

Removal of Organic Contaminant from Wastewater by Applying Fenton Oxidation

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ABSTRACT

This study investigates the effectiveness of Fenton oxidation in treating diethylamine (DEA) contamination in a batch reactor, emphasizing the optimization of key operational parameters. Factors such as reaction time, pH, ferrous ion concentration, and oxidant dosage were systematically analyzed to maximize performance. DEA degradation efficiency significantly improved with extended reaction times (30 min–3 h). Optimal conditions were established at pH 3.0, Fe²⁺ concentrations of 0.77–3.1 g/l, and oxidant dosage of 0.78 g/l. Hydrogen peroxide demonstrated superior performance under these parameters, particularly at 1.5 g/l Fe²⁺. The process achieved a maximum DEA removal efficiency of 83.7%, assessed through chemical oxygen demand (COD) reduction, while minimizing ammonia production (9.5%) and other by-product formation. These findings highlight Fenton oxidation as a promising and sustainable approach for addressing diethylamine pollution in industrial wastewater.

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1. INTRODUCTION

Global economic growth has been accelerating exponentially in the 21st century. However, this rapid expansion, coupled with urbanization and industrialization, has resulted in the release of substantial volumes of wastewater, adversely impacting human health and the environment. Effective mitigation of organic wastewater impacts requires integrated treatment solutions. Regulatory authorities have established stringent disposal frameworks for organic effluents, necessitating rigorous operational protocols to ensure compliance and avoid penalties (Ilhan *et al.*, 2019)(Güneş, Çifçi and Çelik, 2018)(Ebrahiem, Al-Maghrabi and Mobarki, 2017)(Bianco, De Michelis and Vegliò, 2011).

Traditional wastewater treatment methods, including biological processes, adsorption, chemical treatments, filtration, flocculation, activated carbon, and ion exchange resins, are widely used. However, pollutants that resist biological treatments often exhibit high chemical stability and are challenging to mineralize completely. In this scenario, oxidation processes have emerged as preferred solutions for degrading such bio-refractory substances. Nonetheless, the effectiveness of these processes depends on factors such as pollutant load, process constraints, and operating conditions, which must be carefully evaluated to select the most suitable oxidation method for specific compounds. Although direct oxidation processes can achieve high degradation efficiency, they require precise operating conditions, which can significantly increase operational costs(Papić *et al.*, 2009)(Ahmed *et al.*, 2011).

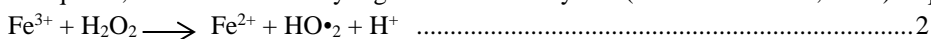
Advanced Oxidation Processes (AOPs), as defined by Glaze *et al.* (1987), are water treatment methods that utilize highly reactive radicals to degrade pollutants under near-ambient temperature and pressure. Hydroxyl radical production in these methods typically relies on oxidizing agents such as ozone and hydrogen peroxide (H₂O₂), which are activated through light exposure, catalytic materials (including Fe²⁺, Fe³⁺, TiO₂), ultrasonic energy, or heat. Other AOP systems, including Fenton, photo-Fenton, UV-peroxidation, and O₃/H₂O₂ systems, have gained greater favor due to their energy efficiency alongside wastewater treatment effectiveness. Fenton and photo-Fenton processes have shown particular success in the degradation of stable pollutants with the benefits of economic viability, reduced energy demands, and environmental amenability. These systems use simple equipment designs, utilize low-hazardous chemicals, and operate in recycles that reduce overall chemical consumption at the same time yielding excellent treatment performance(Nidheesh, 2015)(Chu *et al.*, 2012). A review of literature confirms their extensive use across numerous industries(Bianco, De Michelis and Vegliò, 2011).

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The Fenton reaction is the combined action of hydrogen peroxide (H_2O_2) and ferrous ions (Fe^{2+}) that has been famously established for the oxidation of numerous poisonous organic pollutants in aquatic systems. This is the process of oxidation that was discovered in 1894 by Henry John Horstman Fenton when, in the presence of the solution, he observed the oxidation of tartaric acid by hydrogen peroxide (Zazouli *et al.*, 2012)(da Silva *et al.*, 2015). This was the beginning of the large-scale application of the system of the H_2O_2 -iron for the effective oxidation of numerous organic pollutants. Haber and Weiss proposed that the activation of H_2O_2 by ferrous salts produces highly reactive hydroxyl radicals ($\text{OH}\cdot$)(Mehmet A. and Jean-Jacques, 2014). Equation 1 shows the reaction.



Studies have confirmed the formation of hydroxyl radicals ($\text{OH}\cdot$) in Fenton's reagent solutions through chemical scavengers and spectroscopic techniques. Extensive research and reviews have documented the practical applications of the Fenton process. Pulse radiolysis has provided insights into the reaction rate constants. The Fenton process is highly effective at removing water pollutants under acidic conditions (around pH 3.0), leveraging the catalytic properties of the $\text{Fe}^{3+}/\text{Fe}^{2+}$ couple. Only a minimal amount of Fe^{2+} is required, as it is continuously regenerated in the system (Rush and Bielski, 1985). Equation 2, 3 represent the chemical reactions.



Hydroperoxyl radicals ($\text{HO}_2\cdot$) exhibit lower reactivity than hydroxyl radicals ($\cdot\text{OH}$). Fenton process efficiency depends on multiple parameters: pH, temperature, and concentrations of target pollutant, H_2O_2 , and Fe^{2+} .

Diethylamine is a colorless chemical liquid with odor like ammonia, though commercially it may appear brownish due to impurities. Classified as a flammable and weakly alkaline liquid, it is used in various industries. One important application is as a corrosion inhibitor, protecting materials during manufacturing processes. Additionally, diethylamine acts as a building block in the production of rubber, pharmaceuticals, resins, pesticides, and dyes. However, due to its chemical properties, diethylamine can pose threats to both human health and the environment. Inhalation or contact with the liquid can irritate the skin, eyes, and respiratory system, and in severe cases, lead to fluid buildup in the lungs. Chronic exposure can cause lung damage and irritate the teeth. While the environmental impact of diethylamine is less documented, spills can contaminate soil and water sources. Therefore, proper handling and disposal are crucial to minimize these risks (WEXLER, 2014).

This research is dedicated to understanding how different operational parameters influence the effectiveness of Fenton's process in treating nitrogen-rich wastes and their associated by-products. The study closely analyzes factors such as the duration of treatment, the levels of oxidant and Fe^{2+} , and the pH conditions. The experimental work particularly targets the degradation of diethylamine, a nitrogen-containing compound, using Fenton's reagent.

2. MATERIALS AND PROCEDURE

2.1 Chemical Materials

Diethylamine ($\text{C}_4\text{H}_{11}\text{N}$) molecular weight= 73.14 g/mol, density = 0.707 g/ml is a colorless liquid at ambient temperature. H_2O_2 was used as an oxidant. $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ was used in crystalline form, while H_2SO_4 served as the pH adjustment agent. NaOH, typically available as a powder, was used to adjust the reaction conditions.

2.2 Laboratory Method

All experiments were performed in a 2-L beaker serving as a batch reactor, equipped with magnetic stirring and heating. The procedure commenced by introducing a diethylamine (DEA) solution into the reactor, followed by ferrous sulfate heptahydrate addition. The solution pH was adjusted to 3.0 using H_2SO_4 . The stirring speed was initially set to 60 r.p.m to ensure thorough mixing. Hydrogen peroxide, serving as the oxidant, was then introduced into the reactor. Reaction times ranged from 30 min to 3 hours, with samples collected at 1-hour and 3h intervals to track reaction progress.

This work carried out a systematic investigation of fundamental operating parameters, with levels of oxidant concentration set based on the initial baseline chemical oxygen demand (COD) values. All the experiments were well-performed under conditions of control with the experimental temperature of 25°C. Assessment of performance included COD removal rate along with the measurement of level of pH as well as concentration of nitrogen-containing species such as ammonia, nitrate, and nitrite, using appropriate analytical equipment. This approach allowed for the comprehensive assessment of the effectiveness of Fenton oxidation in DEA degradation processes.

3. RESULTS AND DISCUSSION

3.1 Impact of Time

Reaction duration plays an significant role in Fenton oxidation effectiveness, as demonstrated in previous studies (Ahmadian *et al.*, 2013), (Mirzaei *et al.*, 2017). This investigation steadily analyzed how reaction time influences conversion performance across time periods spanning 30 min to 3 h. COD elimination efficiency exhibited a consistent upward pattern, progressing from 52.0% after 30 min to 78.0% following 3 h of treatment, establishing a direct relationship between extended reaction periods and enhanced COD

reduction. Figure 1 presents this correlation, displaying removal performance as it relates to treatment duration under standard laboratory temperature conditions.

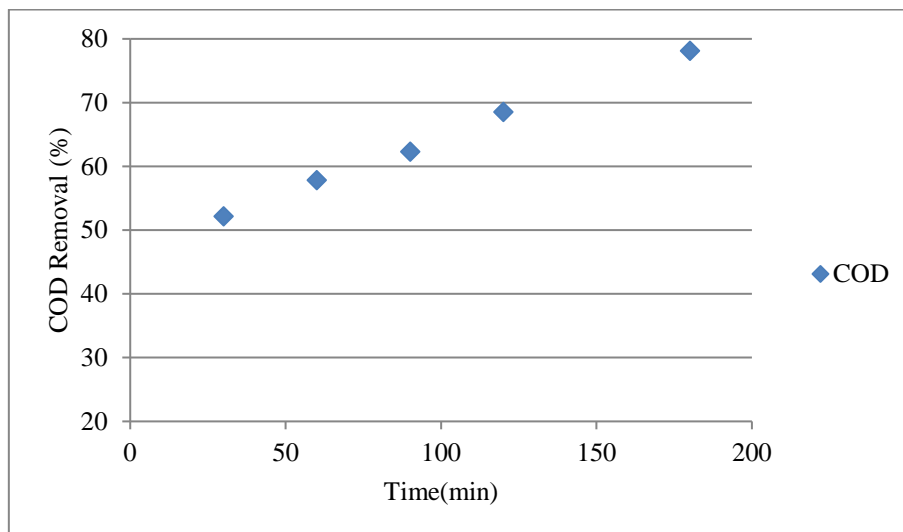


Fig. 1. COD removal efficiency as a function of reaction time under constant conditions: pH 3.0, [H₂O₂] = 1.5 g/l, and [Fe²⁺] = 1.5 g/l.

Concurrently, reaction time demonstrated a pronounced effect on ammonia yield. Prolonged reaction times resulted in a marked decrease in ammonia production, with yields dropping from 76% at 30 min to 19% at 3 hours. These findings suggest that extended reaction times facilitate not only the degradation of diethylamine but also the mitigation of ammonia by-product formation. However, it is noteworthy that while this investigation identified a strong influence of reaction time, some studies have reported minimal or negligible effects, highlighting the potential variability in outcomes based on specific experimental conditions.

Figure 2 depicts temporal variations in ammonia yield, confirming its status as the predominant by-product. Other reaction by-products remained undetectable, with concentrations below the multi-photometer's quantification limits. The Fenton process was conducted under controlled conditions, including a pH of 3, ambient laboratory temperature, and dosages of 1.5 g/l each for ferrous ions and the oxidant. Adjustments to these experimental parameters could enhance the removal efficiency while further reducing ammonia yield.

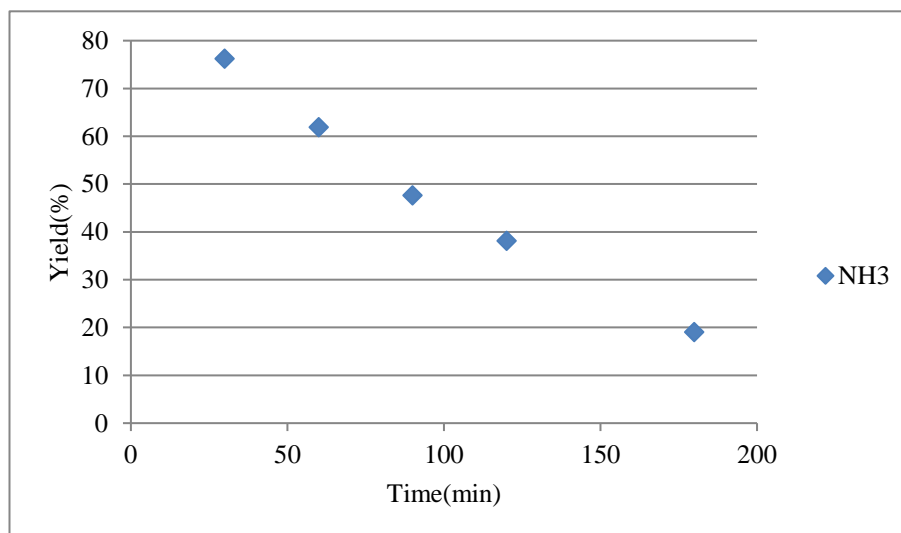


Fig. 2. Ammonia yield as a function of reaction time under constant conditions: pH 3.0, [H₂O₂] = 1.5 g/l, and [Fe²⁺] = 1.5 g/l

3.2 Impact of pH

pH is a critical operational parameter that profoundly influences the reaction between Fe²⁺ and the oxidant. A defined pH range is essential for efficient free radical generation, necessitating strict pH control to maximize process performance (Mirzaei *et al.*, 2017). Consequently, the pH-dependent Fenton process remains integral to numerous applications. (Mohajeri *et al.*, 2010). The Fenton reaction is most effective in an acidic environment, where an increase in pH leads to a reduction in radical concentration. The Fenton oxidation process exhibits pH-dependent hydroxyl radical (•OH) generation, which governs its reaction efficiency. As the pH level

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increases, the reactivity of hydroxyl radicals ($\text{HO}\cdot$) weakens, making the process of Fenton oxidation less effective in alkaline conditions. This method performs optimally in acidic environments, where the oxidation rate is significantly higher. Consequently, maintaining the pH within the range of 2.0 to 6.0 is crucial for maximizing the efficiency of Fenton oxidation (Tercero *et al.*, 2020). Deviations from the optimal pH range adversely affect the efficiency of the Fenton oxidation. At pH levels below 2, the generation of free radicals is inhibited, while at pH levels above 4.5, the efficacy of Fenton oxidation is markedly diminished.

Consequently, extreme pH values reduce the removal efficiency of organic waste and by-products, emphasizing the critical need for pH optimization to maximize free radical generation.

Literature commonly cites a pH range of 2–4 for effective organic waste removal. However, this study investigated an expanded range of 2–6. The results indicated that the COD removal efficiency was highest at pH 3, but gradually declined with increasing pH levels, specifically at 4, 5, and 6. The lowest efficiency, recorded at a pH of 6 with a residence time of 3 hours, was 60.5%. A negative relationship was observed between removal efficiency and the acidity of the medium. Figure 3 illustrates how COD removal efficiency varied with increasing pH levels.

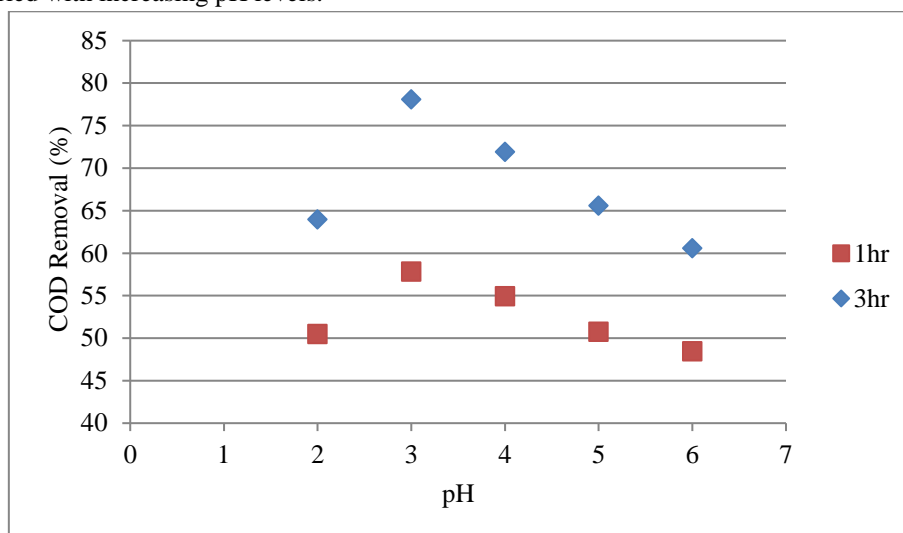


Fig. 3. COD removal efficiency as a function of pH under fixed conditions: $[\text{H}_2\text{O}_2] = 1.5 \text{ g/l}$ and $[\text{Fe}^{2+}] = 1.5 \text{ g/l}$.

Investigating the effect of pH on ammonia, it was observed that its yield decreased at pH 3, but then increased at pH levels of 4, 5, and 6, peaking at 61.9% at pH 6. The findings confirmed that higher pH values had a diminishing impact on ammonia yield. Although nitrate and nitrite were analysed as potential by-products, their minimal yields rendered them negligible and excluded from further analysis. Figure 4 illustrates how pH influences ammonia yield, showing that both low and high pH values do not positively affect ammonia production. The minimum ammonia yield was recorded at the optimal pH value (Ahmadian *et al.*, 2013).

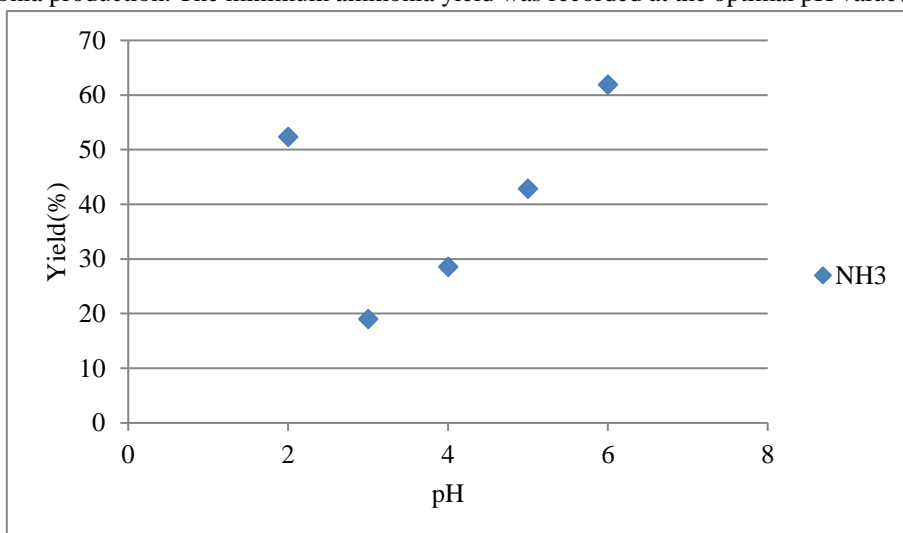


Fig. 4. Ammonia yield as a function of pH under fixed conditions: $[\text{H}_2\text{O}_2] = 1.5 \text{ g/l}$ and $[\text{Fe}^{2+}] = 1.5 \text{ g/l}$.

3.3 Impact of Fe^{2+}

The concentration of Fe^{2+} is an important parameter influencing the efficiency of the Fenton reaction and the generation of hydroxyl radicals ($\cdot\text{OH}$) (Miklos *et al.*, 2018). Elevated concentrations of Fe^{2+} can lead to the premature consumption of hydroxyl radicals,

thereby impeding the complete degradation of organic pollutants. Conversely, insufficient Fe^{2+} concentrations result in a reduced production of hydroxyl radicals. In the Fenton process, Fe^{2+} acts as a catalyst, promoting the decomposition of hydrogen peroxide into highly reactive radicals that drive the oxidation of contaminants. Along with water, these reactive radicals are essential in decomposing organic waste into various organic and inorganic components. The Fenton process has both benefits and drawbacks. Its significant advantages—including high treatment efficiency and operational simplicity—enable enhanced organic waste biodegradability and expanded wastewater treatment applications. Nevertheless, issues of sludge generation, the acidic reaction medium, and the need for an effluent neutralization reactor pose challenges that need to be solved (Domingues *et al.*, 2018).

Figure 5 demonstrates the dose-dependent relationship between Fe^{2+} concentration and COD removal efficiency. Results indicate a significant positive correlation, with peak performance observed at the optimal Fe^{2+} dosage (Ahmadian *et al.*, 2013). A maximum removal of COD of 78.0% was observed at a Fe^{2+} concentration of 1.5 g/l, 3 h reaction time, and pH of 3. The impact of Fe^{2+} diminished at concentrations of 0.77 g/l and 3.1 g/l, and the time of reaction demonstrated a significant positive effect on the conversion rate. Under optimal conditions and at 1 h, the COD removal rate was 57.8%.

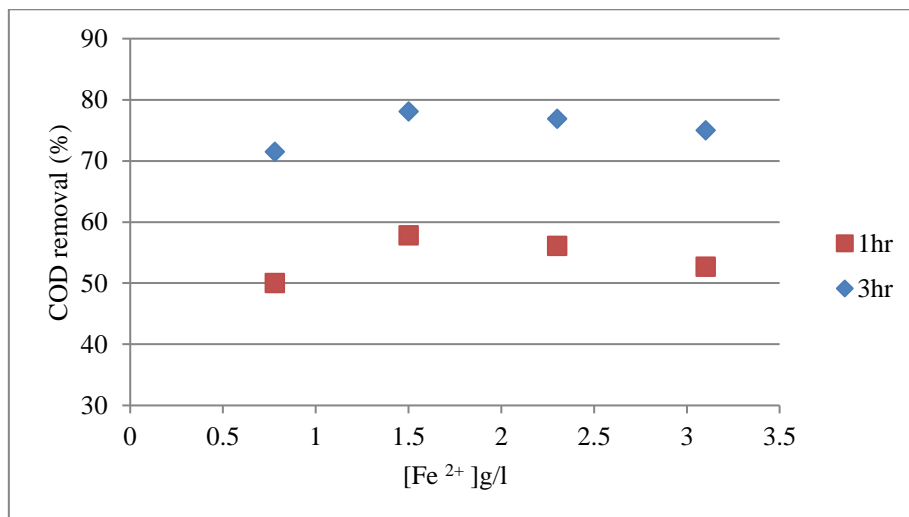


Fig. 5. COD removal efficiency as a function of Fe^{2+} concentration under fixed conditions: $[\text{H}_2\text{O}_2] = 1.5 \text{ g/l}$ and $\text{pH} 3.0$.

Figure 6 reveals ferrous ion-dependent changes in ammonia yield. Previous studies established that excessive free radicals suppress ammonia generation (Pani *et al.*, 2020)(Das and Adak, 2022). In this investigation, Fenton treatment achieved measurable ammonia yield decrease at 1.5 g/l ferrous ion dosage. Varying ferrous ion concentrations, whether low or high, had little to no effect on ammonia yield. Both increasing and decreasing the concentration of ferrous ion in the Fenton process can affect the rate of reaction. While an optimal increase can enhance the degradation of pollutants, too high a concentration can lead to scavenging of hydroxyl radicals, reducing the process efficiency. Conversely, too low a concentration of ferrous ion results in insufficient hydroxyl radical production, slowing down the reaction rate. Maximum ammonia yield (42.8%) was achieved under optimized conditions: pH 3.0, H_2O_2 concentration of 1.5 g/L, and Fe^{2+} concentration of 0.77 g/l. This minimum yield of ammonia was achieved under the same pH and H_2O_2 conditions but with a Fe^{2+} concentration of 1.5 g/l. Contrary to expectations, elevated Fe^{2+} concentrations did not significantly reduce ammonia yield, while nitrate (NO_3^-) and nitrite (NO_2^-) remained undetectable throughout the experiments.

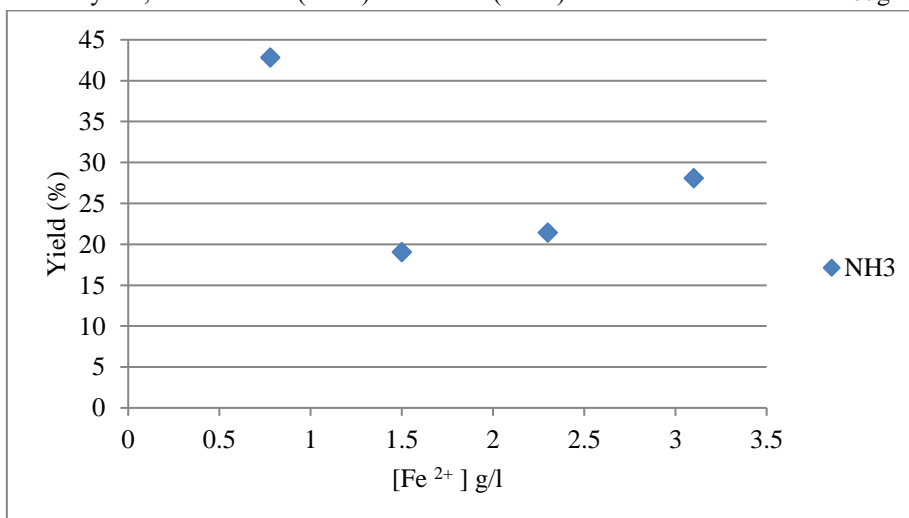


Fig. 6. Effect of Fe^{2+} concentration on ammonia yield under constant conditions: $\text{pH} 3.0$ and $[\text{H}_2\text{O}_2] = 1.5 \text{ g/l}$.

3.4 Impact of Oxidant (H₂O₂)

The Fenton reaction utilizes hydrogen peroxide (H₂O₂) as its main source of radical free oxygen species, also serving as a potent oxidizing agent, making its role crucial to the reaction mechanism. Such reactive species can only be formed in the presence of Fenton's reagent which is constituted of H₂O₂ and a metal catalyst such as iron. An acid medium aids in the cleavage of H₂O₂, which results in the generation of potent hydroxyl HO• radicals which further oxidize (Pani *et al.*, 2020)(Naseem *et al.*, 2019). As with any other oxidant, the efficiency of H₂O₂ is pH dependent, and therefore, its oxidizing power is lower in very acidic and very alkaline pH regions. In addition, with an increase in the concentration of H₂O₂, the rate of oxidation also increases, resulting in a more effective breakdown of pollutants. The Fenton process is dependent on the bottom-up formation of a number of intermediate species and requires the oxidant to be in constant supply in order to effectively and fully eliminate the organic pollutants within the system. This work systematically evaluated the influence of oxidant concentration on COD removal rate and by-product yield. The findings highlight the critical role of radical generation in enhancing the conversion rate. Laboratory experiments were conducted under controlled conditions, maintaining a pH of 3, a Fe²⁺ concentration of 1.5 g/l, and varying reaction times. Hydrogen peroxide dosages between 0.77 g/l and 3 g/l were tested, revealing an improvement in process efficiency up to a certain threshold, beyond which diminishing returns were observed. The maximum COD removal efficiency of 83.7% was achieved at an oxidant concentration of 3 g/l and a residence time of 3 hours. Notably, at a shorter reaction time of 1 hour, the process demonstrated a COD removal efficiency of 63.0% under the same conditions. Extending the reaction duration from 1 to 3 hours markedly boosted DEA removal efficiency by enhancing the conversion rate. Figure 7 illustrates the correlation between oxidant concentration and COD removal efficiency.

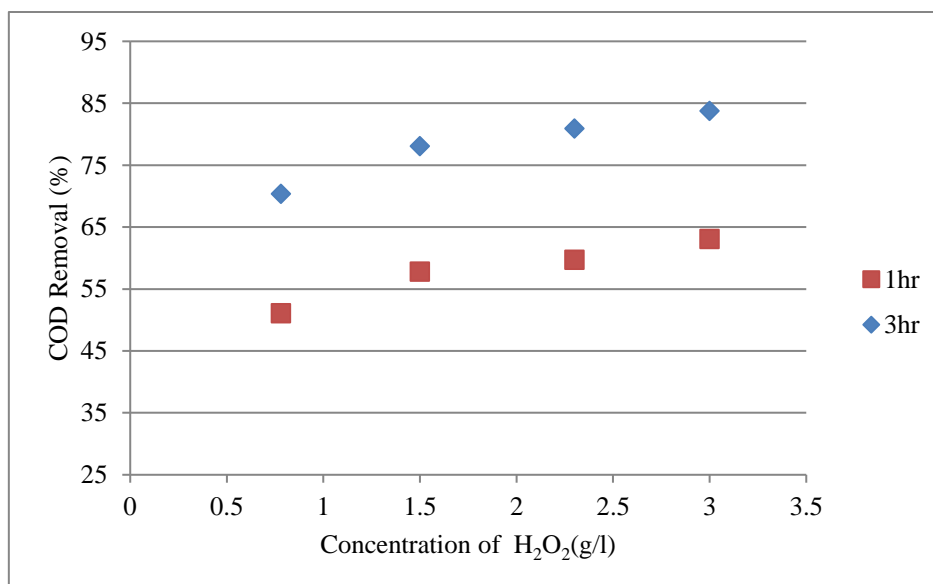


Fig. 7. COD removal rate as a function of oxidant concentration under fixed conditions: pH 3.0 and [Fe²⁺] = 1.5 g/l.

Ammonia was identified as the principal by-product of DEA oxidation, and its formation was significantly influenced by the oxidant concentration. An increase in hydrogen peroxide concentration resulted in a notable reduction in ammonia yield. This trend was attributable to augmented hydroxyl radical (•OH) generation at elevated H₂O₂ concentrations, which enhanced ammonia removal and COD reduction efficiencies. However, at a hydrogen peroxide dosage of 3 g/l, COD removal efficiency did not show a significant improvement, indicating that variations in oxidant concentrations within the range of 0.77 g/l to 3 g/l had a limited impact on the overall COD removal rate. Figure 8 depicts the inverse relationship between oxidant concentration and ammonia yield under fixed conditions (1.5 g/L Fe²⁺, pH 3). Results show ammonia yield decreased with rising oxidant levels, reaching a minimum of 9.5% at 3 g/L after 3 h reaction. Additionally, while nitrite and nitrate ions were investigated, their concentrations were negligible and thus excluded from detailed analysis.

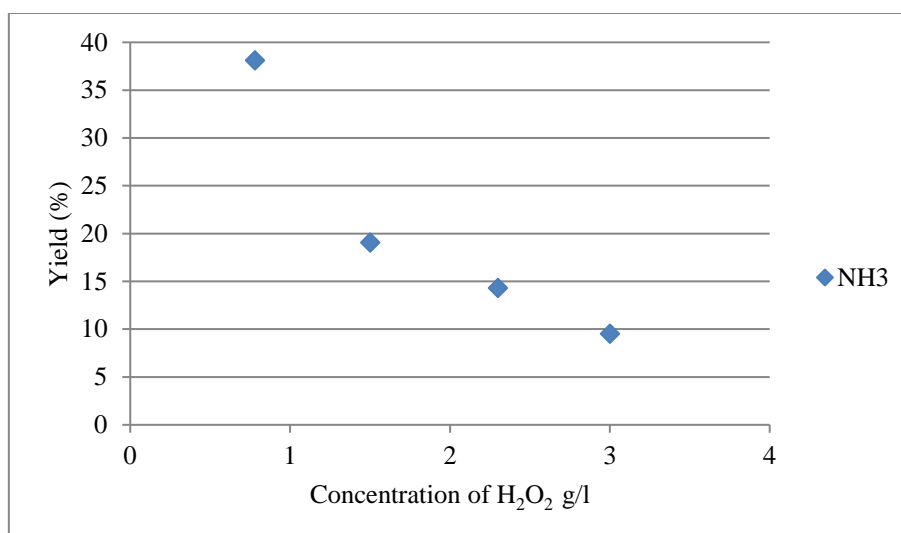


Fig. 8. Effect of oxidant concentration on ammonia yield under fixed conditions: pH 3.0 and Fe²⁺ concentration of 1.5 g/L.

4. CONCLUSION

The DEA oxidation process was effectively carried out using Fenton's reagent, demonstrating its efficiency in improving COD removal under diverse experimental conditions. Key operational parameters demonstrated significant influence on process performance metrics, as confirmed by experimental results. Fenton oxidation proved to be a robust method for degrading organic compounds into simpler substances. Optimal conditions—an oxidant concentration of 3 g/l, pH of 3, reaction time of 3 hours, and Fe²⁺ concentration of 1.5 g/l achieved a maximum COD removal efficiency of 83.7% with a minimal ammonia yield of 9.5%. Higher Fe²⁺ concentrations beyond 1.5 g/l did not yield notable improvements in COD removal, suggesting a threshold effect. Additionally, nitrite (NO₂⁻) and nitrate (NO₃⁻) concentrations remained below quantifiable levels and were thus omitted from quantitative analysis. These findings highlight the potential of Fenton's reagent as a viable and efficient option for wastewater pre-treatment applications.

COMPETING INTERESTS

The author has declared that no competing interests exist.

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