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SUSTAINABLE PHOTOCATALYTIC DEGRADATION OF IBUPROFEN USING GREEN SYNTHESIZED IRON-MOLYBDENUM OXIDE NANOCOMPOSITES UNDER SOLAR IRRADIATION

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Keywords: *Iron-Molybdenum oxide Nanocomposites, Green synthesis, Photocatalysis, Pharmaceutical contaminants*, *Ibuprofen, water treatment.*

ABSTRACT

This study describes a sustainable, highly efficient photocatalytic technique for degrading ibuprofen, a commonly detected pharmaceutical contaminant, using green-synthesised iron-molybdenum oxide nanocomposites (Fe-Mo oxide NCPs) under solar irradiation. As a non-steroidal anti-inflammatory drug (NSAID), ibuprofen poses considerable environmental hazards because of its persistence in aquatic environments and resistance to conventional wastewater treatment processes. The Fe- Mo oxide nanocomposites were synthesised using aloe vera leaf extract and characterised by UV–visible spectroscopy, Fourier Transform Infrared (FTIR) Spectroscopy, X-ray Diffraction (XRD), and Scanning Electron Microscopy coupled with Energy-Dispersive X-ray Spectroscopy (SEM–EDS). The synthesised Fe-Mo oxide NCs exhibited outstanding photocatalytic activity, achieving 98.8% removal of ibuprofen after 60 minutes at pH 3 under simulated solar light. The green synthesis route effectively lowers production costs and minimizes environmental harm, providing a promising, scalable, and eco-friendly strategy for water remediation. This photocatalytic process leverages solar energy to convert organic pollutants into harmless end-products such as carbon dioxide and water, representing an environmentally sustainable solution for the degradation of pharmaceutical compounds.

Keywords: *Iron-Molybdenum oxide Nanocomposites, Green synthesis, Photocatalysis, Pharmaceutical contaminants*, *Ibuprofen, water treatment.*

1.0 INTRODUCTION

Pharmaceutical contaminants, particularly non-steroidal anti-inflammatory drugs (NSAIDs) such as ibuprofen, represent a significant global environmental challenge. Due to its widespread use, ibuprofen is frequently detected as a persistent pollutant in various aquatic ecosystems worldwide. Consequently, conventional wastewater treatment methods are often insufficient for effectively removing these compounds, resulting in their accumulation in surface waters, groundwater, and drinking water sources. This accumulation poses significant ecological risks through bioaccumulation in aquatic organisms and may pose potential health concerns for human populations. Therefore, the development of sustainable and cost-effective treatment technologies for pharmaceutical pollutants remains a critical focus for environmental chemists and water quality specialists (Paul, 2015; Ruziwa *et al*., 2023).

Photocatalysis, an advanced oxidation process, has emerged as a promising technique because it harnesses solar energy to mineralize organic contaminants into harmless substances such as carbon dioxide

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and water. Hence, it offers an environmentally sustainable approach for pharmaceutical degradation (Ruziwa *et al*., 2023).

The recurrent detection of ibuprofen in aquatic environments has driven considerable research into photocatalytic degradation methods, including the use of iron-molybdenum oxide (Fe-Mo) nanocomposites and other nanostructured photocatalysts. Given ibuprofen's persistence and its potential endocrine-disrupting effects, photocatalysis under solar irradiation is recognized as an effective strategy for the breakdown of such pharmaceutical compounds. Specifically, this method generates highly reactive oxidative species capable of mineralizing organic pollutants into non-toxic end products. Moreover, the ability of photocatalysis to operate under visible light enhances its environmental relevance by enabling degradation processes under natural sunlight conditions (Paul, 2015; Tanveer *et al*., 2019).

Traditional photocatalysts, such as titanium dioxide (TiO_2) and zinc oxide (ZnO), have been widely studied for their ability to degrade pollutants under ultraviolet (UV) light. Titanium dioxide, in particular, demonstrates excellent photocatalytic activity; however, its effectiveness is limited because it only responds to UV light, which makes up a small portion of natural sunlight. Consequently, recent research has focused on developing advanced nanocomposites that can utilize a broader spectrum of light, especially visible light, to enhance degradation rates.

Iron oxides like Fe_2O_3 and Fe_3O_4 are notable for their magnetic properties, environmental friendliness, and ability to reduce band gaps, allowing them to work under visible light. In addition, adding iron oxides to semiconductor materials improves charge separation and reduces recombination of charge carriers, which leads to better photocatalytic performance. Similarly, molybdenum oxides (MoO_3) have demonstrated strong oxidative abilities, particularly when combined with silica (SiO_2) to form synergistic nanocomposites that can nearly completely degrade organic dyes within an hour under UV light. Therefore, combining iron and molybdenum oxides into Fe-Mo oxide nanocomposites enhances photocatalytic efficiency by improving charge transfer and expanding light absorption, making them promising candidates for degrading ibuprofen (Afkari *et al*., 2023; Kamoun *et al*., 2020).

Although specific studies on photocatalytic degradation of ibuprofen using Fe-Mo oxide nanocomposites are limited, early research on similar pharmaceutical pollutants is encouraging. Nanocomposites combining molybdenum and iron oxides have shown effective photocatalytic activity for degrading organic molecules under sunlight. Notably, incorporating iron oxides enhances the material's response to visible light, a key feature for solar-driven applications. Furthermore, the formation of heterojunctions between iron and molybdenum oxides improves charge separation, reducing recombination losses and increasing the availability of reactive radicals necessary for pharmaceutical degradation, including ibuprofen (Gong *et al*., 2021; Afkari *et al*., 2023; Bhestar *et al*., 2024). Importantly, photocatalytic efficiency depends on factors such as bandgap engineering, catalyst morphology, composition, and environmental conditions. In this context, iron oxides play an important role in narrowing the bandgap and extending light absorption into the visible range, while molybdenum oxides contribute oxidation potential. Optimizing the iron oxide content is essential, as too much can increase recombination. Additionally, acidic and neutral pH levels favor degradation, whereas complex water matrices may inhibit photocatalytic activity by scavenging reactive species (Saroa *et al*., 2023; Afkari *et al*., 2023).

Iron-based composites, such as Fe_3O_4@MIL-53(Fe) and BN/Fe_3O_4/MIL-

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53(Fe), exhibit impressive efficiency under visible light, achieving nearly complete degradation of ibuprofen within one to three hours. This performance results from the synergistic action of multiple reactive species (h^+, ^{-}OH, e−, O_2^-, SO_4^-) and improved separation of electron-hole pairs. Moreover, the magnetic properties of these composites enable easy recovery and reuse of the catalysts, enhancing their practical applications (Liu *et al*., 2020; Liu *et al*., 2022). Meanwhile, molybdenum-doped photocatalysts, including Mo-doped TiO_2 and Bi_5O_7I-MoO_3, show superior degradation performance due to increased generation of reactive surface species and enhanced visible light absorption (Anucha et al., 2022; Mafa et al., 2020). Other advancements involve modified TiO_2 systems combined with UV-LEDs, MXene, nanofibers, and g-C_3N_4-based composites, which promote rapid ibuprofen degradation while maintaining high stability and recyclability. Similarly, alternative metal oxides such as ZnO (and its doped forms), BiOCl, and CuO-polypyrrole composites have also proven effective, often producing non-toxic byproducts under visible light and exhibiting sustained reusability (Grzegórska *et al*., 2023; Alqahtani *et al*., 2025; Silva *et al*., 2024; Sá *et al*., 2021).

Mechanisms, By-Products, and Environmental Considerations of Photocatalytic degradation of IBU typically follows multi-step pathways involving hydroxylation, decarboxylation, and aromatic ring cleavage, ultimately leading to mineralization into CO_2 and H_2O (Alqahtani *et al*., 2025; Miranda *et al*., 2021). The main reactive species include ^{-}OH, O_2^-, SO_4^-, and h^+, with their relative importance depending on the catalyst system (Liu *et al*., 2020; Nguyen et al., 2023; Liu et al., 2022; Malinowska *et al*., 2022). While toxicity assessments indicate that partial oxidation intermediates may exhibit transient cytotoxicity, complete mineralization generally yields non-toxic products (Jallouli *et al*., 2018; Alqahtani *et al*., 2025; Sá *et al*., 2021). Importantly, most systems demonstrate good reusability and stability over multiple cycles.

Table 1 below depicts the types and photocatalytic activity of various nanocomposite catalysts reported for Ibuprofen degradation in water.

Table 1. Comparison of photocatalyst systems for ibuprofen degradation in water

Photocatalyst System	Light Source	Max. IBU Removal (%)	Time (min)	Key Features/Notes	Citations
Fe_3O_4@MIL-53(Fe)	Visible + H_2O_2	99	60	Magnetic, stable, fast	(Liu *et al*., 2020)
BN/Fe_3O_4/MIL-53(Fe) + Persulfate	Visible	100	180	Multiple radicals, DFT pathway	(Liu *et al*., 2022)
Mo-doped TiO_2	UV	~98	80	Enhanced by Mo doping	(Anucha *et al*., 2022)
Bi_5O_7I-MoO_3	Visible	89.2	-	Z-scheme, stable, visible light	(Mafa *et al*., 2020)
TiO_2/MXene/$MnFe_2O_4$ + PMS	Sim. solar	100	10	Magnetic, PMS activation	(Grzegórska *et al*., 2023)
$NiCo_2O_4$@g-C_3N_4 + PMS	Visible	96	60	Z-scheme, stable	(Nguyen *et al*., 2023)
CuO-polypyrrole/AC hydrogel	Visible	100	40	Fast, sustainable, low toxicity	(Alqahtani *et al*., 2025)
Er-doped ZnO	UV	52.3	120	Green synthesis	(Silva et al., 2024)
ZnO-Ce	UV	60	120	Non-toxic by-products	(Sá *et al*., 2021)

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2.0 MATERIALS AND METHODS

Two – steps Green Syntheses of Fe-Mo Oxide Nanocomposites

Fresh Aloe vera leaves were obtained from White House Residence Garden in Ishaka, Bushenyi district, Uganda. The leaves were washed with tap water, then rinsed with distilled water, chopped into small pieces, and heated in distilled water (20g of the aloe vera in 100 ml of distilled water) at 80°C for 1 hour to extract active phytochemicals. The extract was filtered with No. 1 Whatman filter paper to remove solid residues. Exactly 5.5605 g of ferrous sulphate was dissolved in distilled water (20 mM), and then 60 ml of the Aloe vera extract was added. The solution was heated at 70°C, after which 2M sodium hydroxide was slowly added dropwise under continuous stirring for 20 minutes until the pH reached 9, indicated by a dark green color, signifying the formation of iron oxide nanoparticles (IONPs). The solution was allowed to settle, decanted, dried in an oven at 120°C, ground to a fine powder, and stored (Morales, et al, 2021; Burange *et al*., 2021). Step 2 - For synthesizing iron–molybdenum oxide nanocomposites, the prepared IONPs (4g) were placed in a clean 1000 ml beaker with 100 ml distilled water to form a solution, then heated at 80°C on a magnetic stirrer. While stirring, 50 ml of 0.5M citric acid was added dropwise, maintaining the suspension pH between 10 and 11 by continuous monitoring and adjustment with NaOH and citric acid. Separately, 14.8303 g of ammonium heptamolybdate (AHM) was dissolved in 200 ml distilled water to make a 60 mM solution, of which 100 ml was added to the suspension and allowed to age for 2 hours under stirring. The mixture was dried in an oven at 120 °C, then further heated at 250 °C to remove residual citrate, yielding a black solid which was cooled, crushed with a mortar and pestle into a fine nanoparticle composite powder.

Characterization Techniques

Materials were characterized using various techniques:

Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR analysis was performed using an Agilent Cary 630 FTIR spectrometer, which features a high-emission infrared radiation base system. This system was coupled with a 5-bounce diamond ATR sampling accessory containing an internal reflection element (IRE) crystal. The analysis was conducted in transmittance mode, with data collected over a wavenumber range of 4000 – 650 cm^{-1}. Sample spectra were generated using MicroLab software.

X–Ray powder Diffraction (XRD) Analysis

Powder X-ray diffraction (PXRD) analysis was conducted using a Rigaku MiniFlex 6G diffractometer coupled with Rigaku SmartLab software. The analysis was performed under the following conditions: an X-ray generator operating at 40 kV and 15 mA, with a MiniFlex 300/600 goniometer and a standard sample stage attachment. The diffraction beam was bent, detected by an SC-70S detector, and employed Cu-Kα radiation. Data collection was carried out in continuous scan mode (OD) at a scan speed of 2.00°/min, with a step width of 0.01°, scanning along the θ/2θ/2θ axis over a range of 3° to 90°. A nickel Kβ filter was used, with a divergence slit (DS) of 1.25°, a horizontal incident slit (HIS) of 10 mm, an open scattering slit (SS), and a receiving slit (RS) of 0.3 mm. The cooling fluid used was Innovate, circulated at a flow rate of 1.6 L/min.

Scanning Electron Microscopy (SEM)-Energy Dispersive X-ray Spectroscopy (EDS)

The scanning electron microscopy (SEM) analysis was conducted using cleaned, vacuum-stable samples affixed on aluminium stubs employing conductive carbon tape and securely positioned onto a ZEISS specimen holder. A maximum of eight stubs were incorporated, with thin sections directly mounted. Sample maps were constructed as

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required to facilitate localization within the SEM apparatus. The analytical process encompassed the loading of the specimen holder, the calibration of operational parameters including accelerating voltage, working distance, magnification, focus, brightness, contrast, stigmation, and aperture alignment, along with the selection of the SE2 detector for imaging at working distances ranging from 4 to 30 mm and voltages spanning from 300 V to 30 kV. Images were systematically acquired and archived, and energy-dispersive X-ray spectroscopy (EDS) was conducted for elemental analysis.

UV – Visible Spectroscopic Analysis

The UV- Visible spectroscopic analysis was conducted using the double beam Biolab UV-Visible spectrophotometer model No. BSSBV -202, with a wavelength range between 190 – 1100 nm. The spectrophotometer was used for characterization of the nanocomposites as well as photocatalytic degradation study. A standard calibration curve was plotted using pure Ibuprofen Active Pharmaceutical Ingredient (API), which was dissolved in methanol (1 ml) and water (9 ml) at different concentrations (0.0048 – 3 mg/L). This is to ensure linearity and accuracy of the spectrophotometer (Bio Lab UV-BSSBV-202) response over the concentration range and performance validation.

The Limit of Detection (LOD) and Limit of Quantification were calculated using the equation below;

$$LOD = 3.3 \times \sigma/s \quad \text{..... Equation 1}$$

$$LOQ = 10 \times \sigma/s \quad \text{..... Equation 2}$$

Where σ is the standard deviation of the response and s is the slope of the calibration curve.

Photocatalytic Degradation Experiments

Photocatalytic degradation experiments were conducted under solar irradiation using the Fe-Mo oxide nanocomposites (0.1 – 1g/L) and a double beam Biolab UV-Visible spectrophotometer model No. BSSBV -202, with a wavelength range between 190 – 1100 nm. The initial concentration of Ibuprofen in a methanol-water mixture was 0.6 mg/ml. Various factors like pH, Time and catalyst amount were varied, and the solar irradiated solution after each reaction was centrifuged and filtered with No. 1 Whatman filter paper, and the absorbance was determined at a fixed wavelength of 242 nm.

The % degradation of Ibuprofen was calculated using the equation below;

$$\% \text{ Degradation} = C_o - C_t / Co \times 100 \quad \text{..... Equation 3}$$

The process involves several key steps: photon absorption, charge separation, radical generation, and pollutant degradation. Solar photons excite electrons from the valence band to the conduction band, creating electron-hole pairs. These charge carriers migrate to the catalyst surface, minimizing recombination losses. Surface reactions then produce highly reactive species, such as hydroxyl radicals ($^{\bullet}OH$) and superoxide anions (O_2^-), which attack ibuprofen molecules, leading to complete mineralization through sequential oxidation.

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3.0 RESULTS AND DISCUSSION

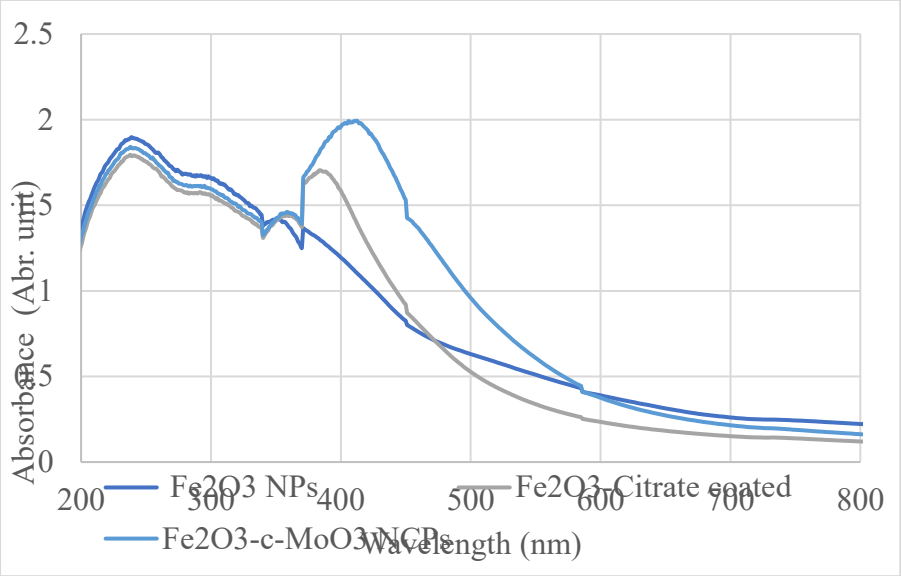

Figure 1: UV-visible Spectra of Fe_2O_3 NPs (blue), Fe_2O_3 – Citrate coated NPs (grey), Fe_2O_3 -c-MoO_3 NCPs (light blue).

The ultraviolet-visible (UV-Vis) spectra illustrated in Figure 1 exhibit distinct absorption maxima at wavelengths of 370 nm, 375 nm, and 414 nm, which have been attributed to the presence of Fe_2O_3 nanoparticles (NPs), citrate-coated Fe_2O_3 NPs, and Fe_2O_3-c-MoO_3 nanocomposites (NCPs), respectively. The observed spectral shift suggests a doping phenomenon that correlates with an augmentation in the size of the nanomaterial and a red shift within the range of 370 nm to 414 nm. This doping effect was previously documented by Shujah *et al*. (2022) in relation to molybdenum-doped iron oxide nanostructures

Fourier Transform Infrared (FTIR) Spectroscopy

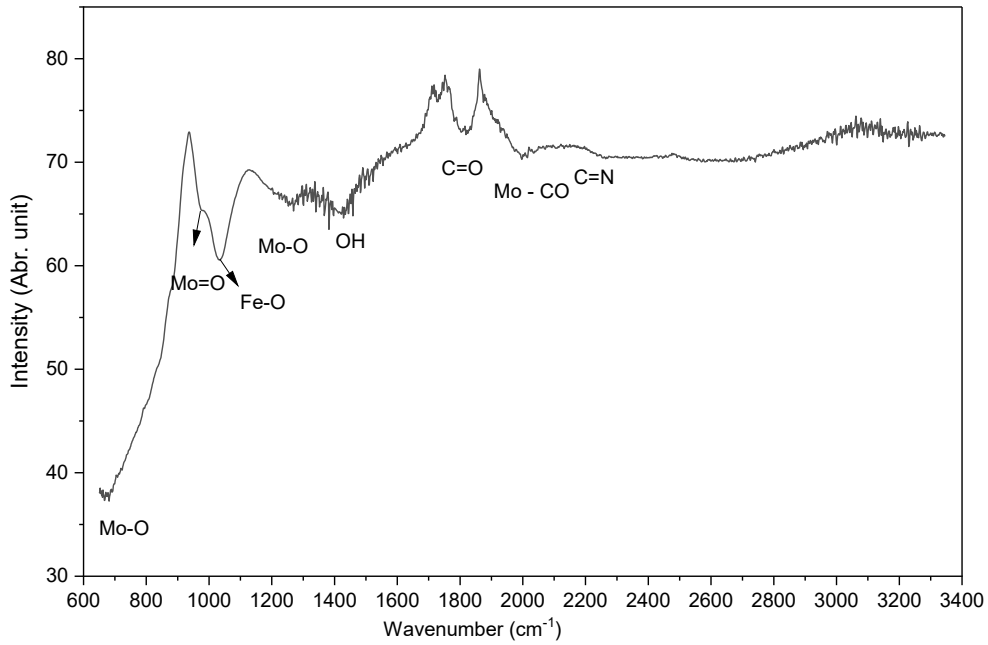

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Figure 2: FTIR Spectrum of Fe-Mo Oxide NCPs

The FTIR spectrum presented in Figure 2 reveals significant absorption bands within the low wavenumber range at 677, 976, 1030, and 1254 cm^{-1}, which correspond to molybdenum-oxygen bridging (Mo-O-Mo) stretching vibrations, molybdenum-terminal oxygen vibrations (Mo=O), as well as Fe-O and Mo-O vibrations, respectively. In contrast, the bands identified at 1427, 1808, 1992, and 2263 cm^{-1} are assigned to O-H stretching, C=O stretching, Mo-CO stretching, and C=N stretching vibrations. The presence of the O-H group may be attributable to adsorbed water molecules or hydroxyl functionalities on the surface of the nanoparticles. The spectrum further indicates the presence of MoO_3 at the surface, suggesting that a junction has formed between the Fe_2O_3 NPs and MoO_3, as evidenced by its visibility on the surface. Analogous findings regarding the dispersion of molybdenum across iron have been previously reported (Brookes *et al*., 2014; Shujah *et al*., 2022; Kumar *et al*., 2024).

X-ray Diffraction (XRD)

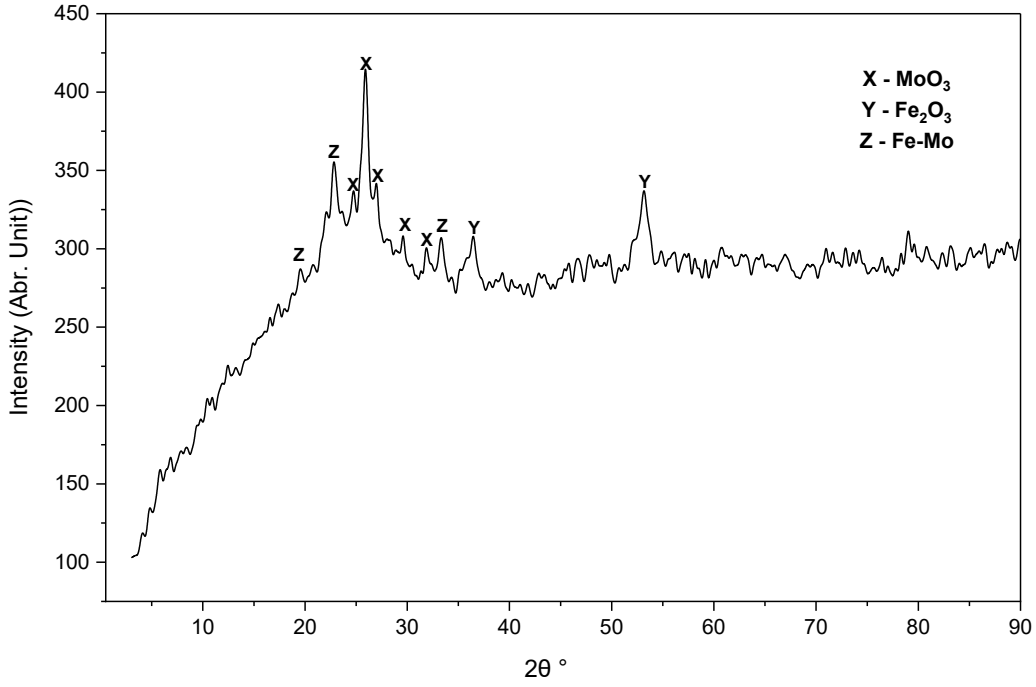

Figure 3; XRD Diffractogram of Fe-Mo oxide NCPs

The XRD diffractogram depicted in Figure 3 illustrates diffraction angles at 19.6°, 22.8°, 24.8°, 25.9°, 26.9°, 31.9°, 33.3°, 36.5°, and 53.2°. The peaks observed at 19.6°, 22.8°, and 33.3° can be ascribed to the Fe-Mo phase, while the peaks at 24.8°, 25.9°, 26.9°, and 31.9° are associated with the MoO_3 phase. The peaks located at 36.5° and 53.2° correspond to the Fe_2O_3 phase.

A notably prominent sharp peak is discerned at 25.9°, indicative of a robust crystalline phase attributed to orthorhombic α-MoO_3 (JCPD No. 01-076-1003), with the peaks at 19.6°, 22.8°, and 33.3° linked to the formation of iron molybdate, β-$Fe_2(MoO_4)_3$, as reported by Parveen *et al*. (2020) and Shujah *et al*. (2022). The peaks at 36.5° and 53.2° further confirm the presence of the α-Fe_2O_3 phase.

Moreover, the crystallite size was computed using Scherrer's equation, yielding an average crystallite size ranging from 41 to 90 nanometers, thereby corroborating the nanostructured morphology, with the

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broadness of the peaks suggesting either smaller crystallite dimensions or structural disorder. This size is notably smaller than that reported by Parveen *et al*. (2020).

Scanning Electron Microscopy (SEM)

The SEM image and EDS spectrum for Fe-Mo oxides NCps in Figure 4 revealed a cuboid and plate-like morphology of the nanocomposites, displaying distinct particles that appear brighter than the black carbon stub background. The average particle size ranged from approximately 150 to 200 nm. EDS analysis confirmed the elemental composition, indicating the presence of Mo, N, C, O, and Fe, with a higher concentration of Mo on the surface compared to Fe. This suggests that Mo predominantly occupies the surface of the composites. These results corroborate findings reported by Parveen *et al*. (2020) and Shujah *et al*. (2022) on Fe doped with MoO_3.

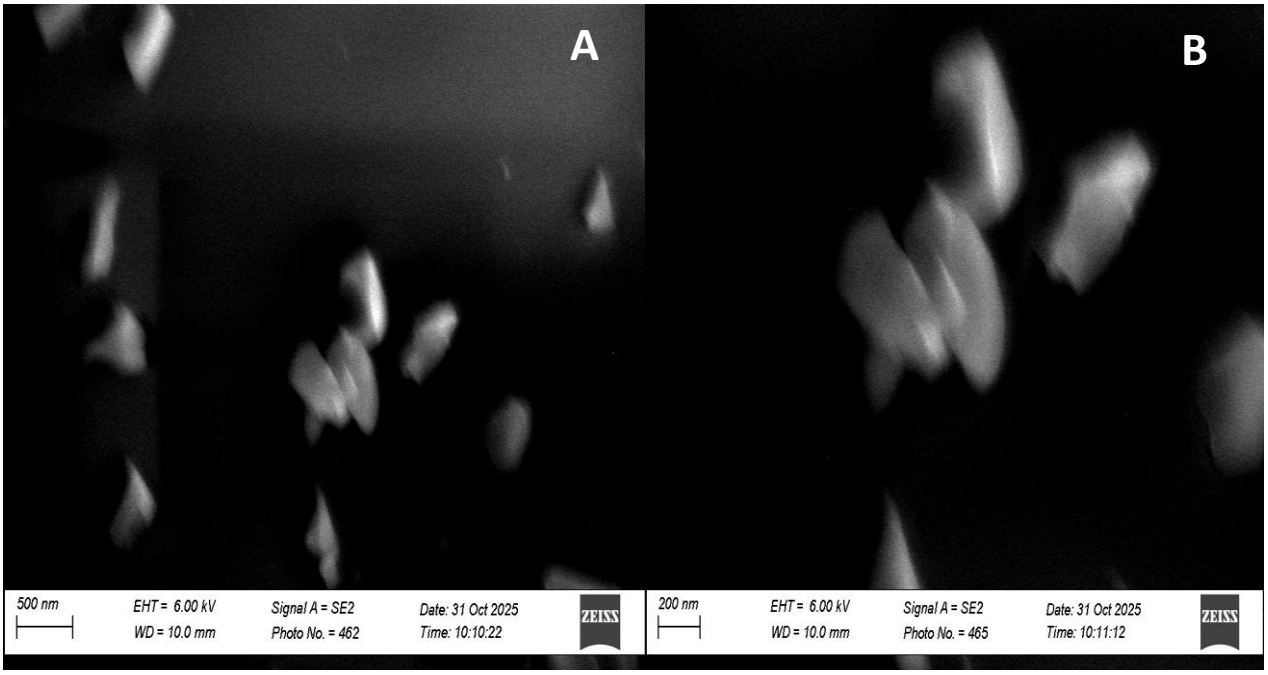
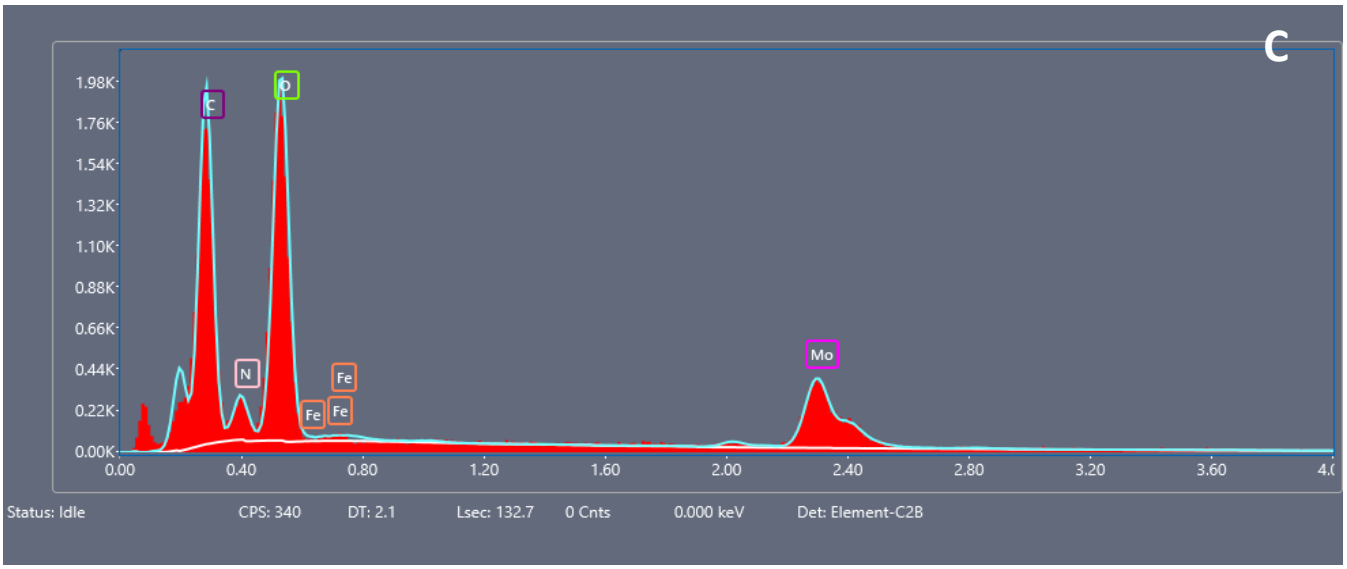


Figure 4: SEM Images for Fe-Mo oxides NCPs (A) 500 nm (B) 200 nm, and (C) EDS spectra of Fe-Mo oxides NCPs



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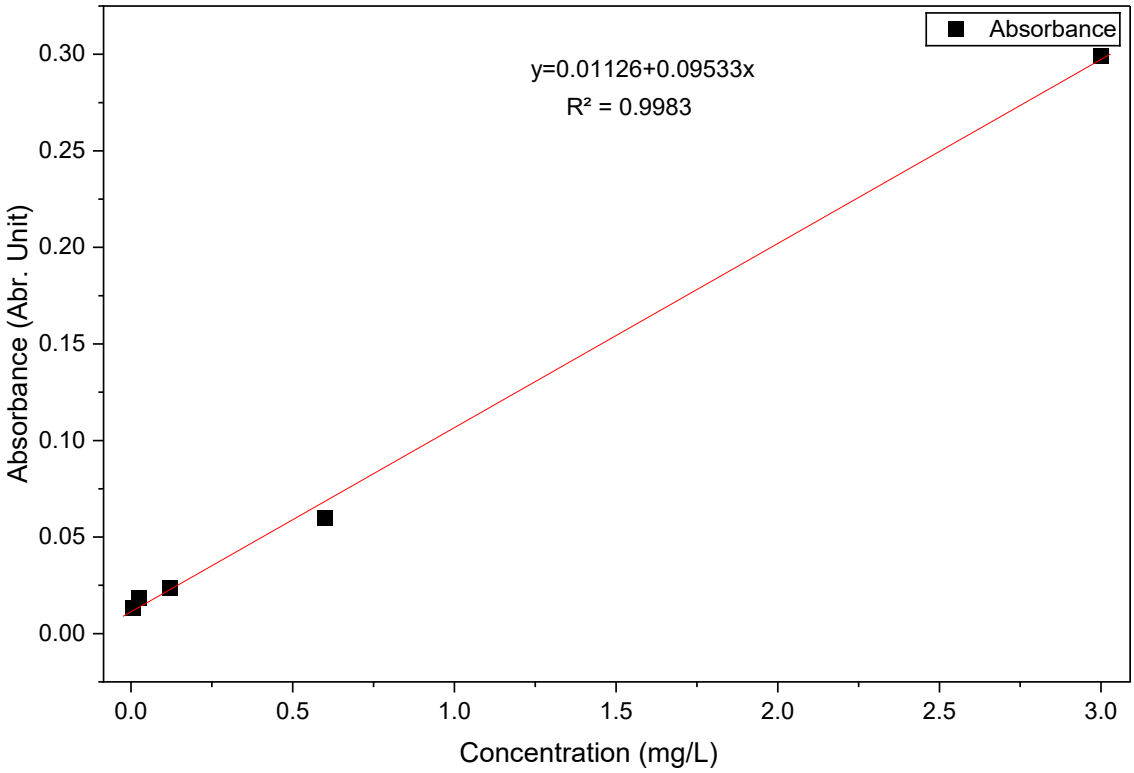

Figure 5. Calibration curve of Ibuprofen in mixture of methanol and water

The plot in Figure 5 depicts that the method for Ibuprofen analysis using UV – Visible spectrophotometer exhibit excellent linearity (R^2 = 0.9983) over concentration range of 0.0048 – 3 mg/L, and LOD and LOQ values of 0.02 and 0.06 respectively. This indicates high accuracy and validity of the method. These findings are similarly to those reported by Jahan *et al* (2014), and Ray, Kachhawa, and Bagherwal, (2025)

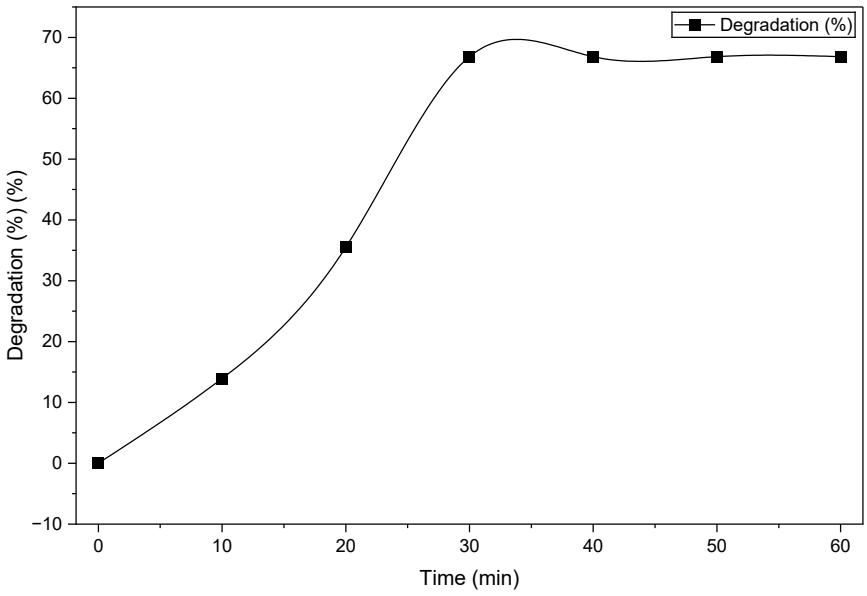

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Figure 6 Effect Time on Ibuprofen Degradation

Figure 6 depict the degradation rate of Ibuprofen under solar irradiation using 0.1 g/L of the catalyst at a pH of 7. The degradation rate increases steadily from 0 – 20 minutes giving a degradation yield of 37 %, which peak at 67 % after 30 minutes. The degradation rate never changes even after 60 minutes. These findings are lower than those reported by Liu *et al* (2020) and Mafa *et al* (2020). However, the pH of the studies was not reported.

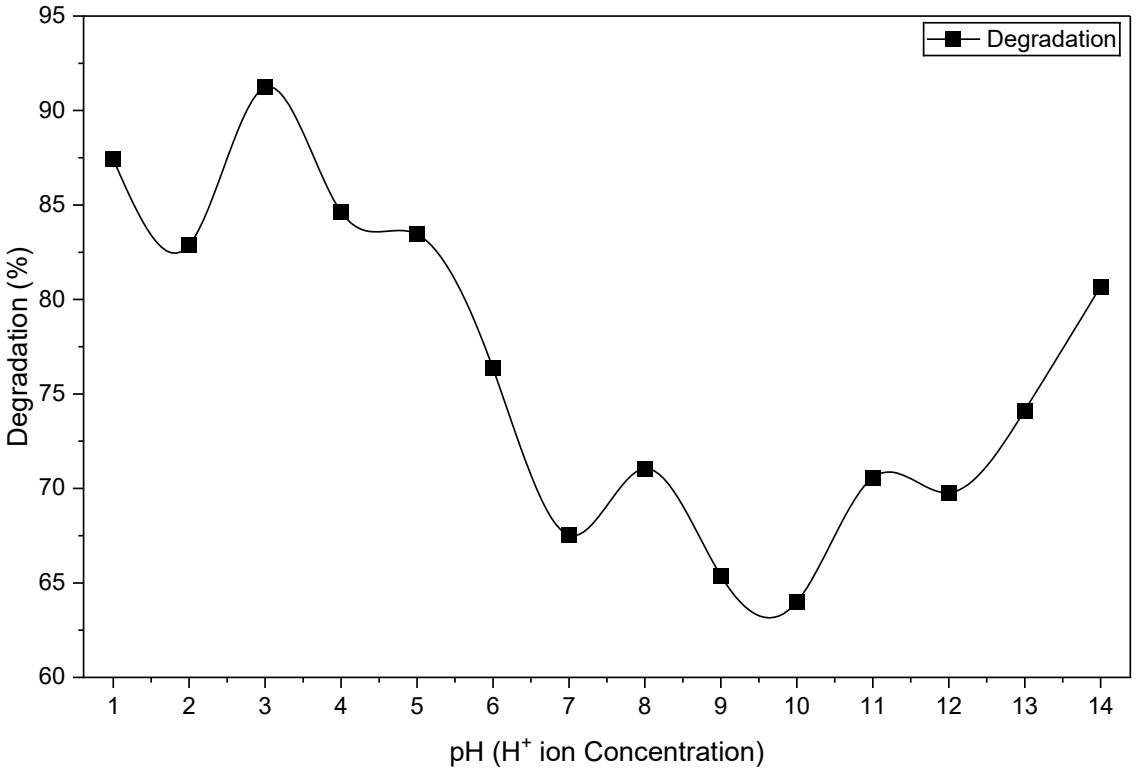

Figure 7 Effect pH on Ibuprofen Degradation

The effect of pH on photocatalytic degradation of Ibuprofen under solar irradiation using 0.1g/L catalyst at 30 minutes was depicted in Figure 7, with highest degradation of 91 % observed at a pH of 3 which declined steadily across neutral pH then slightly increases to 81 % at pH 14. These findings indicate highest Ibuprofen degradation of 91 % at pH of 3, which range is lower than the pH of 6.75 and 7 for optimum degradation rate reported by Mafa *et al* (2021) for Bi_6O_7I-MoO_3 and Pirsahed et al (2023) for CuS-Fe_3O_4/RGO catalysts respectively under visible light

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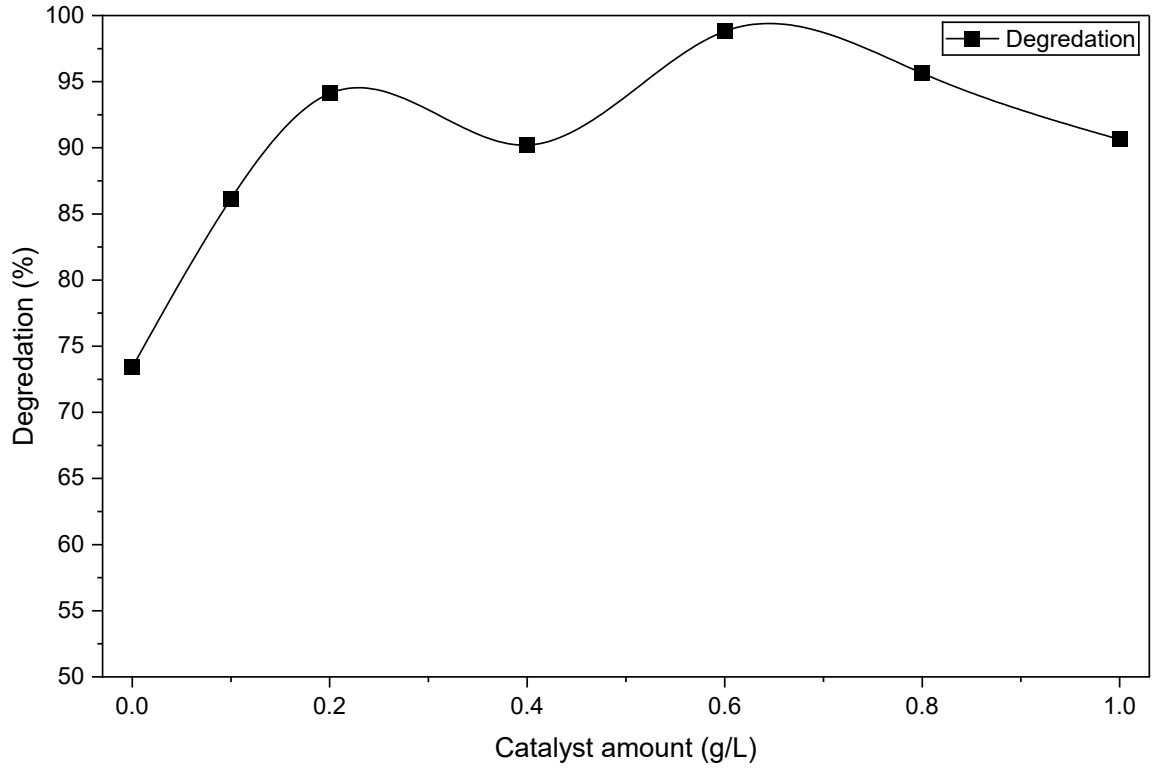

Figure 8: Effect of catalyst amount on Ibuprofen Degradation

Figure 8 depicts the effect of the Catalyst amount on the degradation of Ibuprofen at a pH of 3 for 60 minutes. The degradation rate increases steadily from 86 – 94 % by increasing the amount of catalyst from 0.1 – 0.2 g/L, which declined to 90 % at a catalyst loading of 0.4g/L. Further increase in the catalyst amount to 0.6 g/L gives the highest degradation of 98.8 % which later declines with increasing catalyst loading.

The findings indicate an increase in active catalytic sites for photocatalytic generation of active species, with an optimum loading of 0.6g/L and the highest degradation of 98.8 %. The catalyst loading is above 0.3g/L, reported by Pirsahed *et al* (2023) and slightly above 0.5 g/L reported by Jan *et al* (2025)

4.0 CONCLUSION

This study demonstrates the outstanding photocatalytic performance of green-synthesized Fe–Mo oxide nanocomposites for the degradation of ibuprofen under solar irradiation. The system achieved 98.9% ibuprofen removal at pH 3, offering a sustainable and cost-effective alternative to conventional treatment technologies. The combination of a green synthesis route with the high efficiency and reusability of the nanocomposites provides a solid foundation for the development of sustainable photocatalytic platforms. The heterojunction between iron oxide (Fe_2O_3) and molybdenum oxide (MoO_3) in the nanocomposites facilitates interfacial charge transfer, thereby reducing electron–hole recombination and extending light absorption into the visible region, which markedly enhances photocatalytic activity. Future work should prioritize optimizing reactor design for industrial-scale implementation, evaluating catalyst performance in complex water matrices containing multiple pharmaceutical contaminants, and performing comprehensive toxicity assessments of treated effluents. Overall, this research makes a significant contribution to global

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water-quality management and circular-economy initiatives by utilizing renewable solar energy and environmentally benign synthesis strategies.

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