Optimization of H_2O_2/COD and Fe^{3+}/H_2O_2 Ratios in Combined Electrocoagulation-Fenton System for Petrochemical Wastewater Treatment

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ABSTRACT

Petrochemical wastewater is characterized by a high organic load and complex composition, which can limit the effectiveness of conventional biological treatment and necessitate advanced treatment technologies. This study evaluated an integrated electrocoagulation–Fenton process for petrochemical wastewater treatment, focusing on optimizing the H_2O_2/COD and Fe^{3+}/H_2O_2 ratios and assessing their effects on COD degradation kinetics. Electrocoagulation was initially conducted under previously optimized conditions of 5 V, initial pH 7, and 60 min, followed by Fenton oxidation at pH 3. The H_2O_2/COD ratio was varied at 5, 10, 15, and 20 g/g, while the Fe^{3+}/H_2O_2 ratio was adjusted to 0, 0.013, 0.02, and 0.04 g/g. The optimum H_2O_2/COD ratio of 15 g/g achieved a COD removal efficiency of 48.14%. Further optimization identified an Fe^{3+}/H_2O_2 ratio of 0.02 g/g as the optimal condition, yielding the highest COD removal efficiency of 88.75%. The achieved removal efficiency is comparable to the upper end of the range reported for integrated electrocoagulation–Fenton treatment of refractory industrial wastewater. Kinetic analysis showed that both first-order and second-order models adequately described COD degradation; however, the second-order model provided a slightly better fit, with R^2 values of 0.98–0.99. This result suggests that the oxidation process was governed primarily by interactions between oxidizing species and organic pollutants rather than simple first-order degradation. Overall, optimizing oxidant and catalyst dosages substantially improved the integrated electrocoagulation–Fenton process, demonstrating its potential as an effective treatment strategy for petrochemical wastewater and providing practical operating conditions for advanced wastewater treatment applications.

1. Introduction

Petrochemical wastewater is characterized by high organic loads, refractory compounds, and toxic constituents that severely limit the effectiveness of conventional biological treatment. Wastewater generated from processes such as desalination, hydrocracking, and hydroskimming typically exhibits elevated chemical oxygen demand (COD), biological oxygen demand (BOD), total suspended solids (TSS), oil and grease, sulfides, and ammonia, all of which pose serious environmental risks if not adequately treated. Although physicochemical methods such as adsorption, ozonation, and

photocatalysis have demonstrated high removal efficiencies, their substantial infrastructure requirements, high operational costs, and limited scalability limit their practical implementation [1].

Therefore, electrocoagulation (EC) has emerged as an attractive alternative due to its operational simplicity, lower sludge production, and proven effectiveness in removing a wide range of petrochemical contaminants, including phenols, oils, and petroleum hydrocarbons. However, EC alone is often insufficient to achieve deep oxidation of soluble and refractory organic compounds, leaving residual COD that remains difficult to biodegrade. In this context, advanced oxidation processes, particularly Fenton oxidation, offer a powerful complement by generating highly reactive hydroxyl radicals capable of mineralizing persistent organic matter [1]. Fenton-based AOP approaches are frequently considered, either as a main process or as a pretreatment to reduce the organic load and improve biodegradability before subsequent biological stages [2], [3]. Several previous studies on petrochemical wastewater also show that the application of Fenton, including electro-Fenton, can improve pollutant removal performance through proper control of the catalytic system and operating conditions [4], [5].

Despite having a fairly straightforward mechanism, the Fenton process's actual performance depends heavily on how it is operated. Since it directly affects iron speciation and the stability of hydrogen peroxide, both of which regulate the rate at which hydroxyl radicals form, initial pH is generally acknowledged as the most significant of the different parameters [6]. The best oxidation efficiency is usually attained in acidic environments, especially at pH values near 3, according to numerous studies on petrochemical wastewater systems [7]. Setting the Fe^{3+} and H_2O_2 concentration ratio is very important for maintaining reaction stability. Lower COD removal efficiency can result if a significant portion of the oxidant is consumed in unproductive pathways if this balance is not properly maintained. In this situation, choosing the right operating parameters becomes crucial from the standpoint of performance as well as reagent consumption and process economics [3], [7]. Therefore, to ensure that the Fenton process can be applied effectively and practically at the industrial scale, a systematic evaluation of these parameters is required.

Electrocoagulation (EC) has been widely reported as an effective primary treatment for petrochemical and oil refinery wastewater due to its ability to substantially reduce COD and poorly biodegradable organic compounds. El-Ashtoukhy *et al.* [8] demonstrated that a fixed-bed EC reactor with aluminum Raschig rings achieved 100% phenol removal from a real oil refinery wastewater at a current density of 8.59 mA/cm^2 and pH 7, accompanied by significant COD reduction, while exhibiting lower energy and aluminum consumption compared to conventional EC cell designs. High particulate pollutant removal efficiency has been reported; under operating conditions of 21.64 mA/cm^2, 2 g/L NaCl, and 30 min of electrolysis, the maximum turbidity removal efficiency was 97.43% [9]. However, the effectiveness of EC in degrading dissolved and refractory organic compounds remains limited. Recent studies have shown that COD removal by EC typically ranges from moderate to partial. Abuduaini *et al.* [10] at optimal operating conditions (applied voltage 35 V, working time 30 min, electrode spacing 1.2 cm, and stirring speed 249 rpm), a maximum COD reduction of 52.9% was achieved using a solar-powered EC system, leaving a substantial residual organic fraction—a study by Movahhedtaher *et al.* [1] demonstrated that an Al-Al electrode configuration optimized using response surface methodology achieved 98.09% COD removal from petrochemical wastewater within 10 minutes under 5V operating conditions, highlighting the influence of electrode design and operating parameters on EC performance. Then, a study by Setiadi *et al.* [11] revealed that the optimum conditions of a voltage of 5 V and an initial pH of 7 generated COD removal of about 50.22%—a review by Jing *et al.* [12] Further confirmed that EC can consistently deliver 80- 99% COD removal in various industrial wastewaters, while emphasizing its limitations in fully removing poorly biodegradable dissolved organic compounds and the growing need for advanced EC-oxidation hybrid systems.

Although previous studies have demonstrated the effectiveness of EC and Fenton oxidation individually, studies integrating EC pretreatment with systematic optimization of Fenton operating parameters for petrochemical wastewater remain limited. In particular, limited information is available on the combined optimization of H_2O_2/COD and Fe^{3+}/H_2O_2 ratios, and on their influence on the kinetics of COD degradation in a post-EC Fenton process. No previous study has systematically optimized both H_2O_2/COD and Fe^{3+}/H_2O_2 ratios in a post-EC Fenton system for this specific petrochemical wastewater.

Because the optimal conditions in EC have been identified (a voltage of 5 V and an initial pH of 7), the present study focuses on optimizing the subsequent Fenton process by evaluating the effects of the H_2O_2/COD and Fe^{3+}/H_2O_2 ratios. Furthermore, understanding degradation kinetics is important for relating operating parameters to COD degradation behavior and providing useful information for reactor design, reagent requirement estimation, and future process scale-up. Therefore, the current research aims to investigate the effect of H_2O_2/COD and Fe^{3+}/H_2O_2 ratios on COD removal and degradation kinetics during the Fenton process following EC pretreatment. It is hypothesized that an optimum combination of H_2O_2/COD and Fe^{3+}/H_2O_2 ratios will maximize hydroxyl radical generation while minimizing radical scavenging, thereby enhancing COD removal efficiency and accelerating degradation kinetics. The novelty of this study lies in the systematic optimization of both H_2O_2/COD and Fe^{3+}/H_2O_2 ratios in a post-electrocoagulation Fenton process for real petrochemical wastewater, together with the integration of degradation kinetic analysis to provide practical guidance for process optimization and future industrial scale-up.

2. Research Methodology

2.1. Raw Petrochemical Wastewater

Petrochemical wastewater (PW) was obtained from a petrochemical industry located in Cilegon, Indonesia. The characteristics of the raw PW are summarized in Table 1.

Table 1. Raw Petrochemical Wastewater Characteristics [11]

Parameters	Units	Values
Color	Pt-Co	7,478.9
TSS	mg-dry matter/L	480
Dissolved Iron (Fe^{3+})	mg-Fe/L	<0.09
BOD_5	mg-O_2/L	1,706.00
COD	mg-O_2/L	6,884
pH	-	12.25

2.2. Electrocoagulation Process

The EC was conducted as the first step to reduce the pollutant load in raw petrochemical wastewater before further Fenton oxidation. The EC process was conducted under the optimal conditions reported by Setiadi *et al.* [11]. The 1000-mL PW volume was poured into a 1000-mL glass beaker. The electrodes were immersed, with an active electrode dimension of 9.5 cm × 3.1 cm × 3 mm and an inter-electrode distance of 5 cm. The EC process was carried out at an initial pH of 7, a voltage of 5 V, and a stirring speed of 500 rpm for 60 minutes. In this research, the EC process period was set to 60 minutes, based on a previous study by Setiadi *et al.* [11] the COD removal at minute 80 was not significantly different from that at minute 60. After the EC process, the sludge was separated from the supernatant through sedimentation for 24 hours. Then, the supernatant was collected and analyzed.

In this study, the EC process was performed 4 times, each treating 1000 mL of PW. All EC runs were performed under identical conditions. The supernatant from each EC run (approximately 700 mL per run) was collected and combined, yielding a total supernatant volume of approximately 2800 mL. This combined volume was considered sufficient for the subsequent Fenton process (8 runs). The combined supernatant was homogenized before the Fenton experiments. Therefore, all runs in the Fenton experiments used the same supernatant characteristics. The characteristics of the supernatant are summarized in Table 2.

Table 2. EC-Supernatant Characteristics

Parameter	Unit	Result
Color	Pt-Co	4,383.4
TSS	mg-dry matter/L	340
Dissolved Iron (Fe^{3+})	mg-Fe/L	<0.09
COD	mg-O_2/L	3,410.5
COD removal	%	50.46%

2.3. Experimental Set-up, Design, and Procedures

The experimental arrangement used in this study is illustrated in Fig. 1. Fenton oxidation experiments were performed in a 500 mL glass beaker placed on a magnetic stirrer. For each run, 300 mL of EC-supernatant was transferred into the Fenton reactor, and the initial pH was adjusted to 3.0 by the dropwise addition of concentrated sulfuric acid (98% H_2SO_4). This strongly acidic condition was chosen because it provides the most favorable environment for Fe^{2+}/Fe^{3+} stability and promotes efficient formation of hydroxyl radicals (-OH) during the Fenton reaction, thereby enhancing the oxidation of organic contaminants. All experiments were conducted at ambient temperature and pressure. The temperature was not controlled or monitored during the experiments. The reaction mixture was continuously stirred at 200 rpm to ensure homogeneous mixing throughout the reaction period. The total reaction time was set to 60 minutes. During oxidation, 15 mL of the liquid sample was withdrawn for pH monitoring. Then, the liquid sample was neutralized using NaOH solution to stop the Fenton reaction and transferred into sealed bottles for further COD analysis. In this study, the authors analyzed total COD, so the samples were analyzed directly without filtration using a 0.45 µm membrane. Fig. 1 shows the Fenton experimental setup.

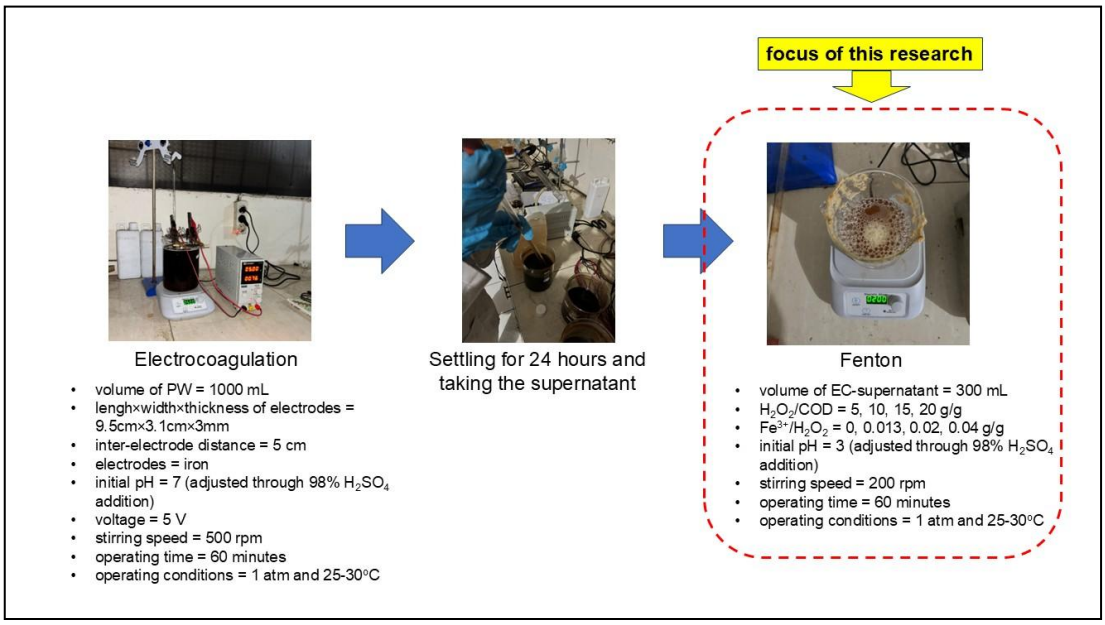

Fig. 1. Experimental set-up for Fenton process

1) Stage 1

In the first experimental stage, hydrogen peroxide (50% H_2O_2) was introduced into the system to get various H_2O_2/COD ratios of 5, 10, 15, and 20 g/g. The moment of H_2O_2 addition was taken as the start of the reaction t = 0 min. The oxidation process was allowed to proceed for 60 min, during which 15 mL liquid samples were taken every 15 min for pH monitoring. Then, the 15-mL samples were analyzed for COD concentrations. Before COD analysis, the collected samples were neutralized to pH 6.5- 7.0 with 5 N NaOH to stop the Fenton reaction. The optimum H_2O_2/COD ratio was selected based on the highest COD removal efficiency achieved.

2) Stage 2

In the second experimental stage, $FeCl_3$ was added to obtain Fe^{3+}/H_2O_2 ratios of 0, 0.013, 0.02, and 0.04 g/g. These ratio values were chosen based on a previous study [13] investigating the Fenton treatment for treating sugarcane vinasse with Fe^{3+}/H_2O_2 ratios of 1:50 – 1:25 g/g (or 0.02 – 0.04 g/g). In this stage, the H_2O_2 was added based on the optimum H_2O_2/COD ratio obtained in the first stage. The experimental procedures were carried out under identical operating conditions, including a reaction time of 60 min and a stirring speed of 200 rpm. During the experiments, 15 mL liquid samples were taken every 15 min for pH monitoring. Then, the 15-mL samples were analyzed for COD concentrations. Before COD analysis, the collected samples were neutralized to pH 6.5-7.0 with 5 N NaOH to stop the Fenton reaction. The optimum Fe^{3+}/H_2O_2 ratio was selected based on the highest COD removal efficiency achieved.

Although Fe^{3+} was used as the catalyst in this study, hydroxyl radical generation proceeds through the Fe^{3+}/Fe^{2+} redox cycle rather than by direct oxidation with Fe^{3+} alone. Initially, Fe^{3+} is reduced to Fe^{2+} by hydrogen peroxide and organic intermediates under acidic conditions. The regenerated Fe^{2+} subsequently reacts with H_2O_2 to produce hydroxyl radicals, while being oxidized back to Fe^{3+} [14], [15]. Therefore, maintaining an appropriate Fe^{3+} concentration is essential to sustain the catalytic cycle. At excessive Fe^{3+} dosage, radical scavenging and iron hydrolysis may become dominant, thereby reducing oxidation efficiency. To allow a systematic assessment of the Fenton oxidation process, a structured experimental scheme was implemented. The operating conditions used in this study are summarized in Table 3.

Table 3. Experimental Design

EC-supernatant (mL)	Initial pH	Speed of stirrer (rpm)	H_2O_2/COD ratios (g/g)	Fe^{3+}/H_2O_2 ratios (g/g)
300	3.0	200	5	0
300	3.0	200	10	0
300	3.0	200	15	0
300	3.0	200	20	0
300	3.0	200	15	0.04
300	3.0	200	15	0.02
300	3.0	200	15	0.013

2.4. Analysis

Parameters analyzed in this study were liquid pH and COD concentration. The analyses were conducted in duplicate. Detailed analysis procedures are explained below.

1) pH

The pH of the process was measured using a digital pH meter. Approximately 15 mL of liquid was withdrawn from the upper section with a volumetric pipette and immediately analyzed.

2) COD

Chemical Oxygen Demand (COD) was analyzed using the closed-reflux digestion method followed by spectrophotometric measurement. In this procedure, 2 mL of the liquid sample was introduced into a COD digestion vial pre-filled with High-Range Plus reagent. The vial was shaken to ensure complete mixing, then heated at 150°C for 2 hours in a COD reactor. After digestion, the vial was cooled to room temperature before being placed into a COD meter for quantification. The COD concentration was calculated using Eq. (1), where c represents the COD reading and f is the dilution factor [16].

$$\text{COD Concentration} = \left(\frac{mg-O_2}{L}\right) = \frac{C}{10} \times f \quad (1)$$

COD removal was determined by subtracting the final COD (effluent) from the initial COD (influent). The removal efficiency, defined as the percentage reduction in organic pollutants during digestion, was calculated using Eq. (2).

$$\text{COD removal (\%)} = \frac{\text{COD}_{influent} - \text{COD}_{effluent}}{\text{COD}_{influent}} \times 100\% \quad (2)$$

2.5. Kinetics

The reduction of COD concentration in petrochemical wastewater was evaluated using empirical first- and second-order kinetic models. The zero-order kinetic model was not evaluated because it assumes that the reaction rate remains constant and is independent of the pollutant concentration. In contrast, the Fenton oxidation process is generally concentration-dependent, as the degradation rate is influenced by the concentrations of both the organic pollutants and the reactive hydroxyl radicals generated during the reaction. Consequently, Fenton oxidation is more commonly described by first-order or second-order kinetics. Therefore, this study focused on first-order and second-order kinetic models, which are considered more appropriate for describing COD degradation during the Fenton process. The mathematical expressions of the first- and second-order kinetic models are presented in Eq. (3) and (4).

$$\ln(COD_t) = \ln(COD_0) - k_1 t \quad (3)$$

$$\frac{1}{COD_t} = k_2 t + \frac{1}{COD_0} \quad (4)$$

In these equations, COD_0 represents the initial COD concentration (g/L), COD_t denotes the COD concentration at time t (g/L), k_1 is the kinetic rate constant of the first-order model, k_2 is the kinetic rate constant of the second-order model, and t is the reaction time (minutes). For the first-order kinetic model, a plot of t versus $\ln(COD_t)$ was constructed, and a linear regression was applied to the data to describe the relationship between t and $\ln(COD_t)$, where the slope corresponds to k_1. For the second-order kinetic model, a plot of t versus $1/COD_t$ was generated, followed by linear regression analysis to express the relationship between t and $1/COD_t$, with the slope representing k_1. Microsoft Excel was employed to perform the kinetic analysis. R^2 and adjusted R^2 were used to assess the model fit. Although R^2 measures the proportion of variance explained, adjusted R^2 accounts for the number of predictors in the model, providing a more accurate estimate of the model's explanatory power. The adjusted R^2 was calculated using Eq. (5).

$$Adjusted\ R^2 = 1 - (1 - R^2)\left(\frac{n-1}{n-p-1}\right) \quad (5)$$

Where n is the number of observations and p is the number of independent variables.

3. Results and Discussion

3.1. Effect of the H_2O_2/COD Ratio

1) COD Removal Efficiency

COD reduction was monitored during the reaction to evaluate the Fenton process' efficacy at different oxidizer dosages. As shown in Fig. 2, the change in COD over time suggests that each H_2O_2/COD ratio affects degradation performance. The following section discusses the reduction patterns to determine the most effective ratio for removing organic pollutants.

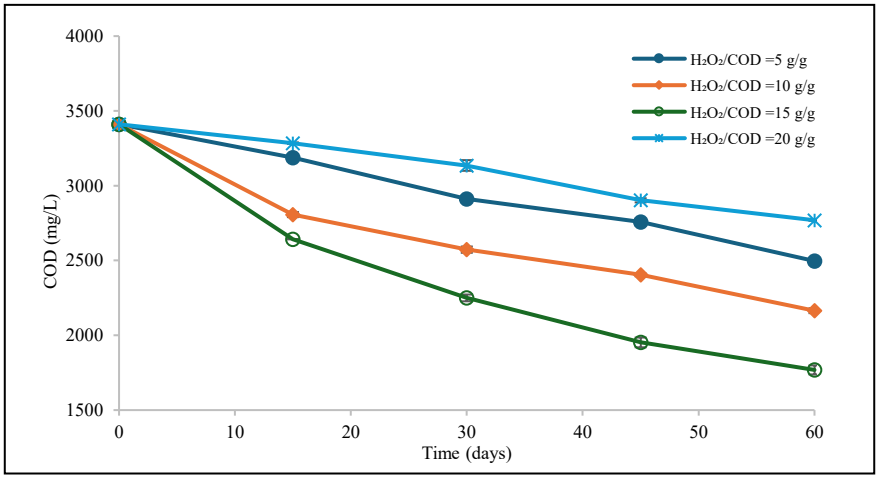

Fig. 2. COD removal in the Fenton process at variations in H_2O_2/COD

Variations in the H_2O_2/COD ratio significantly affected the effectiveness of the Fenton process in reducing COD in EC supernatant. At an H_2O_2/COD ratio of 5 g/g, the average initial COD of 3410.5 mg/L decreased to 3188.5 mg/L at 15 minutes, then gradually decreased to 2910.75 mg/L at 30 minutes, 2757.75 mg/L at 45 minutes, and reached 2496.75 mg/L at 60 minutes. This total decrease of approximately 26.8% indicates that at low ratios, the H_2O_2 dose is still insufficient to produce hydroxyl radicals optimally (•OH), even though this species determines the intensity of organic compound oxidation [17]. Several studies have also confirmed that at too low a ratio, the formation of •OH is limited because the available H_2O_2 is insufficient to drive the iron redox cycle effectively, thereby preventing the degradation rate from increasing significantly [18]. Better performance was seen when the H_2O_2/COD ratio was increased to 10 g/g. The initial COD of 3410.5 mg/L decreased to 2807 mg/L at 15 minutes, 2572.75 mg/L at 30 minutes, 2403.75 mg/L at 45 minutes, and 2164.5 mg/L at 60 minutes. This decrease of around 36.5% confirms that adding H_2O_2 can increase •OH formation and, ultimately, accelerate organic degradation compared to the 5 g/g ratio. The formation

of highly reactive hydroxyl radicals, which can oxidize various organic compounds, depends primarily on the availability of H_2O_2 and ferrous ions under acidic conditions [19].

The highest efficiency was obtained at an H_2O_2/COD ratio of 15 g/g. Under this condition, the average initial COD of 3410.5 mg/L dropped sharply to 2642.5 mg/L at 15 minutes, 2250 mg/L at 30 minutes, 1952.5 mg/L at 45 minutes, and reached 1768.75 mg/L at 60 minutes. In total, this decrease is approximately 48.1%, indicating that a ratio of 15 g/g is the most suitable operating point. H_2O_2 is available in a proportion that is in harmony with the organic load, so that the formation of •OH radicals can be maximized without triggering adverse side reactions [20]. Several studies also confirmed that increasing H_2O_2 concentration tends to increase radical formation and oxidation efficiency, but this positive effect is limited.

At the highest H_2O_2/COD ratio of 20 g/g, process efficiency actually decreased significantly, with a COD removal rate of 18.8%. This sharp decline indicates that excessive H_2O_2 not only generates hydroxyl radicals but also promotes several competing side reactions. At high concentrations, H_2O_2 scavenges hydroxyl radicals (•OH), producing less reactive species such as HO_2•, thereby reducing the overall oxidation potential of the system. By competing with organic pollutants for reactive radicals, excess H_2O_2 in these conditions lowers the fraction of -OH available for effective oxidation [21]. In addition, radical recombination reactions (e.g., •OH + •OH → H_2O_2) further decrease the availability of effective oxidizing species. Moreover, excessive oxidant concentrations may lead to mass-transfer limitations and increased interactions among reactive species, causing self-quenching effects in which radicals preferentially react with one another rather than with organic pollutants. Changes in the reaction medium, such as increased turbidity and the accumulation of intermediate compounds, may also hinder effective contact between •OH radicals and target contaminants. These combined effects result in a non-linear relationship between oxidant dosage and COD removal efficiency, explaining the pronounced decline observed at 20 g/g. Therefore, careful oxidant dosage control is necessary to maintain the efficacy of the Fenton process [22]. The investigation's findings clearly demonstrate that applying H_2O_2 within an optimal concentration window is necessary to maximize oxidation efficiency. Among the tested conditions, an H_2O_2/COD ratio of 15 g/g resulted in the highest COD removal within a 60-minute reaction time, indicating the most efficient use of generated radicals. In light of this finding, a 15 g/g ratio was selected as the reference operating condition for additional experiments examining variations in the Fe^{3+}/H_2O_2 ratio. When oxidant performance has already reached optimal levels, this method allows for a more focused evaluation of iron's catalytic function. The results of the Fenton process at variations in H_2O_2/COD at an operating time of 60 minutes are shown in Table 4.

Table 4. The Results of the Fenton Process at Variation of H_2O_2/COD at an Operating Time of 60 minutes

Initial pH	Speed of stirrer (rpm)	H_2O_2/COD ratios (g/g)	Operating time (minutes)	Initial COD (mg-O_2/L)	Final COD (mg-O_2/L)	COD removal (%)
3.0	200	5	60	3410.5±2.1	2496.75±6.0	26.79
3.0	200	10	60	3410.5±2.1	2164.50±16.3	36.53
3.0	200	15	60	3410.5±2.1	1768.75±30.1	48.14
3.0	200	20	60	3410.5±2.1	2769.25±3.9	18.80

2) pH Profile

Monitoring pH variation during the Fenton reaction is crucial for understanding the reaction environment and oxidation mechanism, as well as for removing COD. At various H_2O_2/COD ratios, pH significantly affects the production of oxidation intermediates, iron speciation, and hydroxyl radicals, all of which influence process efficiency. Thus, Fig. 3 depicts the evolution of pH over the course of the reaction.

Fig.3. shows that the pH remained acidic during the reaction period (0-60 min), with values ranging from 3.00 to 1.86, which is consistent with the requirements of an effective Fenton system. At an H_2O_2/COD ratio of 5 g/g, the pH decreased rapidly, indicating strong oxidation and the generation of acidic intermediates and proton release during H_2O_2 breakdown. When the ratio was increased to 10 g/g, the pH variation became less consistent, suggesting that as the reaction progressed, acid formation and subsequent partial mineralization occurred simultaneously. A slight increase in pH observed after approximately 45 min may be attributed to the gradual consumption of acidic intermediates and the depletion of hydroxyl radicals as the oxidation reaction approached completion. As the concentration

of readily oxidizable organic compounds decreased, the rate of acid formation declined, allowing the pH to recover slightly.

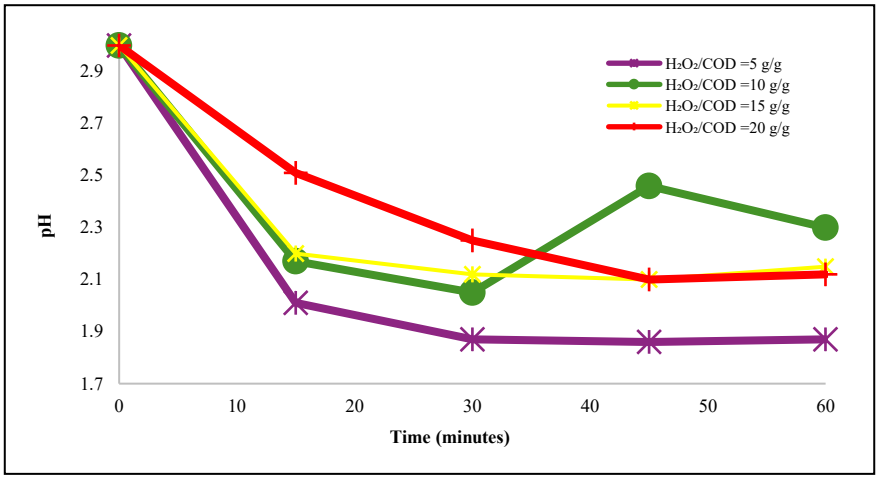

Fig. 3. pH profile in the Fenton process at variation of H_2O_2/COD

A completely different behavior was observed at an H_2O_2/COD ratio of 15 g/g. In these circumstances, there is very little initial drop in pH, and the pH tends to remain mostly steady until the process is finished. The development of more regulated oxidation conditions, in which the rate of formation of acidic intermediates is proportional to their rate of breakdown, is reflected in this pattern. The highest COD removal at this ratio is consistent with this stable pH profile, suggesting effective oxidation without an excessive buildup of acidic intermediates.

The initial pH drop was relatively small at the highest H_2O_2/COD ratio of 20 g/g, and as reaction time increased, it gradually decreased. This pattern suggests that the initial oxidation process was not ideal, perhaps because excessive H_2O_2 reduced the efficacy of hydroxyl radicals via a radical-scavenging mechanism. Oxidation activity began to rise after some of the excess oxidant had broken down, but overall COD removal performance remained below ideal levels. This pH behavior generally supports the idea that oxidant dosage significantly affects the stability and reaction pathway of the Fenton process. Under all conditions tested, an H_2O_2/COD ratio of 15 g/g demonstrated the best balance between oxidation efficiency and pH control, thereby strengthening its position as the most suitable operating condition for post-electrocoagulation treatment of petrochemical wastewater.

The pH profile is also closely related to iron speciation during the Fenton reaction. At acidic conditions (pH 2–3), soluble Fe^{2+} and Fe^{3+} species remain available to catalyze hydroxyl radical generation through the Fe^{2+}/Fe^{3+} redox cycle. As the reaction proceeds, Fe^{2+} is continuously oxidized to Fe^{3+} and subsequently reduced back to Fe^{2+} by hydrogen peroxide or organic intermediates. Maintaining the solution within this acidic pH range prevents excessive precipitation of ferric hydroxides, thereby sustaining catalytic activity. If the pH increases above approximately 3–4, Fe^{3+} tends to hydrolyze and precipitate as $Fe(OH)_3$, reducing the concentration of soluble catalytic iron and consequently lowering the efficiency of hydroxyl radical production.

3.2. Effect of the Fe^{3+}/H_2O_2 Ratio

1) COD Removal Efficiency

The balance between the amount of available oxidant and the efficiency of its catalytic activation is a major factor in COD removal performance, as evidenced by changes in the Fe^{3+}/H_2O_2 ratio. In the end, this equilibrium dictates how quickly hydroxyl radicals are formed during the Fenton reaction and how effectively the oxidation proceeds. The system without Fe^{3+} addition at Fe^{3+}/H_2O_2 = 0 g/g already achieved a significant COD reduction, as shown in Fig. 4, with 48.14% removal after 60 minutes (COD decreased from 3410.5 mg/L to 1768.8 mg/L). But as Fig. 4 illustrates, this performance only represents H_2O_2's self-decomposition and its restricted capacity to produce •OH radicals when the Fe^{2+}/Fe^{3+} catalytic cycle is not present. These results align with the mechanism reported by Riadi *et al.* [23] who emphasized that H_2O_2 alone exhibits poor radical-conversion efficiency because its spontaneous homolysis is slow and thermally limited. Therefore, improved performance upon the addition of Fe^{3+} is expected, as iron catalyzes the redox cycle, accelerating radical formation and increasing the degradation rate.

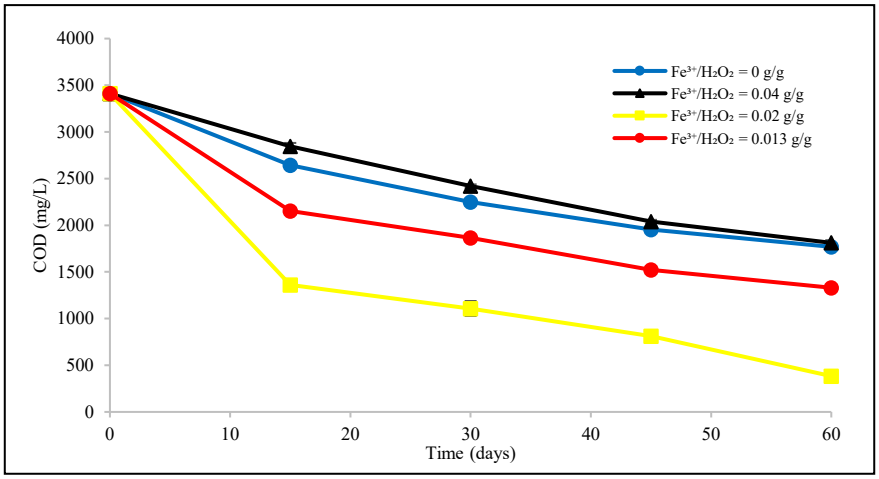

Fig. 4. COD removal in the Fenton process at variation of Fe^{3+}/H_2O_2

When the Fe^{3+}/H_2O_2 ratio was increased to 0.04 g/g, COD removal decreased significantly, with the final COD reaching 1812 mg/L, which corresponds to a removal efficiency of only 46.87%, slightly worse than the Fe-free system. This result appears contradictory but is explained by the radical scavenging behavior described by Zhang *et al.* [24] who showed that excess Fe^{3+} can decrease the net •OH concentration because Fe^{2+}/Fe^{3+} also acts as a radical scavenger at high concentrations through reactions as shown in Eq. (6).

$$Fe^{2+} + \cdot OH \leftrightarrow Fe^{3+} + OH^- \quad (6)$$

The data showed that at Fe^{3+}/H_2O_2 = 0.04 g/g, the iron concentration is high enough to initiate a competitive reaction, in which iron ions consume the generated radicals faster than the radicals oxidize organic compounds. This phenomenon was also explained by Wang *et al.* [25], who showed that excessive catalyst dosage can reduce oxidation efficiency because the reaction pathway shifts to the unproductive iron redox cycle. Consequently, although Fe^{3+} is needed as a catalyst, excessively high dosage actually reduces the availability of reactive radicals and ultimately suppresses the system's oxidative capacity.

In addition, a high Fe^{3+} concentration can accelerate iron hydrolysis and the formation of ferric hydroxide complexes or precipitates, especially as the reaction proceeds and local pH changes occur. These species may reduce the availability of soluble catalytic iron and hinder the Fe^{3+}/Fe^{2+} cycle. Therefore, the lower COD removal at Fe^{3+}/H_2O_2 = 0.04 g/g compared with the treatment without Fe addition suggests that excessive Fe^{3+} caused catalytic inhibition rather than enhancement.

The most significant increase in COD degradation performance occurred at an Fe^{3+}/H_2O_2 ratio of 0.02, where COD decreased from 3410.5 mg/L to 383.8 mg/L, with a very high removal efficiency of 88.75%, the best among all ratios tested. This pattern indicates the achievement of the most balanced conditions, where the amount of Fe^{3+} available is sufficient to accelerate the Fe^{2+} regeneration cycle without causing adverse side effects on the process, resulting in rapid radical formation while remaining within safe limits to prevent radical scavenging. This is in line with the explanation by Sari et al. [26]: at the optimal catalyst level, electron transfer between the Fe^{2+}/Fe^{3+} pair and H_2O_2 becomes more effective, thereby allowing the radical-producing reaction sequence to proceed at higher yields.

An Fe^{3+}/H_2O_2 ratio of 0.02 g/g provides an appropriate balance between oxidant dosage and available catalytic sites, allowing a larger fraction of H_2O_2 to be effectively converted into reactive radicals. The high COD removal seen in this situation is explained by the catalytic turnover being adequate to meet the wastewater's oxidation demand. This finding aligns with the kinetic interpretation presented by Yani *et al.* [27], who highlighted that when the rate of radical generation equals the organic load, maximum pollutant degradation is attained. A rapid reaction rate, indicative of pseudo-first-order behavior under ideal radical availability, is further evidenced by the significant decrease in COD within 15- 60 minutes.

On the other hand, performance clearly declined when the catalyst dosage was lowered to a Fe^{3+}/H_2O_2 ratio of 0.013 g/g. COD only dropped from 3410.5 mg/L to 1330.8 mg/L, indicating a 60.98% removal efficiency. This reduced efficiency suggests insufficient catalytic iron to sustain rapid

Fe^{2+} regeneration, thereby limiting the formation of hydroxyl radicals and slowing the overall oxidation process. Low catalyst levels reduce the acceleration of the radical chain mechanism and increase the system's reliance on the slower, less efficient breakdown of H_2O_2, according to Wang *et al.* [25]. As a result, oxidation continues, but the radical stream is insufficient to mineralize most organic compounds, leading to relatively high residual COD. This pattern is typical of under-catalyzed Fenton systems: the conversion of H_2O_2 to reactive species is slowed, and the degradation pathway tends to terminate at partially oxidized products rather than full mineralization.

Considering this, the Fe^{3+}/H_2O_2 ratio of 0.02 g/g provides the best catalytic balance. It yields the highest COD removal because radical formation is efficient, radical scavenging is suppressed, and the iron redox cycle works synergistically with the available oxidizer. As the ratio shifts above or below the points 0.04 g/g and 0.013 g/g, respectively, performance decreases, either due to excess catalyst promoting scavenging or to too little catalyst limiting radical formation. Meanwhile, the H_2O_2 system alone exhibited oxidizing ability, but the oxidation rate was lower because, without a catalyst, peroxide decomposition could not sustain sufficient •OH flux. Therefore, the data support the conclusion that Fe^{3+}/H_2O_2 = 0.02 g/g is the optimal ratio for COD removal performance. The results of the Fenton process at variations of H_2O_2/COD at an operating time of 60 minutes are shown in Table 5.

Table 5. The Results of the Fenton Process at Variations of Fe^{3+}/H_2O_2 at an Operating Time of 60 minutes

Initial pH	Speed of stirrer (rpm)	Fe^{3+}/H_2O_2 ratios (g/g)	Operating time (minutes)	Initial COD (mg-O_2/L)	Final COD (mg-O_2/L)	COD removal (%)
3.0	200	0	60	3410.5±2.1	1768.75±30.1	48.14
3.0	200	0.013	60	3410.5±2.1	1330.75±23.0	60.98
3.0	200	0.02	60	3410.5±2.1	383.75±47.7	88.75
3.0	200	0.04	60	3410.5±2.1	1812.00±28.3	46.87

2) pH Profile

The pH changes during the Fenton reaction essentially reflect the dynamic interplay between radical formation, the iron redox cycle, and the formation and decomposition of acid intermediates. Across all tested Fe^{3+}/H_2O_2 ratios, the pH remained within a strongly acidic range of approximately 1.74-3.00 (Figure 5), which is typical of conventional Fenton systems. Such acidic conditions are necessary to support effective hydroxyl radical generation and preserve the stability of Fe^{2+} species [23]. To ensure that Fe^{3+} remained soluble and readily accessible for reduction throughout the redox cycle, the system pH was set to 3.0 at the start of the reaction (t = 0). Additionally, catalytic H_2O_2 decomposition is preferred over non-catalytic pathways at this initial pH. These circumstances are in line with the results of Zhang *et al.* [24], who found that a pH near 3 maximizes the production of reactive oxygen species by increasing the rate of Fe^{3+} to Fe^{2+} conversion while inhibiting iron precipitation.

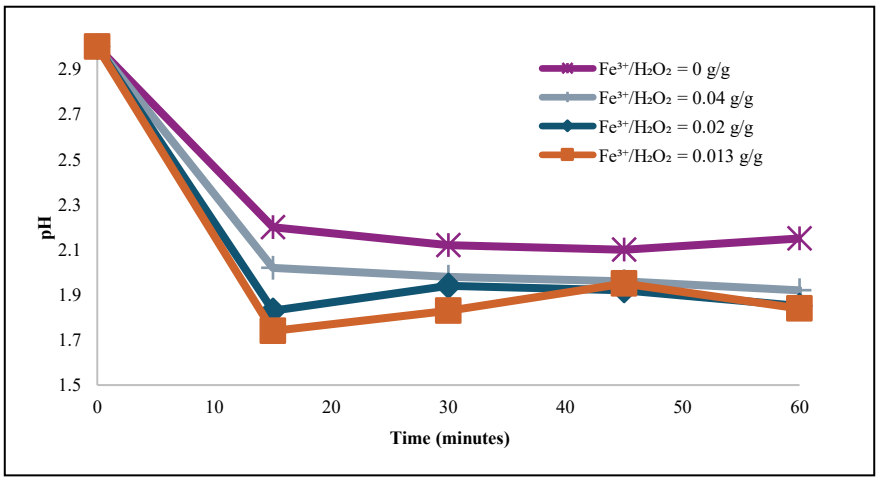

Fig. 5. pH Profile in the Fenton Process with Variation in Fe^{3+}/H_2O_2

When the reaction takes place, the entire system experiences a sharp decrease in pH within the first 15 minutes. The lowest pH value occurred at the highest Fe dosage, Fe^{3+}/H_2O_2 = 0.013, reaching pH

1.74. This rapid acidification was likely triggered by the formation of low-molecular-weight organic acids in the early stages of oxidation. Wang *et al.* [25] explained that strong oxidative cleavage of aromatic and aliphatic structures can produce carboxylic compounds, which then significantly lower the pH. In addition, the catalytic decomposition of H_2O_2 releases protons (H^+), further lowering the pH [27]. Stronger acidification at Fe^{3+}/H_2O_2 = 0.013 g/g is consistent with accelerated catalytic activity; higher Fe^{3+} availability increases the rate of the Fe^{3+}/Fe^{2+} redox cycle, generating more hydroxyl radicals but simultaneously increasing proton-releasing side reactions. A sharp decrease in pH indicates very intense oxidation but also indicates the onset of unproductive radical scavenging [24].

At intermediate Fe doses of Fe^{3+}/H_2O_2 = 0.02 g/g, the pH also decreased but by a smaller amount, stabilizing at 1.83 and 1.94 after 15-60 minutes. The decrease in pH in this system indicates that the oxidation of organic compounds does indeed result in the release of protons. However, this decrease is not continuous because some of the protons are offset during the advanced oxidation stage, when the formed organic acids begin to decompose further into CO_2 and water. The two-stage pattern of a sharp initial decrease followed by a tendency to stabilize was also reported by Sari *et al.* [26], who explained that after the primary acids are degraded, the system approaches a pseudo-steady state, in which the rates of proton formation and consumption are approximately balanced. A more regulated oxidation pathway is indicated by the pH stabilization observed at an Fe^{3+}/H_2O_2 ratio of 0.02, suggesting that intermediate iron dosages favor efficient hydroxyl radical utilization over radical scavenging. The pH varied very little in the absence of an iron catalyst (Fe^{3+}/H_2O_2 = 0 g/g), remaining between 2.10 and 2.20 throughout the reaction. Because its breakdown happens slowly in the absence of catalytic activation, this behavior illustrates H_2O_2's limited oxidative capacity when acting alone. As a result, fewer hydroxyl radicals are produced, thereby reducing the formation of acidic oxidation intermediates. Riadi *et al.* [23] noted a similar pattern, noting that weak organic acid formation in peroxide-only systems resulted in limited pH variation. Therefore, the low oxidizing efficiency of uncatalyzed H_2O_2 is confirmed by the relatively stable pH under Fe-free conditions, which is consistent with the lower COD removal obtained.

At the highest Fe^{3+}/H_2O_2 ratio of 0.04 g/g, the pH decreased moderately from 2.02 to 1.92 and remained relatively stable throughout the reaction. Although this pH profile indicates that the reaction medium remained sufficiently acidic to sustain the Fenton process, the relatively stable pH should not be interpreted as evidence of superior oxidation performance. In fact, the COD removal efficiency under these conditions was only 46.87%, which was substantially lower than that obtained at the optimum Fe^{3+}/H_2O_2 ratio of 0.02 g/g. This finding suggests that maintaining an appropriate pH alone is insufficient to ensure efficient oxidation; the concentration and reactivity of catalytic iron species are equally important.

The superior performance observed at the Fe^{3+}/H_2O_2 ratio of 0.02 g/g indicates that this dosage provided the most favorable balance between hydroxyl radical generation and iron redox cycling. Under this condition, sufficient Fe^{3+} was continuously reduced to Fe^{2+}, allowing efficient regeneration of hydroxyl radicals while minimizing undesirable side reactions. Consequently, acidic intermediates were formed during the initial oxidation stage, leading to a rapid decrease in pH, followed by a relatively stable pH as these intermediates were progressively mineralized into CO_2 and H_2O. This behavior reflects an efficient oxidation pathway in which proton generation and proton consumption gradually approached equilibrium.

By contrast, further increasing the Fe^{3+} dosage to 0.04 g/g did not improve the oxidation process. Instead, excessive iron likely promoted non-productive reactions in which hydroxyl radicals were consumed by Fe^{2+}/Fe^{3+} species rather than reacting with organic contaminants. In addition, the higher concentration of ferric ions may have enhanced iron hydrolysis and the formation of ferric hydroxide complexes, thereby reducing the availability of soluble catalytic iron participating in the Fe^{3+}/Fe^{2+} redox cycle. These phenomena decreased the effective utilization of hydroxyl radicals and explain why the highest iron dosage resulted in substantially lower COD removal despite exhibiting only a moderate change in pH. Similar observations have been reported by Zhang *et al.* [24], who concluded that excessive iron dosage may reduce Fenton efficiency because radical scavenging and iron accumulation outweigh the catalytic benefits.

In contrast, the Fe-free system (Fe^{3+}/H_2O_2 = 0 g/g) exhibited only minor pH fluctuations throughout the reaction because hydrogen peroxide alone decomposes slowly without catalytic activation. The

limited generation of hydroxyl radicals restricted both the oxidation of organic compounds and the formation of acidic intermediates, which explains the relatively stable pH and the lower COD removal observed under this condition. Overall, the pH profiles demonstrate that the optimum Fe^{3+}/H_2O_2 ratio was 0.02 g/g rather than 0.04 g/g, confirming that Fenton performance depends not only on maintaining acidic conditions but also on achieving an appropriate balance between catalytic iron concentration, hydroxyl radical production, and radical consumption.

3.3. Kinetic Analysis

The kinetic behavior of COD removal during the Fenton process with respect to H_2O_2/COD variation was analyzed using first- and second-order models, as shown in Fig. 6 and Fig. 7, with the kinetic parameters summarized in Table 6.

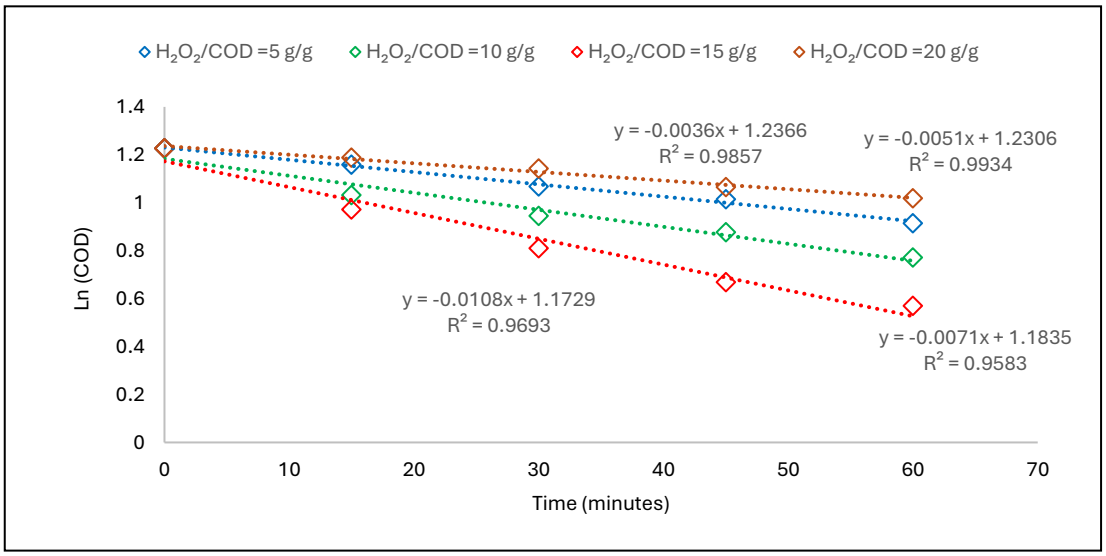

Fig. 6. The results of the first-order kinetic model for various H_2O_2/COD ratios

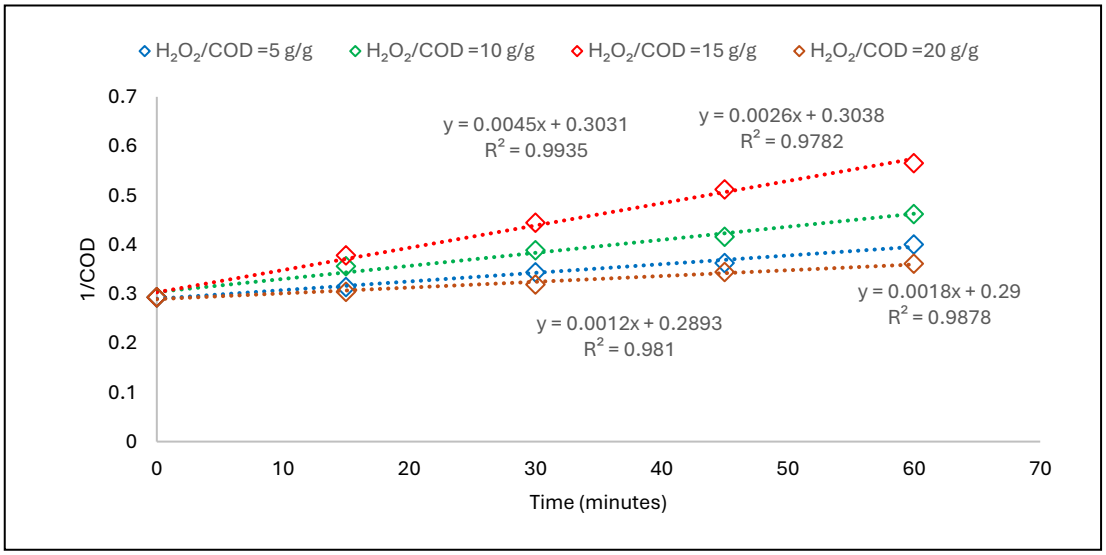

Fig. 7. The results of the second-order kinetic model for various H_2O_2/COD ratios

Table 6. The results of kinetic analysis at H_2O_2/COD ratios

Ratio	First-order			Second-order		
	k_1 *(/min)*	R^2	*Adjusted* R^2	k_2 *(/(g/L.min))*	R^2	*Adjusted* R^2
H_2O_2/COD =5 g/g	0.0051	0.9934	0.9912	0.0018	0.9878	0.9837
H_2O_2/COD =10 g/g	0.0071	0.9583	0.9444	0.0026	0.9782	0.9709
H_2O_2/COD =15 g/g	0.0108	0.9693	0.9591	0.0045	0.9935	0.9913
H_2O_2/COD =20 g/g	0.0036	0.9857	0.9809	0.0012	0.9810	0.9747

Both models exhibited good linearity; however, the second-order model provided a slightly better fit, with higher coefficients of determination (R^2 = 0.98-0.99 and adjusted R^2 = 0.97-0.99) compared to the first-order model (R^2 = 0.96-0.99 and adjusted R^2 = 0.94-0.99). For the first-order model, the rate constant (k_1) increased with increasing H_2O_2/COD ratio up to 15 g/g, reaching a maximum of 0.0108 min-1, then decreased at 20 g/g. This trend indicates that COD degradation is enhanced by increasing oxidant dosage up to an optimum level, beyond which excess H_2O_2 reduces the effective reaction rate due to hydroxyl radical scavenging.

Similarly, the second-order kinetic analysis showed the highest rate constant (k_2) at an H_2O_2/COD ratio of 15 g/g (0.0045 $L \cdot g^{-1} \cdot min^{-1}$), while lower values were obtained at both lower and higher ratios. The superior fitting of the second-order model suggests that COD degradation is governed by interactions between organic compounds and reactive oxidizing species, rather than by substrate concentration alone. Overall, the kinetic results confirm that the H_2O_2/COD ratio of 15 g/g is optimal for COD removal, consistent with the observed experimental degradation trends, and that a second-order kinetic model better describes the Fenton oxidation process.

The kinetic behavior of COD degradation at different Fe^{3+}/H_2O_2 ratios was evaluated using first- and second-order models, as presented in Figs. 8 and 9, with the corresponding parameters summarized in Table 7. Both kinetic models adequately described the experimental data. However, their performance varied with catalyst dosage.

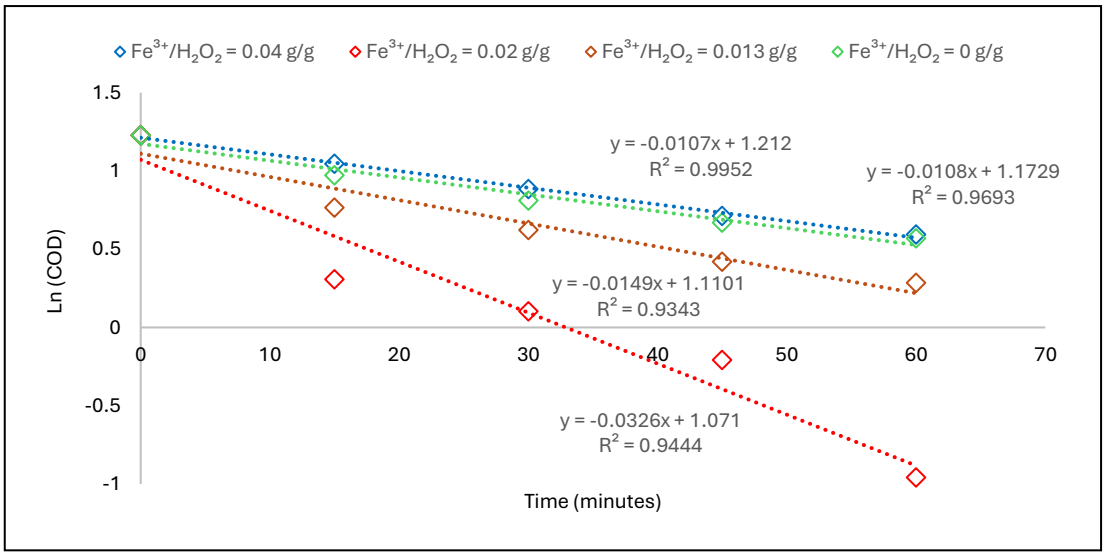

Fig. 8. The results of the first-order kinetic model for various Fe^{3+}/H_2O_2 ratios

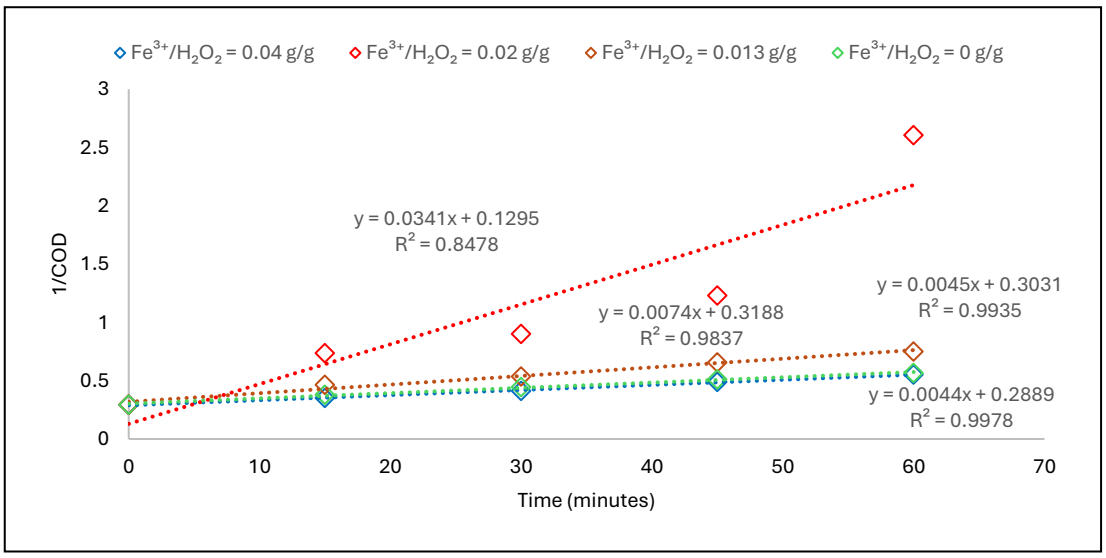

Fig. 9. The results of the second-order kinetic model for various Fe^{3+}/H_2O_2 ratios

Table 7. The results of kinetic analysis at various Fe^{3+}/H_2O_2 ratios

Ratio	First-order			Second-order		
	k_1 (/min)	R^2	Adjusted R^2	k_2 (/(g/L.min))	R^2	Adjusted R^2
Fe^{3+}/H_2O_2 = 0.04 g/g	0.0107	0.9952	0.9936	0.0044	0.9978	0.9971
Fe^{3+}/H_2O_2 = 0.02 g/g	0.0326	0.9444	0.9259	0.0341	0.8478	0.7971
Fe^{3+}/H_2O_2 = 0.013 g/g	0.0149	0.9343	0.9124	0.0074	0.9837	0.9783
Fe^{3+}/H_2O_2 = 0 g/g	0.0108	0.9693	0.9591	0.0045	0.9935	0.9913

Based on the first-order kinetic model, the highest reaction rate constant (k_1 = 0.0326 min^{-1}) was obtained at an Fe^{3+}/H_2O_2 ratio of 0.02 g/g, indicating the fastest COD degradation rate under this condition. Lower k_1 values were observed at ratios of 0.04 g/g (0.0107 min^{-1}) and 0.013 g/g (0.0149 min^{-1}), suggesting that insufficient or excessive iron concentrations reduce the overall reaction rate. The decrease in kinetic performance at higher Fe^{3+} dosage is attributed to hydroxyl radical scavenging and possible formation of iron complexes that limit radical availability. The second-order kinetic model showed a comparable fitting quality, with R^2 values ranging from 0.85 to 0.99. The highest second-order rate constant (k_2 = 0.0341 $L \cdot g^{-1} \cdot min^{-1}$) was also achieved at the Fe^{3+}/H_2O_2 ratio of 0.02 g/g, reinforcing that this condition provides the most favorable balance between oxidant and catalyst. Lower k_2 values at ratios of 0.04 and 0.013 g/g demonstrate that radical generation and utilization are less effective outside the optimum catalyst range. The kinetic analysis confirms that the Fe^{3+}/H_2O_2 ratio of 0.02 g/g is optimal, yielding the highest reaction rate constants in both kinetic models. This result is consistent with the COD removal trends and highlights the critical role of Fe^{3+} concentration in sustaining effective Fenton oxidation kinetics.

4. Conclusion

This study demonstrates that the combined electrocoagulation–Fenton process is an effective treatment for petrochemical wastewater, achieving the highest COD removal at an H_2O_2/COD ratio of 15 g/g and an Fe^{3+}/H_2O_2 ratio of 0.02 g/g. These optimum reagent dosages provided efficient hydroxyl radical generation while minimizing radical scavenging, resulting in superior oxidation performance. The findings confirm that careful optimization of oxidant and catalyst dosages is essential to maximize Fenton efficiency and improve petrochemical wastewater treatment. This study was conducted at laboratory scale and did not monitor the Fe^{2+}/Fe^{3+} speciation during the reaction, which could provide further insight into the iron redox cycle and reaction mechanism. Future research should evaluate the process under continuous-flow conditions, incorporate real-time pH monitoring and control, investigate iron speciation and residual oxidants throughout the reaction, and assess the economic feasibility and sludge management to support large-scale industrial implementation.

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