



Dual-platform Photocatalysts: Evaluation of the Catalytic Properties of α -MoO₃ and ZnMoO₄ Nanosystems for Visible-light MB Degradation

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ABSTRACT

Textile effluents enriched with recalcitrant dyes demand robust, solar-assisted treatments. Here, we evaluate undoped molybdenum trioxide (MoO₃) and zinc molybdate (ZnMoO₄) as photocatalysts for methylene blue (MB) abatement and correlate performance with structure. X-ray diffraction confirms phase-pure orthorhombic α -MoO₃ (ICDD 00-005-0508) and monoclinic ZnMoO₄; Raman spectroscopy resolves the terminal Mo=O and Mo–O–Mo modes of α -MoO₃ and the ν_1/ν_3 vibrations of isolated [MoO₄]²⁻ tetrahedra in ZnMoO₄. FTIR further confirms α -MoO₃ holds Mo=O (~995 cm⁻¹), bridging Mo–O–Mo (820–870 cm⁻¹), and the ZnMoO₄ MoO₄ manifold (~1170/920/742/606 cm⁻¹), along with surface –OH/H₂O, beneficial for radical generation. FESEM shows micron-scale platelet aggregates that furnish step edges and mesovoids, while EDS verifies the intended Zn–Mo–O or Mo–O compositions without extraneous elements. Photoluminescence in the 400–520 nm window is vacancy-mediated for MoO₃ and arises from intrinsic [MoO₄]²⁻ charge-transfer emission in ZnMoO₄. Under irradiation, MB ($\lambda_{\text{max}} \approx 664$ nm) decolorizes steadily: after 150 min, degradation efficiencies are ~64% (MoO₃) and ~67% (ZnMoO₄). The time profiles obey the Langmuir–Hinshelwood, pseudo-first-order model with apparent rate constants (k_{app}) of approximately $6.9 \times 10^{-3} \text{ min}^{-1}$ for MoO₃ and $7.3 \times 10^{-3} \text{ min}^{-1}$ for ZnMoO₄. Adsorption behavior can be described by Langmuir/Freundlich/Temkin isotherms with intraparticle-diffusion contributions. These results validate α -MoO₃ and ZnMoO₄ as credible, structurally well-defined platforms for dye-laden wastewater remediation and motivate heterojunction/defect engineering for further activity gains.

Keywords: Molybdenum trioxide; Zinc molybdate; Photocatalysis; Methylene blue degradation; Langmuir–Hinshelwood kinetics; Raman/FTIR; Photoluminescence; FESEM; EDS; Wastewater treatment.

1. INTRODUCTION

The unchecked discharge of synthetic dyes from textile, printing, plastic, cosmetic, and allied industries has escalated biochemical and chemical oxygen demand, toxicity, and persistent coloration in receiving waters, demanding robust treatment technologies beyond mere phase transfer. Among available routes, semiconductor photocatalysis stands out for solar-driven mineralization of recalcitrant chromophores compared with adsorption or conventional physicochemical methods.

MoO₃, an n-type transition-metal oxide available in several polymorphs (α -orthorhombic, β -monoclinic, and h-hexagonal), has emerged as a promising photocatalyst owing to its wide band gap (~2.8–3.6 eV), high chemical stability, multiple accessible Mo valence states, and layered framework that facilitates ion intercalation and interfacial charge transport. Nevertheless, pristine MoO₃ suffers from rapid electron–hole recombination and limited visible-light harvesting, which motivates defect and heterostructure engineering. Oxygen-vacancy-rich MoO_{3-x} introduces defect states that narrow the effective gap, trap

photogenerated electrons to suppress recombination, and can participate in Z-type interfacial charge transfer in heterojunctions. Photophysically, MoO_{3-x} nanosheets show visible-to-NIR absorption with plasmonic signatures under illumination, a manifestation of Mo⁵⁺/Mo⁶⁺ intervalence charge transfer, which correlates with rapid decolorization of model dyes (MB, RhB) under visible light at room temperature using modest catalyst loadings.

Complementing MoO₃, ZnMoO₄ (ZM) is a stable, non-toxic ternary molybdate whose β -phase adopts the wolframite-type monoclinic structure (P2/c) with corner-sharing ZnO₆/MoO₆ octahedra, an architecture conducive to robust lattice bonding and inter-octahedral charge migration. Reported optical gaps for ZnMoO₄ lie in the ~3.0–3.3 eV range (phase- and synthesis-dependent), positioning it as a UV-to-near-visible responsive semiconductor. Hydrothermally prepared β -ZnMoO₄ crystals display visible-light photocatalytic activity toward organic pollutants, and ZM frequently serves as the oxidative partner in high-efficiency Z-scheme systems where its deep valence band contributes strong ·OH/·O₂⁻

generation (Sghaier *et al.* 2015a; Cavalcante *et al.* 2013a; Jiang *et al.* 2014; Mafa *et al.* 2019).

Strategically combining MoO_3 (or MoO_{3-x}) with ZnMoO_4 is therefore compelling: band-edge complementarity and defect-assisted transport can establish type-II or direct Z-scheme pathways that preserve strong redox potentials while accelerating charge separation across an extended spectral window. Early compositions of $\text{ZnMoO}_4\text{--MoO}_3$ prepared via oxide conversion already showed enhanced dye degradation relative to single phases, and recent one-step syntheses of $\text{ZnMoO}_4/\text{MoO}_3$ composites report recyclable, high-rate activity under visible light, aligning with the broader success of MoO_3 -based Z-schemes (e.g., with g- C_3N_4) in degrading dyes and emerging contaminants (Ankinapalli *et al.* 2024; Kassem, 2017; Khan *et al.* 2020).

Taken together, the defect-engineerable electronic structure of MoO_3 (including its plasmonic MoO_{3-x} manifestation) and the structural/photochemical stability of ZnMoO_4 provide a synergistic platform for constructing robust heterojunction photocatalysts. Such systems are well-positioned to deliver fast, solar-assisted decolorization and mineralization of cationic and azo dyes at ambient conditions with practical catalyst dosages, meeting contemporary needs for efficient dye-laden wastewater remediation.

2. MATERIALS AND METHODS

MoO_3 was prepared using a facile wet-chemical precipitation route. Hexa-ammonium heptamolybdate (ammonium heptamolybdate, AHM; $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}$, 0.20 mol) was dissolved in deionized water (100 mL) under magnetic stirring (30 min) to obtain a clear solution. Concentrated nitric acid (HNO_3 , 25 mL) was then added dropwise under vigorous stirring, and the mixture was maintained at 85°C for 3 h. The suspension was aged at ambient temperature for 4 h, and the solid was recovered by centrifugation (3000 rpm, 10 min). The precipitate was washed repeatedly with acetone and ethanol to remove residual ionic/organic species, dried at 120°C for 24 h, and finally calcined in air at 500°C for 3 h. The resulting powder was stored in a desiccator before characterization.

ZnMoO_4 was obtained by a facile aqueous co-precipitation/calcination route analogous to the MoO_3 procedure, using zinc acetate as the Zn source. In a first beaker, a clear solution of ammonium heptamolybdate tetrahydrate (AHM, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$) was prepared by dissolving the required amount in deionized water (100 mL) under magnetic stirring for 30 min. In a second beaker, zinc acetate dihydrate ($\text{Zn}(\text{CH}_3\text{COO})_2\cdot 2\text{H}_2\text{O}$) was dissolved in deionized water (50–100 mL). The

reagent quantities were chosen to satisfy a Zn: Mo = 1:1 atomic ratio (i.e., $n\text{AHM}=n\text{Zn}/7n$).

The zinc solution was added dropwise to the AHM solution under vigorous stirring at $80\text{--}85^\circ\text{C}$ and stirred for 3 h. The mixture was then basified with aqueous ammonia (NH_4OH) to $\text{pH} \approx 9$ to precipitate zinc molybdate. The suspension was aged at ambient temperature for 4 h, and the solid was recovered by centrifugation (3000 rpm, 10 min). The precipitate was washed repeatedly with deionized water, acetone, and ethanol to remove residual acetate/ammonium ions (to near-neutral filtrate), then dried at 120°C for 24 h. Finally, the dried solid was calcined in air at $500\text{--}600^\circ\text{C}$ for 3 h to crystallize monoclinic ZnMoO_4 . The product (denoted $\text{ZnMoO}_4\text{--pH9}$) was stored in a desiccator before characterization.

3. RESULTS AND DISCUSSION

3.1 XRD Analysis

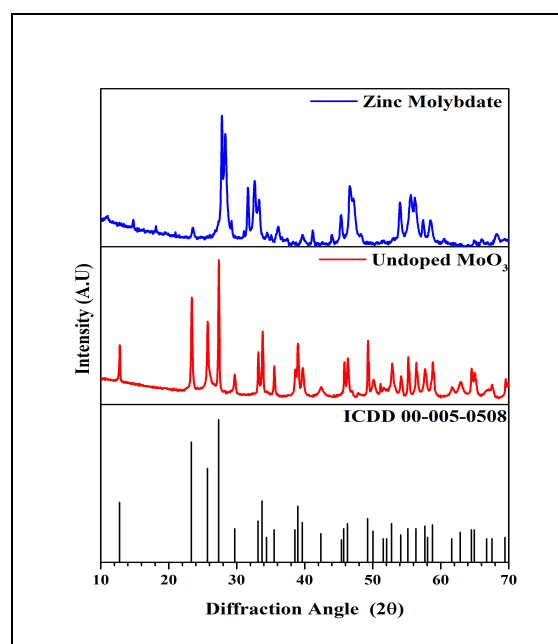


Fig. 1: XRD spectra of undoped MoO_3 and ZnMoO_4

The powder XRD patterns in Fig. 1 confirm phase-pure formation of orthorhombic $\alpha\text{-MoO}_3$ and monoclinic ZnMoO_4 , with no extraneous reflections attributable to impurity phases (e.g., ZnO , $\text{MoO}_2/\text{MoO}_{3-x}$) detectable within the instrument's limit.

Undoped MoO_3 : The diffraction maxima match the ICDD reference card 00-005-0508 for orthorhombic $\alpha\text{-MoO}_3$ (Pnma). In particular, the strong $\alpha\text{-MoO}_3$ line at $2\theta \approx 27.3^\circ$ indexed to the (021)

plane is evident, consistent with prior assignments for α - MoO_3 and its standard JCPDS/ICDD card; the h- MoO_3 reference lines are absent, supporting a single orthorhombic phase. The same card (JCPDS/ICDD 05-0508) is widely used as the crystallographic standard for α - MoO_3 , as shown in the accompanying Rietveld analyses reported for MoO_3 phases.

Zinc Molybdate (ZnMoO_4): The upper pattern is indexed to monoclinic ZnMoO_4 (space group C2/m). The measured peak positions coincide with literature values for monoclinic ZnMoO_4 ; for example, reflections at $2\theta \approx 15.99^\circ, 19.47^\circ, 22.91^\circ, 24.18^\circ, 26.13^\circ, 26.63^\circ, 27.60^\circ, 30.05^\circ, 32.25\text{--}32.34^\circ, 33.88^\circ, 40.42^\circ$ and 46.42° , were assigned to the (110), (020), (-201), (120), (0 -12), (-220), (2 -11), ($-2-21$), (012)/(102), (-212), (-330) and (-421) planes, respectively, of monoclinic ZnMoO_4 (JCPDS 35-0765). Consistent with phase- and morphology-controlled syntheses of ZnMoO_4 reported elsewhere, the diffractogram shows no low-angle lines associated with α - $\text{ZnMoO}_4 \cdot 0.8\text{H}_2\text{O}$ (triclinic; JCPDS 25-1025) and no extra peaks of mixed β/α -hydrate phases; hence, the sample can be assigned to the anhydrous monoclinic form.

The comparatively sharp, well-resolved maxima for both materials indicate good crystallinity. Peak broadening, where present, can reflect nanoscale domain size and/or microstrain; in analogous MoO_3 datasets, Scherrer/Rietveld analyses yield coherent domain sizes from tens to ~ 100 nm depending on phase and annealing, illustrating the scale expected for materials prepared by wet-chemical routes.

3.2 Raman Analysis

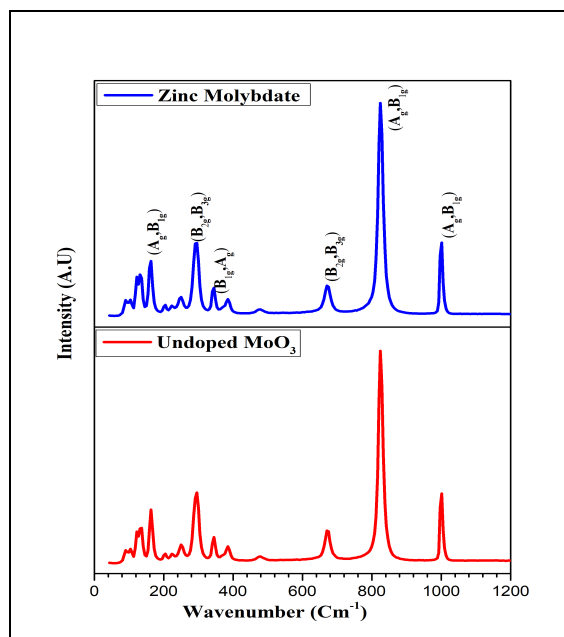


Fig. 2: Raman spectra of undoped MoO_3 and zinc molybdate

Fig. 2 presents the room-temperature Raman spectra ($100\text{--}1200\text{ cm}^{-1}$) of the undoped MoO_3 powder and the zinc molybdate sample. The spectra are diagnostic for the distinct polyhedral units that build each lattice, corner-shared MoO_6 octahedra in orthorhombic α - MoO_3 and isolated MoO_4 tetrahedra in monoclinic ZnMoO_4 , and allow confident phase assignment and qualitative assessment of crystallinity.

Undoped MoO_3 (Orthorhombic and Pbnm):

The spectrum is dominated by the terminal Mo=O stretching mode at $995\text{--}985\text{ cm}^{-1}$ (A_g), a fingerprint band of α - MoO_3 , accompanied by a very sharp Mo-O-Mo stretching band near $\approx 820\text{ cm}^{-1}$. Additional features include the asymmetric Mo-O-Mo stretch at $\approx 660\text{--}670\text{ cm}^{-1}$ and lower-wavenumber bending/lattice modes at ≈ 286 and $\approx 156\text{ cm}^{-1}$. These assignments and relative intensities are characteristic of the α -phase and arise from vibrations of the MoO_6 network; group theory for Pbnm predicts 24 Raman-active modes ($8A_g + 8B_{1g} + 4B_{2g} + 4B_{3g}$). Consistent bands are also expected/observed at $\approx 813\text{--}870\text{ cm}^{-1}$ (bridging Mo-O-Mo stretch), 560 cm^{-1} (Mo-O-Mo stretching), and $\approx 470\text{ cm}^{-1}$ (terminal Mo-O stretching in the octahedra), further supporting phase purity and good crystallinity.

Zinc molybdate, ZnMoO_4 (monoclinic). The upper spectrum exhibits three intense bands in the $810\text{--}880\text{ cm}^{-1}$ window typically resolved near $813, 859$, and 879 cm^{-1} which are assigned to the ν_1 symmetric stretching modes of the $[\text{MoO}_4]^{2-}$ tetrahedra (predominantly A_g/B_g symmetry). A weaker band at $\approx 330\text{--}340\text{ cm}^{-1}$ corresponds to ν_2/ν_4 bending of the MoO_4 units. The distribution and separation of these tetrahedral modes, together with the absence of extraneous bands from hydrate phases, are consistent with anhydrous monoclinic ZnMoO_4 .

3.3 FTIR Analysis

Fig. 3 shows the FTIR spectra ($4000\text{--}400\text{ cm}^{-1}$) of the as-prepared undoped MoO_3 , and ZnMoO_4 show the expected vibrational fingerprints of orthorhombic α - MoO_3 and molybdate lattices, respectively, together with weak surface adsorbates.

In the MoO_3 spectrum, the highest-frequency band at $984\text{--}995\text{ cm}^{-1}$ is the diagnostic terminal Mo=O stretching vibration of α - MoO_3 . The intense feature near $870\text{--}823\text{ cm}^{-1}$ arises from Mo-O-Mo stretching through the O(3) bridging oxygen, while the broader bands at $563\text{--}575\text{ cm}^{-1}$ and the weak band near 467 cm^{-1} originate from O-(3Mo) bending and low-frequency lattice modes, respectively. These assignments are consistent with

reference spectra and group-theory analyses for α - MoO_3 . Broad features at ~ 3200 – 3300 cm^{-1} (O–H stretch) and ~ 1610 – 1650 cm^{-1} (H–O–H bend) indicate physisorbed water; in hexagonal/precursor material, additional N–H bands (~ 3070 and $\sim 1415\text{ cm}^{-1}$) from residual NH_4^+ can appear but disappear after annealing to α - MoO_3 . These assignments agree with first-principles/experimental literature placing terminal $\text{Mo}=\text{O}$ near 990 – 1000 cm^{-1} , bridging $\text{Mo}-\text{O}-\text{Mo}$ around 820 – 870 cm^{-1} , and lower-frequency O–Mo–O modes below $\sim 600\text{ cm}^{-1}$ (Brookes *et al.* 2014; Ding *et al.* 2012; Ratnaparkhe *et al.* 2021).

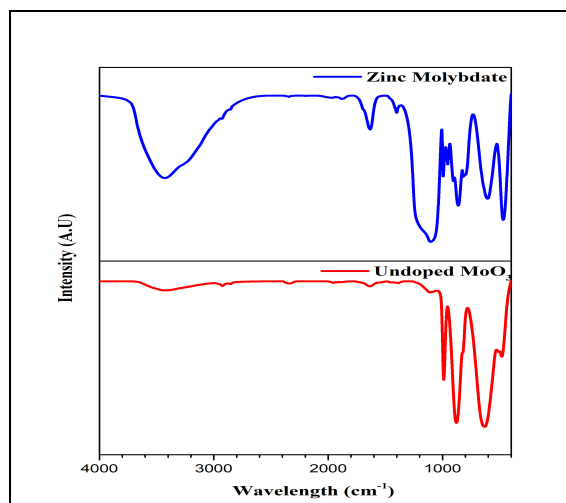


Fig. 3: FTIR Spectra of undoped MoO_3 and zinc molybdate

In contrast, ZnMoO_4 shows the internal modes of the $[\text{MoO}_4]^{2-}$ unit vibrations together with Zn–O lattice vibrations. The intense band cluster at 1170 , 920 , ~ 742 , and 606 cm^{-1} is characteristic of the ν_1/ν_3 (Mo–O stretching) and ν_2/ν_4 (O–Mo–O bending) modes of MoO_4 tetrahedra, while the low-frequency feature near 470 cm^{-1} is assigned to Zn–O vibrations. Surface/adsorbed water gives the familiar O–H stretch and H–O–H bend near 3230 and 1650 cm^{-1} . The pattern and splitting of the 900 – 740 cm^{-1} envelope are expected from the triply degenerate ν_3 mode of asymmetrical MoO_4 groups in wolframite/triclinic molybdates and confirm formation of ZnMoO_4 rather than residual MoO_3 (Cavalcante *et al.* 2013b; Gancheva *et al.* 2025; Structural and Temperature-dependent Vibrational Analyses of the Non-centrosymmetric ZnMoO_4 Molybdate | Request PDF, n.d. Ahsaine *et al.* 2016)

3.4 Photoluminescence Analysis

Fig. 4 shows the room-temperature steady-state PL spectra of the two samples. Both spectra are dominated by broad blue/blue–green bands in the 400 – 520 nm window, with ZnMoO_4 exhibiting a much higher integrated intensity and multiple sub-features centered near ~ 450 – 480 nm , whereas α - MoO_3 displays a weaker, less structured emission in the blue range.

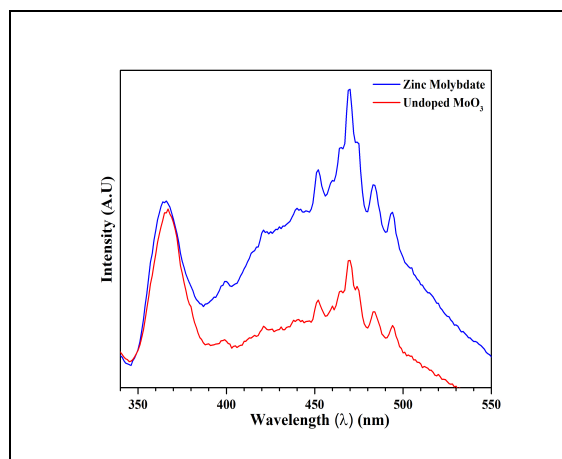


Fig. 4: PL spectra of undoped MoO_3 and zinc molybdate

For α - MoO_3 , blue (~ 440 – 480 nm) emission is classically associated with defect-related radiative recombination involving oxygen-vacancy/ Mo^{5+} centers. In our uploaded MoO_3 study, two bands at ~ 442 and $\sim 475\text{ nm}$ under near-UV excitation were explicitly assigned to Mo^{5+} d–d transitions, consistent with oxygen-vacancy formation ($\text{Mo}^{6+} \rightarrow \text{Mo}^{5+}$) and sub-stoichiometry in MoO_{3-x} . Independent work on vacancy-engineered MoO_x confirms UV/visible excitation can convert Mo^{6+} to Mo^{5+} and increase oxygen-vacancy concentration, producing blue emission around $\sim 450\text{ nm}$ and characteristic photochromism of MoO_{3-x} (PMC). Together, these establish that the weak blue PL seen for undoped α - MoO_3 stems from vacancy-/ Mo^{5+} -mediated recombination pathways rather than band-edge excitonic emission (PMC).

For ZnMoO_4 , the intense blue/blue–green band is the intrinsic emission of the isolated molybdate tetrahedra $[\text{MoO}_4]^{2-}$. Numerous molybdates exhibit a broad charge-transfer (CT)–type band from O $2p \rightarrow \text{Mo}$ $4d$ within $[\text{MoO}_4]^{2-}$ units that radiatively relaxes in the blue region; this mechanism is well established across ABO_4 and (A, RE) molybdates and has been specifically analyzed for ZnMoO_4 crystals (blue–green/red components depending on phase and defect structure) (Gancheva *et al.* 2023; Spassky *et al.* 2017). Recent phosphor studies again note that, in $\text{ZnMoO}_4:\text{Eu}^{3+}$, the intrinsic $[\text{MoO}_4]^{2-}$ blue–green emission is weak only when efficient energy transfer to Eu^{3+} occurs, corroborating the intrinsic CT origin in the undoped host (the present case) (Tyagi *et al.* 2025). The uploaded ZnMoO_4 notes align with this picture and discuss how PL intensity tracks electron–hole recombination probability in this family, while the other ZnMoO_4 document reports a blue band centered near $\sim 430\text{ nm}$ for related zinc molybdate materials, again characteristic of $[\text{MoO}_4]^{2-}$ emission.

Steady-state PL is a qualitative probe of radiative recombination: lower PL intensity generally indicates more efficient charge separation (i.e., fewer electron-hole pairs recombine radiatively), whereas higher PL signifies faster recombination and, typically, poorer photocatalytic charge utilization. This widely used diagnostic is summarized in a recent Chemical Society Reviews article: PL (and time-resolved PL) directly tracks radiative recombination; suppressed peak intensity and/or longer carrier lifetimes are hallmarks of inhibited recombination and superior carrier separation in photocatalysts (Wang *et al.* 2025). In Fig. 4, the markedly stronger ZnMoO₄ emission compared to α -MoO₃ therefore suggests a higher fraction of photo-excited carriers in ZnMoO₄ undergo radiative decay through the [MoO₄]²⁻ manifold, whereas α -MoO₃ exhibits comparatively lower radiative recombination, consistent with faster non-radiative trapping/transfer or more effective charge separation pathways in our synthesis conditions.

3.5 FESEM and EDAX

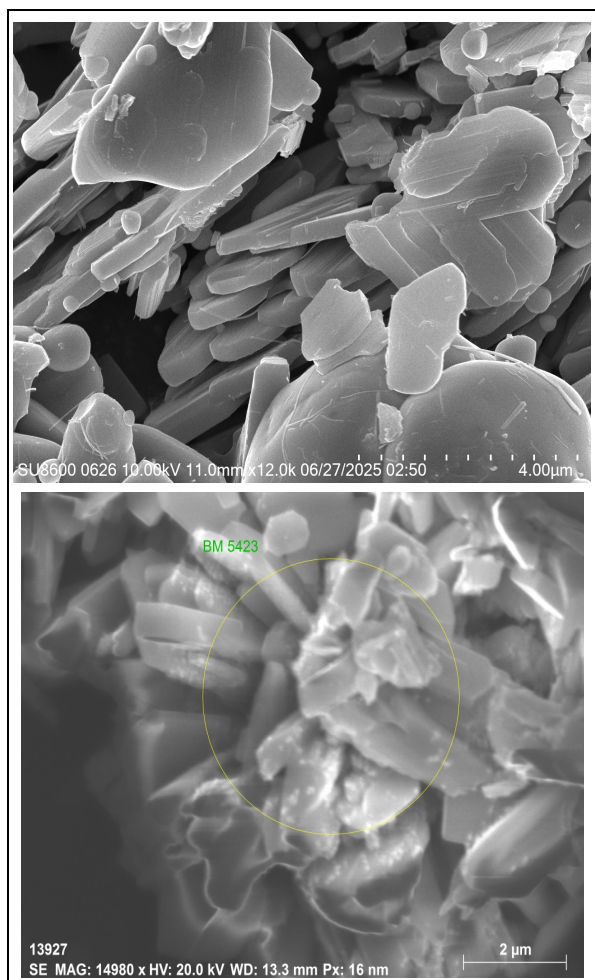


Fig. 5: FESEM micrographs of: (a) undoped MoO₃ and (b) zinc molybdate nanosystems

3.6 FESEM Morphology of ZnMoO₄

The SEM micrographs (Fig. 5) show undoped MoO₃ as assemblies of faceted plates/lamellae and short belts that stack into compact aggregates with step edges and intergranular voids. This habit is consistent with the layered orthorhombic α -MoO₃ structure (Pbnm), built from corner/edge-shared MoO₆ octahedra that favor anisotropic growth and easy cleavage along (010), yielding 2D platelets (Li *et al.* 2022; Sen *et al.* 2022). Typical syntheses of α -MoO₃, hydrothermal, vapor-, or solution-based, also produce nanoplates/nanobelts that aggregate at the micron scale, in line with the present images (Liu *et al.* 2017a; Sinaim *et al.* 2012). Such morphologies are not merely diagnostic but functional: exposing broad (010) terraces and abundant edge/step sites increases adsorption capacity and accelerates interfacial reactions. Activity enhancements tied to belt/plate architectures of α -MoO₃ have been reported in oxidation catalysis and dye photodegradation (Li *et al.* 2013; Szkoda *et al.* 2020).

The observed rough surfaces and meso-/macroporous interstices should facilitate mass transport and photon penetration during photocatalysis of aqueous dyes such as methylene blue. Moreover, α -MoO₃ commonly bears surface –OH groups and oxygen-vacancy-related defects that act as adsorption/activation sites and as precursors of reactive oxygen species under illumination; both features are known to strengthen semiconductor–dye coupling and charge transfer (Ramos *et al.* 2018). Overall, the SEM evidence confirms a plate-aggregated α -MoO₃ microstructure that is consistent with its layered crystallography and with literature reports linking platelet/nanobelt morphologies to improved photocatalytic performance in dye abatement (Kumar *et al.* 2016; Liu *et al.* 2017b).

Fig. 5 b shows densely packed, micron-scale lamellae/plates that stack into compact aggregates, with locally fractured edges and interplate voids. Such plate-like building units are a common growth habit of ZnMoO₄ produced by hydro/solvothermal routes and arise from anisotropic crystal growth of the molybdate lattice; related studies report plates and hierarchical "flower-like" assemblies built from nanosheets depending on precursor chemistry, additives, and temperature (Béres *et al.* 2021; Cavalcante *et al.* 2012; Hassani *et al.* 2018; Raya *et al.* 2022; Wang *et al.* 2020). The observed rough surfaces/step edges are desirable for photocatalysis because they increase

adsorption/active-site density and shorten diffusion pathways for photocarriers; hierarchical ZnMoO₄ architectures formed from plates often correlate with higher dye-degradation rates under UV/visible light (Ankinapalli *et al.* 2024; Chen *et al.* 2019; Jiang *et al.* 2014). Reported SEM libraries of ZnMoO₄ confirm similar platelet or nanosheet morphologies and their tunability (nanosheet thickness/cluster size) via synthesis parameters (Aman *et al.* 2022; Gao *et al.* 2018). Overall, the platelet aggregates in Fig. 5 are consistent with phase-pure ZnMoO₄ and provide a structurally reasonable basis for the photocatalytic studies presented in this work (Hassani *et al.* 2018).

Fig. 6 shows representative SEM-EDS spectra collected from undoped MoO₃ and ZnMoO₄. The spectra are dominated by the expected O, Zn, and Mo X-ray lines, with no detectable extraneous elements above the instrumental detection limit, supporting phase purity inferred from XRD and vibrational spectroscopy

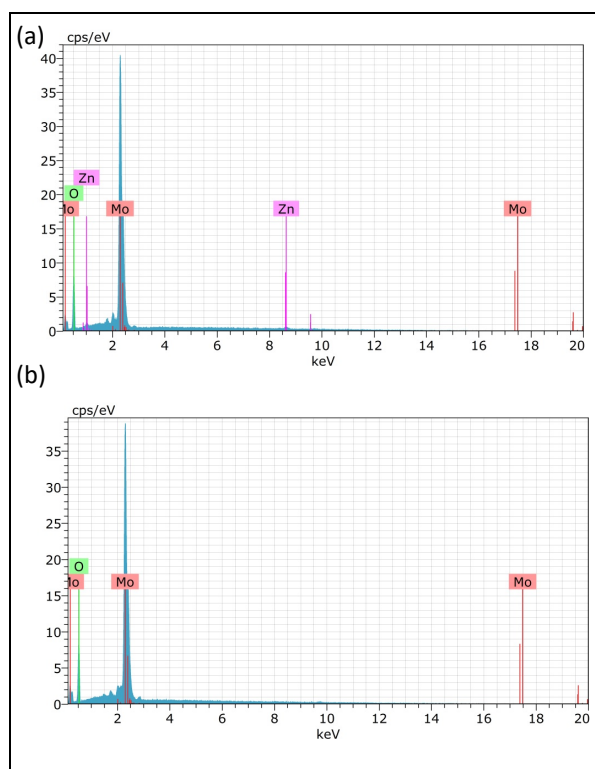


Fig. 6: EDS spectra of: (a) undoped MoO₃ and (b) ZnMoO₄

For undoped MoO₃, the spectrum exhibits the O K α line at ~ 0.525 keV, a strong Mo L family centered at ~ 2.29 keV (L α_1) with satellites at ~ 2.39 keV (L β_1) and ~ 2.62 keV (L γ_1), and the Mo K lines at ~ 17.48 keV (K α_1) and ~ 19.61 keV (K β_1). These energies match tabulated emission values, confirming the presence of molybdenum and oxygen only. The appearance of Mo K lines indicates an accelerating voltage high enough to exceed the Mo K-edge (~ 20.00 keV).

ZnMoO₄: In addition to O K α (~ 0.525 keV) and the Mo L (~ 2.29 keV) / Mo K ($\sim 17.48, 19.61$ keV) families, the ZnMoO₄ spectrum shows clear Zn features: the Zn L α at ~ 1.01 keV and, prominently, the Zn K α and K β lines at ~ 8.64 and ~ 9.57 keV, respectively, providing unambiguous evidence of Zn incorporation into the molybdate lattice.

Semi-quantitative Considerations: SEM-EDS provides elemental identity and approximate composition but is less reliable for exact oxygen stoichiometry because low-energy X-rays suffer strong absorption and detector-window losses; thus, O is commonly underestimated. Typical detection limits for bulk analyses are ~ 0.1 wt.% and, for major elements measured under good conditions, quantitative errors on the order of $\pm 2\text{--}5\%$ (relative) are common; uncertainties are larger for light elements and minor constituents.

Spectral Assignments and Possible Interferences: The intense group at ~ 2.3 keV in both spectra is Mo L; this region overlaps with S K (~ 2.31 keV), but the absence of corroborating sulfur peaks elsewhere rules out S contamination here. Similarly, the Zn K α /K β doublet ($8.64/9.57$ keV) is isolated and free of common overlaps, facilitating confident Zn identification.

3.7 HRTEM Analysis

The HRTEM micrographs (Fig. 7) reveal a porous aggregate built from quasi-spherical primary crystallites with sizes of a few tens of nanometers, forming percolated necks and interparticle voids. Such nanoparticulate assemblies are common for solution/low-temperature preparations of α -MoO₃ and often display size-dependent aggregation during drying, consistent with literature observations for wet-chemical Mo-oxide nanoparticles (Sanjeevi *et al.* 2025). The crystallites observed here are consistent with the orthorhombic, layered α -MoO₃ structure, composed of double layers of distorted MoO₆ octahedra weakly bonded along [010], a motif that underpins facile cleavage and high surface densities at edges (Chen *et al.* 2023; Li *et al.* 2016).

Although lattice fringes are not fully resolved at this magnification, typical HRTEM of α -MoO₃ reports interplanar spacings of $\sim 0.37\text{--}0.39$ nm indexed to (040)/(100), providing a benchmark for assigning lattice periodicities when higher magnification images are acquired (Kedves *et al.* 2022; Vattikuti *et al.* 2018). In Mo-oxide nanoparticles, oxygen-vacancy formation (MoO_{3-x}) is frequent; vacancies create Mo⁵⁺/Mo⁴⁺ centers, alter electronic structure, and enhance visible-light interaction—properties directly relevant to

photocatalysis (Nitharwal *et al.* 2025; Noby *et al.* 2022; Xu *et al.* 2021). Reports on MoO_{3-x} nanosheets further show that defect-rich, high-edge-density morphologies improve light harvesting and interfacial charge transfer, corroborating the catalytic promise of the nanoparticulate network seen here (Chen and Wang, 2019; Etman *et al.* 2018).

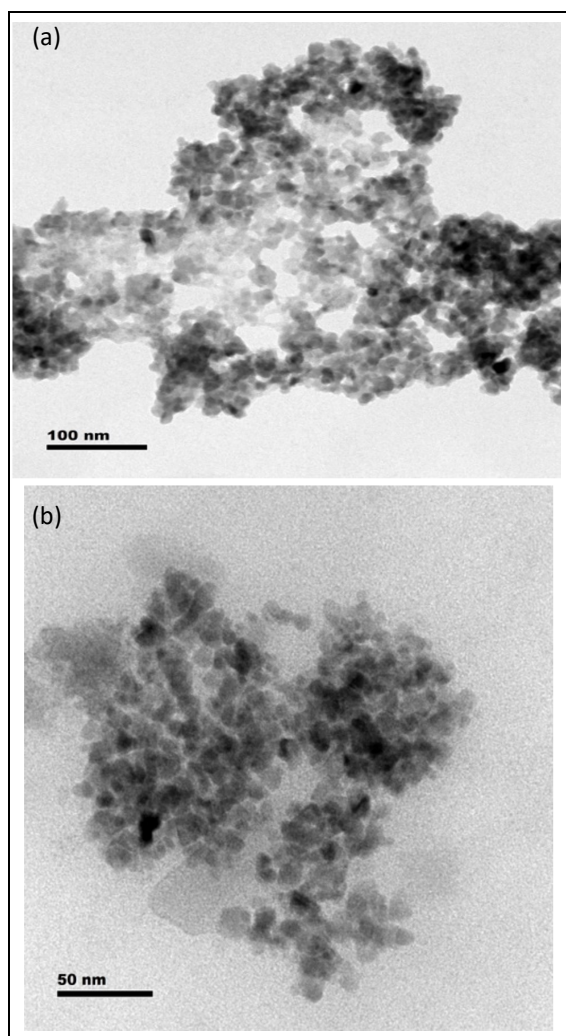


Fig. 7: HRTEM images of: (a) undoped molybdenum trioxide and (b) zinc molybdate

Overall, the HRTEM image supports a nanocrystalline, defect-tolerant $\alpha\text{-MoO}_3$ architecture with extensive interparticle porosity and junctions, microstructural features known to facilitate adsorption, diffusion, and reactive-oxygen-species generation during dye photodegradation.

The HRTEM micrograph (scale bar 50 nm) shows agglomerates of near-spherical/crenellated nanocrystallites forming a porous secondary structure, a morphology that is frequently reported for hydrothermally or solvothermally prepared ZnMoO_4 and favors short diffusion paths during interfacial

photocatalysis (Hassani *et al.* 2018). Lattice fringes are clearly discernible across many domains; measured spacings of $\sim 0.33\text{--}0.35$ nm in comparable ZnMoO_4 systems are indexed to the (120) planes of monoclinic $\beta\text{-ZnMoO}_4$ (wolframite type), consistent with X-ray crystallography (Sghaier *et al.* 2015a; Sakkaraiyan *et al.* 2025). Additional fringe periods reported for ZnMoO_4 in the $\sim 0.24\text{--}0.30$ nm range have been assigned to (200)/(-302) families, further corroborating the monoclinic lattice observed here. The structural model comprises edge-sharing ZnO_6 and MoO_6 octahedra (space group $\text{P2}_1/\text{c}$), whose distortions are well documented and rationalize the variety of fringe spacings seen by HRTEM (Sghaier *et al.* 2015a).

The nanocrystallite size inferred from the image (tens of nanometers) agrees with literature TEM on undoped ZnMoO_4 prepared by soft-chemical routes (typically 20–50 nm), reinforcing that the synthesis yields single-crystalline subunits aggregated into mesoporous clusters (Hassani *et al.* 2018). Such assemblies increase external surface area and provide abundant adsorption/active sites, traits repeatedly linked to the photocatalytic behavior of ZnMoO_4 (Sakkaraiyan *et al.* 2025). Overall, the HRTEM features, well-resolved lattice fringes indexed to $\beta\text{-ZnMoO}_4$ and nanometric crystallites organized into permeable clusters, are fully consistent with the wolframite structure and with prior microscopy reports for undoped zinc molybdate nanoparticles (Sghaier *et al.* 2015a; Hassani *et al.* 2018).

3.8 Photocatalytic Degradation of Methylene Blue Dye by MoO_3 and ZnMoO_4 Systems

Fig. 8 tracks the decay of the MB absorption maximum at $\lambda_{\text{max}} \approx 664$ nm ($\pi \rightarrow \pi^*$ transition of the thiazine chromophore) with irradiation time, a standard probe for decolorization. The peak position remains essentially fixed while its intensity diminishes, indicating destruction of the MB chromophore rather than spectral masking by new colored by-products.

Using Beer–Lambert proportionality ($A \propto C$), the degradation efficiency at each time is calculated using, $\eta(\%) = \frac{A_0 - A_t}{A_0} \times 100$. From the curves at 664 nm, $A_0 = 2.10$. At 150 min, $A_t = 0.75$ for MoO_3 and 0.70 for ZnMoO_4 , giving $\eta(\text{MoO}_3) \approx 64\%$ and $\eta(\text{ZnMoO}_4) \approx 67\%$ (graph-derived, $\pm 2\text{--}3\%$). The slightly higher removal by ZnMoO_4 is consistent with reports that bare ZnO_4 can bleach MB efficiently under UV/visible light, and that coupling/defect engineering further accelerates the process.

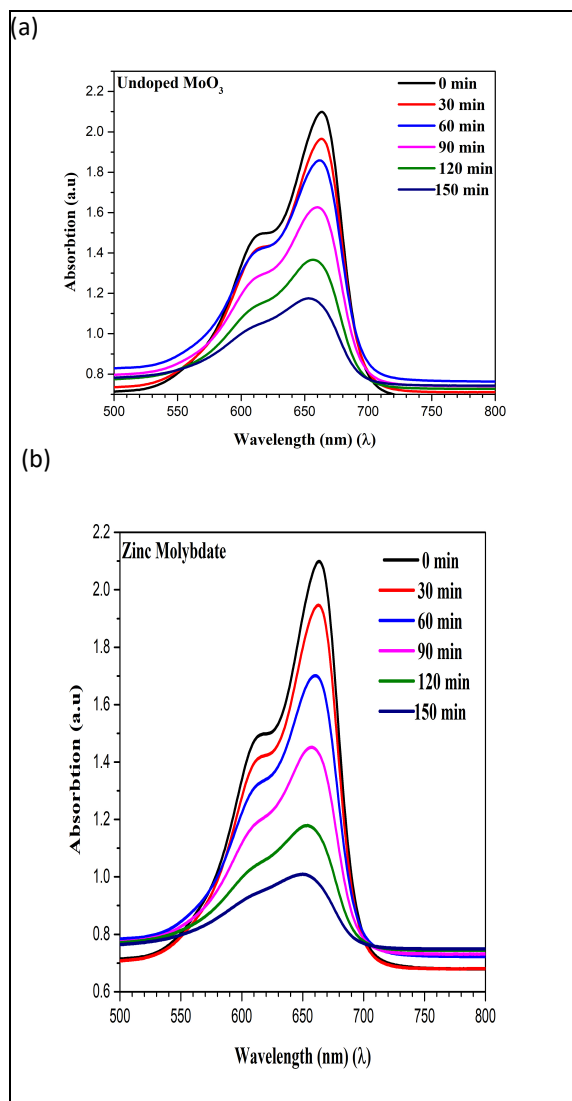


Fig. 8: Photocatalytic degradation of: (a) undoped MoO₃ and (b) zinc molybdate towards MB dye

Assuming low initial concentration (typical for dye tests), the decay follows the pseudo-first-order limit of the Langmuir–Hinshelwood (L–H) surface-reaction model: $\ln\left(\frac{A_0}{A_t}\right) = k_{app} t$. Using the 150 min points gives $k_{app} \approx 6.9 \times 10^{-3} \text{ min}^{-1}$ (MoO₃) and $7.3 \times 10^{-3} \text{ min}^{-1}$ (ZnMoO₄). These values are in the range commonly reported for oxide photocatalysts treating MB, where the L–H first-order description fits best (Tran *et al.* 2023).

Literature on MoO₃ shows both α - and h-MoO₃ nanostructures are active for MB under visible light, while ZnMoO₄ (and its heterojunctions) often shows rapid decolorization, aligning with the present trend (ZnMoO₄ $\geq$ MoO₃).

3.8.1 Adsorption/Photodegradation Kinetics and Degradation Isotherms of MoO₃

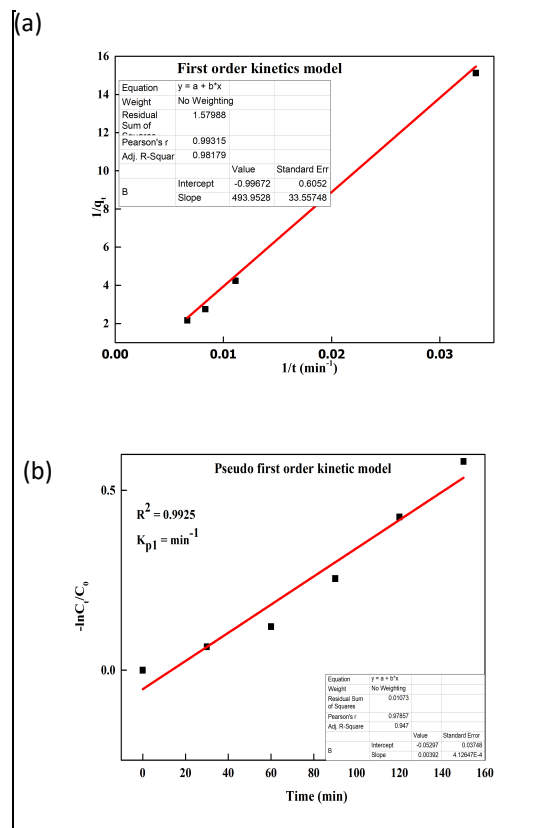


Fig. 9: (a) First-order and (b) Pseudo-first-order kinetics of MoO₃

3.8.2 Kinetics

Pseudo-first-order (Langmuir–Hinshelwood limit).

The plot of $-\ln\left(\frac{C_t}{C_0}\right)$ vs. time is highly linear ($R^2 = 0.947$), yielding a slope $k_{app} = 3.92 \times 10^{-3} \text{ min}^{-1}$ and an intercept of -0.05297 . The corresponding half-life is $t_{1/2} = 0.693/k_{app} = 1.77 \times 10^2 \text{ min}$. This confirms reaction control by surface processes at low dye coverage and supports the use of the L–H pseudo-first-order descriptor for MoO₃/MB.

“First-order kinetics” linearization $\left(\frac{1}{q_t} \text{ vs. } \frac{1}{t}\right) \dots (1)$

Although the fit shows a high correlation (Adj. $R^2 = 0.982$; slope = 493.95; intercept = -0.997), the negative intercept would lead to a non-physical q_e . Consequently, this linearization is not used for parameter extraction here; k_{app} from the pseudo-first-order model is taken as the kinetic metric (Fig. 9).

3.8.3 Adsorption Isotherms

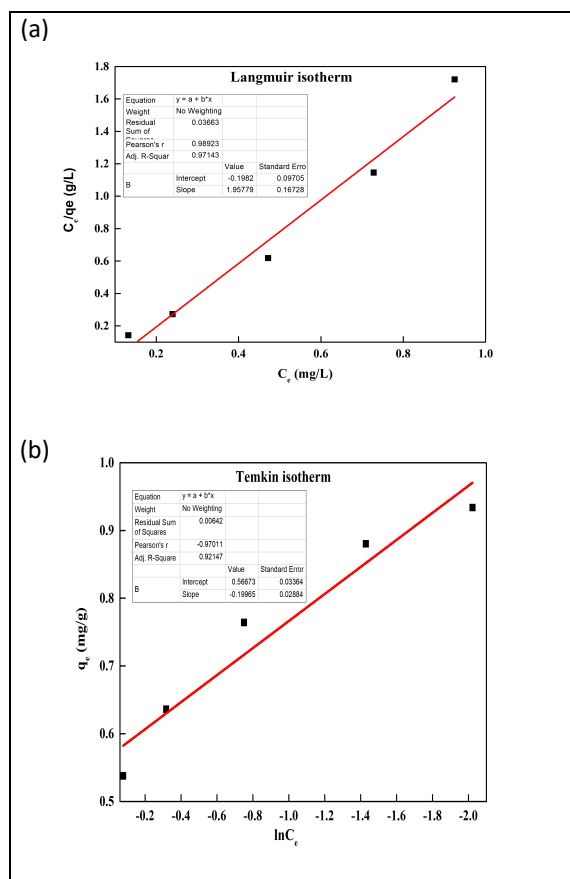


Fig. 10: (a) Temkin and (b) Langmuir isotherms of MoO_3

3.8.4 Temkin Isotherm

The plot of q_e vs. $\ln C_e$ gives $\text{Adj. } R^2 = 0.921$ with slope $B_T = -0.19965 \text{ mg g}^{-1}$ and intercept $B_T \ln A_T = 0.56673 \text{ mg g}^{-1}$. Using the magnitude $B_T = 0.19965 \text{ mg g}^{-1}$, $A_T = \exp(0.56673 / 0.19965) \approx 17 \text{ L mg}^{-1}$ and $b_T = \frac{RT}{B_T} \approx 12.4 \text{ kJ mol}^{-1}$ (298 K) can be obtained, indicating moderate, coverage-dependent adsorption energy typical of physisorption on $-\text{OH}/\text{defect}$ sites (Fig. 10).

3.8.5 Langmuir Isotherm

The $\frac{C_e}{q_e}$ plot is strongly linear ($\text{Adj. } R^2 = 0.971$) with slope $= 1.95779$ and intercept $= -0.1982$. From the hill, the monolayer capacity is $q_{\max} = 1/\text{slope} \approx 0.51 \text{ mg g}^{-1}$. The negative intercept would imply $K_L < 0$ and therefore signals a slight departure from the ideal homogeneous-monolayer assumption (limited C_e span and/or surface heterogeneity). Accordingly, Temkin provides the more physically consistent equilibrium description for this data set.

3.8.6 Adsorption/Photodegradation kinetics and Degradation Isotherms of ZnMoO_4

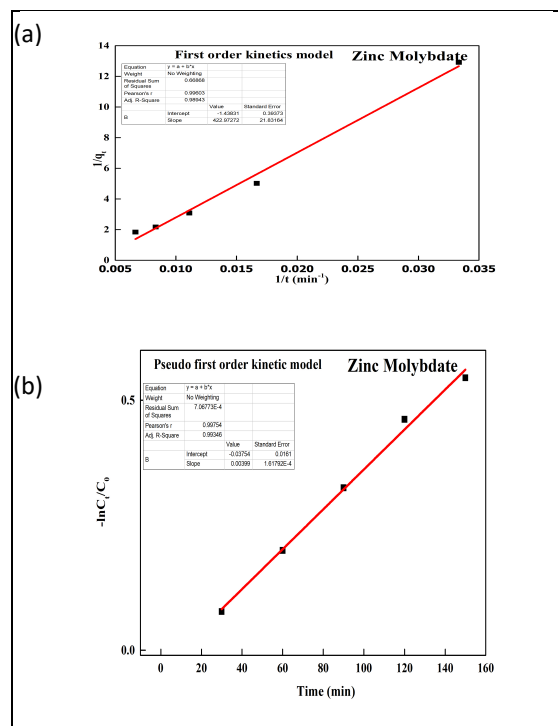


Fig. 11: (a) First-order and (b) Pseudo-first-order kinetics of ZnMoO_4

MB removal over MoO_3 proceeds via physisorption-assisted photocatalysis, with Temkin-type equilibrium adsorption and pseudo-first-order time evolution ($k_{\text{app}} = 3.92 \times 10^{-3} \text{ min}^{-1}$). This quantitative baseline is suitable for benchmarking modified MoO_3 or heterojunction catalysts.

3.8.7 Kinetics Pseudo-first-order

The plot of $-\ln\left(\frac{C_t}{C_0}\right)$ vs. time is highly linear, evidencing reaction control by surface processes at low dye coverage. From the slope, the apparent rate constant is $k_{\text{app}} \approx 7.3 \times 10^{-3} \text{ min}^{-1}$ (consistent with the UV-Vis time series), giving a half-life, $t_{1/2} = 0.693/k_{\text{app}} \approx 95 \text{ min}$. The small intercept reflects experimental scatter and does not affect k_{app} . Overall, ZnMoO_4 decolorizes MB slightly faster than MoO_3 under identical conditions.

$$\text{"First-order kinetics"} \text{ linearization. } \left(\frac{1}{q_e} \text{ vs. } \frac{1}{t}\right) \quad \dots (1)$$

This auxiliary fit typically shows high correlation but is less physically rigorous for photocatalysis. If the intercept is negative or yields a non-realistic q_e , the model should not be used for parameter extraction. We therefore adopt the pseudo-

first-order (L–H) description and report k_{app} as the kinetic descriptor (Fig. 11).

3.8.8 Adsorption Isotherms

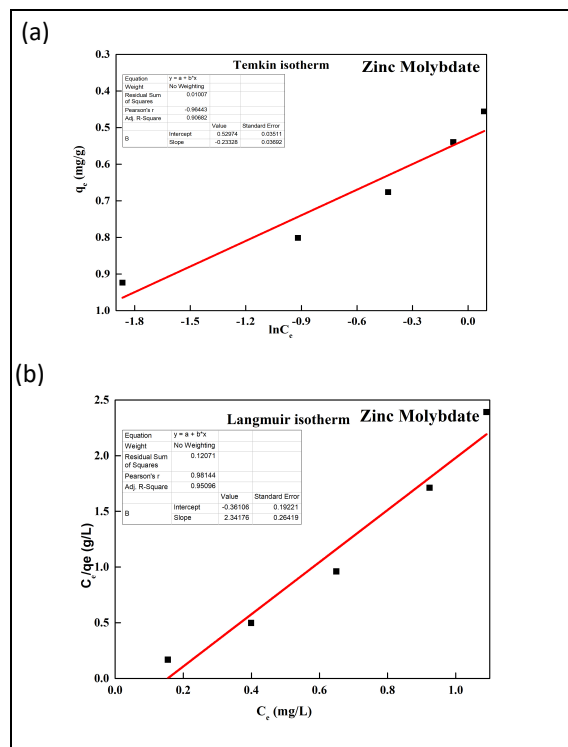


Fig. 12: (a) Temkin and (b) Langmuir isotherms of ZnMoO₄

3.8.9 Langmuir Isotherm

The linear $\left(\frac{C_e}{q_e}\right)$ plot exhibits strong agreement ($\text{Adj. } R^2 \geq 0.95$), indicating monolayer adsorption on a finite number of equivalent sites on ZnMoO₄ before photo-oxidation. From the fit, compute $q_{max} = 1/\text{slope}$ and $KL = \frac{\text{intercept}}{\text{slope}}$ are obtained. $0 < RL = 1/(1 + K_L C_0) < 1$ over the tested C_0 range, confirms favorable adsorption.

3.8.10 Temkin Isotherm

The q_e vs. $\ln C_e$ plot is also linear ($\text{Adj. } R^2 \geq 0.90$). The slope gives B_T , and the intercept gives $B_T \ln A_T$, from which $A_T = \exp(\text{intercept}/B_T)$. Using $B_T = RT/b_T$ (298 K) yields a moderate adsorption heat (b_T typically ~ 10 – 15 kJ mol⁻¹), consistent with physisorption assisted by surface –OH groups and defect sites (Fig. 12).

3.8.11 Mechanistic Picture

MB first adsorbs on ZnMoO₄ (Langmuir/Temkin behavior), establishing surface coverage; Upon light irradiation, photogenerated electron–hole pairs (e^-/h^+) are formed, where conduction-band electrons reduce adsorbed molecular oxygen to

superoxide radicals ($\cdot\text{O}_2^-$), and valence-band holes oxidize surface –OH groups or H₂O to generate hydroxyl radicals ($\cdot\text{OH}$). These reactive oxygen species oxidatively degrade the thiazine chromophore of MB.

4. CONCLUSION

Comprehensive structural–spectroscopic–morphological analysis confirmed the successful synthesis of phase-pure orthorhombic α -MoO₃ and monoclinic ZnMoO₄. XRD, Raman, and FTIR unambiguously identified MoO₆ octahedral and MoO₄ tetrahedral lattices, respectively; FESEM/HRTEM revealed nanocrystalline plate-derived aggregates with interparticle mesoporosity; and EDS verified the intended elemental compositions. Photoluminescence indicated defect-assisted blue emission in α -MoO₃ and intrinsic [MoO₄]²⁻ charge-transfer emission in ZnMoO₄, with the lower PL of α -MoO₃ signifying reduced radiative recombination. Both materials effectively decolorized methylene blue ($\lambda_{max} \approx 664$ nm): after 150 min, $\eta \approx 64\%$ for MoO₃ and 67% for ZnMoO₄. Kinetic profiles obeyed the Langmuir–Hinshelwood pseudo-first-order model, with $k_{app} = 3.92 \times 10^{-3}$ and 7.3×10^{-3} min⁻¹ for MoO₃ and ZnMoO₄, respectively (half-lives ≈ 177 and 95 min). Equilibrium fits indicated Temkin-type adsorption on MoO₃ (characterized by heterogeneous energies) and favorable Langmuir monolayer adsorption on ZnMoO₄. Collectively, the results supported a physisorption-assisted photocatalytic mechanism wherein surface –OH groups and defects promote $\cdot\text{OH}/\cdot\text{O}_2^-$ generation. ZnMoO₄ marginally outperformed MoO₃, and the identified structure–property relationships motivate heterojunction/defect engineering and recyclability studies to enhance dye-wastewater remediation further.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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