



# Investigating the photocatalytic performance of ternary nanocomposite Pd/rGO/MoO<sub>3</sub> under visible light

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## ABSTRACT

Two-dimensional (2D) ternary nanocomposites that integrate noble metal nanoparticles with semiconductor oxides on conductive carbon frameworks have emerged as efficient photocatalysts due to their enhanced charge separation, broad light absorption, and surface reactivity. In this work, a Pd/rGO/MoO<sub>3</sub> ternary nanocomposite was synthesized via a one-step hydrothermal method by anchoring palladium (Pd) nanoparticles and MoO<sub>3</sub> nanorods on reduced graphene oxide (rGO) sheets. Comprehensive structural, morphological, and chemical characterizations (UV-Vis spectroscopy, X-ray diffraction, Raman spectroscopy, X-ray photoelectron spectroscopy, Field-emission scanning electron microscopy, and transmission electron microscopy) confirmed the successful integration and strong interfacial contact among the three components. Photocatalytic tests under visible light demonstrated that the Pd/rGO/MoO<sub>3</sub> nanocomposite exhibited significantly improved degradation of methylene blue (MB) dye (89 % within 60 min) compared with Pd (52 %), Pd/rGO (72 %), and rGO/MoO<sub>3</sub> (77 %) counterparts. This superior activity arises from the synergistic effects of Pd plasmonic enhancement and electron-trapping ability, rGO high conductivity and large surface area, and MoO<sub>3</sub> strong oxidative potential, which collectively promote efficient charge separation and reactive oxygen species generation. Moreover, the catalyst maintained an efficiency of 82–89 % over five reuse cycles, confirming its high stability and recyclability. These findings emphasize the future promise of Pd/rGO/MoO<sub>3</sub> as a robust, visible-light-driven photocatalyst for real wastewater and dye degradation, as well as other environmental remediation applications.

## 1. Introduction

The escalating industrialization in recent times has caused rapid water contamination driven by human activity, resulting in environmental degradation and water scarcity. This requires immediate action to identify particular options for generating potable water for financial advancement and consumption [1,2]. Over the years, various strategies have been utilized to develop reliable water production technologies, with the oxidation process (OP) emerging as the most effective way. The redox process demonstrates exceptional efficacy in water disinfection and the degradation of various water contaminants, including industrial

effluent with toxic colors. Low-dimensional nanostructures have accumulated notable interest in recent decades because of their peculiar electrical and optical properties resulting from size and morphological effects. Consequently, nanostructured materials exhibit extensive potential uses in ion storage, energy conversion, and environmental protection. Specifically, photocatalysts derived from semiconductor nanoparticles have remarkable efficacy in air and water purification, demonstrating exceptional efficiency and durability. Recently, water purification has emerged as a significant domain of study, with photocatalysts increasingly attracting attention for their efficacy in degrading dangerous substances in water. Toxins like metal ions, antibacterials,

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and harmful chemical dyes are difficult to remove, and they build up in living things, causing major health problems for humans. Dye compounds employed in the polymer, paper, chemical beauty products, printing, pharmaceuticals, painting, textiles, and food sectors are the most common chemical contamination in water [2–4]. Diverse procedures have been utilized to remediate dye wastewater, including sorption, decomposition, layered processes, and photocatalysis. The photocatalytic method has garnered significant interest relative to alternative approaches employed in water purification and the degradation of dangerous substances, owing to its high stability, low energy consumption, and environmentally sustainable characteristics [4–6]. Various factors play a crucial role in influencing photocatalytic effectiveness, encompassing optical, electrical, surface, and structural elements [7,8]. Over the last few years, numerous studies have focused on enhancing semiconductor characteristics, with various studies demonstrating promising outcomes for the application of photocatalysis. Numerous metal oxides exhibiting semiconductor properties, including  $\text{MoO}_3$  [9],  $\text{MoO}_3\text{-TiO}_2$  [10],  $\text{rGO-MoO}_3$  [11],  $\text{Pd-BiVO}_4$  [21],  $\text{ZnO-rGO}$  [22],  $\text{SnO}_2$  [57], and  $\text{Fe}_2\text{O}_3$  [58], have been rigorously examined in photocatalytic systems. The attributes, including surface, structural, and electronic properties, morphology, and particle size, have been analyzed to improve photocatalytic activity.  $\text{MoO}_3$  serves as a representative n-type semiconductor and is characterized by three primary crystalline forms [16–19,33,41–44]: orthorhombic ( $\alpha\text{-MoO}_3$ ), monoclinic ( $\beta\text{-MoO}_3$ ), and hexagonal ( $\text{h-MoO}_3$ ). Due to its promising electronic, structural, and surface characteristics,  $\text{MoO}_3$  has been explored for various applications, such as electronic devices [45], photocatalysts [46], and battery energy storage [47]. The layered anisotropic structure makes  $\alpha\text{-MoO}_3$  a promising candidate for photocatalysis [48].  $\text{MoO}_3$  exhibits a band gap of  $\sim 2.7\text{--}3.2$  eV, with the conduction band 4d of Mo considered to be unoccupied, while its valence band is predominantly composed of O 2p states. The n-type semiconductor  $\text{MoO}_3$  presents a fascinating subject of study, characterized by its anisotropic layered crystalline structure. This structure is created by the stacking of bilayers of  $\text{MoO}_6$  octahedrons, held together by Van der Waals forces along the direction [10]. This material exhibits specific physical properties and demonstrates excellent chemical stability, making it suitable for various applications such as solar cells, sensors, supercapacitor electrodes, and catalysis.  $\text{MoO}_3$  has gained attention in the realm of UV- and visible-light-driven photocatalysts due to its effective absorption capabilities for a significant portion of UV light and some visible light [3,5,7].  $\text{MoO}_3$  demonstrated an 81 % reduction of methylene blue (MB) when exposed to UV radiation over 120 min. [3].  $\text{MoO}_3$  was doped with various transition elements such as Au [9] for  $\text{CO}_2$  reduction,  $\text{g-C}_3\text{N}_4$  [17], and  $\text{Pd-MoO}_3$  physicochemical and catalytic properties [18], resulting in some fascinating characteristics. Many methods including chemical vapor deposition [21], chemical vapor transport (CVT) [22], sputtering [23], laser ablation [24], sol-gel [25], and spray pyrolysis thin films made from  $\text{MoO}_3$  [26]. The spray pyrolysis process is widely utilized for the production of oxide thin films, distinguishing itself from alternative methods. The material exhibits a uniform size distribution and offers thin films with grain sizes that can be controlled by adjusting the doping concentration. Furthermore, this approach yields a substantial production area and is distinctive in its ability to produce crystallized thin films without applying heat treatment. [31]. Due to its potentially large specific surface area (approximately  $2630\text{ m}^2/\text{g}$ ), outstanding electrical properties, plentiful functional groups ( $-\text{COOH}$ ), and rapid electron mobility [20–24]. Graphene oxide (GO) is a two-dimensional hybrid material widely utilized as an efficient cocatalyst in photocatalytic systems. Reduced specific surface area results from the notable decrease in oxygen-containing groups during the reduction of graphene oxide (rGO), as well as from the manufacturing and aggregation of graphene [23–25,27,32]. To address this difficulty, the integration of GO with spacing materials has demonstrated a reduction in aggregation. In recent years, hybridization with graphene, graphene oxide (GO), or rGO has emerged as one of the most promising techniques to increase

photocatalytic efficiency. [20–22].

In particular, the semiconductor and graphene have a relatively weak link and inadequate interfacial contact. rGO with oxygen-containing functional groups, however, shows a strong interaction with semiconductors and a tight interfacial contact. In comparison to graphene, graphene oxide (GO) experiences a reduction in conductivity. This issue can be alleviated with a partial decrease in its functional groupings [23]. Consequently, several studies have concentrated on the synthesis of rGO-based semiconductor photocatalysts [12–16]. Visible light-induced Pd catalysis has made notable progress since the first publication by the Gevorgyan group in 2016. By collecting photon energy as a photocatalyst and enabling bond-forming and breaking mechanisms, the Pd catalyst performs two roles: it eliminates the need for an outside photosensitizer. [10] Contemporary excited-state palladium chemistry typically involves single-electron transfer reactions, contrasting with traditional ground-state palladium catalysis, which relies on two-electron redox processes. This approach facilitates innovative, radical transformations through the involvement of hybrid palladium radicals [34]. Recently, the Gevorgyan group presented a series of transformations facilitated by Pd II-H, highlighting an unusual, chemoselective ‘hydridic’ hydropalladation that is driven by an increase in hydricity upon photoexcitation. Pd NPs have demonstrated the ability to enhance photocatalytic activity across multiple applications, including water splitting,  $\text{CO}_2$  reduction, and the breakdown of organic pollutants in both water and air. In this study,  $\text{Pd/rGO}$ ,  $\text{rGO/MoO}_3$ , and  $\text{Pd/rGO/MoO}_3$  nanocomposites are synthesized hydrothermally, and the effectiveness in degrading MB through photocatalytic processes is compared. The hydrothermal method for creating nanocomposites involves the reaction of precursors in an aqueous solution under conditions of elevated temperatures and pressures. XRD, FE-SEM, SEM-EDS, XPS, Raman spectroscopy, TEM-EDS, and UV–vis spectroscopy are among the established techniques that will be used to identify the form, size, morphology, chemical composition, and optical properties of the generated nanocomposites. The absorbance of the MB solution will be measured at various time intervals to assess the degradation efficiency of MB when exposed to visible light irradiation.

In this study, the main aim is to prepare and compare the photocatalytic activity of rGO,  $\text{Pd/rGO}$ ,  $\text{rGO/MoO}_3$ , and  $\text{Pd/rGO/MoO}_3$  nanocomposites for MB dye degradation under visible light. The work targets the improvement of the photocatalytic performance of layered n-type  $\text{MoO}_3$  by using rGO and Pd nanoparticles. The synergism of rGO, with its high electron mobility and large surface area, and Pd, with its electron scavenging ability and plasmonic effect, is believed to contribute to the potential benefits of charge separation and expanded visible light harvesting.

## 2. Experimental materials and methodology

### 2.1. Chemicals and reagents

All compounds were utilized in this work without any additional purification. For the synthesis of rGO, graphite powder (Asbury Carbon, New Jersey, USA) with a particle size of less than  $20\text{ }\mu\text{m}$  was used as the precursor. Sodium nitrate ( $\text{NaNO}_3$ , ACL Labscan, Thailand), sulfuric acid ( $\text{H}_2\text{SO}_4$ , Samchun, Korea, 98.00 vol%), potassium permanganate ( $\text{KMnO}_4$ , Samchun, Korea, 99.30 wt%), hydrochloric acid (HCl, ACL Labscan, 37.00 vol%), and hydrogen peroxide ( $\text{H}_2\text{O}_2$ , QREc, Thailand, 35.00 vol%). Ammonium molybdate tetrahydrate ( $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ , 99.30 wt%, Loba Chemie) was used as the precursor for the manufacture of  $\text{MoO}_3$ , and palladium chloride ( $\text{PdCl}_2$ , Sigma Aldrich, 99.00 wt%) for Pd NPs.

### 2.2. Synthesis of rGO

The production of rGO was successfully prepared with the methodology taken from a modified Hummers approach. In the current study,

rGO was synthesized by a traditional method of chemical oxidation combined with physical exfoliation. A mixture of 1 g pure graphite flakes and 1 g sodium nitrate was well-dispersed at the start, followed by adding 48 mL of  $\text{H}_2\text{SO}_4$  while being continuously magnetically vorticed in an ice bath under cooling temperatures maintained between 0 and  $5^\circ\text{C}$ . 10 min later, mixed slowly and carefully with the addition of 6 g  $\text{KMnO}_4$  into the solution. The reaction mixture was then placed in a  $35^\circ\text{C}$  water bath. It was further agitated for 2 h to form a black, thick paste. At the same time, 100 mL of DI water was added to the reaction mixture, and the temperature was kept at  $90^\circ\text{C}$ . After that, drop by drop, 30 %  $\text{H}_2\text{O}_2$  was added along with stirring of the mixture. The solution was left for some time, and a dark brown solution appeared that was washed several times with 100 mL of distilled water until it was pH neutral. The mixture was filtered through a  $0.2\ \mu\text{m}$  PTFE membrane filter paper and then washed with  $\text{HCl}$ /water. The final step was to let the product dry in the air [28,29].

### 2.3. Synthesis of $\text{MoO}_3$ rods

One-step hydrothermal synthesis of  $\text{MoO}_3$  rods. An experimental procedure involved taking 1.50 g of  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$  in 60.0 mL of DI water and sonicated for 30 min. To the reaction mixture produced above, 0.17 g of sodium (dodecyl sulfate (0.01 M) was added, and it was stirred for approximately 60 min to obtain a homogeneous solution. The pH was then adjusted to 2–3 using 37 %  $\text{HCl}$  with constant stirring. The transparent molybdate solution was charged into a 100 mL Teflon-lined stainless-steel autoclave for the hydrothermal synthesis (24 h at  $180^\circ\text{C}$ ), and after treatment, the transparent solution turned a deep blue color, indicating the formation of  $\text{MoO}_3$  nanorods. After adsorption was completed, anhydrous ethanol and DI water were used to wash the product repeatedly until it was purified, with the final step of drying the product at  $150^\circ\text{C}$  for 6 h. In this synthetic procedure,  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 7\text{H}_2\text{O}$  was first mixed with a surfactant inside a sealed reaction vessel, followed by heating at high temperature and pressure [44].

### 2.4. Synthesis of rGO/ $\text{MoO}_3$ , Pd/rGO, and Pd/rGO/ $\text{MoO}_3$ nanocomposites

Fig. 1 presents the schematic diagram of the Pd/rGO/ $\text{MoO}_3$  nanocomposite synthesis process. In this work, a ternary Pd/rGO/ $\text{MoO}_3$  composite was produced following the procedure. Synthesis of Pd/rGO/

$\text{MoO}_3$ : 75 mg  $\text{MoO}_3$  rods, 75 mg  $\text{PdCl}_2$ , and 480 mg rGO were mixed in DI water [30]. 75 mg  $\text{MoO}_3$  rods were mixed with 480 mg rGO in 60 mL DI water to synthesize the rGO/ $\text{MoO}_3$  composite using the hydrothermal method. Pd/rGO was prepared by dispersing 480 mg of rGO and 150 mg of palladium chloride ( $\text{PdCl}_2$ ) in 60 mL of DI water using the hydrothermal method. After that, for the synthesis of the Pd/rGO/ $\text{MoO}_3$  composite,

previously synthesized  $\text{MoO}_3$  is used to make this nanocomposite. First of all, take 400 mg of synthesized Pd/rGO and give probe sonication for 10 min. After that, synthesized  $\text{MoO}_3$  rod nanomaterials are added and given a magnetic stirrer for one hour; a ternary solution, Pd/rGO/ $\text{MoO}_3$ , is formed. The above mixture solution was sonicated for 1.5 h to obtain a homogeneous solution. After receiving the solution, the mixture was transferred to a conventional Teflon-lined stainless-steel autoclave and subjected to a heat and pressure treatment at  $180^\circ\text{C}$  for 24 h. After the sample was cooled down to room temperature. After that, a centrifuge is used for separating and purifying nanomaterials; it helps in removing impurities. The solid deposit of binary rGO/ $\text{MoO}_3$ , Pd/rGO, and ternary Pd/rGO/ $\text{MoO}_3$  nanocomposite that resulted was washed several times with ethanol and DI water, followed by heating and drying in an air oven maintained at  $80^\circ\text{C}$  overnight (12 h). Finally, powdered samples such as rGO/ $\text{MoO}_3$ , Pd/rGO, and Pd/rGO/ $\text{MoO}_3$  nanocomposites are used for various characterizations.

### 2.5. Photocatalytic properties

A specific quantity of the Pd/rGO/ $\text{MoO}_3$  nanocomposite was introduced into a solution containing MB in a cuvette. This step aimed to assess the overall strength of the nanocomposite in degrading MB through photocatalytic processes. To ensure proper interaction between both the nanocomposite and MB molecules, the mixture was stirred for 30 min in the dark, allowing for the establishment of adsorption-desorption equilibrium. Following the equilibration period, the mixture was exposed to irradiation from an LED light source. In each photocatalytic run, 200  $\mu\text{L}$  of the Pd/rGO/ $\text{MoO}_3$  nanocomposite solution was dispersed in 3000  $\mu\text{L}$  of MB solution. This light exposure started the photocatalytic degradation process, wherein the Pd/rGO/ $\text{MoO}_3$  catalyzes the breakdown of MB molecules under light-induced conditions. UV-vis spectroscopy was utilized at predetermined time intervals, ranging from 0 to 60 min, to analyze the solution and monitor the degradation of MB over time. The extent of degradation could be

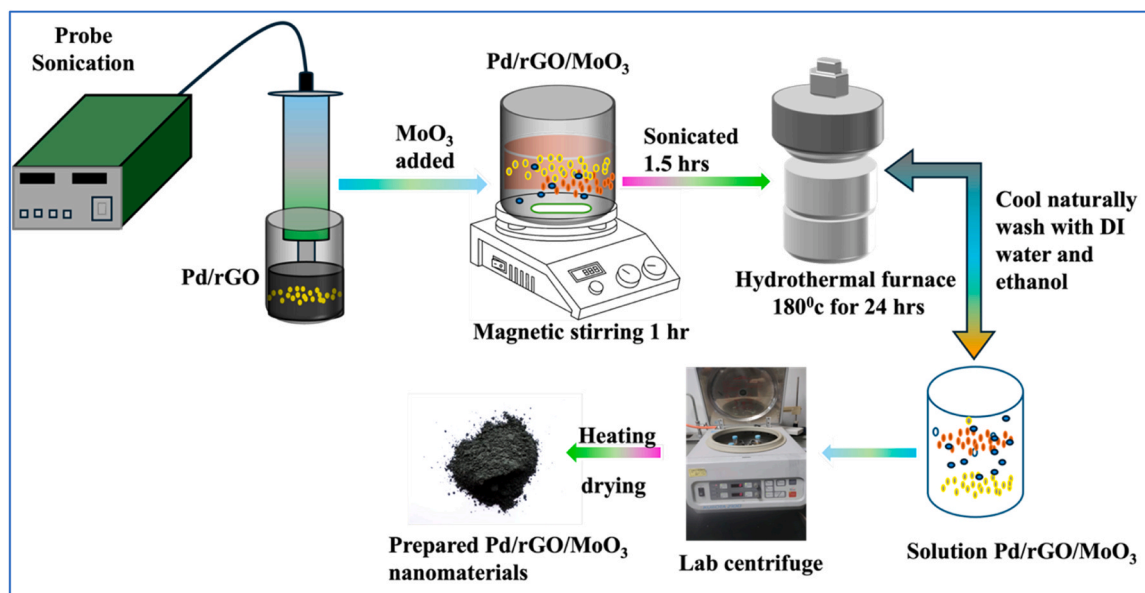


Fig. 1. Schematic representation of the synthesis of Pd/rGO/ $\text{MoO}_3$  nanocomposites.

quantitatively assessed by measuring the changes in MB concentration through spectroscopic analysis. Briefly, photocatalysis was performed under a visible-light LED source (320 mW) equipped with a UV-blocking/UV-B cut filter so that  $\lambda < 400$  nm was effectively removed. The LED reactor distance was fixed at 50 cm.

Controlled photocatalytic degradation experiments were conducted independently using rGO, Pd/rGO, rGO/MoO<sub>3</sub>, and Pd/rGO/MoO<sub>3</sub> nanocomposites to determine the specific contributions of various nanocomposite components to the degradation process. This comparison approach facilitated the evaluation of how each element contributed to improving the degradation efficiency. The experimental protocol included a total duration of 90 min, encompassing both the dark period for equilibrium establishment and the subsequent light-induced degradation process. Finally, the degradation efficiency of MB was calculated using a formula that considers the initial concentration of MB ( $C_0$ ) and its concentration at each time point ( $C_t$ ). This efficiency metric provides insight into the effectiveness of the Pd/rGO/MoO<sub>3</sub> sample in catalyzing MB degradation under photocatalytic conditions.

## 2.6. Characterization

The synthesized nanocomposite underwent comprehensive analysis, scanning electron microscopy (FESEM) for surface morphology, X-ray diffraction (XRD) for crystal structure identification, and UV-Vis spectroscopy for optical properties. The FE-SEM with energy-dispersive X-ray spectroscopy (EDS) for elemental composition and transmission electron microscopy (TEM) for nanoparticle characteristics and crystal structure elucidation. Raman spectra elucidate the structural, vibrational, and phase characteristics of materials, revealing molecular bonds

and the number of layers present. XPS enhances this by examining surface elemental composition, oxidation states, chemical bonding, and electronic structure, providing insights into band alignment and charge transfer across materials. Electrochemical Impedance Spectroscopy (EIS) is used to evaluate the charge transfer resistance, conductivity, and interfacial charge transport in photocatalysts. These approaches offer an in-depth understanding of the structural and electrical characteristics of nanomaterials.

## 3. Results and discussion

### 3.1. UV-vis analysis

Fig. 2 illustrates the UV spectra of all synthesized samples. These include rGO, the rGO/MoO<sub>3</sub> nanocomposite, the Pd/rGO hybrid, and the Pd/rGO/MoO<sub>3</sub> ternary nanocomposite. Fig. 2(a) Incorporation of rGO into MoO<sub>3</sub> (rGO/MoO<sub>3</sub>) resulted in a noticeable red shift of the absorption edge, with the estimated band gap narrowing to  $\sim 2.75$  eV. This shift can be attributed to defect states introduced by rGO as well as strong  $\pi$ - $\pi$  conjugation, which facilitates improved light harvesting in the absorption band centered at 266 nm [24]. Fig. 2(b) depicts MoO<sub>3</sub> exhibiting absorption peaks at 311–353 nm, depending on how many layers of MoO<sub>3</sub> [35]. In Fig. 2(c), the hybrid Pd/rGO, additional absorption is directly linked to the LSPR effect of Pd nanoparticles, which introduce new electronic states and enhance photon electron interactions under visible-light irradiation. Pd nanoparticles not only improve light absorption by amplifying the optical response but also generate energetic hot electrons that can be injected into the rGO/MoO<sub>3</sub> network. This plasmonic contribution is distinct from the defect-induced

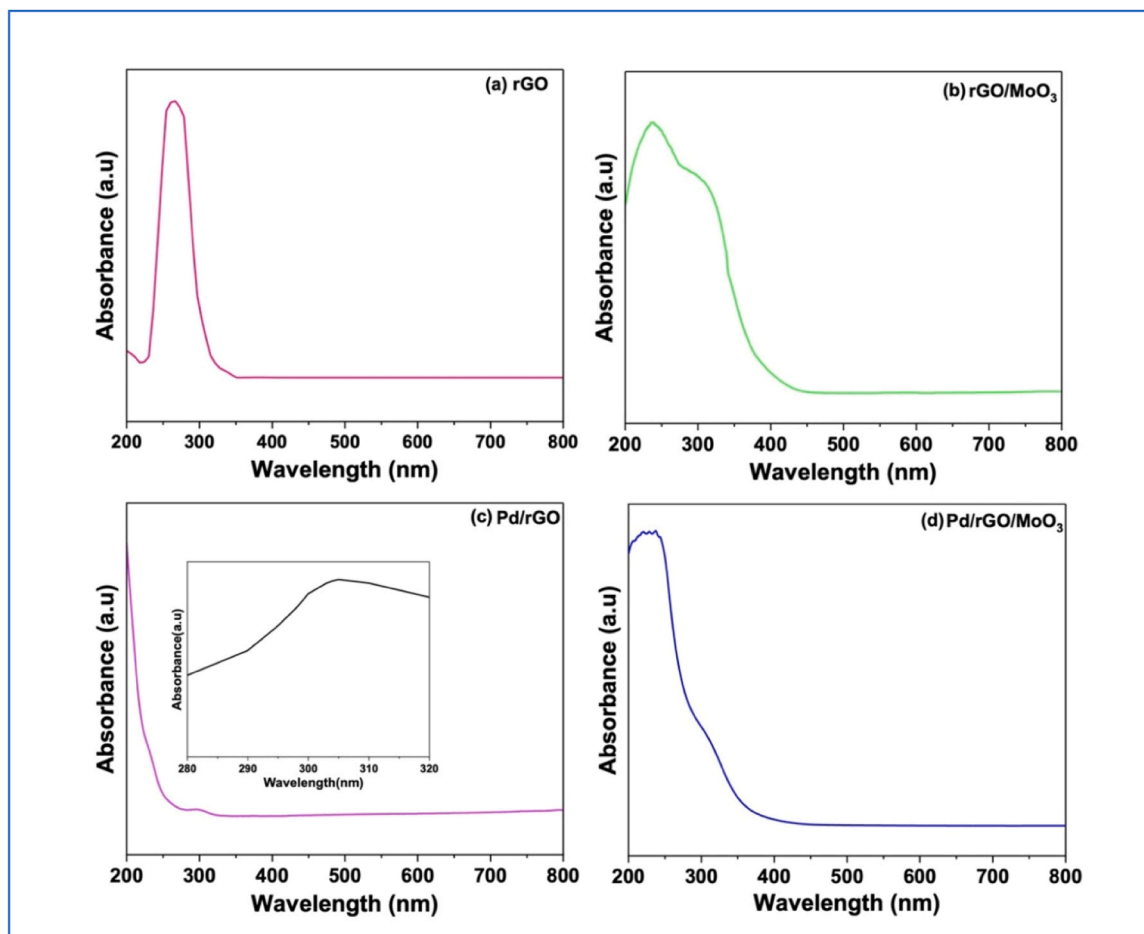


Fig. 2. UV-vis spectra of (a) rGO, (b) rGO/MoO<sub>3</sub>, (c) Pd/rGO, (d) Pd/rGO/MoO<sub>3</sub> nanocomposite.

absorption of rGO; the Pd exhibits a small absorption peak of around 300 nm [36]. Fig. 2(d) presents the UV-visible spectrum of the ternary nanocomposite. The spectrum shows that the overlapping and aligned absorption peaks of the rGO, Pd, and MoO<sub>3</sub> components give it unique properties.

The conduction band (E<sub>cb</sub>) and valence band (E<sub>vb</sub>) edge potentials were calculated using the Mulliken electronegativity method:

$$E_{cb} = \chi - E_e - E_g/2 \quad (1)$$

$$E_{vb} = E_{cb} + E_g \quad (2)$$

Whereas,  $\chi$  is the absolute electronegativity of MoO<sub>3</sub> (6.2 eV),  $E_e$  is the energy of a free electron on the hydrogen scale (4.5 eV), and  $E_g$  is the band gap energy (2.75 eV for Pd/rGO/MoO<sub>3</sub>).

Using Eqs. (1) and (2), the calculated values for the Pd/rGO/MoO<sub>3</sub> composite are  $E_{cb} = 0.33$  eV and  $E_{vb} = 3.08$  eV. Considering the values of  $E_{cb} = 0.33$  eV and  $E_{vb} = 3.08$  eV, it can be concluded that the photocatalyst is capable of producing electrons with enough reducing power and holes that have considerable oxidizing ability. This enables the generation of reactive oxygen species (ROS) for the degradation of dyes, namely •OH and •O<sub>2</sub><sup>-</sup>.

### 3.2. Structural analysis

The crystalline structures and phase composition of all prepared nanocomposite samples were explored via X-ray diffraction (XRD), as shown in Fig. 3. This research aimed to validate the structural phases present in pure-phase MoO<sub>3</sub>, rGO, and hybrid MoO<sub>3</sub>/rGO, Pd/rGO, and Pd/rGO/MoO<sub>3</sub> nanocomposites. The XRD pattern of MoO<sub>3</sub>-NR in Fig. 3(a) displays characteristic diffraction peaks at  $2\theta \approx 9.6^\circ$ ,  $16.7^\circ$ , and other corresponding angles, confirming the successful formation of hexagonal MoO<sub>3</sub> nanorods. These reflections are indexed to the (100), (110), (200), (210), (300), (310), and (410) planes, consistent with the standard crystalline structure of MoO<sub>3</sub>. Notably, the intense peak below  $2\theta = 10^\circ$  is a distinguishing feature of the hexagonal phase, setting it apart from other polymorphic forms of MoO<sub>3</sub>. In the binary rGO/MoO<sub>3</sub>, almost all diffraction peaks are present, but the ternary Pd/rGO/MoO<sub>3</sub> exhibits fewer peaks of MoO<sub>3</sub> present. In Fig. 3(b), the XRD pattern of rGO exhibits a perfect series of the intense and sharp main diffraction peak observed centered at  $26.4^\circ$ , and another small diffraction peak around  $43.60^\circ$  is observed, associated with the in-plane structure of sp<sup>2</sup>-hybridized carbon atoms. Often weak or broad in rGO (corresponding to the (002) and (100) crystallographic planes, respectively [37]. Transitioning XRD spectra exhibit clear peaks typical of the hexagonal crystalline phase of MoO<sub>3</sub> (Fig. 3(c)). Especially, the characteristic

diffraction peaks corresponding to (100), (110), (200), (210), and (300) are seen, which are consistent with the pattern of pure hexagonal MoO<sub>3</sub> (h-MoO<sub>3</sub>) (JCPDS No. 21-0569) [38]. Moreover, Fig. 3(d) proves the existence of peaks of Pd at (111), (200), (220), (311), and (322), which agrees with the (JCPDS No. 46-1043) standard [39]. The XRD pattern of the ternary nanocomposite is presented in Fig. 3(e). In this nanocomposite, all element peaks exhibit the characteristic peaks of Pd, rGO, and MoO<sub>3</sub>, confirming the successful formation of nanocomposite materials.

### 3.3. Morphological analysis

Fig. 4 illustrates the surface topography of several nanocomposites in conjunction with pristine rGO. Fig. 4(a) demonstrates that the rGO displays a consistent, two-dimensional, sheet-like morphology uniformly distributed throughout the substrate. Fig. 4(b) shows the surface characteristics of rGO/MoO<sub>3</sub> nanocomposites, highlighting the formation of nanorod structures typical of MoO<sub>3</sub>. The nanorods interlace or overlap with the rGO sheet, exhibiting lengths of 20–30 micrometers and diameters between 100 and 200 nanometers. Fig. 4(c) depicts the deposition of palladium onto the rGO substrate, resulting in a uniform coating over its surface. The statistical analysis of Pd NPs displays an average size of around 60–100 nanometers, respectively. Fig. 4(d) confirms the integration of all three components, Pd, rGO, and MoO<sub>3</sub>, with the materials intricately combined and aggregated inside the sample, exhibiting a distinctive and complex morphology.

### 3.4. SEM-EDS analysis

Fig. 5 depicts the SEM-EDS spectrum analysis of the Pd/rGO/MoO<sub>3</sub> ternary sample, providing valuable insights into its surface characteristics and elemental composition. Observation of the SEM image reveals a heterogeneous surface morphology characterized by varying particle sizes and distributions, indicative of the nanocomposite composition comprising MoO<sub>3</sub>, rGO, and finely dispersed Pd nanoparticles (NPs). The EDS spectrum complements the SEM image by offering elemental fingerprints. The presence of molybdenum (Mo) and oxygen (O) peaks reveals the existence of MoO<sub>3</sub> within the nanocomposite. Additionally, the detection of carbon (C) signals the presence of rGO, while diverse peaks corresponding to palladium validate the integration of Pd NPs within the nanocomposite structure. Analysis of the EDS spectrum further reveals that the four elements C, O, Mo, and Pd are uniformly spread across the composite, with no evidence of impurities or additional elements apart from silicon. The table shows the elemental composition with carbon (C) 37.58 mass% (60.64 atom%), oxygen (O)

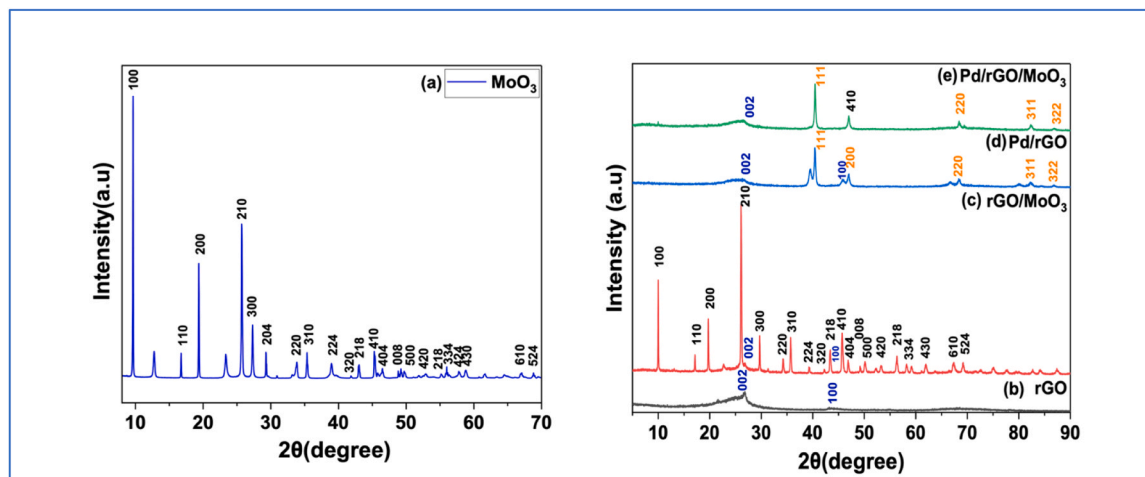


Fig. 3. XRD of (a) MoO<sub>3</sub>, (b) rGO, (c) rGO/MoO<sub>3</sub>, (d) Pd/rGO, and (e) Pd/rGO/MoO<sub>3</sub> nanocomposite.

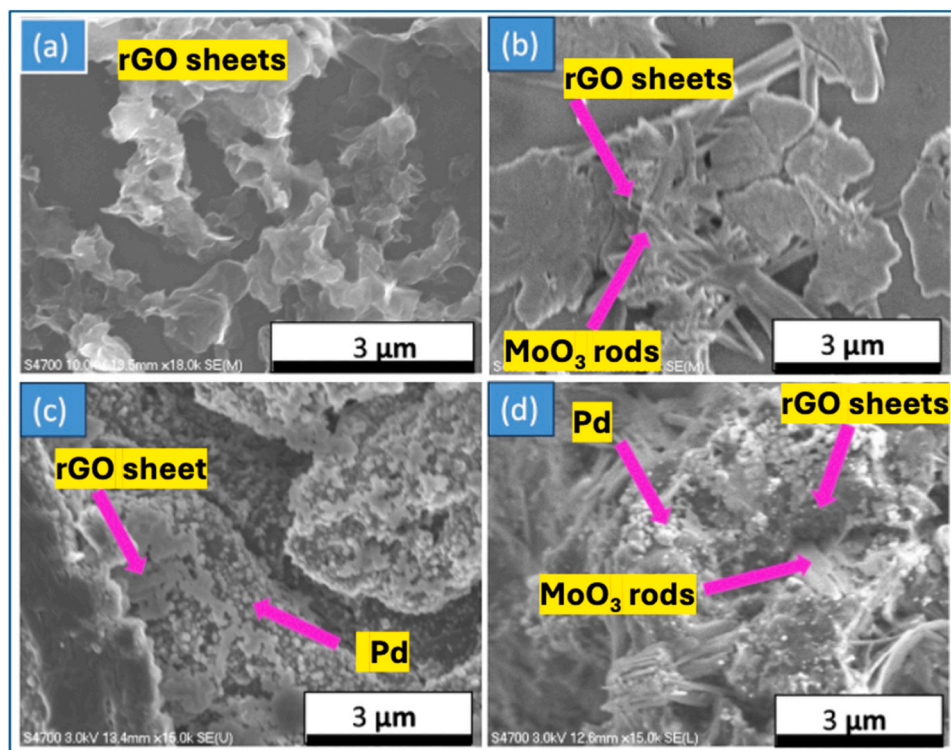


Fig. 4. FESEM of (a) rGO, (b) rGO/MoO<sub>3</sub>, (c) Pd/rGO, (d) Pd/rGO/MoO<sub>3</sub> nanocomposite.

22.48 mass% (27.23 atom%), silicon (Si) 8.61 mass% (5.94 atom%), molybdenum (Mo) 24.68 mass% (4.99 atom%), and palladium (Pd) 6.66 mass% (1.21 atom%). The presence of Mo and Pd reveals the successful doping of rGO, a ternary composite material containing the elements. This ternary material composition is adapted for photocatalytic or electronic applications. This comprehensive SEM-EDS characterization provides crucial insights into the surface properties and elemental composition of the Pd/rGO/MoO<sub>3</sub> ternary composite, essential for understanding its potential applications and performance in various fields.

### 3.5. TEM analysis

Fig. 6 displays the HRTEM images of Pd/rGO/MoO<sub>3</sub> nanocomposite, providing an early insight into the structural arrangement. Nonetheless, high-magnification imaging is essential for a comprehensive knowledge of nanocomposite morphology. Consequently, high-resolution transmission electron microscopy (HRTEM) was employed to achieve enhanced structural resolution. The resultant image, shown in Fig. 6, provides a comprehensive representation of the heterostructure. Upon careful examination, it becomes clear that the delicate and transparent rGO nanosheets serve as a supporting substrate. They are responsible for the distribution of MoO<sub>3</sub> nanorods and Pd nanoparticles, which are shown as smaller spheres distributed across the nanosheets. The incorporation of these components into the heterojunction materials is systematically organized, facilitating efficient electron transport processes. The proper contact enhances charge separation times, thereby improving the electrochemical performance of the materials.

### 3.6. Raman analysis

Raman spectroscopy serves as an effective and non-invasive method for examining the structural and electronic properties of nanocomposites [46–50]. Fig. 7 shows the spectra of MoO<sub>3</sub>, rGO, Pd/rGO, and Pd/rGO/MoO<sub>3</sub> samples analyzed in this study. As shown in Fig. 7(a), the MoO<sub>3</sub> nanostructures possess highly crystallized Raman-active modes ranging from 100 to 1600 cm<sup>-1</sup>. The peaks at

193 cm<sup>-1</sup> (O=M=O winding), 282 cm<sup>-1</sup> (O=M=O oscillating), 335 cm<sup>-1</sup> (O-M-O twisting), 376 cm<sup>-1</sup> (O-M-O slicing), 663 cm<sup>-1</sup> (O-M-O stretching), 819 cm<sup>-1</sup> (M=O symmetrical stretching), and 993 cm<sup>-1</sup> (M=O asymmetric broadening) are characteristics of pure MoO<sub>3</sub>. The peaks indicate the fabrication of MoO<sub>3</sub> nanorods. [48–53]. In detail, two peaks at 148 cm<sup>-1</sup> and 123 cm<sup>-1</sup> relate to the translation of a stiff chain. As such, these results confirm earlier findings and prove the existence of MoO<sub>3</sub> nanostructures. This suggested interaction would be investigated further in the analysis of the Raman spectra of rGO, Pd/rGO, and Pd/rGO/MoO<sub>3</sub>. The Raman spectra for two additional samples, the Pd/rGO and Pd/rGO/MoO<sub>3</sub> samples, showed four additional peaks. Raman spectra of rGO samples are shown in Fig. 7(b). The spectrum of rGO exhibits four characteristic peaks at 1352.96 cm<sup>-1</sup> (D band), 1584 cm<sup>-1</sup> (G band), 2705 cm<sup>-1</sup> (2D band), and 2925 cm<sup>-1</sup> (D + G band). A strong D band is a sign of high structural defects and disorder, whereas the G band is attributed to the graphitic sp<sup>2</sup> carbon network. The wide 2D band indicates a few layers rather than a monolayer of graphene. The existence of the D+G combined band further confirms the presence of structural defects. The high I<sub>D</sub>/I<sub>G</sub> represents the partial reduction of the graphene oxide and the recovery of the connected regions, which is typical for rGO materials. In comparison, the G band appears at around 1584 cm<sup>-1</sup> and corresponds to a first-order E<sub>2g</sub> phonon mode and the stretching vibrations of C-C bonds [53,54]. In Fig. 7(c), although Pd nanomaterials are incorporated, several rGO signals can still be observed (in the 100–3200 cm<sup>-1</sup> range) within the excited state spectra of Pd/rGO. The properties of robust Raman scattering of graphene-based materials, such as rGO, particularly the presence of the distinguishable D and G bands, reduce the characteristic peaks of other components, such as Pd and MoO<sub>3</sub>, from both nanocomposites [55–58]. Due to these interactions, the vibrational spectra of Pd/rGO nanocomposites may have lower intensity than the pure rGO. Raman spectroscopy could not confirm the existence of palladium due to its metallic nature. Therefore, we employed XPS to identify the Pd 3d core-level signals, demonstrating the addition of palladium to the graphene surface. Fig. 7(d) The Raman spectrum of the MoO<sub>3</sub>/rGO nanocomposite exhibits peaks for MoO<sub>3</sub> and rGO. A prominent band at

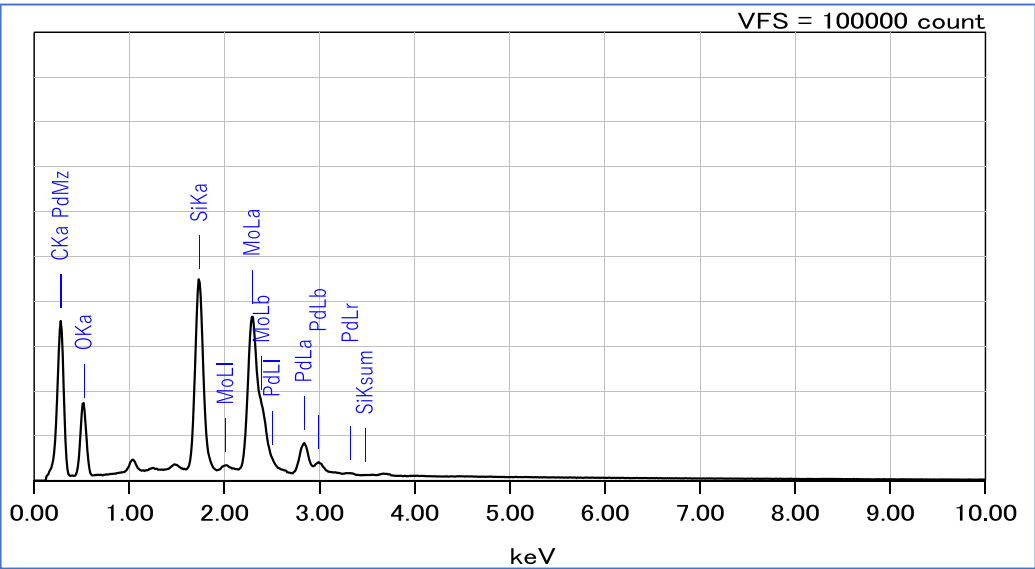
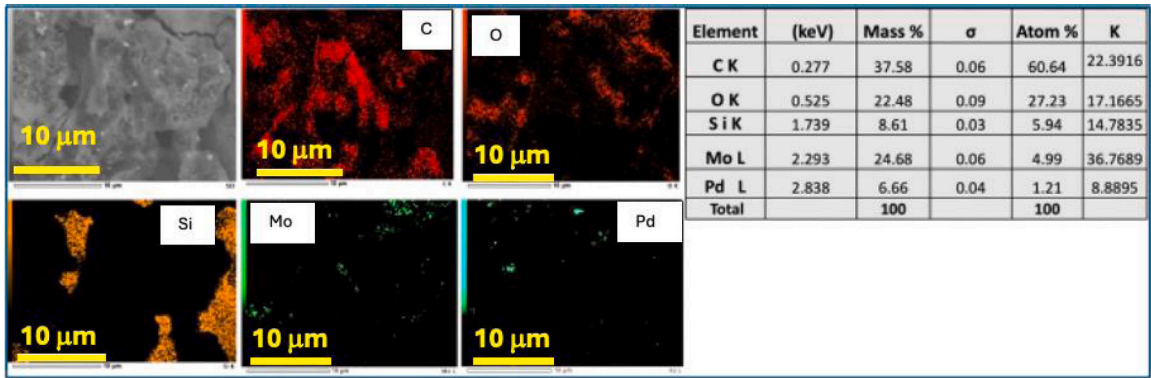


Fig. 5. SEM image, EDS spectra, and elemental mapping of Pd/rGO/MoO<sub>3</sub> nanocomposite.

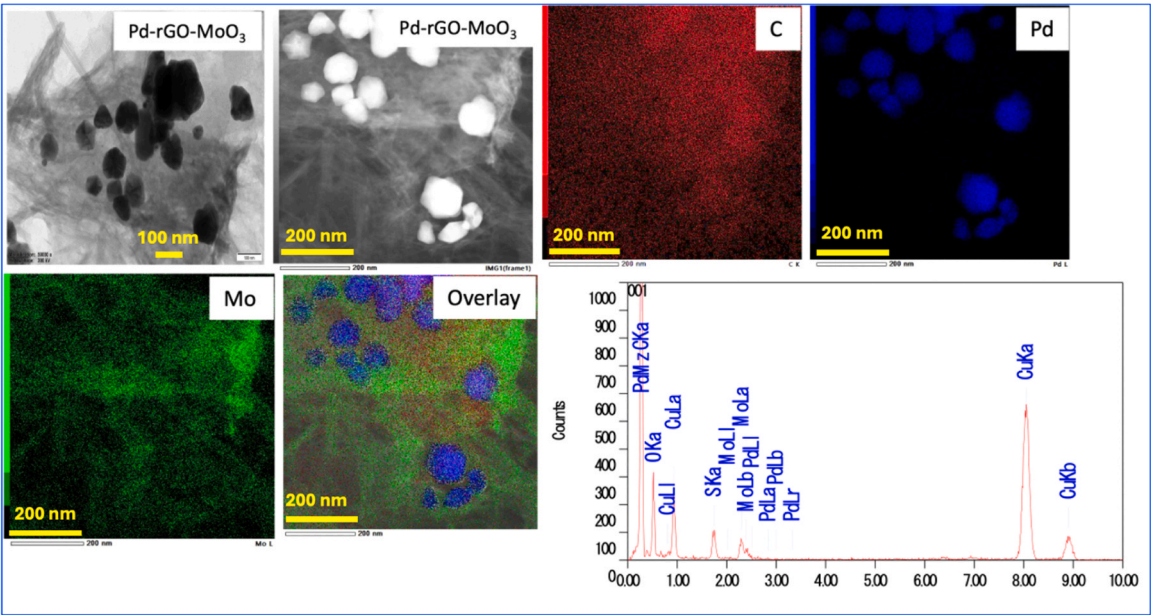


Fig. 6. TEM images, elemental mapping, and EDS spectra of Pd/rGO/MoO<sub>3</sub> nanocomposite.

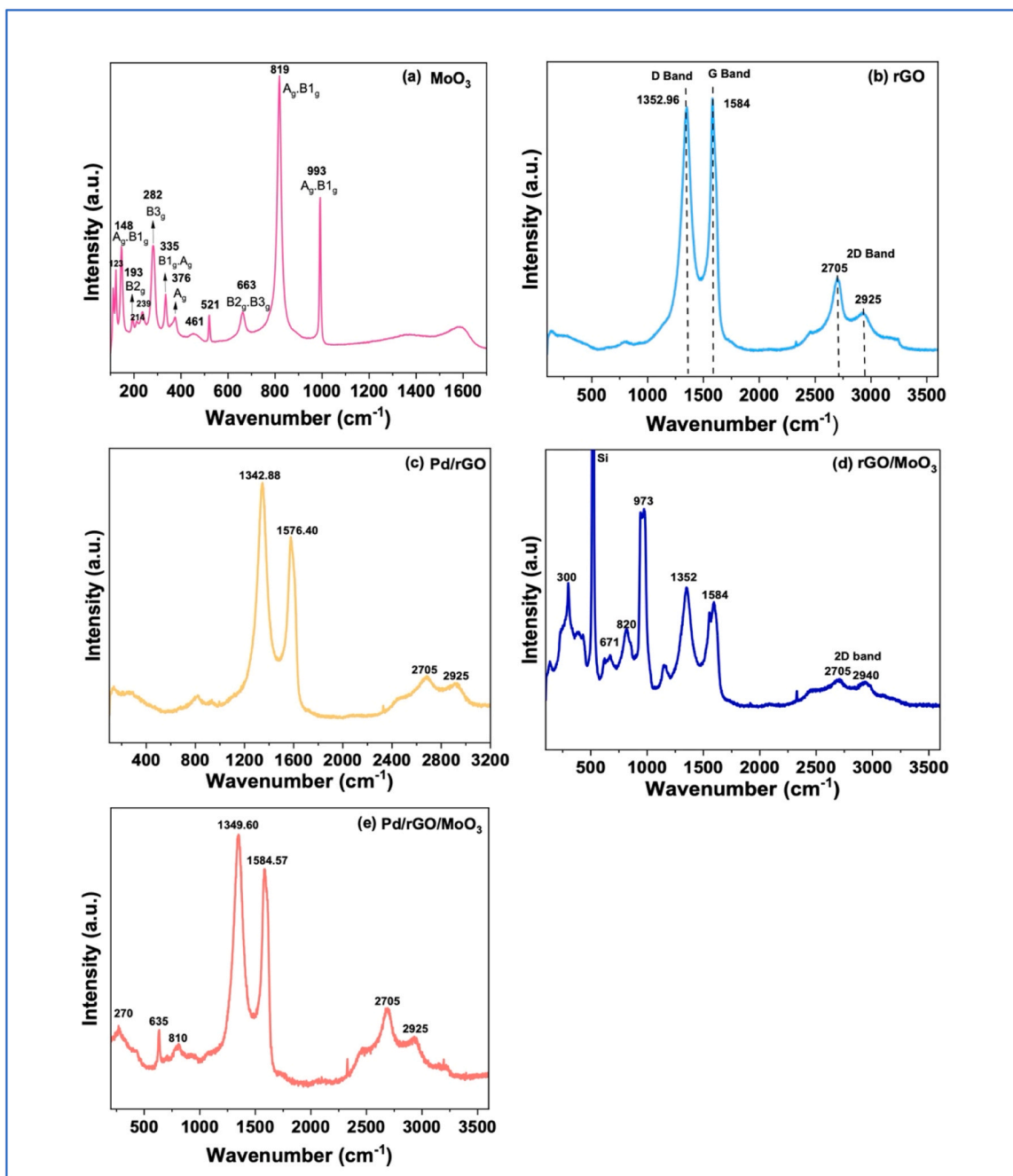


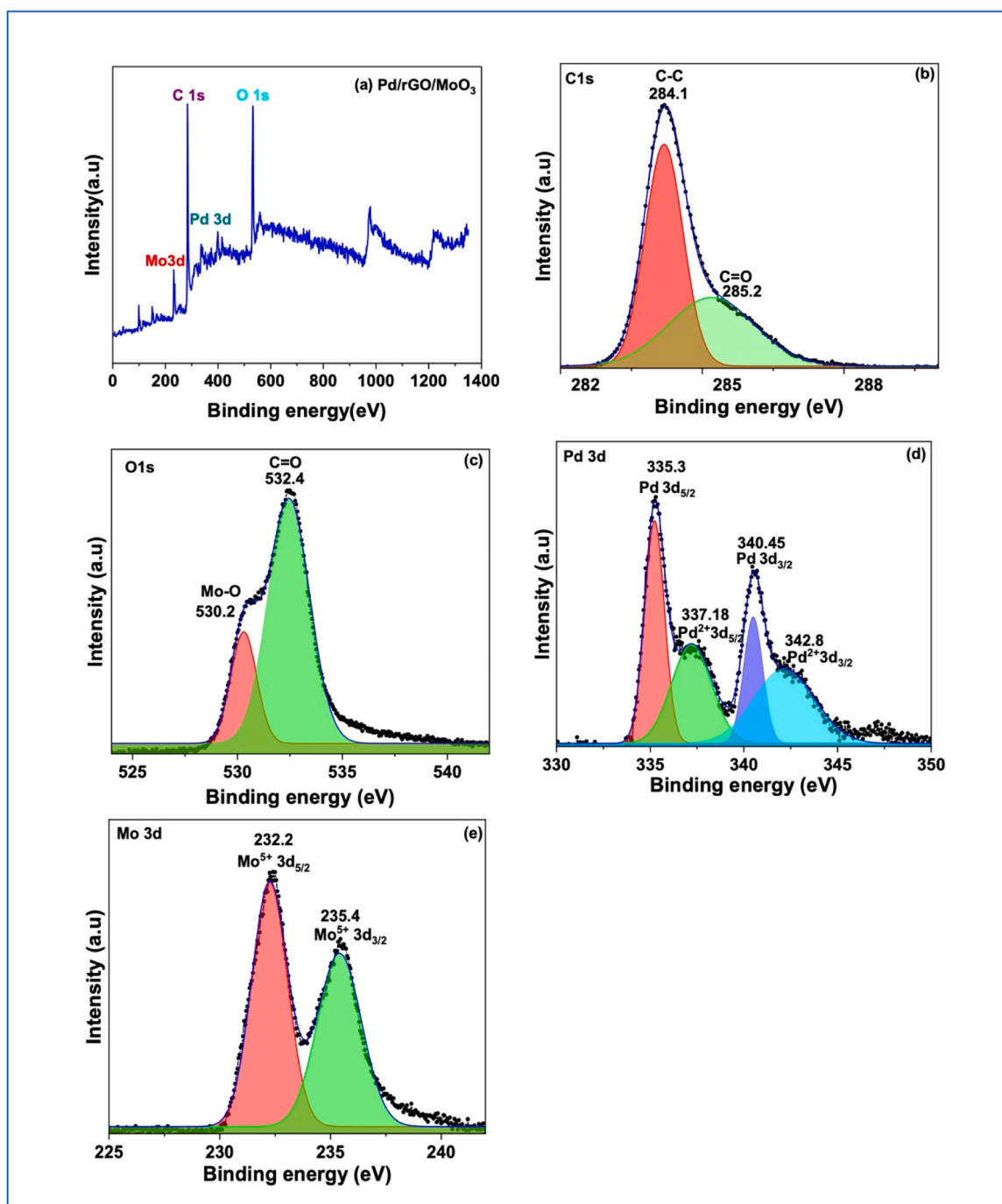
Fig. 7. Raman spectra of Pd/rGO/MoO<sub>3</sub>: (a) MoO<sub>3</sub> nanorods, (b) rGO, (c) Pd/rGO, (d) rGO/MoO<sub>3</sub>, and (e) Pd/rGO/MoO<sub>3</sub> nanocomposite.

~973 cm<sup>-1</sup> can be assigned to the Mo=O stretching vibration, and the bands around 671–820 cm<sup>-1</sup> are related to O–Mo–O bending and stretching vibration that corresponds to MoO<sub>3</sub>. In addition, D and G bands of rGO are observed around 1352 cm<sup>-1</sup> and 1584 cm<sup>-1</sup>. In Fig. 7 (e), the vibrational behavior is indicated qualitatively in Raman spectra of the Pd/rGO/MoO<sub>3</sub> hybrid, which leads to the existence and interplay of all three constituents. The characteristic peaks at approximately 1349.60 cm<sup>-1</sup> (D band) and 1584.57 cm<sup>-1</sup> (G band) could be assigned to the disordered and graphitic structures of rGO, respectively [59–62]. The peaks reflect the sp<sup>2</sup>-hybridized C atoms and a high degree of structural disorder, which are favorable to the anchoring of metal and oxide particles. The emergence of other peaks at 2705 cm<sup>-1</sup> and 2925 cm<sup>-1</sup> corresponds to the 2D band and the D+G combination mode, respectively, which further confirms the few layers of rGO with the partially recovered conjugated  $\pi$ -system. Significant Raman peaks at

270 cm<sup>-1</sup>, 635 cm<sup>-1</sup>, and 810 cm<sup>-1</sup> related to the vibrational modes of hexagonal MoO<sub>3</sub>, including O–Mo–O bending and Mo=O stretching modes, indicate that the metal oxide phase is doped. Although Pd does not have direct Raman-active modes as a metal, the slight displacement of the D and G bands compared to rGO indicates a possibility of the charge transfer interaction or the lattice strain introduced by the Pd NP decoration. Such interactions promote electronic conductivity and synergy between Pd, MoO<sub>3</sub>, and rGO [63,64].

### 3.7. Composition and chemical state analysis

High-magnification XPS analysis of the Pd/rGO/MoO<sub>3</sub> nanocomposite (Fig. 8(a)) gives the peaks obtained from the elemental composition of the elements at 232.2 eV, 284.1 eV, 335.3 eV, and 532.4 eV, respectively. These peaks correspond to the levels of Mo3d, C



**Fig. 8.** XPS spectra of Pd/rGO/MoO<sub>3</sub>: (a) full survey scan, and high resolution scans for (b) C 1 s, (c) O 1 s, (d) Pd 3d, (e) Mo 3d, and (f) elemental composition of materials.

1 s, Pd3d, and O1s [40]. The peaks of C1s illustrated in Fig. 8(b) have undergone deconvolution into two distinct peaks, which represent the graphitic roll floor and the relevant oxygen atmospheres (C=C/C-O at 284.1/285.2 eV). Fig. 8(c) O 1 s peak of Pd/rGO/MoO<sub>3</sub> could also be deconvoluted into two lines corresponding to 530.2 and 532.4 eV. The Mo-O pairs exist in the MoO<sub>6</sub> eight-faced polyhedron, and further oxygen-containing groups (C-O) are present on the rGO surface [43]. Fig. 8(d) shows the Pd 3d region for Pd/rGO/MoO<sub>3</sub>, which displays two peaks for neutral Pd<sup>0</sup> (335.3 and 337.18 eV) and two peaks for bivalent Pd<sup>2+</sup> (340.45 and 342.8 eV) [40–43]. Specifically, the bond energies corresponding to the unbiased Pd 3d band are reduced by 0.8 eV and 0.5 eV from that of Pd/rGO, respectively. This alteration signifies a

shift in the electronic structure of Pd due to the substitution of Mo [44]. The downward shift in the Pd bond energy suggests that Pd donates charge to MoO<sub>3</sub> [45], where the Mo atoms interact with Pd in the nanocomposite. This may reduce the electron concentration surrounding Pd and diminish binding energy. In addition, the peaks of the Mo 3d in the Pd/rGO/MoO<sub>3</sub> sample appear at lower binding energies compared with pristine MoO<sub>3</sub> nanorods. In particular, the peaks at 232.2 and 235.4 eV are assigned to the Mo 3d<sub>5/2</sub> and Mo 3d<sub>3/2</sub> of Mo<sup>5+</sup> in Pd/rGO/MoO<sub>3</sub>, respectively. The Mo 3d peak can be deconvoluted into two separate components, which were observed at 232.2 and 235.4 eV, respectively (Fig. 8(e) shows). These binding energies are lower than those of pure MoO<sub>3</sub>, suggesting a change in the oxidation state of the Mo

3d, and this is ascribed to an electron transfer mechanism compatible with the existence of oxygen vacancies in the Pd/rGO/MoO<sub>3</sub> sample. These vacancies come from annealing in the hydrothermal synthesis of Pd/rGO/MoO<sub>3</sub>. The successful synthesis of the Pd/rGO/MoO<sub>3</sub> ternary nanocomposite and the significant alteration of the electronic structures of Pd and MoO<sub>3</sub> can be confirmed by XPS analysis. It can be anticipated that the charge transfer between the Pd or rGO (Pd/rGO) and MoO<sub>3</sub> is beneficial to enhancing the photocatalytic performance of the Pd/rGO/MoO<sub>3</sub> nanocomposite and broadening photocatalytic applications. In Fig. 8(f), the table of XPS atomic concentration data shows the elemental composition of different material samples. The atomic composition of rGO mainly approximates C1s (85.87 %) and O1s (14.13 %), while the light state MoO<sub>3</sub> contains a high content of Mo 3d (13.40 %), C1s (45.24 %), and O1s (41.36 %). The Pd 3d (0.98 %) content is higher than that of C1s (80.55 %) in Pd/rGO. Mo 3d (20.09 %), C1s (60.33 %), and O1s (19.58 %) were the constituents of the rGO/MoO<sub>3</sub> nanocomposite. Finally, Pd/rGO/MoO<sub>3</sub> presents the most balanced composition with C1s (54.39 %), O1s (18.03 %), Mo 3d (16.69 %), and abundant Pd 3d (10.89 %), confirming the successful embedding of palladium and molybdenum into the surface of the rGO matrix.

### 3.8. Electrochemical impedance spectroscopy (EIS)

The electrochemical impedance spectroscopy (EIS) Nyquist plot (Fig. 9) was carried out in a 1 M KOH electrolyte to study the dynamics of the electrode-electrolyte interface and the charge transfer resistance of the electrocatalysts. The charge transfer resistance (R<sub>ct</sub>), which is a crucial metric showing the rate of electron transfer at the electrode interface, is represented by the diameter of the semicircle in the high-frequency area. Faster reaction kinetics and improved electrocatalytic performance are indicated by a lower charge transfer resistance (R<sub>ct</sub>) value, which is represented by comparatively smaller semicircle. The rGO shows the semicircle with larger diameter, i.e., the highest charge transfer resistance, which is 20.52 Ω. The Pd/rGO composite exhibits a somewhat lower semicircle diameter with comparatively less charge transfer resistance of 20.12 Ω following palladium decoration on rGO, which indicates an improvement in charge transfer. This trend is consistent with studies where supporting Pd nanostructures on graphene led to enhanced electronic conductivities and better charge transfer. The Pd/rGO/MoO<sub>3</sub> nanocomposite exhibits the smallest semicircle, which is noteworthy

because it has the lowest charge transfer resistance i.e. 18.36 Ω.

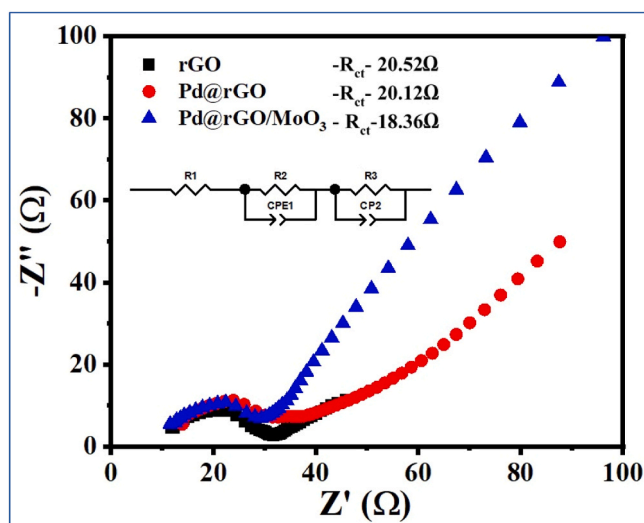


Fig. 9. Electrochemical Impedance Spectroscopy (EIS) Nyquist Plots of rGO, Pd/rGO, and Pd/rGO/MoO<sub>3</sub> Composites.

When compared to both rGO and Pd/rGO, this notable decrease in resistance suggests that the Pd/rGO/MoO<sub>3</sub> composite has the most desirable charge transfer kinetics. This improvement is ascribed to the synergistic interaction between the MoO<sub>3</sub> and the highly conductive palladium nanoparticles, which greatly speeds up the charge transfer process at the electrode-electrolyte interface and produces a more effective electron transfer pathway. The enhanced electrocatalytic activity and lower charge transfer resistance in such nanocomposites is often attributable to interface synergism between the different components.

### 3.9. Photocatalytic dye treatment

Fig. 10(a-d) displays the photocatalytic efficiency of the comparison of unitary, binary, and ternary photocatalysts, namely pure rGO, binary rGO/MoO<sub>3</sub>, binary Pd/rGO, and ternary Pd/rGO/MoO<sub>3</sub> nanocomposites, in the degradation of MB in viscous solutions under visible light. The distinct absorption peak of MB at 750 nm progressively decreased when exposed to light. The time-dependent UV-vis absorption spectra, which monitored MB degradation over 60 min, demonstrated the photocatalytic effectiveness of all catalysts. The fading blue tone of MB indicated its degradation, hastened by the catalysts. The kinetics of photocatalytic degradation of MB were investigated using first-order rate constants following 60 min of light exposure.

The evaluation revealed degradation efficiencies of 55 % for rGO, 72 % for Pd/rGO, 77 % for rGO/MoO<sub>3</sub>, and 89 % for the Pd/rGO/MoO<sub>3</sub> nanocomposite with only 60 min of LED light irradiation. The findings highlight the enhanced photocatalytic characteristics of the ternary Pd/rGO/MoO<sub>3</sub> nanocomposite relative to its equivalents. The increased degradation efficiency indicates synergistic interactions among Pd nanoparticles, rGO, and MoO<sub>3</sub>, resulting in enhanced photocatalytic activity and, therefore, offering prospects for diverse environmental remediation applications.

### 3.10. Photocatalytic kinetic analysis

The first-order rate Eq. 1 was used to investigate the photocatalytic degradation kinetics of MB.

$$\text{Rate} = -dc/dt = KC \quad (3)$$

where K is the rate constant and C is the MB concentration.

The photocatalytic degradation kinetics of MB dye were analyzed using a pseudo-first-order model described by the equation  $\ln(C_0/C_t) = kt$ , where  $C_0$  and  $C_t$  represent the dye concentrations at the initial and irradiation times  $t$ , respectively, and  $k$  denotes the apparent rate constant ( $\text{min}^{-1}$ ). The linearity of the  $\ln(C_0/C_t)$  versus  $t$  plots ( $R^2 > 0.98$ ) confirmed that the degradation of MB followed pseudo-first-order kinetics. The calculated  $k$  values were 0.018, 0.027, 0.032, and 0.042  $\text{min}^{-1}$  for rGO, Pd/rGO, rGO/MoO<sub>3</sub>, and Pd/rGO/MoO<sub>3</sub> nanocomposites, respectively, with corresponding  $R^2$  values of 0.981, 0.986, 0.989, and 0.994. Among all the catalysts, Pd/rGO/MoO<sub>3</sub> exhibited the highest apparent rate constant, approximately 2.3 times greater than that of pure rGO, indicating its superior charge-transfer capability and visible-light-driven photocatalytic activity. This enhanced kinetic performance arises from the synergistic interaction among Pd nanoparticles, which contribute plasmonic hot-electron generation; rGO, which provides high conductivity and a large surface area for rapid charge transport; and MoO<sub>3</sub>, which offers strong oxidative potential, collectively facilitating efficient charge separation and accelerated photodegradation of MB.

## 4. Effect of pH on the photodegradation performance

Fig. 12. (a), (b), and (c) UV-Vis absorption spectra illustrating the effect of pH on the photocatalytic degradation of MB using the Pd/rGO/

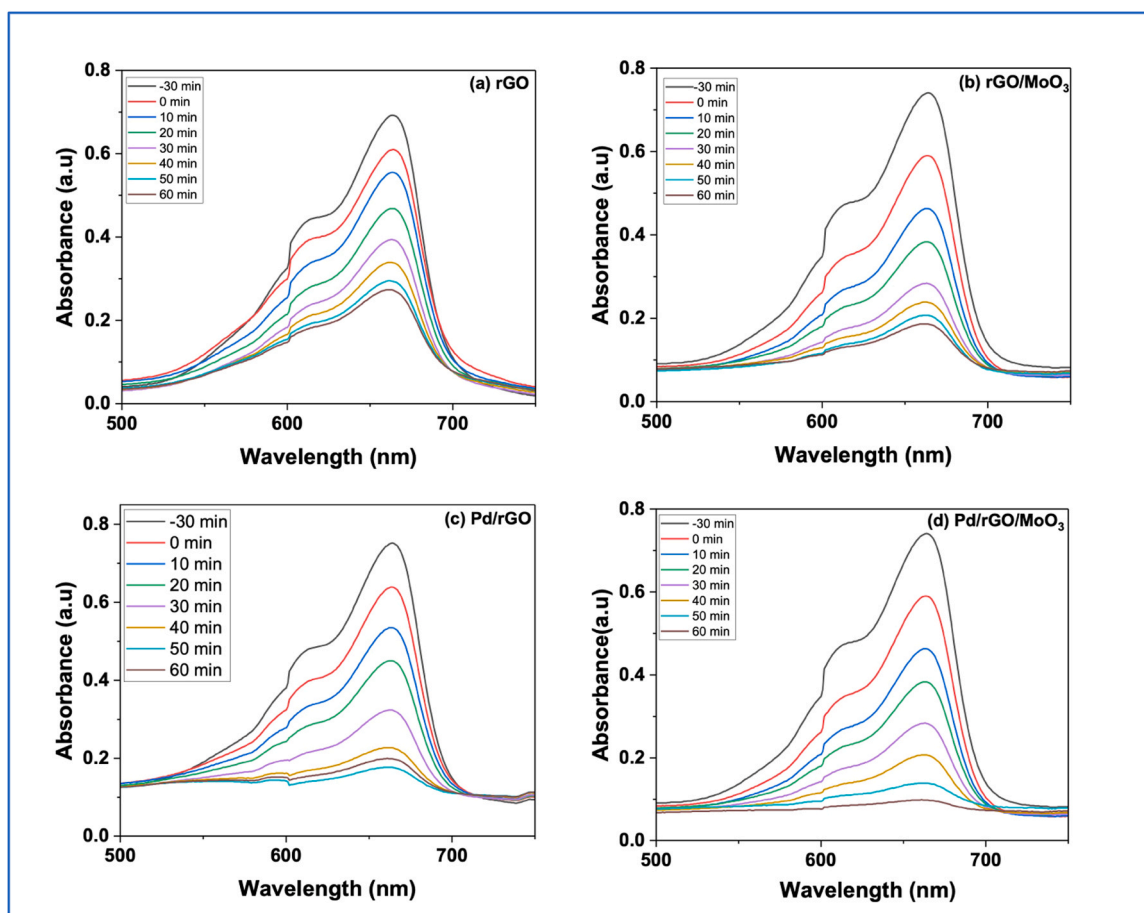


Fig. 10. Photocatalysis degradation of MB (a) rGO (b) rGO/MoO<sub>3</sub> (c) Pd/rGO (d) Pd/rGO/MoO<sub>3</sub> nanocomposite.

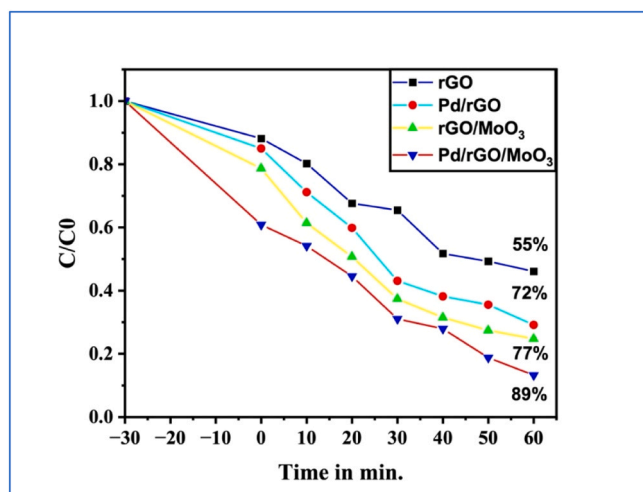


Fig. 11. Degradation efficiency of rGO, Pd/rGO, rGO/MoO<sub>3</sub>, and Pd/rGO/MoO<sub>3</sub> nanocomposite.

MoO<sub>3</sub> catalyst under visible light irradiation. The degradation was examined under acidic (pH 4, adjusted with HCl), neutral (pH 6.5), and basic (pH 8.5, adjusted with KOH) conditions. As shown, the catalyst exhibited the

highest degradation efficiency under basic conditions, which can be attributed to the optimal surface charge interaction and effective charge-carrier separation within 10 min, resulting in 94 % degradation; after that, there is no degradation. In an acidic medium, the degradation rate

of 36 % decreased slightly due to enhanced protonation and competitive adsorption of H<sup>+</sup> ions on the catalyst surface. In neutral conditions, the degradation performance was significantly suppressed, resulting in nearly an 87 % reduction, likely due to dye aggregation and reduced adsorption on the catalyst surface. These results indicate that solution pH plays a crucial role in photocatalytic activity; according to these results, basic pH provides the most favorable environment for efficient photodegradation.

#### 4.1. Reusability and stability of Pd/rGO/MoO<sub>3</sub> nanocomposite

Fig. 13 illustrates the recyclability performance of the Pd/rGO/MoO<sub>3</sub> nanocomposite during the photodegradation of MB dye under simulated sunlight. The catalyst was subjected to five consecutive degradation cycles to evaluate its stability and reusability. Throughout these repeated experiments, no significant decline in photocatalytic efficiency was observed, with degradation rates consistently maintained in the range of 82–89 %. This stable performance highlights the excellent structural integrity and durability of the Pd/rGO/MoO<sub>3</sub> photocatalyst. The sustained activity across multiple cycles confirms that the synthesized nanocomposites can be effectively reused without significant loss of catalytic performance, making them promising candidates for practical wastewater treatment applications.

#### 4.2. Analysis of catalytic process and mechanism

The photocatalytic degradation mechanism of MB dye using Pd/rGO/MoO<sub>3</sub> nanocomposites involves a synergistic interplay of light absorption, charge separation, generation of reactive oxygen species (ROS), and subsequent dye degradation. The Pd/rGO/MoO<sub>3</sub>

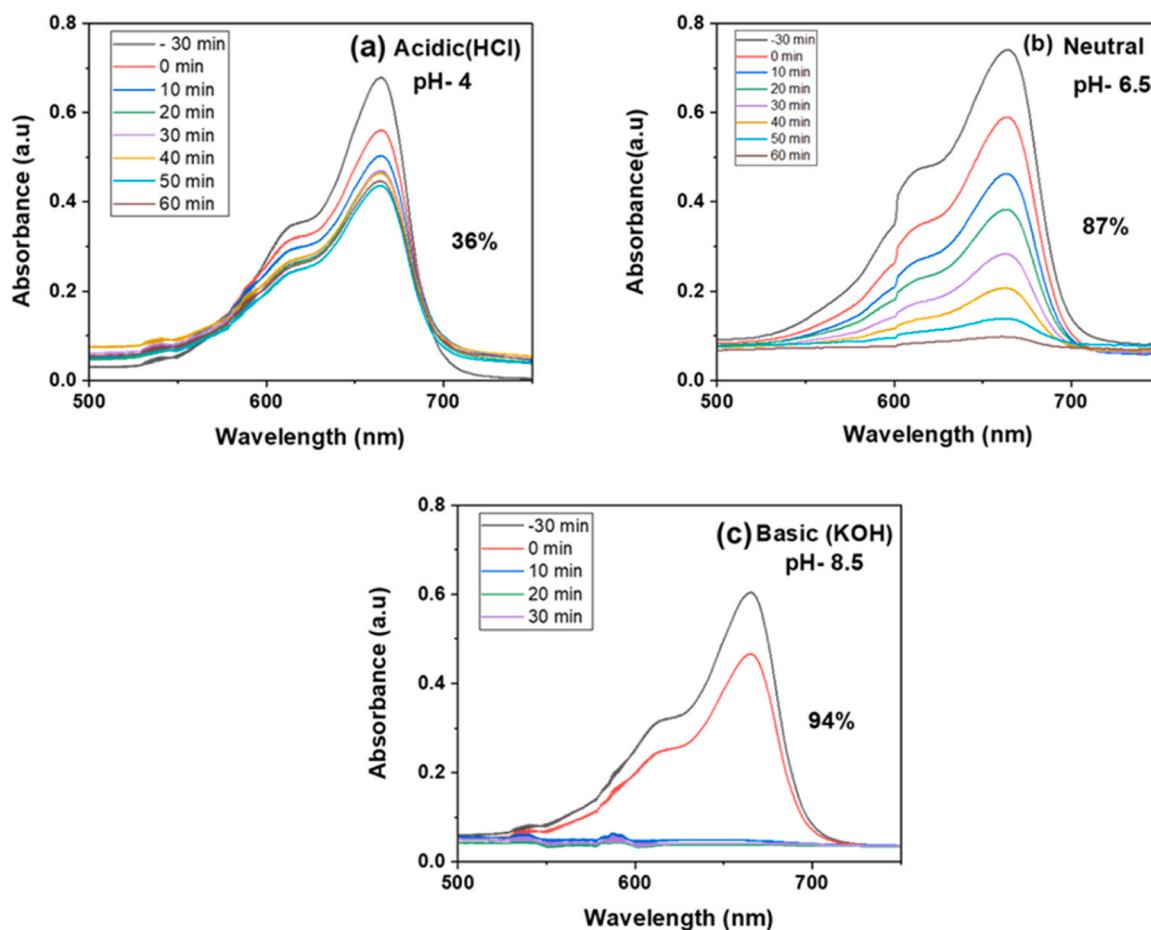


Fig. 12. (a), (b), and (c) UV-Vis absorption spectra showing the effect of pH on the photodegradation performance of the Pd/rGO/MoO<sub>3</sub> catalyst.

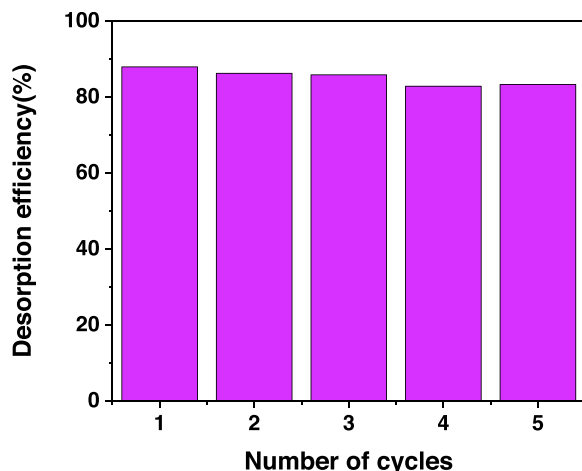
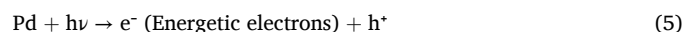
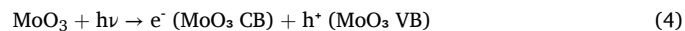


Fig. 13. Cycling runs of the Pd/rGO/MoO<sub>3</sub> nanocomposite for the photodegradation of MB dye under visible-light irradiation.

nanocomposite acts as an efficient photocatalyst due to its unique structural and electronic properties, which facilitate the generation and separation of charge carriers, ultimately leading to the degradation of MB dye. As the light irradiates the Pd/rGO/MoO<sub>3</sub> nanocomposite, the photons are absorbed by the MoO<sub>3</sub> nanorods, generating the electron-hole pairs via excitation of valence band electrons to the conduction band. These photogenerated electrons are sequentially transferred to the graphitic rGO structure.

Meanwhile, the holes in MoO<sub>3</sub> nanorods were available for oxidation reaction. The inherent nature of Pd nanoparticles exhibits the localized surface plasmon resonance (LSPR) effect, which enhances the absorption of visible light. These hot electrons are injected back into the rGO-MoO<sub>3</sub> system, boosting the carrier density and extending the absorption spectrum to a wide visible range [65]. The rGO layered framework serves as a conductive bridge for photogenerated electron transport from MoO<sub>3</sub> to Pd nanoparticles, which act as an active sink, suppressing charge recombination and enabling prolonged charge separation. Meanwhile, the dye adsorbed nanoparticles facilitate a mediator for charge transfer during photocatalysis [68]. The photogenerated electrons react with O<sub>2</sub> molecules in the solution to form ROS such as superoxide radicals (•O<sub>2</sub><sup>-</sup>) and hydroxyl radicals (•OH). These ROS are highly reactive and play a crucial role in the degradation of MB dye molecules [69].

The generated ROS attacks the MB dye molecules, breaking them down into smaller, non-toxic compounds. The high surface area and active sites of Pd/rGO nanocomposite facilitate this process, leading to efficient dye degradation [70]. The ternary Pd/rGO/MoO<sub>3</sub> system outperforms binary and single-component catalysts due to improved charge mobility, extended light absorption, and efficient ROS generation. The multi-component Pd/rGO/MoO<sub>3</sub> nanocomposite highlights the photocatalytic efficiency through improved charge separation and ROS generation [71–73]. The detailed band photocatalytic reaction is highlighted in Eqs. 4–10, respectively.



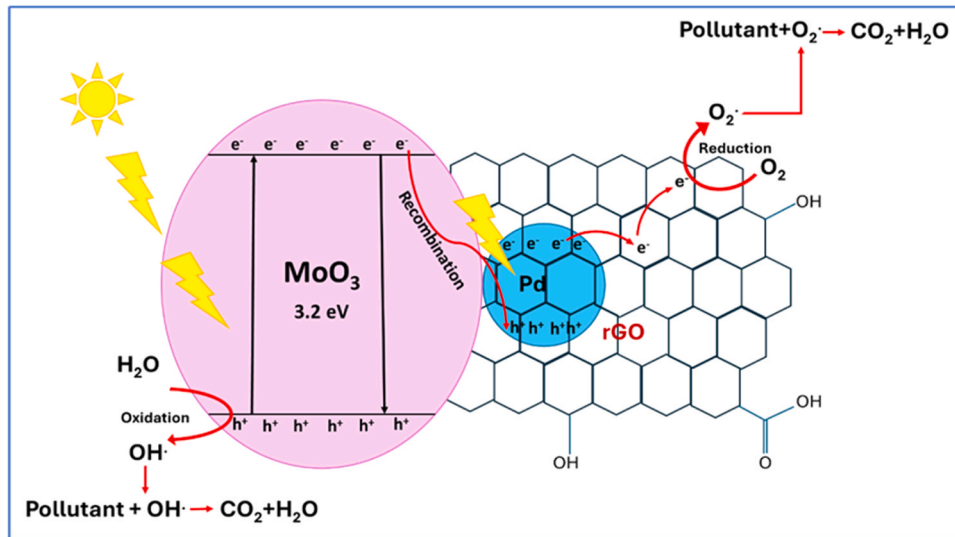


Fig. 14. Possible Photocatalysis Mechanism of Pd/rGO/MoO<sub>3</sub> Nanocomposite.

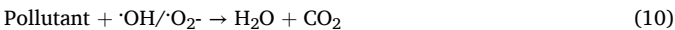


Table 1 summarizes the photocatalytic performance of MoO<sub>3</sub>-based photocatalysts from previously reported results. The photocatalytic efficiency of the Pd/rGO/MoO<sub>3</sub> nanocomposite surpasses all major photocatalysts for degradation rate and catalytic activity due to the synergic effect between the Pd NPs, MoO<sub>3</sub> nanorods, and rGO nanosheet in facilitating the efficient reduction of recombination of photogenerated electron-hole pairs.

## 5. Conclusion

In this work, a facile hydrothermal method was used to prepare two-part and three-part nanocomposites. These include rGO/MoO<sub>3</sub>, Pd/rGO, and Pd/rGO/MoO<sub>3</sub>. The formation of a unique ternary nanocomposite consisting of interconnected MoO<sub>3</sub> nanorods decorated on rGO sheets with Pd nanoparticles was identified by various characterization techniques such as XRD, UV-vis, FESEM, TEM, Raman, and XPS.

Each of the samples was characterized by X-ray diffraction to confirm both the crystalline nature and the states of the phases in the samples. FESEM and TEM examine the morphologies of every composition of the samples. In addition, the different vibrational modes in the samples were identified and confirmed through Raman analysis, making Raman measurement helpful in the phase confirmation. Optical absorption spectra showed that the nanocomposite is well dispersed in visible light with a smaller bandgap. On comparing the photocatalytic degradation studies, it is observed that all the samples have a good tendency to decompose the MB organic dye in repeated reactions. In photocatalytic tests, Pd/rGO/MoO<sub>3</sub> had a greater degradation than that of pure rGO, rGO/MoO<sub>3</sub>, and Pd/rGO. The results indicate that Pd/rGO/MoO<sub>3</sub> holds great potential as a photocatalytic material. This means improved utilization of nanomaterials in disinfecting various sectors of water, the environment, and energy. The Pd/rGO/MoO<sub>3</sub> photocatalyst shows strong visible-light activity, efficient dye degradation, and good recyclability, making it promising for real-world water treatment. It can be used for polishing textile effluents, decentralized dye removal with LED reactors, and continuous-flow systems with immobilized catalysts. Its effectiveness extends beyond MB to various dye pollutants, and future work will focus on real wastewater testing, fouling resistance, and scalable reactor designs.

Table 1

Summary of previously reported MoO<sub>3</sub>-based photocatalysts and their nanocomposite photocatalysis performance [66,67].

| Photocatalyst                                                        | Synthesis method                           | Degradation Efficiency (%) | Degradation duration | Dye       | Year        | Ref.              |
|----------------------------------------------------------------------|--------------------------------------------|----------------------------|----------------------|-----------|-------------|-------------------|
| h-MoO <sub>3</sub> nanocrystals                                      | Chemical precipitation                     | 80 %                       | 120 min              | MB        | 2013        | 57                |
| MoO <sub>3</sub> -supported SnO <sub>2</sub> nanocomposites          | Wet chemical                               | 83.97 %                    | 25 min               | RhB       | 2016        | 58                |
| MoO <sub>3</sub> /Fe <sub>2</sub> O <sub>3</sub> /rGO nanocomposites | In-situ hydrothermal                       | 99.47 %                    | 60 min               | MB        | 2018        | 59                |
| MoO <sub>3</sub> -x nanosheets                                       | Liquid exfoliation                         | 80 %                       | 60 min               | RhB & MB  | 2018        | 60                |
| 3D rGO/MoO <sub>3</sub> aerogels                                     | Self-assembly                              | 90 %                       | 120 min              | MB        | 2019        | 61                |
| MoO <sub>3</sub> nanosheets                                          | CVD                                        | 85 %                       | 50 min               | Cr (VI)   | 2019        | 62                |
| rGO/MoO <sub>3</sub> nanobelt-slab                                   | Two-step microwave-assisted hydrothermal   | 95 %                       | 120 min              | RhB       | 2023        | 63                |
| rGO/MoO <sub>3</sub> nanosorbent                                     | Facile hydrothermal                        | 99.5 %                     | 120 min              | MB        | 2024        | 64                |
| Z-scheme MoO <sub>3</sub> /Fe <sub>2</sub> O <sub>3</sub>            | Two-step microwave heating and calcination | 81.4 %                     | 180 min              | MB        | 2024        | 65                |
| Fe <sub>3</sub> O <sub>4</sub> /RGO                                  | Sol-gel and Hummers methods                | 93.3 %                     | 90 min               | MB        | 2019        | 66                |
| Ag-CoFe <sub>2</sub> O <sub>4</sub> /rGO                             | Hydrothermal process                       | 93.25 %                    | 100 min              | MB        |             | 67                |
| <b>Pd/rGO/MoO<sub>3</sub></b>                                        | <b>Hydrothermal Method</b>                 | <b>89 %</b>                | <b>60 min</b>        | <b>MB</b> | <b>2025</b> | <b>This study</b> |

## CRediT authorship contribution statement

**Akash Sawate:** Methodology, Characterization, Writing; **Gaurav Kumar Yogesh:** Methodology, Writing, review and Editing, **Tetsuro Katayama:** Characterization, review & editing, **Matin Patel** and **Bhaskar R. Sathe:** Electrochemical impedance studies and interpretations; **Rungsima Yeetsorn:** Writing, Review & editing, Supervision **Akihiro Furube:** Review & editing, Supervision, Funding acquisition; **Pankaj Koinkar:** Writing: Review & editing, Supervision, Conceptualization.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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