

RESEARCH ARTICLE

DEGRADATION OF PROCION RED MX-5B REACTIVE DYE COUPLING A PHOTO FENTON SYSTEM

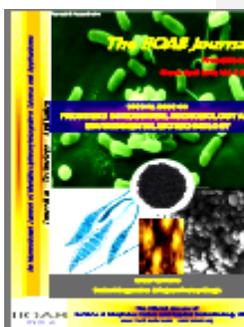
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ABSTRACT



The oxidative degradation of Procion Red MX-5B by solar Fenton's has been investigated using sunlight as irradiation source in presence of hydrogen peroxide and Fe²⁺. Maximum decolorization (99%) and COD removal (97%) for MX-5B was obtained at H₂O₂ dose of 16.6 mM and at 30 min irradiation time. Fe²⁺ dose of 0.36 mM was optimum for Procion Red MX-5B degradation. The solar degradation mechanism occurs in three steps in which initially the more active bonds were hydroxylated. The N=N and the C-S bonds of sulphonate group form organic acids, SO₄²⁻ and NH₄⁺. At the same time, the aromatic acids produced initially were further oxidized to aliphatic acids. MX-5B undergoes oxidation leading to non toxic and biodegradable ultimate breakdown products, such as, oxalic acid and acetic acid. Solar Fenton process not only oxidizes but also helps in decolorization and nearly mineralization of the dyes at low dose of reactants.

Key words: Solar Fenton's degradation; solar energy; Procion Red MX-5B; H₂O₂; decolorization; product of degradation

[1] INTRODUCTION

Wastewaters generated from textile and dyeing industries are highly colored, rich in organic and inorganic materials and saline. With the presence of complicated color causing compounds, decolorization and degradation of these wastes is a difficult and challenging task. Hence, it is essential to remove/destroy the refractory/ recalcitrant pollutants before its discharge into the surrounding water bodies. In recent years, new technologies such as Advanced Oxidation Processes (AOP) show great potential for the treatment of wastewater containing toxic organic compounds. The AOPs involve generation of hydroxyl radicals (HO), which non selectively attack the organic compounds faster than the commonly known oxidizing agents. The radicals can be produced on site, in a reactor where the radicals can contact the organics in the wastewater.

Fenton's process, one of the oldest advanced oxidation processes that use a mixture of Fe²⁺ and H₂O₂ as the oxidizing agent, has been proven as a powerful oxidant of organic compounds and has attracted interest recent years in wastewater treatment. Hydrogen peroxide reacts with ferrous ions in water

and generates free hydroxyl radicals, which have high oxidation potential and can oxidize wide range of organic compounds [1].



Studies have shown that textile and commercial dyes can be successfully treated by Fenton's oxidation [2,3,4]. Fenton's process has been applied for the pretreatment of organic compounds to reduce toxicity and improve biodegradability [5].

In recent years, many studies have shown that the oxidizing power of the Fenton type system can be enhanced greatly under UV light irradiation and can be used for the degradation and mineralization of toxic organic contaminants present in wastewater [6, 7]. The efficiency of degradation of organic compounds was considerably improved due to the continuous regeneration of Fe (II) via photo reduction of Fe (III) by Fenton's reagent combined with UV-visible irradiation [8].

Solar energy was successfully used for the photo catalytic degradation of dyes [9,10]. Bandara et al.[11] reported that the degradation and decolorization of Orange II could be achieved

in less time in the presence of natural sunlight via Fenton type reaction. Solar assisted Fenton's process was efficient to decolorize and mineralize reactive dyes used in the textile dyeing industries [1,12]. The objective of the work is to study the efficiency of solar Fenton's process for the degradation of MX-5B in the presence of natural sunlight

[II] MATERIALS AND METHODS

2.1. Reagents

FeSO₄ · 7H₂O (MERCK, purity 96%) was prepared at a predetermined concentration of 10 g l⁻¹. H₂O₂ (30%) and other chemicals used in the experiment were of analytical grade. Commercial grade dyes Procion Red MX-5B obtained from Color Chem (India) Ltd. were used without any purification. A known concentration of the dye was prepared in deionized water and used for all studies. [Supplementary figure-1](#) gives the structure of dyes Procion Red MX-5B.

2.2. Experimental procedure

Solar experiments were carried out on sunny days of April to June 2004 between 11 AM and 2 PM. A known volume dye solution was taken in an open borosilicate glass tray of 500ml capacity, to which known volume of FeSO₄ was added. The addition of H₂O₂ to the reacting solution marks the beginning of the reaction. The solution was mixed with the magnetic stirrer and exposed to sunlight. Solar light intensity was measured for every 1 hour and the average light intensity over the duration of each experiment was calculated. The intensity was 825x100 Lux. The experiments were carried out at pH 3.0. Samples were collected at regular time interval and analyzed immediately.

2.3. Analysis

COD and pH were measured as per Standard Methods [13]. The UV-VIS spectra of the samples were recorded from 200 to 800 nm using spectrophotometer (Shimadzu Model UV 160A). Color removal was measured for each dye at the wavelength in the visible range, where maximum absorbance was obtained. Residual hydrogen peroxide was determined by potassium iodide titration method [14]. Accordingly, correction was made in the COD determination for residual H₂O₂ [15].

Low molecular weight organic acids formed during AOP processes were analyzed by ion chromatography. Ion chromatography employed to analyze organic acids was equipped with an Anion Ion 12 (AS-4) column (Dionex) and operated in suppressed conductivity detection mode. The filtered eluent used was 3.3 mM Na₂CO₃/1.0 mM NaHCO₃ at a flow rate of 0.7 ml/min.

Fourier Transform Infrared (FTIR) spectroscopy was carried out using a Perkin Elmer Brucker Vector 22 FTIR instrument for identification of functional groups. The samples were prepared for FTIR analysis by drying at 90°C and mixing the powdered samples with dried KBr. The resulting powder was then pressed to produce a pellet for analysis. IR data were collected over the wave number range of 700 – 4000 cm⁻¹.

[III] RESULTS AND DISCUSSION

3.1. Effect of hydrogen peroxide

Decolorization and degradation of Procion Red MX-5B for

different H₂O₂ dose from 8.83 mM to 33.2 mM at constant iron dose (0.36 mM) are presented in [\[Supplementary figure-2\]](#) and [Supplementary figure-3](#). Maximum decolorization (99%) and COD removal (97%) for MX-5B was obtained at H₂O₂ dose of 16.6 mM and at 30 min irradiation time. The enhancement in the removal rate is due to increase in hydroxyl radical production by solar light. Light intensity determines the amount of photons absorbed by the catalyst. With increase in the solar power, Fenton's generate more photons and produce more hydroxyl radicals, which help in improving the degradation efficiency. But at high dosage of H₂O₂ the decrease in decolorization is due to the hydroxyl radical scavenging effect. Costa et al. [12] reported complete decolorization of Bright Blue Ramazol and Red Procion H-E713 in the mixture on exposure to solar and ultraviolet radiation. Hung et al. [15] observed that increase in H₂O₂ concentration from 3.75 mM to 42.8 mM resulted in 90% color removal of azo dye Acid Black 1 by the UV/H₂O₂ process. Murganandham et al. [16] also reported that solar Fenton process was found to be more effective during the decolorization of Reactive Yellow 14.

3.2. Effect of ferrous dosage

Effect of iron dose on decolorization and COD removal for Procion Red MX-5B is presented in [Supplementary figure-4](#) and [Supplementary figure-5](#). H₂O₂ dose was fixed at 16.6 mM and ferrous sulphate dose as Fe²⁺ was varied from 0.18 mM to 0.72 mM. Optimum ferrous iron dose observed was 0.36 mM with 92.4% decolorization and 89.4% COD removal at 30 min irradiation time. Solar light has the largest fraction of photons with the energy needed to drive photoreaction. The photo generated ferrous ion participates in Fenton's reaction generating additional .OH radicals thereby accelerating the oxidation process compared to Fenton's process. Kavitha and Palanivelu [17] reported that catalytic nature of iron helps in enhancing the reaction occurring at lower concentration of iron in solar Fenton's process. Selvam et al. [7] reported 70% decolorization and 51.3% degradation of Reactive Orange 4 by solar light at lower iron dose.

3.3. Decolorization and degradation by solar Fenton's process

The decolorization of MX-5B by solar Fenton's process at optimum conditions is shown in [\[Supplementary figure-6\]](#). Color removal was very fast as the solution was decolorized in less than 5 min in solar Fenton's process the color completely changed from dark brown to colorless. The UV-visible spectral changes of Procion Red MX-5B at different irradiation time are presented in [\[Supplementary figure-6\]](#). The absorption band at 538 nm decreased in 3 min irradiation and the solution turned from dark red to colorless. The faster decolorization of dye is due to the initial electrophilic cleavage in chromophoric azo (-N=N-) bonds attached to the naphthalene ring. The absorption

band corresponding to MX-5B at 538 nm completely disappeared indicating degradation and disappearance of the conjugated structure. Chacon et al. [18] observed that the intensity of absorption at 430 nm decreased as the irradiation time increased during solar photo catalytic degradation of Acid Orange 24. Similar results are reported by Murganandham et al. [17] Light plays two different roles that lead to an improvement of the reaction yield. It drives solar-Fenton's reaction, producing extra hydroxyl radicals and the recovery of Fe (II), which helps in improving the degradation and decolorization efficiency. The use of sunlight and Fenton's reagent may become an effective way of eliminating color and organic load. Selvam et al. [7] reported that Reactive Orange 4 was almost decolorized and degraded in 30 min by solar Fenton's process.

Torrades et al. [19] stated that Procion Red H-E7B, and Red Cibacron FN-R could be efficiently decolorized by solar photo-Fenton's process. Chacon et al. [18] reported that photo-Fenton's process by sunlight was effective for the degradation and mineralization of Acid Orange 24. The results obtained demonstrate that use of solar Fenton's process is more effective in decolorization and degradation of both compounds as compared to Fenton's process alone.

To obtain detailed information on the reaction during solar Fenton's process the resulting oxidation products were determined. Conductivity increased from 1490 $\mu\text{S cm}^{-1}$ to 2300 $\mu\text{S cm}^{-1}$ for MX-5B [Supplementary figure-7]. A rapid increase in the conductivity indicates the formation of acidic by-products during the degradation of MX-5B by solar Fenton process. Release of inorganic ions at different irradiation time is presented Supplementary figure-8. Sulphate concentration increased from 124 mg L⁻¹ to 286 mg L⁻¹ for MX-5B. During oxidation process, sulphonic group may be substituted by .OH at the initial stage resulting in release of sulphate in the solution. The formation of chloride from the mineralization of organic chlorine linked to the triazine ring. Nam et al. obtained similar results during the study on azo dye oxidation by the FeIII-EDTA-H₂O₂ system. MX-5B contains three sources of nitrogen including an azo group, a triazine ring and a nitrogen atom linking naphthalene ring to the triazine ring. It was found that primary aromatic amine would disappear with the decolorization of MX-5B [20]. Ammonia was detected in traces from the oxidation of primary aromatic amine. Nitrate concentration increased from 60 mg L⁻¹ to 110 mg L⁻¹ for MX-5B. Chloride concentration increased from 83 mg L⁻¹ to 105 mg L⁻¹ from 5 min to 30 min of reaction time for MX-5B. The degradation leads to conversion of organic carbon to inorganic ions such as nitrate and sulphate and chloride. Houas et al. [21] reported complete mineralization of carbon, nitrogen, sulfur, and heteroatom of Methylene Blue (MB) by TiO₂/UV process into CO₂, NH₄⁺, NO₃⁻ and SO₄²⁻. Stylidi et al. [22] reported that during solar induced photocatalytic degradation of azo dyes in aqueous TiO₂ suspensions generates sulphate and nitrate as oxidation products.

3.4. Ion chromatography analysis

The data on organic acids formed during solar Fenton's process is presented in Supplementary figure-9. For MX-5B, oxalate formed during initial stages of reaction was oxidized at 30 min irradiation time whereas acetate was remaining at the end of reaction. It was observed that nearly complete oxidation of acids was achieved in solar Fenton's process. The lower values of acetate and oxalate obtained in solar Fenton process indicates that these compounds are oxidized under the influence of solar light leading to higher mineralization efficiency. Aromatic and aliphatic acids were identified during photo catalytic degradation of Acid Orange 7 before complete mineralization to water and CO₂ [7, 23]. Herrmann et al. observed formic and lactic acids as intermediates on solar photo catalytic irradiation of Amaranth indicating fast and easy naphthalene ring rupture [9]. Kavitha and Palnaivelu [17] reported that carboxylic acids like acetic and oxalic acids were formed as the end products during the degradation of phenol by solar Fenton processes. Mahamoodi et al. [23] detected format, acetate, and oxalate as aliphatic intermediates during the photo catalytic degradation of Reactive dyes RB8 and RB 220. They have reported that the amount of inorganic ions reached the maximum when solution color completely disappears.

3.5. Degradation kinetics by solar Fenton's process

The pseudo first order kinetics of MX-5B during solar Fenton's process is presented in the following reaction.

$$-dc/dt = K'd [CA] C.OH]$$

At constant hydroxyl radical concentration C.OH should be constant hence, the above equation becomes

$$-dc / dt = K[CA]$$

K is the pseudo first order rate constant at time, and CA is the concentration. COD removal was used to study the degradation kinetics for MX-5B at optimum reaction conditions Fe²⁺/H₂O₂ and irradiation time. The plot of -ln(CODt/COD₀) vs. reaction time is presented in Supplementary figure-10. The data fitted linearly with the coefficient values of 0.96 for MX-5B. The rate constant for MX-5B was 0.1214 min⁻¹. Shu and Chang [24] reported pseudo first order rate constant on decolorization of azo dyes by UV /H₂O₂ process. Feng et al. [25] observed pseudo first order reaction on discoloration of dye by solar photolysis of ferri-oxalate.

3.6. Biodegradability assays

The data presented in Supplementary figure-11 show that after solar Fenton's process the ratio of BOD₅/COD was increased. The ratio before treatment was 0.13 for MX-5B. After treatment the ratio increased to 0.45 for MX-5B indicating an effective increase of biodegradability. Torrades et al. [19]

reported that solar Photo Fenton has proven beneficial in increasing BOD5/COD ratio of Procion Red H-E7B and Red Cibacron FN-R. The increase of BOD5/COD value 0.4 is considered as the quantitative index for complete biodegradability of organic matter [26].

3.7. FTIR analysis

FTIR spectra of MX-5B before and after Fenton's treatment are presented in [Supplementary figure-12](#). Majority of peaks observed before Fenton's process disappeared during the reaction. The bands observed in the raw MX-5B in the range at 1370 cm^{-1} to 1042 cm^{-1} disappeared after 15 min of reaction time. The new peaks formed at 1709 cm^{-1} (oxalate), 1403 cm^{-1} (formate) and 1062 cm^{-1} (carboxylic acid) disappeared after 30 min indicating the decrease in the aromaticity. The sample at this stage showed the presence of bands at 1666 cm^{-1} , 1147 cm^{-1} and 803 cm^{-1} may be due to stretching vibrations of O-H bond, apparently became strong. A new peak at 1403 cm^{-1} may be formed due to interaction between the C-O stretching and O-H bending in a carboxylic group from acetic acid. Galindo et al. [26] reported that bands corresponding to oxalate (1709 cm^{-1} and 1690 cm^{-1}) and acetate (1416 cm^{-1}) were observed during photo degradation of Acid Blue74. Formation of carboxylic acid was reported by others [22, 23]. Based on the IR data it can be concluded that the naphthalene ring in the dye is oxidized, and broken down to carboxylic acid, which is in accordance with oxalate and acetate identified by ion chromatography. Zhao et al. [27] reported formation of carboxylic acid as acetic acid by IR spectroscopy indicating the breaking of -N=N- during photo degradation of an azo dye, Mordant Yellow 10 (MY10), under UV irradiation. By comparing IR data of MX-5B, it was observed that some of the carboxylic acids were formed in the early stages of MX-5B of solar irradiation and were oxidized completely at 30 min of reaction time. Similar results are reported by Hu et al. [28] during photo degradation of Procion Red dye.

[IV] CONCLUSION

The decolorization and degradation of MX-5B was studied using solar Fenton's process. The optimum dose of H₂O₂ for MX-5B was 16.6 mM. 0.36 mM Fe dose of was optimum for MX-5B. For MX-5B almost 99% decolorization 97% COD removal was achieved by solar Fenton's process in 30 min of irradiation time.

Study of FTIR and IR results shows that, solar degradation mechanism follows in three steps in which initially the more active bonds were hydroxylated. The N=N bond linked to the benzene ring or the naphthalene ring, and the C-S bond of sulphonate group form organic acids, SO₄²⁻ and NH₄⁺. The triazine ring further oxidized to form NO₃⁻ and Cl⁻. At the same

time, the aromatic acids produced initially were further oxidized and led to the cleavage of the aromatic ring opening to form aliphatic acids. MX-5B undergoes oxidation leading to non toxic and biodegradable ultimate breakdown products, such as, oxalic acid and acetic acid. Solar Fenton process at reduced Fe²⁺ and H₂O₂ concentration not only oxidizes but also helps in decolorization and nearly mineralization of the dyes at low dose of reactants. By solar Fenton's process better biodegradability of MX-5B could be achieved. The study shows that solar Fenton process can be prove an effective advanced oxidation process

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- via Fenton type reagents. *Journal of Photochem. and Photobio. A: Chemistry* 99(1-13): 57-66.
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SUPPLEMENTARY FIGURES
(Not verified by Journal. Authors are responsible for any error)

Fig. 1. Structure of Procion MX-5B

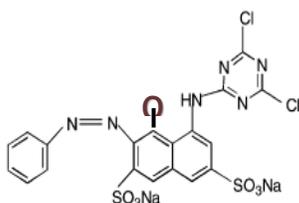


Fig. 4. Effect of Fe²⁺ dose on decolorization of MX-5B by solar Fenton's process

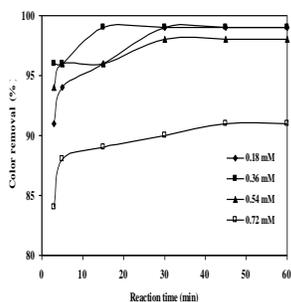


Fig. 7. pH and conductivity of MX5B during solar Fenton process

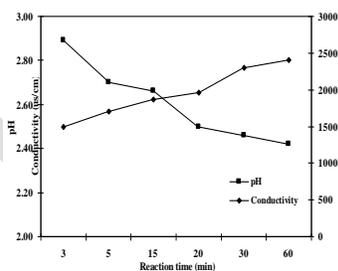


Fig. 2. Effect of H₂O₂ dose on decolorization of MX5B by solar Fenton's process

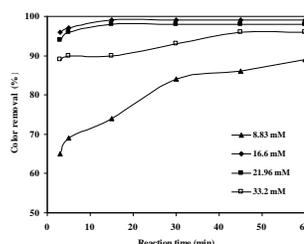


Fig. 5. Effect of Fe²⁺ dose on COD removal of MX-5B by solar Fenton's process

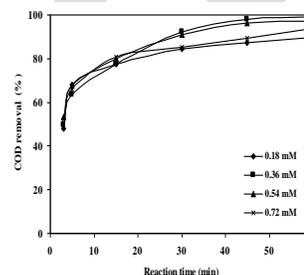


Fig. 8. Release of inorganic anions of MX5B by solar Fenton Process

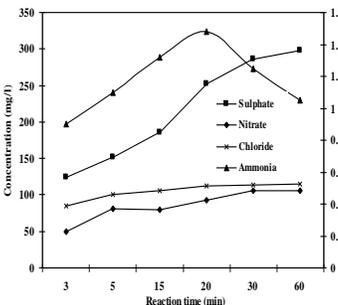


Fig.3. Effect of H₂O₂ dose on COD removal of MX5B by solar Fenton process

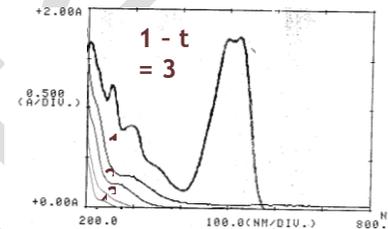


Fig. 6. UV –Visible spectra of MX-5B during solar Fenton process with different irradiation time

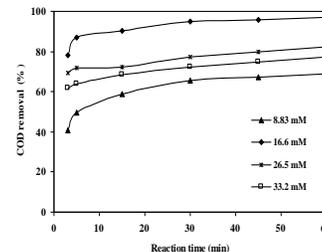


Fig. 9. Formation of acetate and oxalate of MX5B by solar Fenton's process

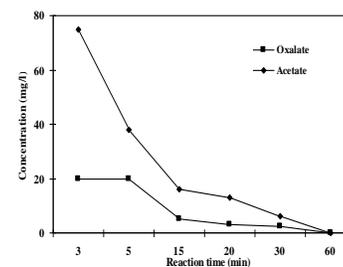


Fig. 10. Degradation kinetics of MX-5B by solar Fenton's process

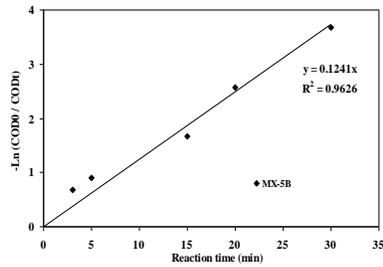


Fig. 11. BOD₅/COD ratio by solar Fenton's process

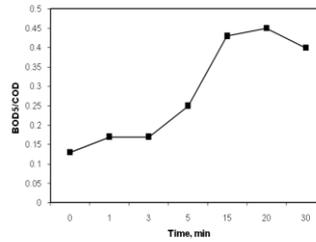


Fig. 12. FTIR spectra of MX-5B during solar Fenton process with different time. a – before treatment, b, c, after treatment. (15min, 30 min)

Fig. 10. Degradation kinetics of MX-5B by solar Fenton's process

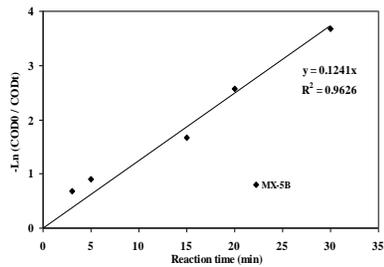


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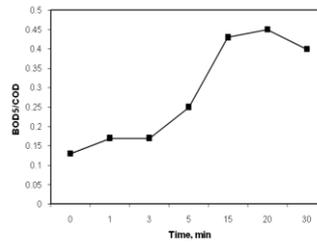


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