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Research Paper

Degradation of Rhodamine B by $FeSO_4$-Catalyzed Sulfite Activation Using Oxalic Acid as a Chelating Agent

Asghar Abbas [a], Ahmad Faraz Ali [a], Maria Sophy [b], Meesam Ali [a,*], Usman Saeed [a]

[a] Department of Chemical Engineering and Technology, Muhammad Nawaz Sharif University of Engineering and Technology, Multan 60000, Pakistan.
[b] Department of Physics, University of Agriculture, Faisalabad, Pakistan.

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*CORRESPONDING AUTHOR

Meesam Ali
Department of Chemical Engineering and Technology, Muhammad Nawaz Sharif University of Engineering and Technology, Multan 60000, Pakistan
Email meesam.ali@mnsuet.edu.pk

ABSTRACT

Purpose: Synthetic dyes such as Rhodamine B (RhB) in textile effluents pose serious environmental and health hazards due to their toxicity, carcinogenicity, and resistance to biodegradation. Since conventional treatment methods often fail to achieve complete removal, this study aims to develop and evaluate an efficient, sustainable catalytic system for RhB degradation in wastewater.

Design/methodology/approach: RhB degradation was investigated using a metal-based catalytic system composed of ferrous sulfate ($FeSO_4$), sodium sulfite (Na_2SO_3), and oxalic acid as a chelating agent. The process relies on sulfite activation to generate reactive radicals ($SO_4^{\bullet-}$, $SO_3^{\bullet-}$, and •OH) that attack the chromophoric structure of RhB. Key operational parameters—catalyst dose, Na_2SO_3 concentration, oxalic acid addition, RhB loading, and pH—were systematically varied to determine optimal degradation conditions.

Findings: Under optimal conditions ($FeSO_4$ = 5 mM, Na_2SO_3 = 7 mM, oxalic acid = 3 mM, RhB = 5 mg/L, pH ≈ 5.9), the system achieved a maximum degradation efficiency of 90.3%. Oxalic acid stabilized the Fe^{2+}/Fe^{3+} redox cycle through chelation, sustaining radical generation. The observed RhB degradation and decolorization demonstrated the effectiveness of the developed system for pollutant removal, without directly confirming complete mineralization or detoxification.

Originality: This study presents a novel, cost-effective $FeSO_4/Na_2SO_3$/oxalic acid advanced oxidation process (AOP) for dye wastewater treatment, offering a practical alternative to conventional methods, especially for textile-producing regions such as Pakistan.

Practical implications: The system offers a low-cost, scalable option for treating dye-laden industrial effluents, supporting cleaner production practices in the textile sector.

***Keywords*:** Rhodamine B, $FeSO_4$, Sodium Sulfite, Oxalic Acid, Wastewater Treatment, Advanced Oxidation Processes

1. Introduction

The expansion of industrialization, particularly in the textile, printing, paper, and cosmetic industry, has resulted in an escalation in the release of synthetic dyes into water (Xu et al., 2024). These dyes which are required for certain industries, have now found their way into the environment in large quantities due to their toxic nature and slow biodegradation, making them one of the major environmental pollutants. From the vast array of commercially available dyes, Rhodamine B (RhB) is a notorious one (Pathakoti et al., 2018). RhB is a xanthene dye, it is highly water soluble and good photostability and bright fluorescent, so it is widely used in textile dyeing and printing and cosmetic (Sutanto et al., 2020). Nevertheless, it's high stability and resistance to biodegradation lead to long environmental persistence and the subsequent release into the environment results in considerable ecologic and health risk (Umair Arshad et al., 2019).

Risk of RhB is the subject of Regulation (EC) No 1272/2008 (Umair Arshad et al., 2019). It is considered as a carcinogenic, mutagenic substance which causes liver, kidney and reproductive toxicity on exposure (Nguyen et al., 2018). In aquatic environments RhB not only colors receiving waters—reducing light availability for photosynthesis but also bioaccumulates in organisms, jeopardizing biodiversity and food (Lops et al., 2019). Its aromatic and intricate molecular structure makes it non-biodegradable by traditional wastewater treatment methods and a stubborn pollutant at the global scale. In Pakistan particularly, this would be cause for great concern now that the textile industry reigns supreme in the national economy. Almost 60% of the country's exports are linked to the textile industry, which is the largest employer of the industrial workforce, accounting for 45% (Pang et al., 2019). However, this industry produces huge amount of wastewater which contains synthetic dyes, salts and auxiliaries. It has recently been estimated that the textile industry in Pakistan generates in excess of 500 million gallons of wastewater a day, more than 180 billion gallons annually (Larasati et al., 2020). With dye losses in processing of 10–15%, it is calculated that annually 250–500 tons of RhB might be released in aqueous environment of Pakistan, implying values at ~0.7–1.4 tons per day. These discharges have a double problem: they are contaminating surface waters in an aesthetic sense and are generating a cancer risk for populations using these waters to irrigate and drink (Pratiwi et al., 2020).

Several methods, including adsorption, membrane filtration, coagulation–flocculation and biological degradation, are also available for the removal of dyes (Sun et al., 2020). However, for synthetic dyes like RhB, these techniques have significant limitations. Adsorptive techniques merely relocate contaminants from one phase to another phase, rather than degrading them, resulting in a secondary solid waste that must be disposed of (Suharyadi et al., 2020). Coagulation and flocculation processes also produce substantial amounts of chemical sludge, adding to the cost of treatment and environmental stress (Z. Wang et al., 2024). Although membrane-based separations are very efficient in removing dyes, they are costly and suffer from fouling, which restricts their use in large-scale application (N. Wang et al., 2025). RhB is xenobiotic in nature, structurally complex and toxic towards microbial consortia and hence biological treatments are usually not efficient for RhB (Zhuang et al., 2024). In addition, the aromaticity and electron-withdrawing substituents present in the dye make it extremely difficult to be degraded by enzymatic pathways.

These limitations have raised an urgent need of alternate treatment strategies which can remove as well as mineralize such contaminants to non-toxic end products (Huang et al., 2024).

Advanced oxidation processes (AOPs) are considered an excellent choice for refractory organic pollutants degradation (Zhuo et al., 2023). AOPs permit the generation of a series of reactive oxygen species (ROS) such as hydroxyl radicals (•OH, E0 = 2.7 V), sulfate radicals ($SO_4^{\bullet-}$, E_0 = 2.5–3.1V) and other radicals that could non-selectively oxidize organic compounds (Behnajady et al., 2011). Sulfate radical-based (SR)-AOPs are favored compared to hydroxyl radical systems as they provide higher selectivity, better stability at neutral/mild conditioning, and decreased scavenging with background matrices (Sakthivel et al., 2003). Among SR-AOPs, the sulfite (SO_3^{2-}) activation route is receiving much attention since, in catalytic mode, it produces multiple ROS i.e., $SO_4^{\bullet-}$, $SO_3^{\bullet-}$ and •OH. The sulfite activation can be catalyzed by transition metals like Fe^{2+}, to generate a cocktail of oxidants that can be used to degrade complicated organics such as the discs of RhB (Bica and de Melo, 2020). Moreover, contrary to classic Fenton reactions, which have to be performed in strongly acidic media (pH ≈ 3), the sulfite-based approaches are neutral pH feasible. Hence, the activation of sulfite is also promising for treatment of large-scale textile wastewater (Hidayanto et al., 2017).

Ferrous sulfate ($FeSO_4$) is an inexpensive, widely accessible, and non-toxic metal salt used as a catalyst in AOPs. Fe^{2+} ions, released from $FeSO_4$, are involved in electron transfer processes, which lead to sulfite activation and production of reactive oxygen species (ROSs). The major advantage of Fe^{2+} catalysis is its redox cycling: during ROS production, Fe^{2+} is converted to $Fe^{3+,}$ which is then reduced back to Fe^{2+} by sulfite or other reducing agents to keep the catalytic cycle running with very little external supply (Shah et al., 2021). However, a great challenge in iron based system is that Fe^{3+} tends to hydrolyze and precipitate as ferric hydroxide [Fe $(OH)_3$] around neutral pH, which leads to the loss of catalyst. For this purpose, oxalic acid ($H_2C_2O_4$) is used as a chelator. Oxalate may associate with Fe to give iron–oxalate complexes, which are known to be rather stable and to effectively resist precipitation maintaining Fe in soluble and catalytically active forms (Sutanto et al., 2016). In addition, oxalic acid promotes the Fe^{3+} reduction to Fe^{2+}, increasing the redox cycling and the ROS generation lifetime (Yao et al., 2017). As a photoreductant, oxalic acid can also enhance light-assisted conversion of Fe^{3+} to Fe^{2+}, thus be accelerating Fe^{2+} regeneration.

Given that Fe(II)/sulfite systems as well as Fe–oxalate complexes alone have shown potential for degrading a range of contaminants, there are no or very few reports on the combined use of the two for RhB degradation at room temperature. The current literature has largely concentrated on UV-based or photo-Fenton systems which are efficient but involve additional energy consumption, and rigorous pH regulation (Sreedhar et al., 2018). A knowledge gap remains to be filled in terms of finding green, cheap, and scalable advanced oxidation processes (AOPs) that can efficiently function at mild pH conditions in the absence of external energies.

This work fills this gap by investigating for the first time the degradation of RhB in a synergistic $FeSO_4$/Na_2SO_3/oxalic acid system. The study focused on the following: 1) to evaluate the impact of $FeSO_4$, Na_2SO_3, and oxalic acid dosages on the efficiency of RhB degradation; 2) to investigate the effect of initial RhB concentration and solution pH; 3) to suggest a mechanistic pathway for RhB degradation based on the generation of radicals and cycling of iron redox; 4) to discuss the relevance of the experimental findings to literature and the potential upscaling to the treatment of textile wastewater. This work is directed to that from the synergistic impact of $FeSO_4$, Na_2SO_3 and oxalic acid, it demonstrated an efficient and economical environmental friendly method to treat dye wastewater in this study. Since the results in this paper are of prime importance in emerging textile producing countries like Pakistan, the low-cost and simple scalable treatment technology for sustainable wastewater management are dire.

2. Materials and Methods

2.1. Chemicals and reagents

Rhodamine B (RhB, $C_{28}H_{31}ClN_2O_3$, ≥99% purity) was obtained from Nitin Dye Chem Pvt. Ltd., India, and used as the target pollutant without further purification. Ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$, ≥99%), sodium sulfite (Na_2SO_3, ≥98%), and oxalic acid ($H_2C_2O_4$, ≥99%) were purchased from Sigma-Aldrich (USA). Deionized water with a resistivity of 18.2 MΩ·cm was used for the preparation of all solutions throughout the study. n-Butanol (≥99%) was used as a radical scavenger to quench residual radicals during sampling. H_2SO_4 (0.1M concentration) and NaOH (1M concentration) were used for pH adjustments.

2.2. Solutions Preparation

A 100 mg/L stock solution of RhB was prepared by dissolving 100 mg of RhB powder in 1 L of deionized water. Working solutions of desired concentrations (5–40 mg/L) were prepared by serial dilution of the stock solution. A 1 M n-butanol solution was prepared as a quenching agent by diluting the appropriate amount of reagent in deionized water. Solutions of $FeSO_4$, Na_2SO_3, and oxalic acid were freshly prepared prior to each experiment to minimize oxidation and degradation.

3. Experimental setup

Batch degradation experiments were performed in a 500 mL borosilicate glass beaker containing 200 mL of RhB solution. The mixture was continuously stirred at 250 rpm using a magnetic stirrer and maintained at room temperature (~25 ± 1 °C). The standard reaction condition was as follows: RhB = 5 mg/L, $FeSO_4$ = 5 mM, Na_2SO_3 = 7 mM, oxalic acid = 3 mM, and initial pH = 5.9 (unless otherwise specified). The pH of solutions was adjusted using 0.1 M H_2SO_4 or 1 M NaOH and monitored using a calibrated pH meter.

3.1. Degradation experiments

Effect of catalyst concentration The concentration of $FeSO_4$ was varied as 3, 5, 7 and 10 mM for study the influence of catalyst concentration, while the concentrations of RhB (5 mg/L), Na_2SO_3 (7 mM), and oxalic acid (3 mM) were kept constant. Similarly, the impact of Na_2SO_3 was examined at concentrations of 3, 5, 7, and 10 mM, with the concentrations of $FeSO_4$ (5 mM), oxalic acid (3 mM), and RhB (5 mg/L) held constant. The effect of oxalic acid was investigated at 3, 5, 7, and 10 mM in the presence of the same fixed parameters. Effect of initial dye concentration The initial dye concentration effect was studied in the range of RhB 5–40 mg/L at constant concentration of $FeSO_4$ (5 mM), Na_2SO_3 (7 mM) and oxalic acid (3 mM). Finally, the effect of pH was investigated by setting the solutions pH to 3, 5.9, 7, and 11 according to the standard conditions, respectively.

All reactions were started by the single addition of $FeSO_4$, Na_2SO_3 and oxalic acid to the RhB solution. At given time intervals (0–120 min), aliquots of 5 mL were collected and immediately quenched with 1 mL of n-butanol, to arrest any further reactions.

4. Analytical methods

The residual RhB concentration was determined using a UV–Vis spectrophotometer (Shimadzu UV-1800, Japan) at a maximum absorbance wavelength of 554 nm. Standard calibration curves were prepared for RhB concentrations ranging from 0 to 100 mg/L (R^2 = 0.9997). The degradation efficiency (%) was calculated using Eq. (1):

$$\text{Degradation Efficiency (\%)} = \frac{C_o - C_t}{C_o} \times 100 \quad (1)$$

where C_o (mg/L) is the initial RhB concentration and C_t (mg/L) is the concentration at time *t*.

4.1. Quality assurance and reproducibility

All experiments were conducted in triplicate, and average values are reported. Variations in experimental results were within ±3%. Control experiments without $FeSO_4$ or Na_2SO_3 were conducted to confirm that RhB removal was attributable to radical oxidation rather than adsorption or photolysis.

5. Results and Discussion

5.1. Calibration curve for Rhodamine B

The calibration curve of RhB (0–100 mg/L) was prepared by plotting absorbance at 554 nm against concentration (Fig. 1). The curve displayed excellent linearity with a regression coefficient of R^2 = 0.9997, confirming the reliability of UV–Vis spectrophotometry for quantifying RhB concentration during degradation experiments.

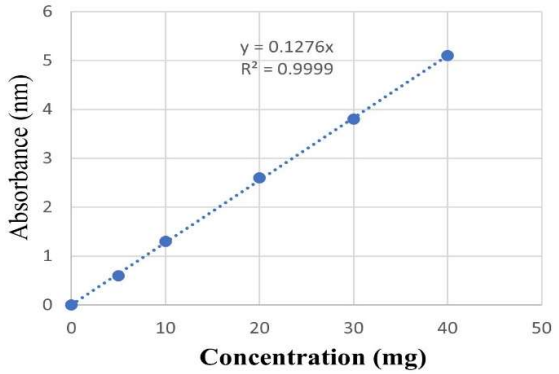

Figure 1. Standard calibration curve of Rhodamine B at 554 nm.

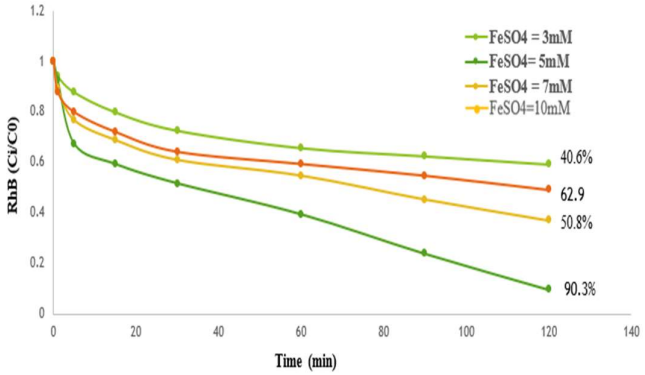

Figure 2. Effect of $FeSO_4$ concentration on RhB degradation efficiency.

5.2. Effect of $FeSO_4$ dosage

The concentration of $FeSO_4$ plays a critical role in sulfite activation. Figure 2 demonstrates that the degradation efficiency of RhB improved as the $FeSO_4$ concentration increased, reaching a maximum of 90.3% at an $FeSO_4$ concentration of 5 mM within 120 min. At 3 mM $FeSO_4$, only 40.6% degradation was observed, reflecting insufficient radical generation due to lower catalyst availability. Further increases to 7 mM and 10 mM resulted in decreased efficiency (62.9% and 51.2%, respectively), likely due to radical scavenging by excess Fe^{2+} ions and precipitation of $Fe(OH)_3$ under near-neutral conditions. These results indicate that 5 mM $FeSO_4$ is the optimal catalyst concentration for the $FeSO_4/Na_2SO_3$/oxalic acid system.

5.3. Effect of Na_2SO_3 dosage

Sodium sulfite serves as the activator and radical precursor in this system. As illustrated in Fig. 3, degradation efficiency improved with increasing Na_2SO_3 dosage up to 7 mM, achieving 90.3% removal of RhB. At lower concentrations (3–5 mM), insufficient sulfite availability limited radical production. Conversely, excess Na_2SO_3 (10 mM) reduced degradation efficiency (72.5%) due to scavenging of generated radicals by surplus sulfite ($SO_3^{2-} + \bullet OH \rightarrow SO_3^{\bullet-} + OH^-$). Thus, 7 mM was identified as the optimum Na_2SO_3 concentration.

5.4. Effect of oxalic acid dosage

Oxalic acid enhances Fe^{2+} availability by stabilizing iron through chelation and promoting Fe^{3+} reduction. As shown in Fig. 4, the addition of oxalic acid significantly enhanced RhB degradation, with maximum efficiency (90.3%) achieved at 3 mM. At higher concentrations (5–10 mM), degradation efficiency decreased slightly, which may be attributed to oxalate ions competing with RhB molecules for available radicals and little absorption. The results confirm the dual role of oxalic acid as both a stabilizing agent for iron and a co-catalyst in sustaining radical generation.

To assess the influence of substrate loading, the initial RhB concentration was adjusted between 5 and 40 mg/L, and the corresponding results are shown in Fig. 5. The system exhibited maximum degradation efficiency (90.3%) at 5 mg/L. As the concentration increased, degradation efficiency decreased significantly (e.g., 61.2% at 20 mg/L and 39.8% at 40 mg/L). This behavior is related to the fixed concentration of radicals being insufficient to completely oxidize the higher pollutant load, as well as inner filter effects reducing light penetration and radical interactions at elevated dye concentrations.

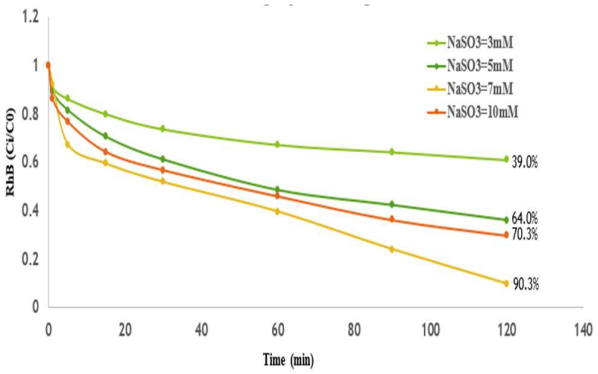

Figure 3. Effect of Na_2SO_3 concentration on RhB degradation efficiency.

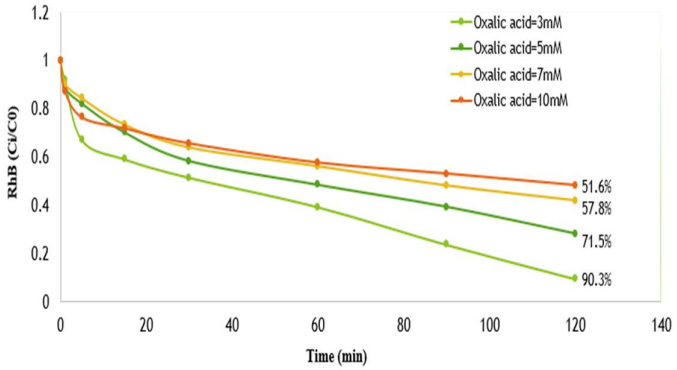

Figure 4. Effect of oxalic acid concentration on RhB degradation efficiency.

5.5. Effect of pH

pH of solutions has a great impact on the speciation of Fe and the production of radicals. As shown in Fig. 6, the decolorization of the $FeSO_4/Na_2SO_3$/oxalic acid system was the best at near neutral pH (5.9) with the value of 90.3%. Degradation was less (65.4%) at pH 3 under acidic conditions because of partial activation of sulfite. At alkaline pH (7–11), the efficiency were dramatically decreased (52.1% at pH 7 and 28.3% at pH 11) because of the fact that precipitation dFe 3+ as ferric hydroxide and the instability of sulfite in high pH environment. These findings indicate that the system has a good operating efficiency under near neutral pH conditions, which is desired for application since standard Fenton process requires strong acidic media.

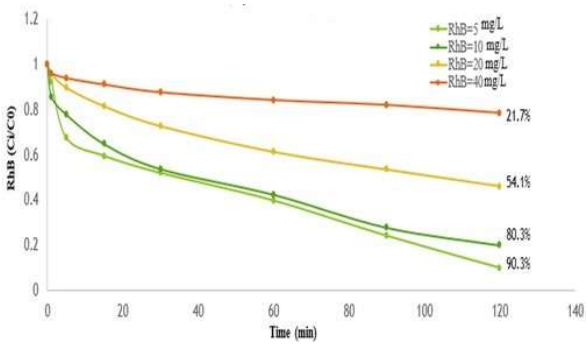

Figure 5. Effect of initial RhB concentration on degradation efficiency.

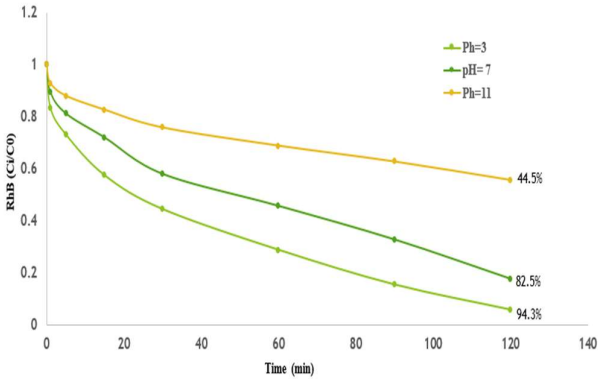

Figure 6. Effect of solution pH on RhB degradation efficiency.

5.6. Proposed degradation mechanism

A plausible mechanism for RhB degradation was developed based on the experimental results and relevant literature, as illustrated in Fig. 7. In the presence of Fe^{2+}, sulfite undergoes activation to generate reactive radicals such as $SO_4^{\bullet-}$, $SO_3^{\bullet-}$, and •OH. Oxalic acid stabilizes Fe^{2+}/Fe^{3+} through chelation, preventing precipitation and promoting redox cycling. The generated radicals attack the conjugated chromophoric structure of RhB, leading to de-ethylation, cleavage of aromatic rings, and eventual mineralization into CO_2, H_2O, and SO_4^{2-}.

$$Fe^{2+} + SO_3^{2-} \rightarrow Fe^{3+} + SO_3^{\bullet-}$$

$$SO_3^{\bullet-} + O_2 \rightarrow SO_5^{\bullet-}$$

$$SO_5^{\bullet-} \rightarrow SO_4^{\bullet-} + \bullet OH$$

This synergistic process ensures sustained radical production and efficient RhB degradation under mild conditions.

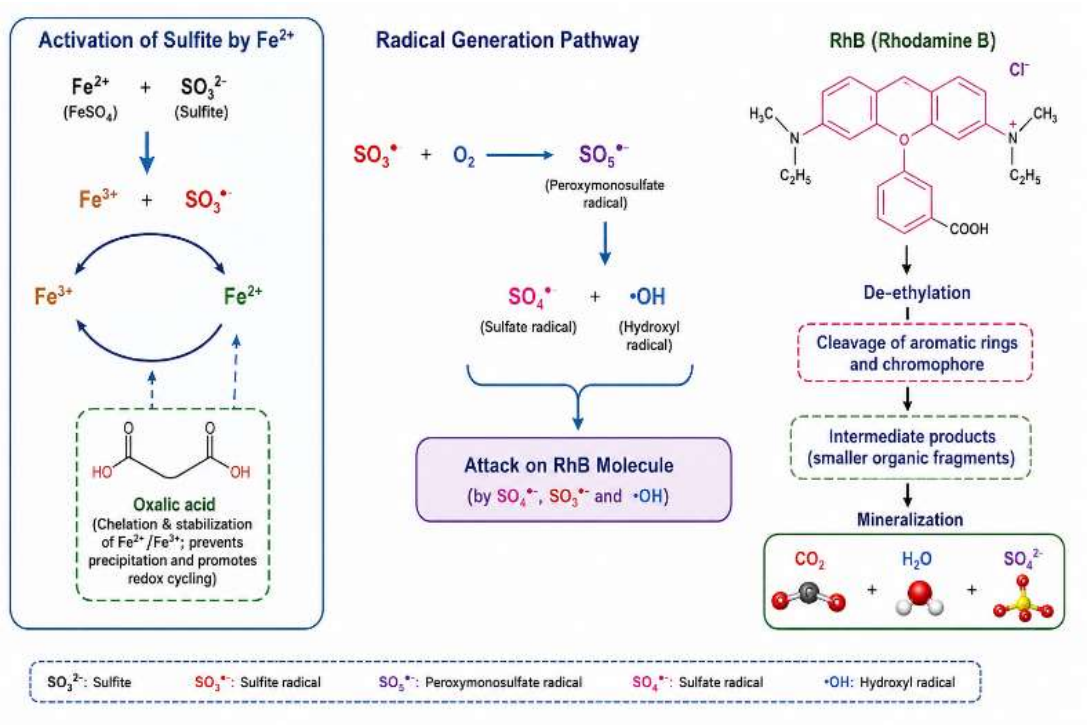

Figure 7. Proposed mechanistic pathway of RhB degradation in $FeSO_4/Na_2SO_3$/oxalic acid system.

5.7. Comparative analysis with literature

The current system exhibited excellent performance with more than 90% degradation at neutral pH in contrast to the conventional Fe(II)/sulfite system. Chen et al. (2018) found only 72% rhodamine B (RhB) degradation in Fe(II)/sulfite systems without addition of oxalic acid, while Zhang et al. (2018) found superior performance under UV-assisted conditions. The presence of oxalic acid in the present investigation was critical for stabilizing Fe ions and for remaining radical production, so external energy was not necessary. These results demonstrate that from a practical point of view, the $FeSO_4/Na_2SO_3$/oxalic acid system is a cheap and green process which can be possibly applied to treat real wastewater.

The fouling operational parameters and the optimal conditions for Rhodamine B degradation found are summarized in Table 1. The findings indicated that the maximum degradation (%) was attained under the concentration of 5 mM $FeSO_4$, 7 mM Na_2SO_3, 3 mM oxalic acid, an initial RhB concentration of 5 mg/L and pH 5.9 at 120 min of reaction and it was 90.3%.

Table 1. Summary of the investigated operational parameters and optimum conditions for Rhodamine B degradation using the $FeSO_4/Na_2SO_3$/oxalic acid system.

Parameter	Range Investigated	Optimum Value
$FeSO_4$ concentration	3–10 mM	5 mM
Na_2SO_3 concentration	3–10 mM	7 mM
Oxalic acid concentration	3–10 mM	3 mM
Initial RhB concentration	5–40 mg/L	5 mg/L
Initial pH	3–11	5.9
Maximum RhB degradation	90.3%	(120 min)

Conclusion

In this work, the $FeSO_4/Na_2SO_3$/oxalic acid system was proven as an efficient AOP to degrade Rhodamine B (RhB) in aqueous solution. The influence of the process parameters on degradation efficiency was examined systematically with regard to $FeSO_4$, Na_2SO_3, and oxalic acid dosages. The best conditions were $FeSO_4$ = 5 mM, Na_2SO_3 = 7 mM, oxalic acid = 3 mM, RhB = 5 mg/L, and pH ≈ 5.9, at which 90.3% RhB removal was obtained in 120 min. Optimal concentrations of activator or catalyst are required, and excessive activator or catalyst concentrations are deleterious to treatment efficiency because available radicals are scavenged by them, and the excessive dye loading also adversely affects the degradation efficiency due to limited radical supply. Importantly, the system exhibited excellent activity at near-neutral pH, overcoming the key drawback of traditional Fenton-based systems that need very acidic environment.

Mechanistic investigation revealed that Fe^{2+}-activated sulfite produces a series of reactive species, such as $SO_3^{\bullet-}$, $SO_5^{\bullet-}$, $SO_4^{\bullet-}$, and •OH radicals, which combinedly attack the chromophoric structure of RhB. Oxalic acid had a twofold effect: it protected Fe ions from precipitation as well as after the conversion of Fe^{2+} to Fe^{3+}, it aided in the reduction of Fe^{3+} to Fe^{2+}, thus maintaining the redox cycling and the continuous generation of radicals. The combination of all these compounds resulted in the efficient removal of RhB de-ethylation, breaking aromatic rings, then mineralizing RhB into CO_2, H_2O and sulfate ions.

In comparison with previously reported Fe(II)/sulfite systems, which achieved only moderate efficiency (~70%) or required UV assistance, the present work highlights the significance of incorporating oxalic acid as a redox-mediating and chelating agent. The ability to achieve >90% RhB degradation under ambient conditions without external energy input underscores the cost-effectiveness and environmental compatibility of this process. This is particularly relevant for textile-producing countries such as Pakistan, where untreated dye effluents pose major ecological and health challenges.

Future work should focus on expanding this approach to real textile wastewater containing mixed dyes and co-contaminants to evaluate system robustness (Naseer et al., 2020). Additional studies on by-product identification and toxicity assessment will further ensure environmental safety (Fu et al., 2011; Kavitha et al., 2013). Furthermore, integrating this process with renewable energy-driven systems (e.g., solar-assisted activation) and scaling up to continuous flow reactors will provide valuable insights for industrial applications (Ratshiedana et al., 2024).

Author Contributions

Asghar Abbas: Writing-Original Draft, Methodology, Investigation. **Ahmad Faraz Ali:** Validation. **Maria Sophy:** Data Curation. **Meesam Ali:** Supervision, Writing-Review & Editing **Usman Saeed:** Project Administration.

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Conflicts of Interest Statement

The authors declare no competing financial or non-financial interests, nor any personal relationships, that could influence the work reported herein.

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