Synthesis, characterization and photocatalytic performance of iron molybdate (Fe$_2$(MoO$_4$)$_3$) for the degradation of endosulfan pesticide

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Abstract
Iron molybdate was prepared via simple solution chemistry method and the photocatalytic degradation of a pesticide (endosulfan) was investigated under visible light irradiation. As-prepared (Fe$_2$(MoO$_4$)$_3$) was characterized using scanning electron microscope (SEM), x-ray diffraction (XRD), energy dispersive x-ray spectra (EDX), diffused reflectance spectroscopy (DRS) and Zeta particle sizer techniques. The iron molybdate crystallite size was 36 nm, while grain size was in the range of 160–340 nm. The particles of polymetallic compound were spherical, highly porous and with fluffy texture indicating high surface area. DRS revealed Fe$_2$(MoO$_4$)$_3$ was active under visible region since band gap value calculated was 2.7 eV. Response surface methodology (RSM) was employed for the optimization of photocatalytic activity (PCA) of Fe$_2$(MoO$_4$)$_3$ as a function of catalyst dose, H$_2$O$_2$ dose, solution pH and concentration of endosulfan and up to 77% degradation was achieved at optimum conditions, which was monitored by UV/vis spectroscopy. In response to endosulfan degradation, the chemical oxygen demand (COD) and total organic carbon (TOC) were reduced up to 76% and 67%, respectively. Results revealed that iron molybdate is highly efficient photocatalyst for the degradation of endosulfan under solar light irradiation and could possibly be used for the treatment of endosulfan containing wastewater.

1. Introduction

Pesticides applications have been increased to many folds since last three decades to manage pests and to enhance the crops yield. Complex structure makes the pesticides persistent organic pollutants, which are responsible for diverse genotoxic and cytotoxic effects to living organisms. Endosulfan (C$_8$H$_4$Cl$_3$O$_2$S) is an organochlorine pesticide, which is a highly stable and has been detected in the environment at elevated concentration [1–4]. Endosulfan is responsible for various ill effects i.e., endocrine disruptor human primary hepatocytes, convulsions, restlessness, induce seizures, cause cancer, affect central nervous system, reproductive system disorders, gastrointestinal diseases, physiological disorders and muscular hyper twitching. Endosulfan is banned in various countries and endosulfan (isomers and sulfate) are added to the Stockholm convention list of persistent organic pollutants and suggested to stop the application of endosulfan [5].

Different techniques have been employed for the removal of endosulfan such as biological methods (bacterial degradation, fungal biodegradation, phytoremediation), photocatalysis, ozonation, photocatalysis, adsorption (organic and inorganic materials-derived adsorbents) and membrane separation [6–18]. A comparative efficacies for endosulfan degradation are presented in table 1. The reported techniques showed promising efficiency for endosulfan removal. However, all the techniques are not equal regarding cost and...
efficiency [19–23]. Some techniques are responsible for secondary pollution and produce toxic degradation intermediate. Advanced oxidation processes (AOPs) has been emerged as an one of efficient techniques for the destruction of pollutants [24, 25].

Semiconductor oxides, metal oxides and their composites have been used as photocatalysts to carry out AOPs. AOPs degrade the pollutants by generating highly reactive oxidizing specie (‘OH) at ambient conditions. However, band gap of semiconductor is important regarding excitation using UV or solar light and materials bearing small band gap are active under visible light, which is of paramount importance in photocatalysis for destruction of organic pollutants [43, 44]. Heterostructured metal oxides constituting molybdates, tungstate and vanadates are important families that exhibit narrow band gap for harvesting solar light for catalysis. Molybdates adopt tetrahedral coordination with relative large bivalent cations; (Sr, Pb, Ba and Ca) ionic radius (>0.99 Å) and exists in Scheelite type structure and have relative ionic radius (<0.77 Å) with bivalent cation (Co, Ni, Mg, Mn, Zn) and overall, it adopt six-fold coordinated octahedral geometry and constitute Wolframite geometry [45]. Previously, iron molybdate shown promising efficiency as photocatalyst for the removal of endosulfan, heptachlor and Rhodamine B dye and the efficiency was reported to be very promising [28, 46–49].

In view of aforementioned facts, present study was focused on the preparation and characterization of iron molybdate and techniques i.e., scanning electron microscope (SEM), x-ray diffraction (XRD), energy dispersive x-ray spectra (EDX) and Zeta particle sizer were employed for the characterization . Response surface methodology (RSM) was employed for the optimization of photocatalytic activity (PCA) for the degradation of endosulfan as a function of catalyst dose, H2O2 dose, solution pH and concentration of endosulfan (figure 1), a pesticide that is banned due to its high toxicity to human being and other living organisms.

| S. no | Treatment methods | Endosulfan removal | References |
|-------|-------------------|---------------------|------------|
| 1     | Trichosporon cutaneum | A 60.36% α-endosulfan, 70.73% β-endosulfan and 52.08% endosulfan sulfate was removed | [26] |
| 2     | *Pseudomonas* sp. | A 70%–80% endosulfan removal within three days | [27] |
| 3     | Fe2 in iron turning waste is responsible | A 85% of endosulfan (20 μg l−1) in 10 min | [28] |
| 4     | *Paenibacillus* sp. JTTSM08, *Bacillus* sp. PRB77 and *Bacillus* sp. PRB101 | A degradation of 92% at 5 mg kg−1 endosulfan conc. was observed for PRB101 after 120 days | [29] |
| 5     | *Psillimonas* sp. JW2 and *Bordetella* petrii NS | JW2 degraded 100% and 91.5% of α- and β-endosulfan, and NS degraded 95.1% and 90.3% of α- and β-endosulfan, respectively. | [30] |
| 6     | *Rhodococcus koreensis* strain S1-1 | Endosulfan sulphate reduced to 2.11 μM from 12.25 μM in 14 d at 30 °C | [31] |
| 7     | *Pseudomonas* aeruginosa. | A 96% of endosulfan after 288 h under static conditions and complete removal after 28 days | [32] |
| 8     | Gamma irradiation induced degradation | A 99.5% (1.30 μM) endosulfan sulphate was observed at an absorbed dose of 1020 Gy and removal efficiency of endosulfan sulphate was affected by the pH removal efficiency of 99.5%, 98.3% and 31.3% at pH 6.2, pH 10.0, and pH 2.6, respectively | [33] |
| 9     | Zero-valent zinc | >90% endosulfan was degraded in 180 min. The degradation was accelerated under acidic conditions | [34] |
| 10    | TiO2/UV radiation | Endosulphan removal efficiency 80%–99% | [35] |
| 11    | TiO2/solar radiation | Endosulphan complete mineralization | [36] |
| 12    | UV/HSO3−/Fe2+ | Degradation efficiency of 99.0% at UV fluence of 360 mj cm−2 | [37] |
| 13    | UV/S2O82−, UV/HSO3−, and UV/H2O2 | A significant removal, 91%, 86%, and 64%, of endosulfan, at an initial concentration of 2.45 μM and UV fluence of 480 mj cm−2, was achieved, respectively, | [38] |
| 14    | Ag doped nano TiO2 | Complete endosulfan removal | [36] |
| 15    | Oxonation | A 97% of beta-endosulfan 6-mg min −1 ozone dosages and at a pH of 4 for 60 min | [39] |
| 16    | Phytoremediation- vegetative filter strips | A 98% endosulfan removal | [40] |
| 17    | TFC-SR2 and TFC-SR3 nanofiltration membrane | A 80%–95% endosulfan removal | [41] |
| 18    | Adsorption- lateritic red and latosol soils | A 0.209 and 0.186 mg g−1 for α-endosulfan in lateritic red and latosol soil, respectively, and 0.148 and 0.140 mg g−1 for β-endosulfan in lateritic red and latosol soil, respectively. | [42] |
| 19    | Fe3(MoO4)2/ solar light | A 77% endosulfan removal | Present study |
2. Material and methods

2.1. Chemicals and reagents
The chemicals and reagents used were of analytical grade and procured from Sigma-Aldrich Chemical Co, USA i.e., metal nitrates (Fe), ammonium salts, ethylene glycol. The solutions were prepared in deionized water (Millipore Corp., Milli-Q, 18.2 MΩ cm at 25 °C) and working standards of desired concentration was prepared by dilution method.

2.2. Synthesis procedure
Iron molybdate was prepared following reported methods with slight modification [50, 51]. Briefly, a 7.51 g of iron nitrate and 4.94 g of ammonium molybdate were dissolved in 200 ml deionized water separately (ammonium heptamolybdate and iron nitrate were mixed at ratio of 1:5) and both solutions were mixed drop wise with continuous slow stirring. Later, 5 ml of ethylene glycol was added and stirred the solution for 120 min. The sample was washed with water and ethanol several times to remove the impurities. The precipitate was dried at 70 °C for 5 h. The characterization of synthesized material was carried out by XRD (Joel JDX-3532 diffractometer), SEM (Quanta 250 FEI, USA), EDX (INCA Oxford instruments, UK) and Zeta particle size analyzer (Malvern).

2.3. Photocatalytic activity
The PCA was evaluated by degrading endosulfan (pesticide) under solar light irradiation. For solar light generation, solar simulator (150 W Xe lamp having cutoff filter (λ > 420 nm)) was used. For the optimization of PCA, response surface methodology was employed and central composite design (CCD) was used for experimental runs (table 2). The experimental conditions (catalyst load, pH, H2O2 concentration and endosulfan concentration are shown in table 3). Before irradiation, the mixture was stirred for 30 min in dark to attain the adsorption–desorption equilibrium between catalyst surface and endosulfan. The sample was irradiated for 1 h under ambient conditions. Then, 2 ml sample was withdrawn, filtered by Millipore filter and analyzed for endosulfan residual concentration by UV/Visible spectrophotometry (CE Cecil 7200, UK) and HPLC, which are regarded as efficient analytical techniques [52–54]. The percentage degradation was estimated using relation shown in equation (1). To evaluate the pure photolysis effect, blank experiment was also performed under similar conditions. The endosulfan percentage degradation was estimated by concentration difference method, while TOC was measured at 590 nm as already reported [55]. The COD and TOC were measured of those samples treated at optimum conditions. High performance liquid chromatographic (HPLC) was performed to analyze the endosulfan and its metabolites. The system equipped with C18 column and UV detector at 240 nm (50 Hz, 0.05 s) was used. Eluents (water and acetonitrile) were used as mobile phase after

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**Table 2. Experimental ranges of under study parameters.**

| Name             | Units | Symbols | Low (-1) | Middle(0) | High (+1) |
|------------------|-------|---------|----------|-----------|-----------|
| Catalyst load    | g L⁻¹ | A       | 0.5      | 1         | 1.5       |
| pH               | —     | B       | 3        | 6         | 9         |
| H2O2 conc.       | %     | C       | 3        | 5         | 7         |
| Endosulfan conc. | mg L⁻¹| D       | 10       | 20        | 30        |

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**Figure 1.** Structure of endosulfan (C9H6Cl6O3S) used for PCA evolution of Fe2(MoO4)3.
filtration and degasification at 70:30 volume ratio with flow rate \(0.6 \text{ ml min}^{-1}\) and injection volume 1 \(\mu\text{l}\). The highest detection of endosulfan and its metabolites was achieved at 243 nm.

\[
\text{Degradation}(\%) = \frac{C_o - C_f}{C_o} \times 100 \tag{1}
\]

3. Results and discussion

3.1. Characterization

The XRD pattern of iron molybdate synthesized by precipitation is shown in figure 2. XRD patterns revealed sharp and well defined peaks without any impure phase. The diffraction pattern obtained are attributed to the pure form of monoclinic iron molybdate (ICPDS 35-0183) \[56\]. The highest and broadest peak appeared at 22 degree \(2\theta\) indicating small crystallite size. Estimation of average size of crystallite \(L\) of iron molybdate particles was done using Debye-Scherer’s relation (equation (2)).

\[
L = \frac{0.9\lambda}{\beta \cos \theta} \tag{2}
\]

Where, \(k = 0.9\) (constant), \(\lambda = 1.54 \text{ Å}\), \(\beta = \text{FWHM}\), \(\theta = 2\theta/2\). \(L\) corresponds to the average size of crystallite (nm), \(\beta\) represents the full width at half maximum peak intensity (0.3944) and \(\theta\) is the Bragg’s angle of diffraction. The average crystallite size of iron molybdate particles was found to be 36 nm.

The elemental analysis of iron molybdate was performed by EDX and results are shown in figure 3. The peaks obtained correspond to the elements O, Mo and Fe. The percentages of O, Mo and Fe were 65.90, 18.75 and 15.35 (\%), respectively. The EDX analysis also revealed the purity of the prepared iron molybdate since no additional peak was observed the spectrum.

SEM analysis was also performed to investigate the morphology and surface texture properties of iron molybdate and results are shown figure 4. SEM images obtained clearly revealed the highly porous and fluffy texture ranging in size of 50–55 nm. These fluffy and small grain shaped iron molybdate particles have great potential for catalytic properties. The photocatalytic properties strongly relates with particle size, geometry and morphology. Therefore, the grain size distribution was investigated by Zeta particle sizer and response is shown.

| Run | Catalyst load (g l\(^{-1}\)) | pH | \(\text{H}_2\text{O}_2\) | Endosulfan conc. mg l\(^{-1}\) | Degradation (\%) |
|-----|-------------------------------|----|----------------|-----------------------------|----------------|
| 1   | 1.5                           | 9  | 7             | 10                          | 64             |
| 2   | 1.5                           | 9  | 3             | 10                          | 54             |
| 3   | 1.5                           | 9  | 3             | 30                          | 64             |
| 4   | 0.5                           | 9  | 3             | 30                          | 25             |
| 5   | 1.5                           | 3  | 3             | 30                          | 51             |
| 6   | 0.5                           | 3  | 7             | 10                          | 64             |
| 7   | 0.5                           | 9  | 7             | 30                          | 45             |
| 8   | 0.5                           | 3  | 3             | 10                          | 42             |
| 9   | 0.15                          | 6  | 5             | 20                          | 37             |
| 10  | 1.84                          | 6  | 5             | 20                          | 73             |
| 11  | 1                             | 0.95| 5             | 20                          | 54             |
| 12  | 1                             | 11.04| 5             | 20                          | 38             |
| 13  | 1                             | 6   | 1.63         | 20                          | 45             |
| 14  | 1                             | 6   | 8.36         | 20                          | 73             |
| 15  | 1                             | 6   | 5             | 31.8                        | 87             |
| 16  | 1                             | 6   | 5             | 36.81                       | 59             |
| 17  | 1                             | 6   | 5             | 20                          | 77             |
| 18  | 1                             | 6   | 5             | 20                          | 77             |
| 19  | 1                             | 6   | 5             | 20                          | 77             |
| 20  | 1                             | 6   | 5             | 20                          | 78             |
| 21  | 1                             | 6   | 5             | 20                          | 79             |

Table 3. Experimental layout and percentage degradation of endosulfan (predicted versus observed degradation).
in figure 5. The grain size distribution was in the range of 160–340 nm (with average particle size of 250 nm). The large grain size indicated the polycrystalline nature of the compound synthesized consisting of many crystallites. The large particle size also revealed the high surface charge of iron molybdate which caused agglomeration of particles.

Diffused reflectance spectrum of iron molybdate was recorded in the range of 200–800 nm and maximum absorption capacity of iron molybdate particles (upto 50%) was found in visible region >380 nm (figure 6). The astounding absorbing capacity was found in visible region, which anticipated the remarkable photocatalytic property under solar light [57]. The band gap was calculated using data of diffused reflectance spectra following Kubelka Munk relationship as shown in equation (3). The band gap calculated was 2.7 eV which supported the results of DRS for harvesting sunlight in visible region.

\[
[F(R) \times E]^\frac{1}{2}
\]  

(3)
**Figure 4.** SEM analysis of iron molybdate.

**Figure 5.** Zeta sizer distribution of iron molybdate.

**Figure 6.** (Left) Diffused reflectance spectrum and (right) band gap energy of Fe₂(MoO₄)₃.
3.2. RSM for PCA optimization

Central composite design (CCD) was employed for the optimization of PCA under response surface methodology. Effects of various operational parameters were studied to deduce the optimal conditions for maximum degradation of pesticide (endosulfan). The experimental conditions are shown in table 2 and experimental run along with degradation of endosulfan is shown in table 3. The residual values indicate a good relationship between predicted and observed values. A polynomial equation elaborating the effectiveness and individual and cumulative influences of process variables. Where $Y$ is the percentage degradation, whiles $A$, $B$, $C$ and $D$ are coded values for the influencing parameters (concentration of endosulfan, catalyst load, $H_2O_2$ and pH).

Fitting of the model was carried out through sequential process and selection of model summary was carried out through model summary statistics. The elaboration of the optimal response and model fitting was assessed through lack of fit test (table 4) and ANOVA is shown in table 5.

\[
\text{Degradation}(Y) = +A77.42 + 10.7A - 4.39B + 8.21C - 8.32D - 3.45AB - 2.38AC + 3.58BD - 7.79A^2 - 10.97B^2 - 6.38C^2 - 1.43D^2
\]  

(4)

The efficacy of the model was also evaluated on the basis of $F$ and $P$ values. Smaller the $P$ value and larger value of $F$ indicates the best fitting of the model [58, 59]. From ANOVA lower value of prob. $P < 0.005$ indicates that variables have significant effect on response [58]. In similar fashion, other variables also ensured significance of the model since $F$ value has 0.01% of noise value. The regression analysis reveals that $A$, $B$, $C$, $D$, $AB$, $AD$, $A^2$, $B^2$, $C^2$ and $D^2$ variables have significant effect on degradation, while $BC$, $CD$, $BD$ and $AC$ are insignificant with

| Source | Sum of squares | Df | Mean square | Std. Dev. | Pred. R$^2$ | PRESS | F-value | p-value |
|--------|---------------|----|-------------|-----------|-------------|--------|---------|---------|
| (A) Linear | 3092.17 | 12 | 257.681 | 13.9135 | 0.164 41 | 4875.33 | 198.216 | <0.0001 |
| 2FI | 2960.63 | 6 | 493.438 | 17.2216 | −6.6307 | 44 521.8 | 379.568 | <0.0001 |
| Quadratic | 2.067 15 | 2 | 1.033 57 | | | | 0.795 06 | 0.512 |
| Cubic | 0 | 0 | | | | | |
| Pure Error | 5.2 | 4 | 1.3 | |

| Source | Sum of Squares | Df | Mean Square | F- value | p-value |
|--------|---------------|----|-------------|---------|---------|
| Mean versus Total | 75 960.4 | 1 | 75 960.4 | | |
| Linear | 3092.17 | 12 | 257.681 | 198.216 | <0.0001 |
| Quadratic versus 2FI | 2960.63 | 6 | 493.438 | 379.568 | <0.0001 |
| Cubic versus Quadratic | 2958.56 | 4 | 739.64 | 610.672 | <0.0001 |
| Cubic | 0 | 0 | | | |
| Pure Error | 5.2 | 4 | 1.3 | | |

Table 4. (A) lack of fit tests and model summary statistics of model compound degraded by iron molybdate (B) Sequential model sum of squares (Type I) of model compound degraded by iron molybdate.

Table 5. Analysis of variance (ANOVA) of endosulfan degradation using iron molybdate.
P > 0.05. Model fitting was further verified by $R^2$ which endorses quadratic model with $R^2$ values 0.999. Predicted and experimental values relies very close to each other, which revealed that the CCD is very effective for the optimization of conditions for endosulfan degradation and at optimum conditions of process variable, up to 79% degradation of endosulfan was observed. The optimum conditions were; 1.00 g l$^{-1}$ catalyst dose, pH 6, 5% H$_2$O$_2$ and endosulfan concentration of 20 mg l$^{-1}$. Verification of as predicted values was done by running experiments under the optimum conditions and up to 77% degradation of the endosulfan was observed, which is in good agreement with predicted values by RSM.

3.3. Effect of process variables on degradation

3.3.1. Effect of catalyst dosage

The 3D response surfaces and contour plots of pH, H$_2$O$_2$ and endosulfan concentration and catalyst load are shown in figure 7. In advanced oxidation processes, catalysts with narrow band gap was employed, which is active under solar light that promotes the electrons to conduction bands and resultantly, hydroxyl radicals are generated, which oxidize the organic pollutants in aqueous media. Iron molybdate on irradiation leads to the promotion of electron from valence to conduction band leaving behind a hole bearing positive charge, which is principally responsible for generation of hydroxyl radicals by reacting with water $[44, 60–62]$. Moreover, conduction band promoted electrons also become a source for the generation of strong oxidizing species such as superoxide and per hydroxyl radical on reacting with dissolved oxygen $[63]$. Dissolved oxygen plays a dual role in PCA; firstly, it accepts a photo-promoted free electrons from conduction band and inhibits the recombination of free electron to hole. Secondly, it generates per hydroxyl and super oxide radicals and these species have strong potential to completely degrade the organic pollutant in aqueous environment (equations (5)–(8)) $[44, 57, 60, 62]$. Seeking the optimum concentration of catalyst, different concentration of the catalyst were employed ranging from (0.5–1.5 g l$^{-1}$) and catalyst dose of 1.0 g l$^{-1}$ was found optimum for the degradation of the endosulfan. Beyond this, the PCA did not change considerably.

\[ \text{Fe}_2(\text{MoO}_4) + h\nu \rightarrow h^+ + e^- \]  \hspace{1cm} (5)

\[ \text{O}_2 + e^- \rightarrow \text{O}_2^- \]  \hspace{1cm} (6)

\[ h^+ + \text{H}_2\text{O} \rightarrow \text{OH}^- \]

Figure 7. 3D-response surface curves and contours depicting effect of process variables on endosulfan degradation (A) catalyst load versus pH (B) H$_2$O$_2$ versus catalyst load (C) pH versus conc. of endosulfan.
3.3.2. Effect of pH
The pH of solution is an important parameter for photocatalysis, which controls the generation of reactive species. The PCA was enhanced due to the formation of hydroxyl radicals in the acidic medium range of pH 2.5–3.5. Under basic condition, the OH decreased; hence the PCA was also decreased. Moreover, the decomposition of H₂O₂ into O₂ and H₂O is accelerated under alkaline condition instead of OH generation [43]. Also, at pH < 3.0, hydrogen peroxide converted in to a stable oxonium ion (e.g., H₃O₂⁺) and the scavenging effect of the OH by H⁺ is enhanced. To evaluate the influence of initial pH of solution on PCA, endosulfan was degradation in the pH ranging of 3–9. Under alkaline conditions, the degradation rate was slow due to electrostatic repulsion between anionic structure and negatively charged catalyst surface [64]. This adsorption took place which was probably due to negatively charged oxygen over carboxylic group with the Fe³⁺ or Mo⁶⁺ cations through a Lewis acid–base reaction. It is found that the degradation efficiency of endosulfan was 79% for 1.0 g L⁻¹ catalyst dose, pH 6, 5% H₂O₂ and endosulfan concentration of 20 mg l⁻¹. It can be explained by the solid acid characteristics of Fe₂(MoO₄)₃, which are proved by both the low PZC and the presence of strong acid sites. The PZC of Fe₂(MoO₄)₃ is 2.94, beyond this deprotonation occurs and an acidic microenvironment formed on the surface of Fe₂(MoO₄)₃ particles. Moreover, by increasing the solution pH, more protons are subtracted from the surface of the Fe₂(MoO₄)₃, leading to a more acidic environment. It compensates the negative influence due to increase in solution pH. In this case, the surface of Fe₂(MoO₄)₃ plays an acid–base buffering role. However, it was observed in the present study that the PCA decreases apparently when the solution pH raised to 8.0. This phenomenon could be supported by description of strong alkaline condition that is resulted due to lower oxidation ability of OH, the reduction of OH due to the decomposition of H₂O₂ into O₂ and H₂O [24].

3.3.3. Effect of endosulfan concentration
The influence of the initial endosulfan concentration on the attainment of steady state for maximum degradation was studied by varying the initial concentration (10 to 30 mg l⁻¹). Results revealed that an increased endosulfan initial concentration did not affect the degradation rate; however, it appears to have a pronounced effect on TOC mineralization. Indeed, at 180 min of reaction time the median TOC percentage removal was 67%, 62% and 52% for initial endosulfan concentration, respectively. The irradiation time necessary to achieve complete mineralization tends to increase with the pollutant concentration. Moreover, a higher concentration of the pollutant may saturate the active sites on photocatalyst surface and reduce the photonic efficiency leading to photocatalyst deactivation [43]. Apparently, this was the case with the endosulfan mineralization, and results showed that higher endosulfan concentration require longer reaction period (at a given catalyst dose) for achieving sufficient mineralization.

![Figure 8. Percentage reduction in COD of endosulan treated using iron molybdate under solar light irradiation.](image_url)
3.4. Water quality parameters

The COD and TOC of endosulfan was also evaluated before and after treatment and results are shown in figures 8–9. The removal of both TOC and COD was found correlated with PCA. Initially, the degradation rate was low, which was enhanced considerably since TOC and COD removal enhanced with reaction time. COD removal of 10 mg l\(^{-1}\) concentration was found to be 76% and by increasing concentration, COD removal decreased and 64% COD removal was observed for 20 mg l\(^{-1}\) of endosulfan concentration. However, at initial concentration of 30 mg l\(^{-1}\), the rate of mineralization slowed down and COD removal reduced to 51%. Similarly, TOC was also evaluated of endosulfan treated at optimized conditions and in correlation with mineralization of the endosulfan, the TOC was reduced and up to 67% when treated at optimum conditions. At higher initial concentration, the PCA was reduced and TOC removal was also reduced. For 20 mg l\(^{-1}\) of endosulfan concentration, 62% TOC removal was observed and for 30 mg l\(^{-1}\) initial concentration, the COD removal was 52%. The improvement in water quality parameters indicates that the iron molybdate is efficient to treat the wastewater containing pesticides, which are otherwise difficult to treat. Under the current scenario of environmental pollution [4, 15–18, 65–68], there is need to develop and utilize efficient materials that are active under solar light and iron molybdate proved to be highly efficient since the activity was considerable under solar light that could possibly be used for the degradation of pesticides in wastewater.

4. Conclusion

Iron molybdate was successfully prepared by precipitation route, which was characterized by SEM, XRD, EDX and Zeta particle sizer. The PCA was evaluated by degrading endosulfan pesticide under solar light irradiation. The iron molybdate size was in the range of 160–340 nm with average particle size of 250 nm. The particles were highly porous with fluffy texture having band gap value of 2.7 eV. The iron molybdate PCA was significantly affected by catalyst dose, H\(_2\)O\(_2\) dose, solution pH and concentration of endosulfan and at optimum conditions, up to 77% endosulfan degradation was achieved along with 64% COD and 67% TOC reductions. Results revealed that iron molybdate is highly efficient catalyst for the degradation of endosulfan and could possibly be used for the degradation of wastewater contains endosulfan under solar light irradiation.

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