5 Surface morphological image of MoO_3 at different scales a-c, d) EDS of MoO_3

3.5 Photocatalytic Dye Degradation

By using the absorption spectra under solar radiation, the photocatalytic activity was studied in terms of rate order kinetics. The MB dye degradation by the sample under solar radiation is illustrated in Figs. 6 and 7. For every quarter of an hour, 3 mL solution was taken for measurement of activity.

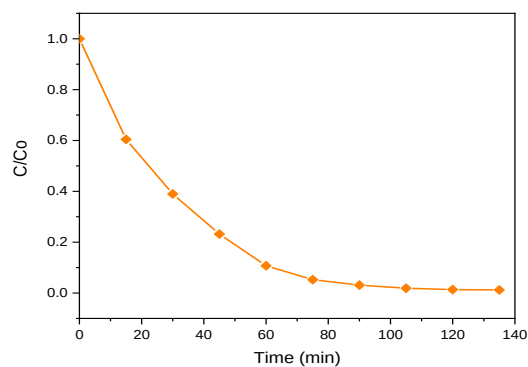


Fig. 6 The plot of $\ln(C/C_0)$ versus time for MB dye degradation

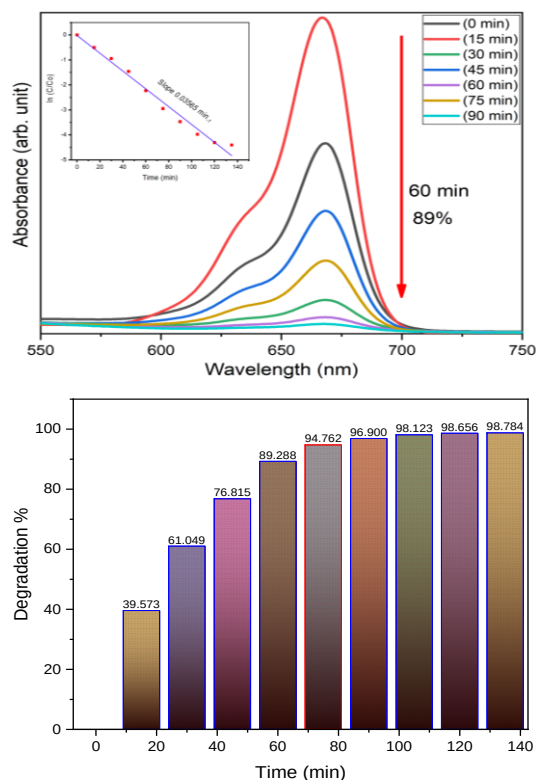


Fig. 7 Absorption spectra of MB Degradation using MoO_3 and inset of pseudo-first-order rate constant time vs $\log(C_0/C)$

In each sample, the peak was observed at 666 nm, and the first order constant was evaluated from Fig. 6; at first, the solution of dye, distilled water, and sample was kept in the dark for 10 min. Nearly negligible photoactivity was observed during this span, and subsequent large changes were observed when the solution was kept under the sun. It was observed that the reaction follows pseudo-first-order rate kinetics and is given by,

$$\log \frac{C_0}{C_t} = kt \quad (6)$$

where, C_t is the concentration at 80 mins, and C_0 is the initial concentration taken at 0 min. k represents the rate order constant. As seen from the figure, the slope specifies the rate constant and is given as ($k = 0.03565 \text{ min}^{-1}$) in Fig. 6.

Note that there is no use of any scavengers of external agents to speed up the process [28] comparatively, more efficient results are obtained. The photocatalytic degradation efficiency was calculated by the formula,

$$\text{Photodegradation efficiency(\%)} = \left[\frac{C_0 - C_{80}}{C_0} \right] * 100 \quad (7)$$

where, C_{80} is the concentration at 80 mins, and C_0 is the initial concentration taken at 0 min. It is clear from the graph that the efficiency within 80 min time is nearly 94 %. The results are compared from reports and concluded to be of higher efficiency [11,14,18,21,24,27].

3.6 Mechanism

The possible mechanism of MB degradation over MoO_3 under visible-light irradiation is proposed in Fig. 8. Since the band gap energy of MoO_3 was found to be 2.93 eV, the photoexcitation of electrons from the valence to the conduction band occurs under visible-light irradiation. As a result, holes are formed in the valence band of MoO_3 , which can oxidize the MB molecules adsorbed on the surface of the catalyst. On the other hand, the photoexcited electrons can reduce the dissolved oxygen molecules to form superoxide anions, which also contribute to the degradation process. The oxidation and reduction reactions ultimately lead to the complete mineralization of MB into CO_2 and H_2O , as shown in the proposed mechanism in the Fig. 8.

Furthermore, according to the Fig. 8, the dye-sensitized mechanism might also contribute to the observed photocatalytic activity. Indeed, under visible-light irradiation, the MB molecules can undergo electronic transitions from the highest occupied molecular orbital (HOMO) to the lowest unoccupied molecular orbital (LUMO). As a result, an electron transfer from the sensitizer to the conduction band of MoO_3 becomes possible. Therefore, both direct photoexcitation of MoO_3 and sensitization by MB can lead to photocatalytic degradation of the dye under visible light.

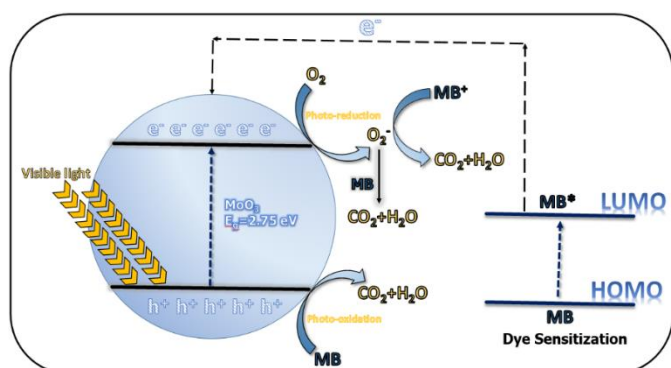


Fig. 8 Mechanism for the photocatalytic degradation of MB dye with MoO_3 NPs

3.7 Comparative Performance Analysis

Comparison of the photocatalytic performance of the present catalyst with those reported in the literature is presented in Table 2. The green-synthesized material that was obtained in the current study degraded 94% of methylene blue (10 ppm) within 80 min under natural solar radiation with the catalyst dosage of 0.05 g/L. Compared to conventional hydrothermal synthesis approaches [33,35], which typically require artificial visible light irradiation and higher amounts of a catalyst (0.1–0.5 g/L) during longer periods of time (90–210 min), the prescribed catalyst demonstrated higher material efficiency under solar radiation. The preparation of catalyst composite by microwave irradiation under UV light requires longer exposure time (300 min) and higher catalyst dosage (0.16 g/L) to achieve comparable 93% degradation of the pollutant [37]. On the other hand, previous reports on green synthesis of photocatalyst composite [38] demonstrated similar efficiency in terms of dye

degradation but have been used with artificial light sources while applied to lower concentrations of methylene blue. Thus, the key advantages of the synthesized catalyst include the lower amount of the material required for photocatalytic water treatment, higher rate of pollutant degradation within a shorter period of time, and the ability to use natural solar light instead of artificial radiation sources.

Table 2 Comparison of photocatalytic activities in current and reported studies of various studies of MoO_3 based catalysts

Synthesis Method	Dye	Radiation Source	Dye Conc (ppm)	Catalyst Conc. (g/L)	%Degradation	Time (min)	Ref.
Hydrothermal	Methylene Blue	Visible	50	0.5	~95	~210	[33]
Microwave	Ibuprofen	Visible	15	0.2	37.3	120	[14]
Hydrothermal	Methyl Orange	Visible	10	~0.6	50.7	100	[34]
Hydrothermal	Methylene Blue	Visible LED	10	0.1	99	90	[35]
Chemical Bath Deposition	Methylene Blue	UV	-	-	70	60	[36]
Microwave Assisted	Methylene Blue	UV	10	0.16	93	300	[37]
Green Synthesis	Methylene Blue	Visible	5 μM	0.08	98	90	[38]
Hydrothermal	Rhodamine B	NTP	10	0.5	97	180	[39]
Green Synthesis	Methylene Blue	Solar	10	0.05	94	80	This Work

4. Conclusion

The current investigation deals with the structural, chemical, morphological, optical, and photocatalytic aspects of the transition metal oxide MoO_3 . XRD revealed the crystalline orthorhombic structure with a little bit of amorphosity due to a stacking defect and a crystallite size of 24 nm. SEM-EDS displayed the pure composition and sheet-like structure formed in the synthesized material comprising Mo and O atoms. The oxygen vacancies are confirmed by Raman spectroscopy, leading to a decrease in the bandgap. The exchange interactions between localized electrons spin moments arising from oxygen vacancies at the surfaces were sufficiently reduced, obliterating the concentration of intrinsic oxygen vacancies. The optical studies resulted in a bandgap of 2.93 eV, which align well with the degradation results. The observed red shift in the UV-vis absorption spectrum suggests the presence of defect-related electronic states, which is consistent with the oxygen-vacancy-related features observed in the Raman analysis. These abundant active sites prove to be having assistance for photo-degradation and with 94% degradation in 80 min time. The degradation was observed under solar radiation by MB dye with a rate order constant of 0.03565 min^{-1} . Further, it was seen that the nanomaterial acts as an efficient photocatalyst and degrades the dye positively.

Acknowledgment

The authors are thankful to the Department of Physics Dr. Babasaheb Ambedkar Marathwada University Aurangabad, for giving us the lab facility to complete this work.

Funding

One of the authors Prof. Ramphal B. Sharma is thankful to the CURIE project (Ref. No. DST/CURIE-02/2023/IISU) funded by GOI DST New Delhi for the financial support.

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