JETIR.ORG ISSN: 2349-5162 | ESTD Year : 2014 | Monthly Issue
JOURNAL OF EMERGING TECHNOLOGIES AND INNOVATIVE RESEARCH (JETIR)
An International Scholarly Open Access, Peer-reviewed, Refereed Journal

SYNTHESIS AND APPLICATION OF IRON (II) SCHIFF BASE COMPLEX FOR REMEDIATION OF WASTE WATER OF CAR WASH FACTORY

[1]Mohammad Mudassir, [2]Ashish Kumar, [3]Shivalini Singh, [4]Himani Pachouri

[1]Research Scholar, [2]Professor, [3]Research Scholar, [4]Research Scholar
Department of Chemistry, Agra College, Agra
Dr. Bhimrao Ambedkar University, Agra, India

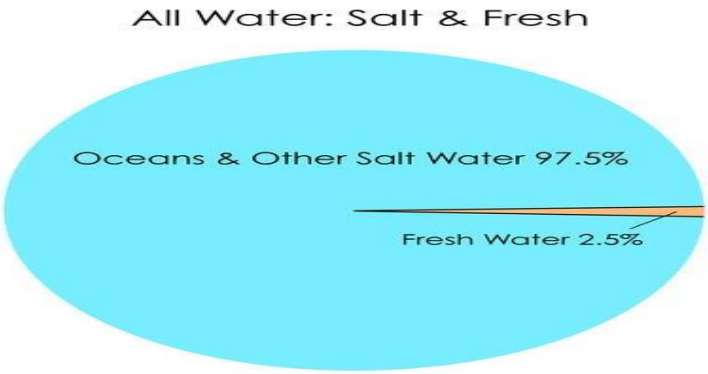
***Abstract*:** We report the synthesis and characterization of iron (II) coordination complex designed for environmental cleanup. By condensing 6-aminopenicillanic acid (6-APA) with salicylaldehyde, we developed a stable tridentate ONO-donor ligand, which was subsequently bound to Fe (II) core. The structure was verified using FTIR, ^{1}H-NMR, and elemental analysis. We then tested this complex as a photocatalyst on tough, real-time car wash wastewater (initial COD = 650 mg/L, TOC = 150 mg/L). The results show that a catalyst dose of 0.5 g/L works best, destroying 78.5% of the COD and mineralizing 66.7% of the TOC within 180 minutes. The degradation process follows pseudo-first-order kinetics ($k = 0.0060\ min^{-1}$). This confirms that modifying antibiotic cores is a highly practical strategy for building heavy-duty industrial water purification systems.

***Keyword*:** Schiff base, Photocatalysis degradation, wastewater treatment, Advanced oxidation process

INTRODUCTION

The basic requirements to life of a human being are air, water, and food. Water is the one of the most essential thing required to survive on this planet earth. Only 2.5% of water is in pure state out of its total weight age. Due to industrialization more and more unwanted substances are added to water bodies which lead to water pollution. In Present time dyes, pigments, paints are universal in industries like textile, leather, food, paper and plastic. The effluents from these industries act as pollutant and are dangerous due to its adverse effect on the environment [1]**.** The water pollutants from the industries are having an adverse effect on water bodies and to avoid the effective development of such pollutant in the environment it is important to take an active approach to reducing these pollutants to smaller amount of harmful compounds or minerals the process [2].For environmental remediation, major challenges are effective and cheap oxidative removal of organic pollutants from waste water [3]. In current years process of advanced oxidation show a strong action for the removal of organic pollutant from waste water [4]. For the treatment of toxic organic pollutants from waste water metal oxide like TiO2 and photo fenton are powerful advanced oxidation processes [5]**.** In these type of waste water treatment methods hydrogen peroxide activated by dissolved iron or UV irradiation to produce high reactive species mainly ·OH radical, which oxidize the organic compound [6]. Photo fenton process has some limitations such as working at the low pH (pH≤3), costly UV light source and cannot be restored easily [7]. Wastewater can also be treated by using enzyme based catalysis for the conversion of toxic substrate into less toxic. This method is also ecofriendly as compared to other one [8]. Peroxidases and laccases are oxidative enzymes which work without any other cellular component. These enzymes can remove many toxic compounds [9].**)**. The enzymes based catalysis method has drawback of being expensive due to which the commercial implementation of these biocatalyst is very low. Due to their expensiveness biocatalyst treatment is replaced by the O_2 and H_2O_2 oxidants [10]. **(**Some metal organic complex for example metal porphyrins and metal pathalocyanines are very good photo catalyst [11]. These metal organic complexes are very effective photo catalyst because they are good electron transfer agents and have strong absorption in the visible light. By this type of environmental friendly metal-organic complex O_2 for H_2O_2 molecules are activated [12]. Due to relative ease of synthesis, metal Schiff base complexes have been recognized in the field of catalysis [13]**.** Metal Schiff base complexes have attracted much attention because of their stunning electronic characteristics and stable structure. Selectivity and stability of Schiff base metal complexes can be renovated by the help of optimization of ligand or put up an extra subsequent [14]. The organic compounds can be easily degraded by photo catalytic action of Schiff base complexes of copper, iron [15].

Selective catalytic oxidation has been performed by metal Schiff base complexes in homogeneous reactions [16]. But in some researches it is reported that in visible light heterogeneous photo catalytic execution for treatment of water which contain toxic organic contaminants. Benefits of heterogeneous catalysts are, these can be discarded easily from the reaction solution. It is more pleasant that this hydrophobic micro environmental simulation enzyme can produce catalytic properties and prevent aggregation and degradation [17]. Photo catalysis is an important method in which naturally available sunlight is an excellent source of energy for rapid breakdown of chemical molecules.



Industrialization has brought about extraordinary economic prosperity, but also caused serious environmental problems, of which water contamination is one of the most serious. It is considered as one of the biggest polluters of water among the industrial sectors, contributing for around 20% of industrial wastewater globally. The textile sector has specialized manufacturing units such as the shoe lace, ribbon and narrow fabric industries, which generate effluents with peculiar characteristics, requiring specific treatment procedures.



Some water Pollution's pictures

Materials and method

Salicaldehyde, 6-Aminopenicillanic Acid (6APA), methanol, ethanol, iron acetate, Hydrochloric acid, sodium hydroxide. All reagents used were analytical grade, procured from CDH, Sigma Aldrich, all synthesis work was carried out in the Department of Chemistry, Agra College, Dr. Bhim Rao Ambedkar University, Agra.

Synthesis of Schiff base and metal complex

Dissolve salicylaldehyde (1.22 g, 10 mmol) in 20 mL of dry methanol, Dissolve 6-APA (2.16 g, 10 mmol) in 30 mL of dry methanol, Mix both solutions in a 100 mL round-bottom flask ,Add 2-3 drops of glacial acetic acid as a catalyst, Reflux at 60°C for 4 hours under

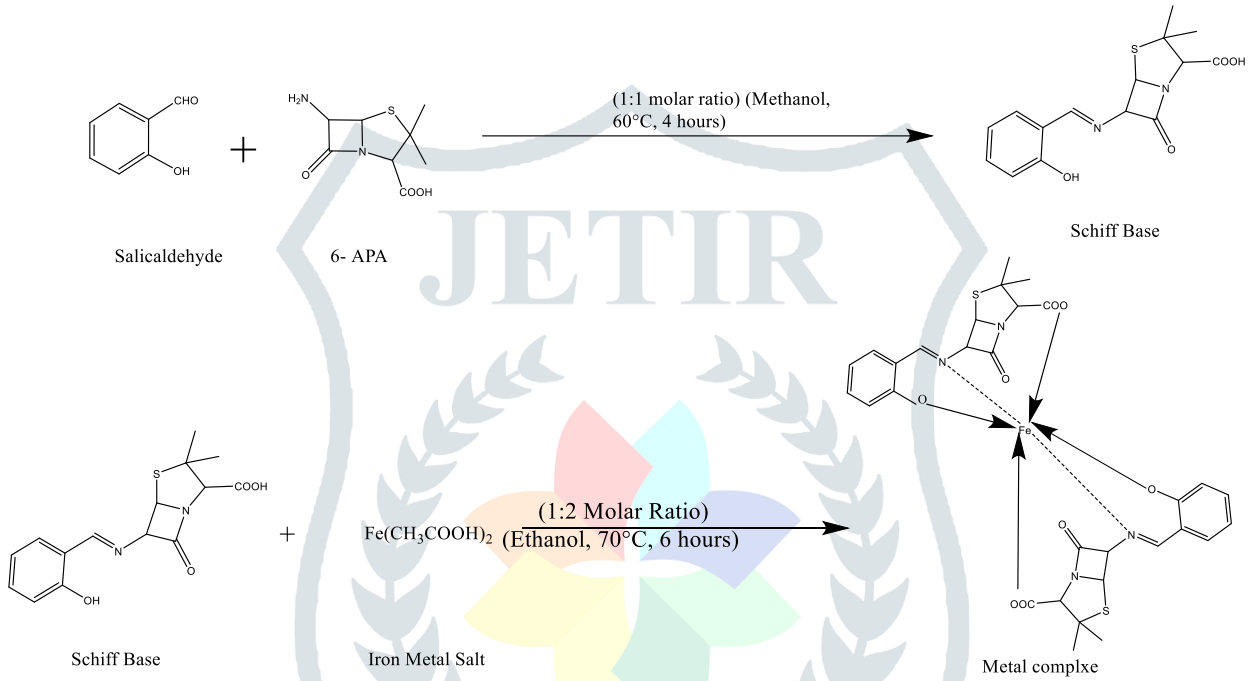
magnetic stirring, Cool to room temperature Concentrate the solution under reduced pressure (rotary evaporator),Precipitate with cold diethyl ether, Filter and wash with cold methanol Recrystallize from ethanol Dry.

IUPAC NAME**:** (*Z*)-6-((2-hydroxybenzylidene) amino)-3,3-dimethyl-7-oxo-4-thia-1-azabicyclo[3.2.0]heptane-2-carboxylic acid

Yield: 2.83 g (85%) Color: - Lemon Yellow

Dissolve Schiff base ligand (3.34 g, 10 mmol) in 30 mL of absolute ethanol, Dissolve iron (II) acetate (1.43 g, 5 mmol) in 20 mL of absolute ethanol, Mix the two solutions in a 100 mL round-bottom flask, Heat at 70°C for 6 hours under reflux with continuous stirring, Cool to room temperature, The complex precipitates spontaneously, Filter and wash with ethanol, Recrystallize from methanol, Dry in vacuum oven at 50°C for 4 hours.

Yield: 3.42 g (78%), Color: Deep brownish-red.



Elemental Analysis of Schiff Base and Metal Complex in (%)

Ligands/Metal complexe	Colour	M.P. (°C)	C Found (Calcd)	H Found (Calcd)	N Found (Calcd)	O Found (Calcd)	S Found (Calcd)	Fe Found (Calcd)	Mol. Wt. Found (Calcd)
6-APA Salicylaldehyde ($C_{15}H_{16}N_2O_4S$)	Lemon Yellow	110	55.98 (56.24)	4.78 (5.03)	8.27 (8.74)	19.36 (19.98)	9.75 (10.01)	---	318.76 (320.36
6-APA Salicylaldehyde $Fe(CH_3COOH)_2$ $(C_{15}H_{15}N_2O_6S)_2$	Brownish yellow solid	132	51.26 (51.88)	4.09 (4.35)	7.95 (8.07)	18.12 (18.43)	9.23 (9.23)	7.88 (8.04)	690.32 (694.55)

FT-IR: 3440 cm^{-1} (phenolic O–H stretching), 2870 cm^{-1} (carboxylic O–H stretching), 1772 cm^{-1} (β-lactam C=O stretching), 1718 cm^{-1} (carboxylic C=O stretching), 1628 cm^{-1} (azomethine C=N stretching), 1515 cm^{-1} (aromatic C=C stretching), 1290 cm^{-1} (C–N stretching), 1205 cm^{-1} (phenolic C–O stretching), and 785 cm^{-1} (C–S stretching).

^{1}H NMR: DMSO (500 Hz): d 12.55 (s), 8.69 (s), 7.89-7.70 (m), 4.88 - 4.35 (m), 1.69 (s)
^{13}C***NMR***: DMSO (500 Hz): d 174.70, 166.80, 160.80, 129.20, 79.50, 64.10, 62.50, 24.00

IR Spectra: ν CO (Acidic): 3325 cm^{-1}; ν C=N: 1620cm^{-1} ν C=O: 1755 $cm^{-1;}$ ν C6H5: 2995 cm^{-1}; ν CH3: 1480 cm^{-1}; ν C-OH (Acidic): 2815 cm^{-1}; ν C-N: 1295 cm^{-1}; ν C-S: 785 cm^{-1}; ν Fe-N: 855 cm^{-1}

NMR Data: *^{1}H NMR:* DMSO (500 Hz): d 12.55 (s), 8.69 (s), 7.89-7.70 (m), 4.88- 4.35 (m), 1.69 (s)

***^{13}C NMR*:** DMSO (500 Hz): d 174.80, 166.90, 160.85, 129.30, 79.60, 64.15, 62.58, and 24.18

Table 1. Industrial Waste water Matrix for automotive (car wash)

Parameter	Value	Indication
pH	8.1	The wastewater was slightly alkaline in nature.
COD	650 mg/L	The high COD value indicates a significant organic load due to the presence of detergents, oil and grease, and hydrocarbons.
TOC	150 mg/L	The wastewater contained a considerable amount of organic carbon.
TSS	750 mg/L	The high concentration of total suspended solids suggests the presence of a large amount of particulate matter, which is typical of car wash wastewater.
TDS	1500 mg/L	The elevated total dissolved solids indicate a high concentration of dissolved salts, detergents, and other soluble contaminants.

Interpretation of Physicochemical Characteristics of Car wash wastewater. The wastewater showed a high COD (650 mg/L) and TOC (150 mg/L). The COD/TOC ratio (~4.3) indicates the presence of large amounts of chemically oxidizable pollutants such as detergents, surfactants, oils, and petroleum hydrocarbons, requiring advanced treatment. High TSS and TDS: The TSS (750 mg/L) reflects the presence of suspended particles like soil, sand, and vehicle debris, while TDS (1500 mg/L) confirms high concentrations of dissolved salts and cleaning chemicals. The wastewater had a pH of 8.1, due to the use of alkaline detergents and commercial cleaning agents during vehicle washing. The high organic load, elevated solids, and alkaline pH indicate that car wash wastewater requires efficient treatment. These characteristics support the application of Schiff base metal complex-based photocatalytic oxidation for effective pollutant degradation.

The Fe(II)-6APA–salicylaldehyde Schiff base complex exhibited efficient photocatalytic activity through ligand-to-metal charge transfer (LMCT) under light irradiation. Photoexcitation generated reactive oxygen species (ROS), particularly superoxide ($O_2^{\bullet-}$) and hydroxyl (•OH) radicals, which enhanced pollutant degradation. Stable coordination of the ligand with the Fe(II) center facilitated rapid electron

transfer and efficient Fe(II)/Fe(III) redox cycling. Consequently, organic contaminants in car wash wastewater were effectively mineralized into CO_2 and H_2O.

$[Fe^{II}(L)] + hv \rightarrow [Fe^{III}(L)] + e^-$ .....(1)

$O_2 + e^- \rightarrow O_2\bullet^-$ ..... (2)

$O_2\bullet^- + H^+ \rightarrow HO_2\bullet$ ..... (3)

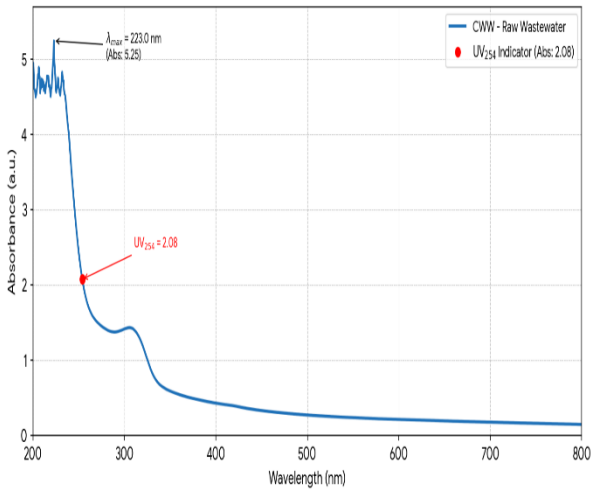
$2HO_2\bullet \rightarrow H_2O_2 + O_2$ ..... (4)

$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + \bullet OH + OH^-$ .....(5)

$\bullet OH$ + Organic Pollutants $\rightarrow CO_2 + H_2O$ + Mineralized Products ... (6)

Photocatalytic Reactor and Experimental Design

Photocatalytic degradation experiments were carried out in a flat-bed, open-dish batch photo reactor housed inside a HEPA-filtered laminar air-flow (LAF) chamber to minimize external contamination. The reactor was irradiated using two 15 W UV-A lamps (λ = 365 nm). For each experiment, 100 mL of car washing factory wastewater was treated with the synthesized Fe-6-APA–salicylaldehyde Schiff base complex at catalyst dosages of .1, .3, and .5 g per 100 ml. The reaction mixture was continuously stirred, and aliquots were collected at 60, 120, and 180 min for physicochemical analysis. All experiments were conducted in triplicate (n = 3). Control experiments included: (i) a blank run without catalyst and UV irradiation to evaluate wastewater stability, (ii) a UV photolysis experiment without catalyst to determine direct photo degradation, and (iii) a dark adsorption experiment with the Fe(II) Schiff base complex under continuous stirring in the absence of UV light to assess adsorpon. The net photocatalytic degradation efficiency was calculated using the relationship: net photocatalytic removal (%) = total observed removal (%) − dark adsorption removal (%) − direct photolysis removal (%).



UV- visible Absorption spectrum of raw water

Table 2. Photocatalytic UV_{254} Absorbance Degradation Analysis Matrix

Photocatalyst Complex	Dosage (g/L)	UV removal (%) at 60 min	UV removal (%) at 120 min	UV removal (%) at 180min
Fe(6APA-Salicylaldehyde)	.1	27.5%	43%	58.5%

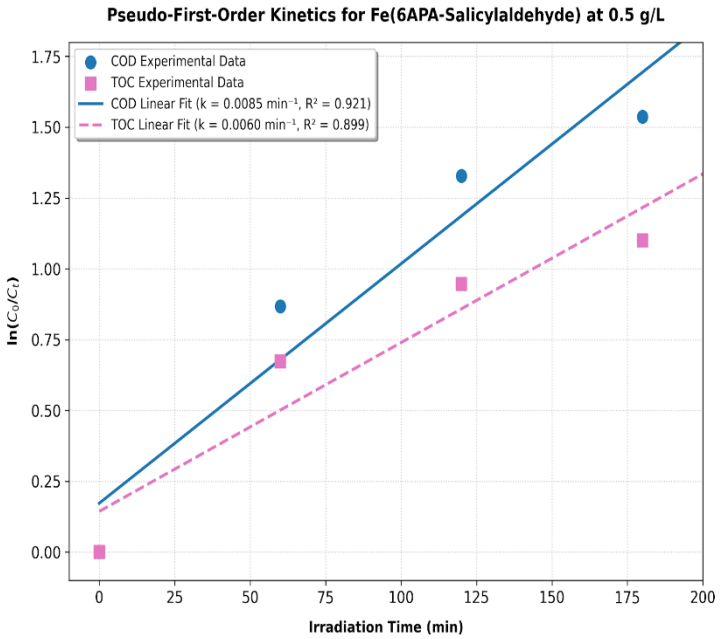
Fe(6APA-Salicylaldehyde)	.3	46.8%	63.3%	72%
Fe(6APA-Salicylaldehyde)	.5	72.9%	72.9%	82.6%

Kinetic and Statistical Analysis

The photocatalytic degradation efficiency of car washing factory wastewater was evaluated using the pseudo-first-order kinetic model based on the Langmuir–Hinshelwood (L–H) equation:

$$\ln(C_t/C_0)=kt$$

Where C_0 is initial chemical oxygen demand (COD), Ct is COD at irradiation time t (min), k is apparent pseudo-first-order rate constant (min-1), and t (min) is the irradiation time. The kinetic rate constant k was determined from the slope of the linear plot of in (C_0/Ct) against irradiation time. The goodness of fit of the kinetic model was evaluated using the coefficient of determination (R^2). All photocatalytic degradation experiments were carried out in triplicate (n=3), and the results are expressed as mean ± standard deviation (SD). Statistical analysis was performed using one-way analysis of variance (ANOVA) to compare the degradation efficiencies of different Schiff base metal complexes. Statistical significance was considered at a confidence level of 95% $p<0.05$. Optimization Matrix Showing the Effect of Complex, Dosage, and Irradiation Time on COD Removal Efficiency (%).



Photocatalytic Degradation Efficiency Report

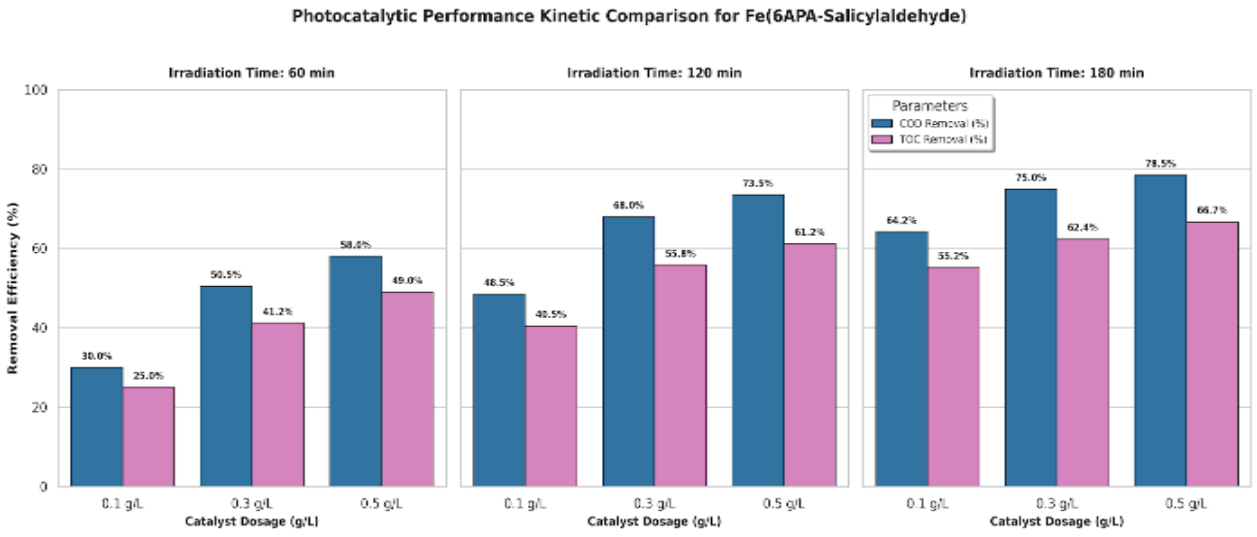
COD Removal (%) Kinetic Study
Catalyst Core: Fe(II) Metal Complex with 6-APA-Salicylaldehyde Schiff Base Ligand
This technical section presents the comparative optimization data regarding the treatment of organic load (COD) using the synthesized Iron(II) Schiff base complex under variable catalyst dosages. The evaluation covers a structural kinetic monitor over a maximum time frame of 180 minutes to establish the optimal quantitative parameters for degradation.

Table 3. Chemical Oxygen Demand (COD) Degradation Analysis Matrix

Photocatalyst Complex	Dosage (g/L)	COD Removal (%) at 60 min	COD Removal (%) at 120 min	COD Removal (%) at 180 min
Fe(6APA-Salicylaldehyde)	0.1	30.0%	48.5%	64.2%
Fe(6APA-Salicylaldehyde)	0.3	50.5%	68.0%	75.0%
Fe(6APA-Salicylaldehyde)	0.5	58.0%	73.5%	78.5%

Table 4. Total Organic Carbon (TOC) Degradation Analysis Matrix

Photocatalyst Complex	**Dosage (g/L)**	**TOC Removal (%) at 60 min**	TOC Removal (%) at 120 min	**TOC Removal (%) at 180 min**
Fe(6APA-Salicylaldehyde)	0.1	25.0%	40.5%	55.2%
Fe(6APA-Salicylaldehyde)	0.3	41.2%	55.8%	62.4%
Fe(6APA-Salicylaldehyde)	0.5	49.0%	61.2%	66.7%



CONCLUSION

6-APA-salicylaldehyde Schiff base ligand-coordinated Iron (II) complexes, synthesized via a controlled condensation and reflux route, were systematically evaluated as heterogeneous photocatalysts for the remediation of real-time industrial car wash wastewater. Coordination of the Fe(II) metal core with the rigid ONO-donor Schiff base scaffold significantly enhanced the structural and electronic characteristics, facilitating efficient ligand-to-metal charge transfer (LMCT) and suppressing photogenerated electron-hole recombination. Under optimized operational parameters at a catalyst loading of 0.5g/L and 180 min of light exposure, the engineered Fe(II) complex achieved a peak Chemical Oxygen Demand (COD) degradation efficiency of 78.5% and a Total Organic Carbon (TOC) mineralization efficiency of 66.7% (reducing residual organic carbon from an initial 150 mg/L down to 50 mg/L. UV_{254} reduced to 82.6%. Kinetic evaluation confirmed that the organic breakdown strictly conformed to the pseudo-first-order Langmuir-Hinshelwood model $R^2 = 0.899$), yielding an apparent mineralization rate constant (k) of 0.0060 min^{-1}. Control experiments comprising blank stability runs, direct UV photolysis, and dark adsorption checks—confirmed that the observed organic pollutant removal reflects genuine semiconductor-mediated heterogeneous photocatalysis rather than non-catalytic photolysis or primary surface adsorption. Statistical validation via one-way ANOVA further established significant differences across variable catalyst dosages ($p < 0.05$). These results identify the synthesized Fe(II)-6-APA-salicylaldehyde complex as a highly effective, robust photocatalyst for the advanced oxidation treatment of recalcitrant industrial effluents,

ACKNOWLEDGMENT

The authors thanks to the Department of Chemistry, Agra College, Dr. Bhimrao Ambedkar University, Agra, for synthesis facilities, and Jiwaji University.

REFERENCES

- Ahmed, B., Limem, E., Abdel-Wahab, A. and Nasr, B. (2011). Photo-Fenton treatment of actual agro-industrial wastewaters. *Industrial & Engineering Chemistry Research*, ***50*(11)**, 6673-6680.
- Araya, T., Quan, S., Man-ke, J., Wan-hong, M., Johnson, D. and Ying-ping, H. (2016). Selective Photocatalytic Degradation of Organic Pollutants Using a Water-Insoluble Zn–Schiff Base Complex. *Water, Air, & Soil Pollution*, ***227*(8)**, 284.
- Basak, T., Bhattacharyya, A., Harms, K., & Chattopadhyay, S. (2018). The ability of a trinuclear zinc (II) Schiff base complex to act as a photocatalyst for the degradation of methylene blue and to mimic phosphatase. *Polyhedron*, ***157***, 449-457.
- Cao, T. T., Zou, C. Q., Luo, G. F., Chen, D. X., Zhao, X. R., Li, R. P., & Huang, Y. P. (2011). Heterogenous Degradation of Toxic Organic Pollutants by Hydrophobic Iron (III) Schiff Base Complex Under Visible Irradiation [J]. *Chemical Journal of Chinese Universities*, ***1*** ,105-112
- Chowdhury, P., Athapaththu, S., Elkamel, A. and Ray, A. K. (2017). Visible-solar-light-driven photo-reduction and removal of cadmium ion with Eosin Y-sensitized TiO2 in aqueous solution of triethanolamine. *Separation and Purification Technology*, ***174***, 109-115.
- Dodwad, S. S. and Sawant, A. S. (1994). Study of diamagnetism in uranyl complexes of some Schiff bases. *Asian Journal of Chemistry*, ***6*(4)**, 877-883.

- El-sharkawy, R. G., El-din, A. S. B. and Etaiw, S. E. D. H. (2011). Kinetics and mechanism of the heterogeneous catalyzed oxidative decolorization of Acid-Blue 92 using bimetallic metal–organic frameworks. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, ***79*(5)**, 1969-1975.
- Ghosh, M. K., Pathak, S. and Ghorai, T. K. (2019). Synthesis of Two Mononuclear Schiff Base Metal (M= Fe, Cu) Complexes: MOF Structure, Dye Degradation, H_2O_2 Sensing, and DNA Binding Property. *ACS omega*, ***4*(14)**, 16068-16079.
- Guguloth, V., Ahemed, J., Subburu, M., Guguloth, V. C., Chetti, P. and Pola, S. (2019). A very fast photodegradation of dyes in the presence of new Schiff's base N4-macrocyclic Ag-doped Pd (II) complexes under visible-light irradiation. *Journal of Photochemistry and Photobiology A: Chemistry*, ***382***, 111975.
- Hei, J., Yang, L. and Li, J. (2019). Synthesis and Photocatalysis of Metal Complexes with Schiff Base Derived from 2-Thiophene Carboxaldehyde and Ethylenediamine. *Chinese Journal of Applied Chemistry*, ***36*(8)**, 949-957.
- Hong, X. J., Liu, X., Zhang, J. B., Lin, C. L., Wu, X., Ou, Y. J. and Cai, Y. P. (2014). Two low-dimensional Schiff base copper (I/II) complexes: synthesis, characterization and catalytic activity for degradation of organic dyes. *CrystEngComm*, ***16*(34)**, 7926-7932.
- Kianfar, A. H., Dehghani, P. and Momeni, M. M. (2016). Photo-catalytic degradation of methylene blue over nano titanium/nickel oxide prepared from supported Schiff base complex on titanium dioxide. *Journal of Materials Science: Materials in Electronics*, ***27*(4)**, 3368-3375.
- Lamsal, R., Walsh, M. E. and Gagnon, G. A. (2011). Comparison of advanced oxidation processes for the removal of natural organic matter. *Water research*, ***45*(10)**, 3263-3269.
- Li, J., Ma, W., Huang, Y., Tao, X., Zhao, J. and Xu, Y. (2004). Oxidative degradation of organic pollutants utilizing molecular oxygen and visible light over a supported catalyst of Fe (bpy) 32+ in water. *Applied Catalysis B: Environmental*, ***48*(1)**, 17-24.
- Li, X., Zhang, D., Liu, Z., Xu, Y. and Wang, D. (2018). Synthesis, characterization of a ternary Cu (II) Schiff base complex with degradation activity of organophosphorus pesticides. *Inorganica Chimica Acta*, ***471***, 280-289.
- Liu, S., Peng, J., Yang, H., Bai, Y., Li, J. and Lai, G. (2012). Highly efficient and convenient asymmetric hydrosilylation of ketones catalyzed with zinc Schiff base complexes. *Tetrahedron*, ***68*(5)**, 1371-1375.
- Manimohan, M., Pugalmani, S., Ravichandran, K. and Sithique, M. A. (2020). Synthesis and characterisation of novel Cu (ii)-anchored biopolymer complexes as reusable materials for the photocatalytic degradation of methylene blue. *RSC Advances*, ***10*(31)**, 18259-18279.