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Bench-to-budget evaluation of Fenton oxidation for real citrus processing wastewater: Process optimization, full-scale costing, and decision support

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ABSTRACT
This study aims to optimize the Fenton treatment of real citrus-processing wastewater (CPWW) using response surface methodology (RSM) and to develop a techno-economic assessment for a treatment capacity of 200 m^3 day^{-1}. The experimental design, analysis of variance, and response-surface plots were performed using Design-Expert v.12 software. Under the optimal conditions—2,500 mg L^{-1} H_2O_2, 750 mg L^{-1} $FeSO_4$, pH 3, and a reaction time of 90 min—a maximum chemical oxygen demand (COD) removal of 38.2% was achieved. The relatively moderate COD removal obtained in the present study should be interpreted in the context of the real wastewater matrix, considering the possible presence of recalcitrant citrus-derived compounds, radical scavengers, and constituents capable of complexing iron. For a full-scale plant with a capacity of 66,000 m^3 $year^{-1}$, the estimated treatment cost was \$7.563 m^{-3}, equivalent to \$5.04 per kilogram of COD removed. Overall, the estimated cost lies between the higher reported costs of photo-Fenton treatment and the lower reported costs of biological-treatment technologies. Operational expenditures (OPEX) accounted for 98% of total costs, with hydrogen peroxide alone contributing 89.9% of annual OPEX. Sensitivity analysis revealed reagent costs as the most influential parameter, where a ±20% fluctuation led to a ±18.82% variation in total costs, highlighting the vulnerability of Fenton economics to chemical supply markets. Interest rate variations similarly impacted annualized capital expenditures (CAPEX), with ±20% fluctuations resulting in ±19.6% changes. This study provides a transparent cost model, identifies reagent consumption as the primary economic bottleneck, and highlights opportunities to integrate Fenton oxidation with pre- and/or post-treatment processes to reduce oxidant demand and improve COD removal beyond that achievable by stand-alone Fenton treatment. This study establishes a scalable techno-economic framework for Fenton oxidation of CPWW; it highlights limitations in pollutant removal efficiency and identifies hydrogen peroxide as the primary cost driver, which significantly increases sensitivity to market fluctuations.

Keywords: Citrus processing, Advanced oxidation, Fenton, Wastewater treatment, Techno-economic analysis.

INTRODUCTION
Citrus fruits rank among the most valuable tropical and subtropical crops globally, with approximately 31.2 million tons processed annually worldwide (Raimondo *et al.* 2018). Over half of global citrus production originates in Asia, where China leads as a top producer. Iran also plays a significant role, ranking among the top ten citrus-producing countries. In 2020, Iran produced 3.74 million tons of citrus, down slightly from 4.07

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million tons in 2019, reflecting an average annual growth rate of 6.19% since 1970. That year, citrus cultivation spanned 290,000 hectares, yielding 4.7 million tons, with a per capita production of 56 kg. The provinces of Mazandaran, Golestan, and Guilan contribute approximately 80% of Iran's total citrus output (Goodarzian *et al.* 2023). Citrus fruit processing produces two main by-products: A solid or semi-solid fraction, composed of peels and fruit residues, and liquid citrus wastewater. The citrus industry generates substantial volumes of wastewater, known as Citrus Processing Wastewater (CPWW), as a natural outcome of its diverse operations. CPWW primarily arises from activities such as washing fruits with water sprays, cleaning facilities and equipment, cooling processes, extracting essential oils, and drying peels (Zema *et al.* 2019). Depending on the technologies and methods employed, CPWW may also include discharges from producing citric acid, pectin, citrus molasses, and peel oil. On average, processing one ton of citrus fruit produces 1 to 17 cubic meters of CPWW (Di Trapani *et al.* 2019; Zema *et al.* 2019; Lucia *et al.* 2022), and in some cases, the wastewater volume may exceed ten times that of the citrus juice extracted (Güneş Durak *et al.* 2023). Several factors influence CPWW production, including water usage per unit of fruit, the total mass of citrus processed, and water management practices, such as recirculation systems or the type of cooling process (open-cycle versus closed-cycle). Additionally, CPWW's volume and composition fluctuate significantly, showing seasonal, yearly, monthly, weekly, and even daily variations (Zema *et al.* 2019).

Management of wastewater from citrus juice production is a particular problem due to both its high volume and complex nature. Globally, over 700 million cubic meters of CPWW are generated annually as a by-product of citrus fruit processing (Lucia *et al.* 2022). With a low pH, elevated organic matter content, and essential oils, it can cause considerable environmental harm if not properly managed (Zema *et al.* 2012; Calabrò *et al.* 2016; Corsino *et al.* 2021). Meeting increasingly strict environmental regulations requires advanced treatment technologies, which impose substantial costs on the industry. However, in some developing countries, simpler treatment systems are allowed due to differing regulations and socioeconomic conditions (Zema *et al.* 2019). Improper handling of CPWW can lead to severe environmental consequences, such as pollution of surface and groundwater sources, degradation of aquatic habitats, increased risk of waterborne diseases, and greenhouse gas emissions. To meet rigorous environmental standards, industries must adopt robust treatment technologies. Additionally, CPWW holds potential for resource recovery, enabling a sustainable bio-economy by extracting energy, nutrients, and valuable materials to create useful products. For example, treated CPWW can serve as irrigation water, delivering both hydration and nutrients to crops. The fluctuating volume and chemical composition of CPWW, however, demand adaptable and efficient treatment approaches (Lucia *et al.* 2022). The treatment methods used for citrus wastewater have evolved from simple disposal techniques to more advanced treatment and valorization procedures, driven by environmental regulations and the possibility of resource recovery. Various physical, chemical, and biological treatment processes, as well as combined strategies, have been explored to improve the efficiency of citrus wastewater treatment (Lucia *et al.* 2022; Kato & Kansha 2024). Biological treatment methods are widely used for CPWW due to its high organic content. Common approaches include aerobic treatments, such as activated sludge systems (Osorio *et al.* 2006; Navarro *et al.* 2008) and Sequencing batch reactors (SBRs; Guzmán *et al.* 2015; Corsino *et al.* 2018), as well as anaerobic digestion using up-flow anaerobic sludge blanket (UASB) reactors (Elnekave *et al.* 2012; Güneş Durak *et al.* 2023). Aerobic-anaerobic lagooning systems also play a role in CPWW management (Zema *et al.* 2012, Andiloro *et al.* 2013; Zema *et al.* 2016). However, conventional biological processes face significant challenges. The variable characteristics and seasonal production of CPWW, along with toxic essential oil (EO) compounds, nutrient imbalances, and high energy demands, complicate treatment. Additionally, the long retention times necessitate large footprints, and biochemical instability can disrupt performance (Zema *et al.* 2019; Lucia *et al.* 2022). Over the past decade, researchers have explored integrating membranes into biological processes to address the limitations of conventional CPWW treatment. Membrane bioreactors (MBRs), which combine biological degradation with advanced filtration, have emerged as a promising alternative (Di Trapani *et al.* 2019; Corsino *et al.* 2022). MBRs offer improved effluent quality and a compact design compared to traditional methods. However, challenges such as membrane fouling and reduced long-term filtration efficiency hinder their adoption in large-scale applications (Lucia *et al.* 2022).

In recent years, researchers have investigated advanced oxidation processes (AOPs) to improve the treatment of various wastewater types, either as a final step to ensure effluent quality for disposal or reuse, or as a pretreatment to enhance biodegradability for biological processes (Ma *et al.* 2021; Babu Ponnusami *et al.*

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2023; Kato & Kansha 2024). Numerous studies have explored AOPs for food industry wastewater treatment (Heponiemi & Lassi 2012; Krzemińska *et al.* 2015; Amor *et al.* 2019; Dave & Das 2021). However, only a few studies have focused on CPWW treatment using AOPs, highlighting a research gap in addressing its treatment challenges (Miklos *et al.* 2018; Zema *et al.* 2019; Lucia *et al.* 2022).

AOPs generate powerful hydroxyl radicals to break down organic pollutants, using methods such as ozone-based, UV-based, electrochemical, catalytic, or physical processes like ultrasound (Miklos *et al.* 2018). These techniques offer promising solutions for managing CPWW's complex composition.

Fenton processes, a widely adopted type of AOP, rely on iron-catalyzed breakdown of hydrogen peroxide (H_2O_2) to produce hydroxyl radicals (•OH), which effectively degrade organic pollutants non-selectively. Their performance can be enhanced by integrating techniques like electrical current, ultraviolet light, or ultrasound, which generate additional •OH radicals and improve Fe^{2+} regeneration from Fe^{3+}. Beyond breaking down persistent compounds, Fenton processes offer key benefits: they operate at ambient temperature and pressure, use accessible and manageable reagents, and achieve rapid reaction times compared to other AOPs. These processes are straightforward to implement, requiring no specialized equipment, and can be seamlessly combined with existing treatment methods, such as coagulation, filtration, or biological systems. However, the homogeneous Fenton process has notable limitations, including ongoing iron ion loss and the production of solid sludge, which demands additional handling. The high cost of reagents, especially H_2O_2, is another challenge, with operating costs ranging from 0.2 to 17.7 € m^{-3}, highlighting the influence of operating conditions on cost-effectiveness and overall efficiency (Cañizares *et al.* 2009; Ribeiro & Nunes 2021; Saravanan *et al.* 2022). The advantages of the Fenton process make it a promising treatment for agro-industrial wastewaters, such as CPWW, due to its ability to tackle complex organic loads (Krzemińska *et al.* 2015; Amor *et al.* 2019; Dave & Das 2021). Guzmán *et al.* (2015) demonstrated that combining photo-Fenton pretreatment with a SBR effectively treats synthetic citrus wastewater with high initial chemical oxygen demand (COD) (up to 10,000 mg L^{-1}). The photo-Fenton process, conducted in a solar chamber using a xenon lamp to simulate sunlight, achieved 61% dissolved organic carbon (DOC) removal. After neutralization and clarification, the SBR further reduced organic matter, yielding 93% DOC removal and a final COD of 120 mg L^{-1}. Using response surface methodology, the study optimized operating parameters (initial COD: 1000–10000 mg L^{-1}; H_2O_2: 425–4250 mg L^{-1}; Fe^{3+}: 85–850 mg L^{-1}), identifying initial COD and H_2O_2 concentration as the primary factors influencing DOC removal. Higher initial COD led to reduced degradation efficiency, while increased H_2O_2 consistently enhanced performance. Fe^{3+} effects varied, decreasing efficiency at low COD, improving it at moderate COD, and exhibiting nonlinear behavior at high COD. In another study, Guzmán *et al.* (2016) evaluated ozone-based AOPs—including O_3, O_3/OH^-, O_3/UV, O_3/H_2O_2, and $O_3/UV/H_2O_2$—and solar photo-Fenton as pretreatments for synthetic citrus wastewater. Ozonation processes effectively reduced color and turbidity but achieved limited organic matter removal, with $O_3/UV/H_2O_2$ at pH 7 yielding the highest performance (15.7% COD and 10.9% DOC removal). In contrast, solar photo-Fenton pretreatment, optimized at pH 3.8, 15,937 mg L^{-1} H_2O_2, 510 mg L^{-1} Fe^{3+}, and a 30-minute reaction time, removed 76.9% COD and 53% DOC. Despite a higher treatment cost of 13.8 € m^{-3} compared to biological treatments, solar photo-Fenton offers a viable pathway to meet stringent organic matter discharge standards, enhancing CPWW treatment sustainability. It is noteworthy that this cost estimation only accounts for operating costs and excludes capital expenditures associated with the treatment plant. In a recent study, Vilar *et al.* (2024) applied electrochemical oxidation with a boron-doped diamond anode to reduce the toxicity and organic load of biodegraded citrus pulp wash effluent. Electrolysis at 20 mA cm^{-2} for 6 hours achieved 99.7% TOC and 70% COD removal. Additionally, the energy consumption was estimated at 2.93 kWh per gram of TOC removed, suggesting economic potential for high-efficiency treatment. Can (2014) investigated three electrochemical processes—electrooxidation (EOX), electrocoagulation (EC), and electro-Fenton (EF)—for treating fruit-juice production wastewater. The electro-Fenton process achieved the highest COD removal, reaching 84.4% after 25 minutes with 10 mL H_2O_2 addition, a constant current density of 200 A m^{-2}, and a wastewater pH of 3.5. Despite EF's superior efficiency, its specific energy consumption (SEC) of 0.767 kWh per kg COD removed was relatively moderate compared to EOX and EC. These findings highlight EF's potential for effective organic matter removal, though high energy costs pose challenges for sustainable treatment. In a study by Rodrigues *et al.* (2022), the effectiveness of different AOPs including electro-Fenton (EF), electrochemical oxidation (ECO), activated persulfate (APS), and combined EF/APS treatments for treating fruit processing wastewater was

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evaluated. The results indicated that while EF and combined EF/APS achieved the highest organic load removals (> 90%), ECO demonstrated the most significant improvement in biodegradability and reduction in toxicity, particularly at higher applied current densities, without requiring chemical addition. Single APS treatment was deemed unfeasible due to the high chemical requirements, resulting in toxicity, and low biodegradability improvement. The study highlights the trade-off between achieving high organic load removal, reducing toxicity, and minimizing energy consumption across these methods.

The comprehensive literature review highlights that while Fenton oxidation has been widely studied in laboratory settings, most reports are limited to synthetic wastewaters or partial parametric optimization without linking findings to economic feasibility. To strengthen the bridge between bench-scale experimentation and industrial application, this study makes the following contributions:

- It applies systematic RSM optimization to the treatment of real citrus processing wastewater, capturing the complexity and inhibitory effects absent from synthetic matrices.
- It develops a full techno-economic model, covering both CAPEX and OPEX, for a scaled-up 66,000 m^3 $year^{-1}$ plant, thereby moving beyond the preliminary or partial cost analyses common in previous studies.
- It conducts sensitivity analysis to quantify the influence of chemical prices on total treatment costs, revealing reagent supply as the dominant risk factor.
- It provides decision-support insights by identifying reagent costs as the main economic bottleneck and pointing to hybridization strategies (e.g., pre-coagulation, biological polishing, or electro-Fenton variants) as promising directions to reduce oxidant demand and enhance overall feasibility.

By integrating experimental optimization with full-scale cost modelling, the present work offers a transparent and transferable framework that supports both industrial stakeholders and researchers in evaluating the practical potential of advanced oxidation for challenging food-industry effluents.

1. MATERIALS AND METHODS

1.1. Wastewater characteristics

To account for variability in wastewater composition and ensure reliable results, 40 L of CPWW was collected daily over one month (August 2023) from a citrus processing facility in Chaboksar, Guilan Province, Iran. The samples were stored at 4 °C to preserve their characteristics until analysis or treatment. The sample characterization, including key parameters such as COD, biological oxygen demand (BOD_5), total suspended solids (TSS), total dissolved solids (TDS), total nitrogen (TN), total phosphorus (TP), and pH is presented in Table 1.

1.2. Analytical methods

COD, BOD_5, TSS, TDS, TN, TP, and pH were measured in triplicate following Standard Methods for the Examination of Water and Wastewater (Federation and Association, 2023). COD was determined using a thermoreactor (Aqualytic AL 125, Dortmund, Germany) via the closed-reflex method (Method 5220). pH was measured with a pH meter (3510, Jenway, UK; Method 4500-H). BOD_5 was assessed through incubation (Method 5210), and TSS was quantified by filtration (Method 2540-D). TN and TP were measured after digestion followed by colorimetric analysis (Methods 4500-TN C and 4500-TP E, respectively).

Table 1. Characteristics of citrus processing wastewater.

Parameter	Value
COD (mg L^{-1})	3850 ± 150
BOD_5 (mg L^{-1})	1040 ± 10
TSS (mg L^{-1})	250 ± 2
pH	4.8 ± 0.2
TDS (mg L^{-1})	1850 ± 50
TN (mg L^{-1})	0.5 ± 0.1
TP (mg L^{-1})	1.5 ± 0.25

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1.3. Fenton oxidation procedure

Fenton process parameters were adjusted according to the experimental design presented in Table 2. Experiments were conducted using a jar-test apparatus (Zag Chemie Co., Iran) with six 250-mL beakers at ambient temperature (25 ± 2 ºC). Each beaker was filled with 200 mL CPWW and stirred continuously at 120 rpm. The pH was adjusted to the desired values using 1 M H_2SO_4 or 1 M NaOH. Predetermined amounts of $FeSO_4 \cdot 7H_2O$ and 30 w/w% H_2O_2, based on the experimental design, were added sequentially ($FeSO_4 \cdot 7H_2O$ first, Followed by H_2O_2). The reaction time was controlled as per the design. At the end of the designated reaction time, the Fenton reaction was terminated by rapidly adjusting the solution pH to 8 ± 0.1 using NaOH solution. This sudden increase in pH effectively quenched hydroxyl radical generation and initiated the precipitation of dissolved iron species as ferric hydroxides. Stirring was then stopped, and the samples were allowed to settle for 4 hours to complete phase separation. Finally, the supernatant from each beaker was collected and filtered through Whatman No. 42 filter paper, followed by filtration through a 0.45-µm filter, prior to COD analysis. COD removal efficiency was calculated using Equation 1:

$$COD\ removal\ efficiency\ (\%) = (1 - \frac{COD_f}{COD_i}) \times 100 \qquad (1)$$

where COD_i and COD_f are the initial and final COD concentrations, respectively. Noteworthy, the dilution caused by the addition of H_2O_2 and $FeSO_4$ solutions was taken into account in the COD calculations. The measured COD concentration was corrected using the ratio of the final reaction volume to the initial wastewater volume. To ensure reproducibility, experiments were conducted in triplicate, and mean values were reported. All reagents were of analytical grade, purchased from Merck, and used without further purification.

1.4. Experimental design and statistical analysis

A Central Composite Design (CCD) within Response Surface Methodology (RSM) was implemented using Design Expert software (version 12, Stat-Ease Inc., USA) to optimize the Fenton process. A five-level, four-factor CCD was used to evaluate the effects of H_2O_2 concentration (X_1), $FeSO_4$ concentration (X_2), pH (X_3), and reaction time (X_4) on COD removal efficiency (%) and to determine optimal conditions for maximizing COD removal. The ranges of the independent variables were selected based on operating conditions commonly reported in the literature for Fenton oxidation and were chosen to encompass conditions expected to provide measurable COD removal while avoiding excessive reagent consumption. The experimental ranges and coded levels of each independent variable are presented in Table 2.

Table 2. Levels of independent variables used in CCD-RSM for predicting COD removal efficiency.

Independent variable	Symbol	Coded levels				
		-α	-1	0	+1	+α
H_2O_2 (mg L^{-1})	X_1	500	1500	2500	3500	4500
$FeSO_4$ (mg L^{-1})	X_2	250	500	750	1000	1250
pH	X_3	1	2	3	4	5
Time (min)	X_4	30	60	90	120	150

A total of 30 experiments, including 16 factorial points, 8 axial points, and 6 center points, were conducted based on the CCD matrix (Table 3). Experimental data were analyzed using analysis of variance (ANOVA) to assess the significance of main effects, interactions, and quadratic terms. Quadratic models were developed to predict COD removal efficiency as a function of the independent variables, with model quality evaluated using the coefficient of determination (R^2) and adjusted R^2. Statistical significance was determined using *p*-values at a 95% confidence level ($p < 0.05$).

1.5. Economic study

AOPs, such as the Fenton process, are emerging and efficient wastewater treatment methods, offering high mineralization efficiency and minimal secondary pollution, although Fenton processes generate iron sludge. However, their practical application is limited by harsh reaction conditions (e.g., low pH) and high treatment costs (e.g., reagents, and energy). Comprehensive economic analysis is thus critical to evaluate the technical and economic feasibility of AOPs for CPWW treatment, guiding decision-makers in selecting cost-effective, sustainable solutions (Ma *et al.* 2021). The cost of wastewater treatment systems depends on factors such as plant

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size, system configuration, wastewater characteristics, required effluent quality (e.g., COD limits), and local chemical and electricity prices. Consequently, treatment costs vary significantly across regions and technologies. The economic feasibility of the Fenton system was evaluated by calculating the total treatment cost per cubic meter of treated CPWW, comprising both capital expenditures (CAPEX) and operating expenses (OPEX). CAPEX was itemized based on a 200 m^3 day^{-1} plant capacity and included:

1) Mechanical Equipment: Pumps, dosing systems for reagents (H_2SO_4, $FeSO_4$, H_2O_2, and NaOH) including metering pumps and storage tanks, electro-mixers, and a multi-disc screw press for sludge dewatering.
2) Civil Works: Concrete structures for the reaction, pH-adjustment, and gravity settling tanks.
3) Infrastructure: Electrical distribution, process instrumentation, and piping networks.

OPEX was calculated based on annual operating costs, including chemical consumption (H_2SO_4, NaOH, H_2O_2, and $FeSO_4 \cdot 7H_2O$), electricity for electromechanical equipment, maintenance and repair, and labor. Annual maintenance and repair costs were assumed to be 2% of the total CAPEX (Jordá *et al.* 2011). The total capital cost was annualized using the Capital Recovery Factor (CRF) to account for the interest rate and plant lifetime, as follows (Economics & Center 1999; Mahamuni & Adewuyi 2010):

$$\text{Annualized capital cost} = \text{CAPEX} \times \text{CRF} \qquad (2)$$

The CRF was calculated based on a 25-year system lifetime and a 23% interest rate, as shown in Equation 3 (Economics & Center 1999; Mahamuni & Adewuyi 2010). While this interest rate is higher than those typically used in international studies (5–10%), it was selected to align with the actual cost of industrial capital and prevailing lending rates in Iran, where high inflation and central bank policies result in higher nominal interest rates for infrastructure projects (Economics 2024). This conservative approach ensures that the annualized CAPEX reflects the real-world financial conditions of the local market. Furthermore, the 25-year lifetime represents the design life of the primary civil structures (e.g., concrete reaction and settling tanks, which are protected with anti-corrosive liners). To account for the shorter service life of mechanical and electrical components operating under corrosive acidic conditions and handling abrasive chemical sludge, these assets are assumed to be constructed from corrosion-resistant materials (e.g., PVDF, and PTFE) and are sustained by a dedicated annual maintenance and repair allowance.

$$\text{CRF} = \frac{\text{rate} \times (1 + \text{rate})^{\text{life}}}{(1 + \text{rate})^{\text{life}} - 1} \qquad (3)$$

Annual chemical and electricity consumption was calculated using the optimum Fenton operating conditions and a design flow rate of 200 m^3 d^{-1}. Considering 90% system availability, the annual operating period was assumed to be 330 days per year, corresponding to an annual treatment volume of 66,000 m^3 $year^{-1}$. The annual consumption of each reagent was calculated from its optimum dosage, whereas electricity consumption was calculated from the system's energy demand. Annual operating costs were then calculated by multiplying the calculated consumption of H_2O_2, $FeSO_4 \cdot 7H_2O$, NaOH, H_2SO_4, and electricity by their respective unit prices in Iranian market: \$0.8 L^{-1} for H_2O_2, \$0.6 kg^{-1} for $FeSO_4 \cdot 7H_2O$, \$0.4 L^{-1} for NaOH, \$0.25 L^{-1} for H_2SO_4, and \$0.04 kWh^{-1} for electricity. The resulting annual consumption and cost values are presented in Table 6. Labor costs were estimated based on two full-time operators, each receiving a monthly salary of USD 354. Therefore, the total annual labor cost was calculated as about USD 8,500 $year^{-1}$ and included in the operating expenditure. The estimated OPEX includes the costs of chemicals, electricity, labor, maintenance, and sludge dewatering. However, off-site transportation and final disposal of the dewatered iron-rich Fenton sludge were not included, since a reliable and standardized local disposal tariff was unavailable and such costs are highly site-specific, depending on sludge characteristics, disposal route, transport distance, and local regulatory requirements. Therefore, the reported unit treatment cost should be interpreted as excluding final off-site sludge management.

Noteworthy, all capital and operating costs were initially estimated based on actual quotations obtained from local suppliers in Iran for the base year 2025. For international comparability, costs were converted to US dollars (USD) using the prevailing open-market exchange rates of 1,200,000 IRR/USD and 0.86 EUR/USD.

To assess the economic viability and sensitivity of the Fenton treatment system, a sensitivity analysis was performed by evaluating ten one-at-a-time scenarios. These scenarios corresponded to a ± 20% variation in five

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key parameters affecting the overall treatment cost: system lifetime, interest rate, reagent prices (H_2O_2 and $FeSO_4 \cdot 7H_2O$), electricity tariffs, and labor costs. For each scenario, the annualized capital cost, annual operating cost, and resultant total treatment cost per cubic meter of wastewater were systematically recalculated. The subsequent variations in treatment cost were rigorously analyzed to identify the critical economic drivers, providing valuable insights for decision-makers seeking cost-effective and sustainable solutions for CPWW treatment.

2. RESULTS AND DISCUSSION

The physicochemical characteristics of CPWW, as summarized in Table 1, reveal a BOD_5/COD ratio of 0.27, indicating poor biodegradability. This is potentially due to the presence of inhibitory citrus-derived compounds, including essential oils, that may interfere with biological treatment processes.

Table 3. Central composite design matrix and results.

Exp. no.	H_2O_2 (mg L^{-1})	$FeSO_4$ (mg L^{-1})	pH	Time (min)	Initial COD (mg L^{-1})	Final COD (mg L^{-1})	COD removal efficiency (%)
1	1500	1000	4	120	3850	2840	26.2
2	2500	750	5	90	3850	2780	27.8
3	3500	1000	4	60	3850	2840	26.2
4	3500	500	2	60	3850	2640	31.4
5	4500	750	3	90	3850	2940	23.6
6	1500	1000	4	60	3850	2960	23.1
7	2500	750	3	150	3850	2800	27.3
8	2500	750	3	30	3850	2740	28.8
9	1500	1000	2	60	3850	2718	29.4
10	3500	1000	2	60	3850	2522	34.5
11	2500	750	3	90	3850	2440	36.6
12	2500	750	3	90	3850	2400	37.7
13	500	750	3	90	3850	3400	11.7
14	3500	500	2	120	3850	2700	29.9
15	3500	1000	2	120	3850	2580	33
16	1500	500	4	120	3850	2918	24.2
17	2500	750	3	90	3850	2380	38.2
18	1500	500	2	60	3850	2880	25.2
19	2500	750	3	90	3850	2400	37.7
20	1500	500	4	60	3850	2840	26.2
21	2500	1250	3	90	3850	2440	36.6
22	3500	500	4	120	3850	2800	27.3
23	2500	750	3	90	3850	2400	37.7
24	1500	500	2	120	3850	3042	21
25	3500	500	4	60	3850	2680	30.4
26	2500	250	3	90	3850	2600	32.5
27	1500	1000	2	120	3850	2760	28.3
28	3500	1000	4	120	3850	2740	28.8
29	2500	750	3	90	3850	2440	36.6
30	2500	750	1	90	3850	2640	31.4

In addition, the low concentrations of total nitrogen (TN = 0.5 mg L^{-1}) and total phosphorus (TP = 1.5 mg L^{-1}) indicate that conventional biological treatment would require substantial nutrient supplementation to sustain microbial activity. These combined constraints necessitate the exploration of alternative treatment strategies, such as AOPs, specifically Fenton oxidation, to achieve effective organic load reduction. Table 3 presents the CCD-RSM design matrix used in this study, with the corresponding measured values of COD removal efficiency for each experiment.

The results revealed that the efficiency of treating real CPWW using the Fenton process strongly depended on reaction conditions, with COD removal efficiencies ranging from 11.7% to 38.2% (initial COD = 3850 ± 150 mg L^{-1}). The highest efficiency (38.2%) was achieved at 2500 mg L^{-1} H_2O_2, 750 mg L^{-1} $FeSO_4$, pH 3, and 90 min, which favored hydroxyl-radical generation. The lower COD removal efficiency observed in the present study, compared with the efficiencies reported for synthetic CPWW in previous studies, e.g., 61% (Guzmán *et al.* 2015),

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76.9% (Guzmán [et al.] 2016), 84.4% (Can 2014), and > 90% (Rodrigues *et al.* 2022)) can likely be attributed to both the use of real, chemically complex CPWW and the application of a stand-alone conventional Fenton process rather than a hybrid treatment system. Real CPWW contains high concentrations of organic matter, recalcitrant citrus-derived compounds, radical-scavenging species, and iron-complexing constituents, all of which can reduce Fenton performance by limiting the availability of hydroxyl radicals. This finding is consistent with Guzmán *et al.* (2015), who observed reduced degradation efficiency at higher COD levels.

2.1. Model development and statistical evaluation of experimental results

2.1.1. Quadratic regression model for experimental data

To investigate the effects of key process variables on COD removal from CPWW, a second-order polynomial model was developed using the response surface methodology. The model captures the influence of hydrogen peroxide concentration (X_1, mg L^{-1}), ferrous sulfate concentration (X_2, mg L^{-1}), pH (X_3), and reaction time (X_4, min) on the COD removal efficiency (%). The fitted equation (Equation 4) is expressed as follow:

$$\begin{aligned} COD\ removal\ officiency\ (\%) &= -48.7747 + 0.0303\,X_1 + 0.0294\,X_2 + 14.4447\,X_3 + 0.2954\,X_4 \\ &- 7.44 \times 10^{-4}\,X_1X_3 - 5.375 \times 10^{-3}\,X_2X_3 + 1.16 \times 10^{-4}\,X_2X_4 \\ &+ 0.0185\,X_3X_4 - 4.8822 \times 10^{-6}\,{X_1}^2 - 1.1 \times 10^{-5}\,{X_2}^2 - 1.8947\,{X_3}^2 \\ &- 2.536 \times 10^{-3}\,{X_4}^2 \end{aligned} \quad (4)$$

This model accounts for linear, quadratic, and interaction effects, providing a comprehensive description of variable influences on the Fenton process.

2.1.2. Goodness-of-fit and predictive capability

The strength and reliability of the model were evaluated through several statistical metrics, detailed in Table 4.

Table 4. Statistical validation parameters for the quadratic model.

Response	R^2	Adjusted R^2	Predicted R^2	Adequate precision	Standard deviation	CV (%)
COD removal efficiency (%)	0.988	0.9768	0.94	38.22	0.9128	3.08

The model explained approximately 99% of the observed variability in COD removal (R^2 = 0.988). The adjusted R^2 value of 0.9768, which corrects for the number of predictors, remained close to R^2, confirming the model's validity without evidence of overfitting. In terms of predictive power, the model yielded a predicted R^2 of 0.94. The small gap between adjusted and predicted R^2 values demonstrates the consistency of the model and suggests that it generalizes well beyond the experimental dataset. The adequate precision value of 38.22, substantially higher than the recommended threshold of 4, signifies a robust signal-to-noise ratio, which is critical for navigating multi-variable optimization spaces. The overall model exhibited a standard deviation of 0.9128 and a coefficient of variation (CV) of 3.08%, while the six center-point replicates showed a mean COD-removal efficiency of 37.42%, a standard deviation of 0.65 percentage points, and a CV of approximately 1.7%. This scatter may reflect the combined effects of the heterogeneous composition of real CPWW and normal experimental variability associated with sample handling, reagent dosing, pH adjustment, mixing, reaction timing, filtration, and COD analysis. The wastewater matrix may contain variable concentrations of organic constituents, suspended matter, radical-scavenging compounds, and iron-complexing species. Accordingly, the variation should not be attributed solely to wastewater instability.

2.1.3. ANOVA and significance of model terms

The model's statistical validity was assessed through analysis of variance (ANOVA), summarized in Table 5.

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Table 5. ANOVA results for COD removal quadratic model in Fenton process.

Response	**COD removal efficiency (%)**				
Source	**Sum of squares**	**Degrees of freedom**	**Mean square**	***F*-value**	***p*-value**
Model	1029.08	14	73.51	88.21	8.42E-12
X_1-H_2O_2	158.62	1	158.62	190.36	6.28E-10
X_2-$FeSO_4$	20.35	1	20.35	24.42	1.77E-04
X_3-pH	31.51	1	31.51	37.82	1.86E-05
X_4-Time	4.77	1	4.77	5.72	3.03E-02
X_1 X_2	2.98	1	2.98	3.57	7.83E-02
X_1 X_3	8.85	1	8.85	10.62	5.28E-03
X_1 X_4	0.03	1	0.03	0.04	8.51E-01
X_2 X_3	28.89	1	28.89	34.67	2.98E-05
X_2 X_4	12.08	1	12.08	14.49	1.72E-03
X_3 X_4	4.95	1	4.95	5.94	2.77E-02
X_1^2	653.81	1	653.81	784.62	2.29E-14
X_2^2	11.85	1	11.85	14.22	1.85E-03
X_3^2	98.48	1	98.48	118.18	1.65E-08
X_4^2	142.87	1	142.87	171.46	1.30E-09
Residual	12.50	15	0.83		
Lack of Fit	10.31	10	1.03	2.36	1.78E-01
Pure Error	2.19	5	0.44		
Cor Total	1041.57	29			

The model was highly significant, with an F-value of 88.21 and a *p*-value of 8.42×10^{-12}, well below the 0.05 threshold, indicating that the observed variance is highly unlikely to result from random error. The lack-of-fit test yielded a non-significant F-value of 2.36 ($p = 0.178$). A non-significant lack-of-fit ($p > 0.05$) is statistically desirable, as it indicates that the residual variance–the error that the model cannot explain–is primarily attributable to pure experimental error rather than model inadequacy. Consequently, this result confirms that the quadratic model effectively describes the relationship between the operational parameters and COD removal, ensuring the model is robust and reliable for predicting optimal conditions within the studied experimental design space. The low residual sum of squares (12.50) and minimal pure error (0.44) further confirm the precision of the experimental design and the reliability of the measurements. Among the main effects, hydrogen peroxide concentration (X_1) emerged as the most influential factor ($F = 190.36$, $p < 0.0001$), followed by pH (X_3) and ferrous sulfate concentration (X_2). The quadratic term of hydrogen peroxide (X_1^2) showed the highest significance (F= 784.62), reflecting a strong nonlinear relationship—consistent with hydroxyl radical scavenging at excessive H_2O_2 levels (Bello *et al.* 2019; Krupińska 2024). Significant curvature was also observed for the quadratic terms of pH (X_3^2), reaction time (X_4^2), and $FeSO_4$ (X_2^2), highlighting the importance of optimizing these parameters to maximize pollutant degradation efficiency.

Several interaction effects were statistically significant. The $FeSO_4$–pH interaction (X_2X_3) was particularly pronounced ($F = 34.67$), suggesting that iron speciation and catalytic performance are strongly pH-dependent (Bokare & Choi 2014). The H_2O_2–pH interaction (X_1X_3) was also significant ($F = 10.62$), indicating that acidity affects peroxide decomposition and radical formation (Babuponnusami & Muthukumar 2014; Bello *et al.* 2019). Interactions between $FeSO_4$ and time (X_2X_4), and pH-time (X_3X_4), also influenced COD removal to a lesser extent. In contrast, the H_2O_2–time interaction (X_1X_4) was statistically insignificant ($F = 0.04$, $p = 0.851$), indicating negligible synergistic effects between oxidant dose and reaction time under the studied conditions, likely due to the predominant role of initial H_2O_2 concentration in driving reaction kinetics.

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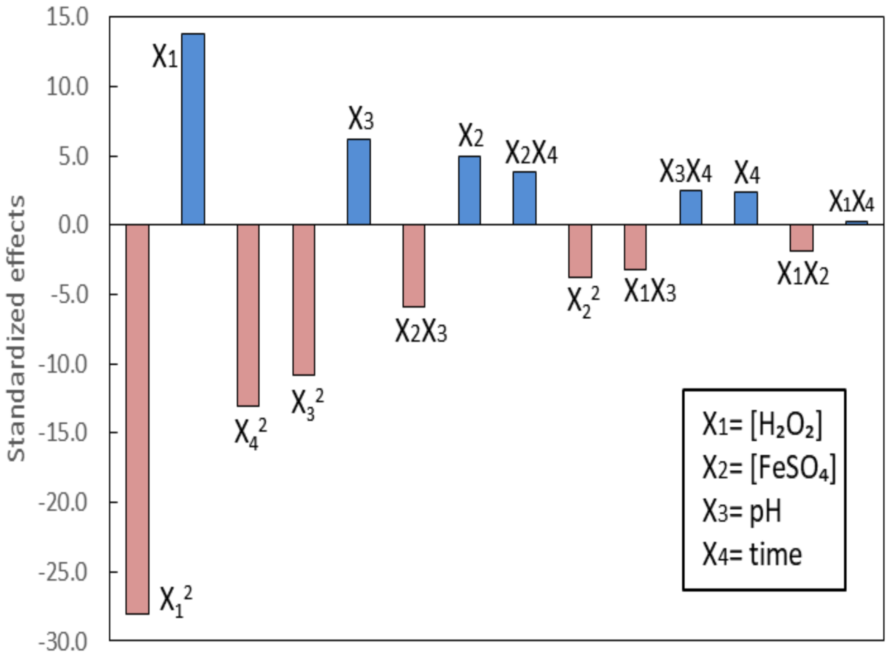

Fig. 1. Pareto chart of standardized effects for the quadratic regression model of COD removal from real CPWW using the Fenton process.

To visualize the relative significance and directionality of each factor, a Pareto chart of standardized effects was plotted (Fig. 1). The chart reveals that the quadratic term of H_2O_2 concentration (X_1^2) exerted the most pronounced negative influence on COD removal efficiency, indicating a performance decline at elevated peroxide levels—likely due to hydroxyl radical scavenging by excess H_2O_2. Conversely, the linear term of H_2O_2 (X_1) had a strong positive effect, highlighting its critical role in initiating oxidative reactions through hydroxyl radical formation. These trends are consistent with the fundamental kinetics of Fenton chemistry, as previously reported by Bello *et al.* (2019) and Krupińska (2024). Similarly, the quadratic term for pH (X_3^2) exhibited a notable negative effect, indicating the existence of a narrow optimal pH range that maximizes Fe^{2+} solubility and catalytic activity (Bokare & Choi 2014; Krupińska 2024). Among the interaction effects, the $FeSO_4$–pH interaction (X_2X_3) showed substantial statistical significance, highlighting the pH-dependent reactivity of ferrous ions and their influence on hydroxyl radical production. Additional interactions such as $FeSO_4$-time (X_2X_4) and pH- time (X_3X_4) contributed moderately, suggesting time-dependent synergistic effects in catalyst performance and acidity control. In contrast, term of H_2O_2-time (X_1X_4) was statistically insignificant and could be excluded from a simplified model without compromising predictive accuracy. These findings emphasize the critical importance of precise control over hydrogen peroxide dosage and pH regulation when applying the Fenton process to complex industrial wastewaters, such as real CPWW, characterized by high COD loads and variable matrix compositions, where nonlinear and interactive effects can significantly influence treatment efficacy.

2.2. Influence of operational parameters (H_2O_2, $FeSO_4$, pH, and reaction time) on Fenton process performance

The 3D surface plots presented in Fig. 2 provide critical insights into how key operational parameters influence the efficiency of COD removal during the Fenton treatment of real CPWW. These visualizations clearly illustrate the interactions between variables and facilitate the identification of optimal conditions for maximizing pollutant degradation.

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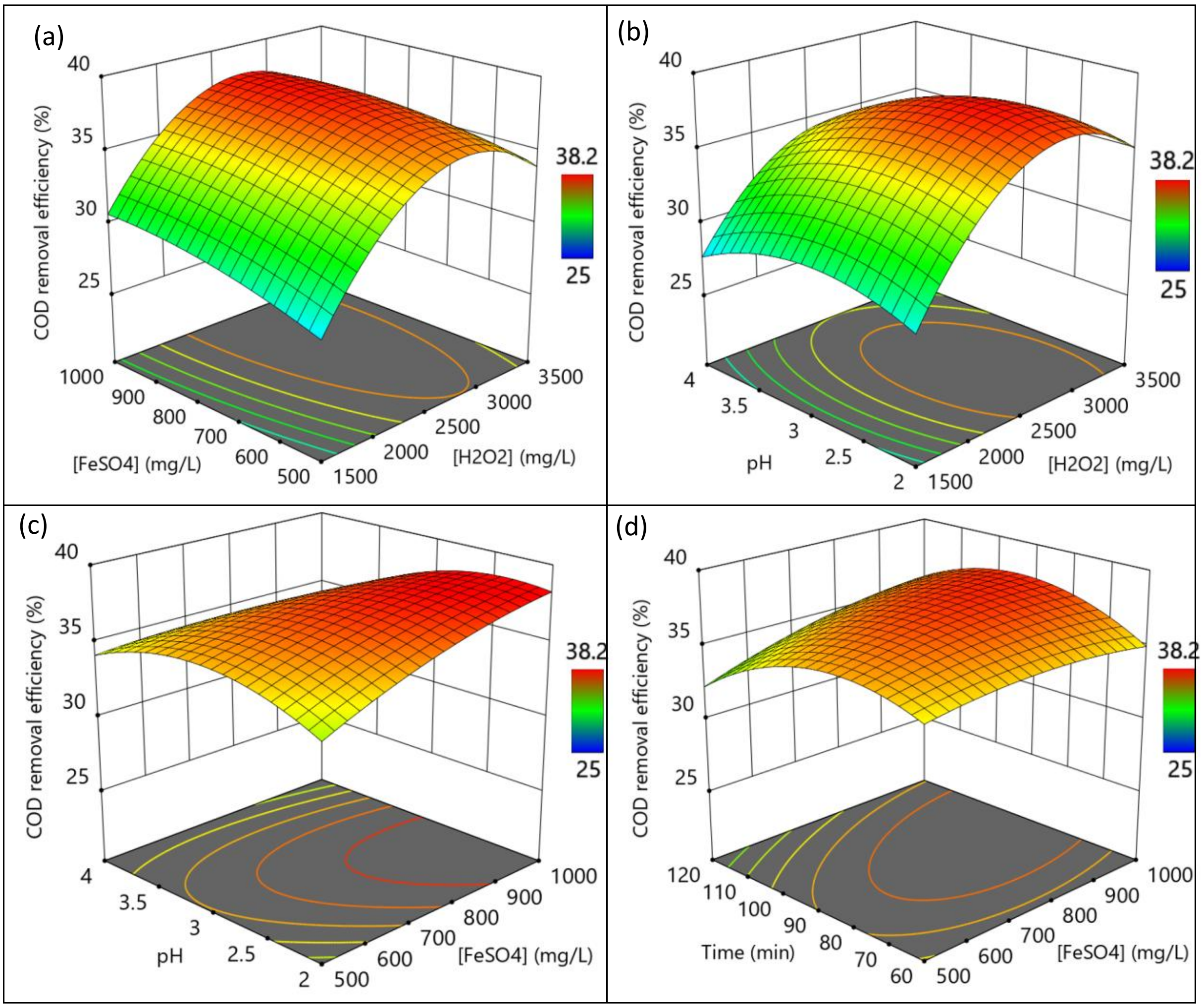

Fig. 2. Three-Dimensional surface plots illustrating interactions on COD Removal Efficiency in the Fenton process for CPWW: (a) $FeSO_4$ vs H_2O_2 concentration (b) pH vs H_2O_2 concentration (c) pH vs $FeSO_4$ concentration (d) reaction time vs $FeSO_4$ concentration.

2.2.1. Effects of $FeSO_4$ and H_2O_2 concentrations

As shown in Fig. 2a, increasing both $FeSO_4$ and H_2O_2 concentrations led to a progressive increase in COD removal efficiencies, peaking at approximately 38.2% under intermediate reagent dosing. The model predicted that elevating $FeSO_4$ from 600 mg L^{-1} to 750 mg L^{-1}, combined with an optimized H_2O_2 concentration of around 2500 mg L^{-1}, significantly enhanced oxidation capacity. This observation aligns with the fundamental chemistry of the Fenton process (Bello *et al.* 2019), where Fe^{2+} catalyzes the decomposition of H_2O_2 to produce hydroxyl radicals (•OH):

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^{-} + OH^{\cdot} \quad (4)$$

However, further increases in either reagent beyond these optimal levels resulted in diminished returns, with the COD removal efficiency plateauing or slightly declining. This trend reflects the onset of radical scavenging reactions at elevated H_2O_2 concentrations:

$$OH^{\cdot} + H_2O_2 \rightarrow H_2O + HO_2^{\cdot} \quad (5)$$

This side reaction reduces the availability of •OH radicals for pollutant oxidation, thereby impairing treatment performance. Similar observations have been reported by Babuponnusami and Muthukumar (2014), Guzmán *et al.* (2015) and Krupińska (2024), who attributed such losses to competitive radical quenching pathways occurring at high oxidant doses.

An optimal H_2O_2 to Fe^{2+} ratio is critical for maximizing efficiency in the Fenton process. Generally, lower H_2O_2:Fe^{2+} ratios tend to favor chemical coagulation, while higher ratios are more conducive to chemical oxidation (Amaral-Silva *et al.* 2016; Ribeiro & Nunes 2021). Achieving this optimal ratio is critical, as excess H_2O_2 and

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Fe^{2+} can adversely affect performance due to scavenging effects. The most efficient and cost-effective conditions for Fenton oxidation of CPWW can be determined using the H_2O_2: Fe^{2+} and H_2O_2:COD ratios. These ratios vary widely in the literature, likely due to differences in the types of pollutant characteristics treated in the Fenton process. For example, Amaral-Silva *et al.* (2016) reported an optimal H_2O_2:Fe^{2+} molar ratio of 5 for elderberry juice wastewater treatment. Lucas & Peres (2009) identified an optimal molar ratio of 15 for olive mill wastewater, while Guzmán *et al.* (2015) observed the highest efficiency at a H_2O_2:Fe^{3+} mass ratio of 31.25 for photo-Fenton treatment of synthetic citrus wastewater. In the current study, the optimal H_2O_2: Fe^{2+} ratio was determined to be 16.67 (w/w), corresponding to a molar ratio of 27.35, achieved at 2500 mg L^{-1} H_2O_2 and 750 mg L^{-1} $FeSO_4$.
The dosage of H_2O_2 relative to the pollutant load, often expressed as the H_2O_2:COD ratio, is another key parameter influencing Fenton process performance. H_2O_2 concentration directly affects the generation of hydroxyl radicals (•OH). Generally, pollutant degradation increases with higher H_2O_2 dosages up to an optimal threshold. In the study by Amaral-Silva *et al.* (2016), the optimal mass ratio of H_2O_2 to COD was 5.48 for elderberry effluent. Lucas & Peres (2009) reported an optimal H_2O_2/COD mass ratio of 1.75 for olive mill wastewater treatment. In another study, Guzmán *et al.* (2015) observed maximum COD removal in the photo-Fenton process from synthetic citrus wastewater at a H_2O_2/COD mass ratio of 1.59. In the current study, the optimal H_2O_2/COD mass ratio for maximum COD removal (38.2%) was found to be 0.65. This demonstrates the Fenton process's ability to degrade a substantial amount of the oxidizable organic matter present in real CPWW.
A closer inspection of the 3D surface plot (Fig. 2a) reveals a parabolic relationship among $FeSO_4$, H_2O_2, and COD removal efficiency. At lower $FeSO_4$ concentrations (e.g., < 600 mg L^{-1}), changes in H_2O_2 had minimal impact on degradation efficiency, indicating that Fe^{2+} availability was the limiting factor. As $FeSO_4$ concentration increased to approximately 750 mg L^{-1}, a sharper rise in COD removal was observed across the mid-range H_2O_2 concentrations (2000–3000 mg L^{-1}), highlighting the critical synergy between Fe^{2+} and H_2O_2 in driving radical generation. Beyond this threshold, however, further increases in either reagent particularly H_2O_2 resulted in reduced efficiency, suggesting that excessive Fe^{2+} may promote non-productive pathways consuming H_2O_2 without effective •OH formation. Additionally, at higher pH levels, excess Fe^{2+} can rapidly oxidize to Fe^{3+}, forming insoluble $Fe(OH)_3$ precipitates that deactivate the catalyst.

The inflection point observed at 750 mg L^{-1} $FeSO_4$ and 2500 mg L^{-1} H_2O_2 corresponds closely to the statistical findings from the ANOVA and Pareto analysis (Fig. 1). These analyses confirmed the dominant positive linear effects of H_2O_2 and $FeSO_4$ concentrations, as well as the significant negative quadratic impact of their concentrations on COD removal efficiency. These results emphasize the importance of precisely optimizing reagent dosages to maintain oxidative efficiency while mitigating potential side effects, such as scavenging and iron loss through precipitation.

2.2.2. Effect of pH

Fig. 2b depicts the interaction between pH and H_2O_2 concentration on the COD removal efficiency. This surface plot reveals a clear correlation between pH and COD removal efficiency, particularly at optimal H_2O_2 concentrations (approximately 2500 mg L^{-1}). As pH increases from 2 to 3, COD removal efficiency improves. However, further increases in pH lead to a decline in efficiency. This trend is consistent with the Pareto analysis in Fig. 1, which identifies both a dominant positive linear effect of pH (X_3) and a significant negative quadratic effect (X_3^2) on COD removal efficiency. The maximum COD removal efficiency (38.2%) was achieved at pH 3 and H_2O_2 concentration of 2500 mg L^{-1}. This pH-dependent behavior aligns with the principles of the Fenton process, where acidic conditions enhance the stability and solubility of Fe^{2+} ions, which are essential for generating hydroxyl radicals. In acidic environments, H_2O_2 decomposes more efficiently to produce •OH, thereby improving oxidation efficiency. The optimal pH approximately 3 corresponds to findings from previous studies (Amaral-Bokare & Choi 2014; Babuponnusami & Muthukumar 2014; Silva *et al*. 2016; Krupińska, 2024), which emphasize the importance of maintaining pH near 3 to maximize the catalytic activity of Fe^{2+} ions and promote •OH formation.
Fig. 2c further highlights the role of pH in optimizing the Fenton process by illustrating the relationship between COD removal efficiency, pH, and $FeSO_4$ concentration. The plot shows that the highest COD removal efficiency occurs at pH 3 and an $FeSO_4$ concentration of 750 mg L^{-1}, indicating that moderately acidic conditions (pH = 3) combined with an optimal $FeSO_4$ concentration are most effective for COD degradation. This finding is consistent with the Pareto chart (Fig. 1), where the linear effect of $FeSO_4$ concentration (X_2) is positive, but the quadratic

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term (X_2^2) has a negative impact, suggesting diminishing returns when $FeSO_4$ concentration exceeds 750 mg L^{-1}. Additionally, the positive linear effect of pH (X_3) on COD removal is confirmed, as increasing pH from 2 to 3 enhances the oxidation process. However, as pH increases beyond 3, COD removal efficiency decreases, a trend supported by the significant negative effect of the quadratic term for pH (X_3^2) in the Pareto chart. This reduction in efficiency can be attributed to the decreased solubility of Fe^{2+} ions and impaired •OH formation at elevated pH levels, as documented in the literature (Bokare and Choi, 2014; Babuponnusami and Muthukumar; 2014; Amaral-Silva et al. 2016; Krupińska 2024). These findings highlight the importance of precisely optimizing pH and $FeSO_4$ concentration to achieve maximum COD removal in the Fenton process.

2.2.3. Effect of reaction time

Fig. 2D illustrates the impact of reaction time and $FeSO_4$ concentration on COD removal efficiency, showing a gradual increase in efficiency as reaction time extends from 60 to 90 minutes, with a peak efficiency of 38.2% at 90 minutes and 750 mg L^{-1} $FeSO_4$. This trend highlights the importance of providing sufficient reaction time for hydroxyl radicals to fully degrade organic contaminants, with no notable improvement observed beyond 90 minutes. This behavior aligns with the findings from the Pareto chart (Fig. 1), where the positive linear effect of reaction time (X_4) on COD removal efficiency is significant, but the quadratic term for reaction time (X_4^2) indicates that further increases in time do not result in proportional gains. The negative quadratic effect suggests that, after the 90-minute threshold, extended reaction times do not enhance organic matter degradation and may even hinder the process due to depletion of available H_2O_2 or formation of stable reaction intermediates. These findings are in agreement with previous studies, including Krupińska (2024), which observed similar trends in Fenton oxidation.

Fig. 2d further indicates that increasing $FeSO_4$ concentration from 500 mg L^{-1} to 1000 mg L^{-1} positively influences COD removal efficiency, which aligns with the positive linear effect of $FeSO_4$ concentration (X_2) observed in the Pareto chart (Fig. 1). However, similar to the trends in Fig. 2c, the increase in efficiency plateaus at higher $FeSO_4$ concentrations, reinforcing the conclusion that excessive $FeSO_4$ results in diminishing returns in COD removal efficiency. This observation is further validated by the negative quadratic term for $FeSO_4$ concentration (X_2^2) in the Pareto chart, indicating an optimal concentration beyond which additional $FeSO_4$ does not significantly enhance the process. Overall, the plot demonstrates that both reaction time and $FeSO_4$ concentration contribute to the efficiency of the Fenton process, but their effects are limited by optimal thresholds, as evidenced by the statistical analysis and model outcomes.

In conclusion, this study demonstrates that optimizing key operational parameters—H_2O_2 concentration, $FeSO_4$ dosage, pH, and reaction time—is critical for achieving high COD removal efficiency in the Fenton process for real CPWW treatment. The highest efficiency of 38.2% was attained under optimal conditions, which include 2500 mg L^{-1} of H_2O_2, 750 mg L^{-1} of $FeSO_4$, a pH of 3, and a reaction time of 90 minutes. These findings provide valuable insights for scaling up the Fenton process in industrial applications, particularly in the citrus processing industry, where wastewater treatment is a critical challenge. By precisely optimizing these parameters, industries can not only enhance the efficiency of their wastewater treatment systems but also reduce the consumption of reagents and minimize operational costs, making the process more sustainable and economically viable. The results underline the importance of balancing reagent concentrations to avoid overuse, which could lead to inefficiencies and increased operational costs. This study lays a foundation for future research that can explore the integration of the Fenton process with other treatment technologies, such as membrane filtration or biological treatment, to further improve the overall sustainability of wastewater management systems in agro-industrial sectors.

2.3. Economic analysis

The flow rate of CPWW generated in the citrus processing facility studied in this research was approximately 200 cubic meters per day. Based on this capacity, all necessary equipment for the Fenton process to treat CPWW was designed. A schematic diagram illustrating the designed Fenton process for the plant is presented in Fig. 3. The Fenton process comprises pH adjustment with H_2SO_4 dosing, catalyst mixing, H_2O_2 dosing and oxidation reaction, neutralization using NaOH dosing, and solid–liquid separation.

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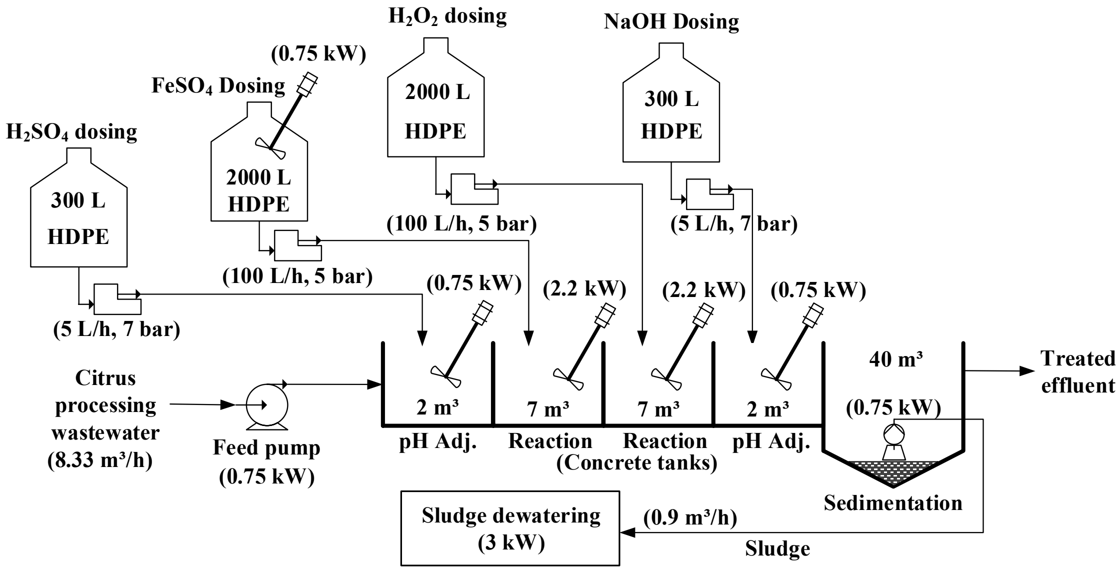

Fig. 3. Schematic diagram of the Fenton process for the treatment of citrus processing wastewater.

Table 6. Costs associated with the Fenton-based CPWW treat plant.

Item	Value	Note
Plant capacity (m^3 d^{-1})	200	-
Total annual treated wastewater (m^3 $year^{-1}$)	66,000	200 m^3 d^{-1} × 330 day $year^{-1}$
Capital costs		
Feed pumps ($)	1190	2 units, 200 m^3 day^{-1} each
H_2SO_4 dosing package price ($)	1930	Includes metering pumps & tank
$FeSO_4$ dosing package price ($)	5400	Includes metering pumps, tank, mixer & hopper
H_2O_2 dosing package price ($)	4320	Includes metering pumps & tank
NaOH dosing package price ($)	1930	Includes metering pumps & tank
Sludge dewatering system price ($)	8300	Multi-disc screw press for approximately 0.9 m^3 h^{-1} produced sludge
Concrete structure price (reaction tank & pH adjusting tanks with electro-mixers, settling tank) ($)	15600	Includes 2 reaction tanks, 2 pH-adjusting tanks, electro-mixers, settling tank
Electrical, instrumentation and control devices price ($)	4150	PLC, sensors, wiring
TOTAL CAPITAL COST ($)	42820	-
Capital recovery factor	0.23	Based on a 25-year system lifetime and a 23% interest rate
Annualized capital cost ($ $year^{-1}$)	9912	-
Annualized capital cost ÷ Total annual treated wastewater ($ m^{-3})	0.15	-
Annual operating costs		
Cost of H_2SO_4 ($ $year^{-1}$)	1375	5500 L $year^{-1}$
Cost of $FeSO_4$ ($ $year^{-1}$)	29700	49500 kg $year^{-1}$
Cost of H_2O_2 ($ $year^{-1}$)	440,000	550,000 L $year^{-1}$
Cost of NaOH ($ $year^{-1}$)	5280	13200 L $year^{-1}$
Cost of electricity ($ $year^{-1}$)	3513	87,833 kWh $year^{-1}$
Annual labor cost ($ $year^{-1}$)	8500	2 operators, each with a monthly salary of USD 354
Annual maintenance and repair Cost ($ $year^{-1}$)	856	2% of total capital costs
TOTAL OPERATING COST ($ $year^{-1}$)	489,225	-
Total operating cost ÷ Total annual treated wastewater ($ m^{-3})	7.413	-
TOTAL TREATMENT COST ($ m^{-3})	7.563	-

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Based on laboratory-scale data, the optimal operational parameters selected for scaling up to full-scale plant operation were 2500 mg L^{-1} of H_2O_2, 750 mg L^{-1} of $FeSO_4$, pH of 3, and reaction time of 90 minutes. These conditions were obtained using CCD-RSM to maximize COD removal. The economic evaluation was subsequently performed for the full-scale system designed under these process-optimum conditions. Its purpose was to estimate the CAPEX, OPEX, and unit treatment cost of the resulting full-scale design, rather than to identify a separate economically optimal operating point.
Table 6 details the costs associated with the Fenton process for treating CPWW at a flow rate of 200 m^3 day^{-1}. Notably,t the cost analysis is based on prices observed in the Iranian market, and these costs may vary in different regions.

Based on the data from Table 6, Fig. 4 presents the cost breakdown of the Fenton-based CPWW treatment plant.

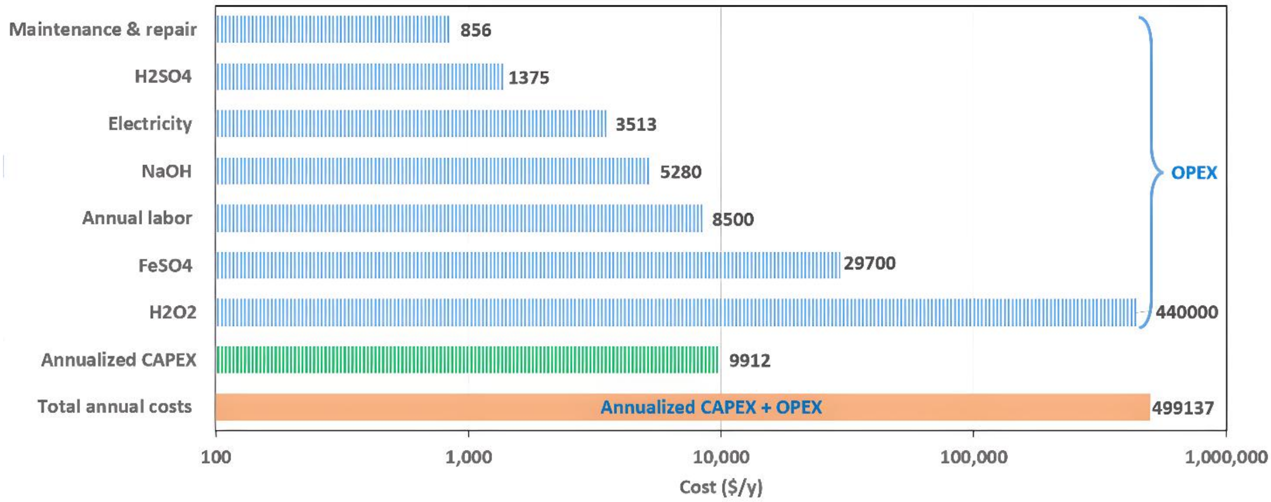

Fig. 4. Cost breakdown of the Fenton-based CPWW treat plant.

2.3.1. Capital costs considerations

The CAPEX for the Fenton treatment plant was $42820, which, when annualized using a capital recovery factor of 4.32, results in an equivalent yearly cost of $9912. The primary investment component was concrete structures, including reaction tanks, pH-adjustment tanks, and electro-mixers, accounting for 36.4% of total CAPEX. This highlights the need for durable infrastructure to withstand acidic conditions and ensure efficient mixing during the oxidation reactions. The sludge dewatering system accounted for 19.4% of CAPEX, while the $FeSO_4$ dosing system contributed 12.6%, emphasizing the importance of solids handling and precise reagent delivery in process design. Alternative Fenton variations, such as fluidized bed Fenton and heterogeneous Fenton, offer potential reductions in sludge generation. These systems utilize solid catalysts or immobilized iron species, reducing iron-rich sludge production compared to conventional homogeneous Fenton processes (Bello *et al.* 2019). However, their adoption may increase CAPEX due to specialized reactor designs or catalyst materials. Annualized capital costs amount to only $0.15 m^{-3} of treated wastewater, constituting a minor fraction of the overall treatment cost (~2%) compared to operating expenses. This low CAPEX is attributed to the chemical-intensive nature of the Fenton process, which requires minimal specialized or expensive equipment (Ameta *et al.* 2018). This highlights the fact that the system's long-term sustainability relies more on reducing ongoing operating costs—especially chemical usage—than on minimizing infrastructure investments. Notably, regional variations in equipment pricing and construction costs could alter these cost proportions, emphasizing the need for localized economic assessments for full-scale deployment.

2.3.2. Operating costs considerations

The OPEX of the Fenton process was dominated by H_2O_2 expenses ($440000 $year^{-1}$), which accounted for 89.9% of annual expenditures ($489225 $year^{-1}$). The high H_2O_2 demand results from the complex matrix of CPWW, containing radical scavengers and non-biodegradable organics, which reduce hydroxyl radical availability and necessitate elevated oxidant doses. $FeSO_4$ contributes 6.1% of OPEX ($29700 $year^{-1}$), reflecting its critical

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catalytic role but relative affordability compared to H_2O_2. The combined costs associated with labor, pH adjustment chemicals (NaOH and H_2SO_4), energy consumption, and routine maintenance collectively represented a marginal 4% of the total OPEX, indicating their minimal impact on the overall operational costs. Considering a COD removal efficiency of 38.2% (equivalent to 1470.7 ppm COD removed from an initial 3850 mg L^{-1}), the total OPEX translates to \$7.413 m^{-3} of treated wastewater, or more specifically, \$5.04 kg^{-1} COD removed. This notably higher operational cost, particularly when compared to reported costs for treating petrochemical wastewaters using the Fenton process (\$1.78 kg^{-1} COD) and ozonation (\$1.96 kg^{-1} COD; Cheng *et al.* 2022), indicates the inherent complexity and heightened resistance of CPWW to oxidative degradation via the Fenton process. This resistance, in turn, contributes to a reduced overall treatment efficiency for this specific wastewater stream. The calculated total OPEX of \$7.41 m^{-3} for the Fenton process in this study, falls within the previously reported range of 0.2 to 17.7 € m^{-3} (approximately 0.23 to \$20.5 m^{-3}, using the exchange rate adopted in this study for 2025) for similar treatment processes (Cañizares *et al.* 2009; Ribeiro & Nunes 2021). This consistency indicates that the observed costs are broadly aligned with the economic landscape of AOPs. A direct comparison of these results with the OPEX of solar photo-Fenton treatment for a synthetic citrus wastewater offers further insight. Guzmán *et al.* (2016) reported an OPEX of 13.8 € m^{-3} (approximately \$16 m^{-3}) for solar photo-Fenton, achieving a higher COD removal efficiency of 76.9%. While the conventional Fenton process employed in our study yielded a lower COD reduction efficiency—attributable to the inherently complex matrix and high initial COD concentration of the real CPWW—it concurrently demonstrated a significantly lower operational expenditure. This trade-off suggests that despite its reduced efficiency for challenging real-world wastewaters, the conventional Fenton process remains a cost-effective option for bulk COD reduction under certain economic considerations.

2.3.3. Total treatment cost considerations

The total annual cost of \$499137 for the Fenton process, designed to treat 66000 m^3 $year^{-1}$ of CPWW, translates to \$7.56 m^{-3} of treated wastewater. Annualized CAPEX (\$9912 $year^{-1}$) constitutes just 2% of total costs, highlighting that long-term sustainability of Fenton process hinges on optimizing reagent efficiency rather than minimizing infrastructure investment. This contrasts with membrane-based systems, where CAPEX predominates due to high equipment costs. Strategies to reduce the total costs associated with Fenton processes largely focus on mitigating the dominant expenses of chemicals and sludge management. Optimizing the ratio of Fe^{2+} to H_2O_2 can help in minimizing chemical consumption and reducing sludge production. Processes like Electro-Fenton and Bio-electro-Fenton offer the potential for *in-situ* production of hydrogen peroxide, which could significantly lower costs by eliminating the need to purchase and transport external H_2O_2 (Asghar *et al.* 2015; Wang *et al*. 2017). Moreover, integrating AOPs like Fenton with biological post-treatment is encouraged as a method to potentially decrease overall treatment costs by enhancing biodegradability.

2.3.4. Sensitivity analysis

To comprehensively evaluate the economic resilience of the Fenton process for CPWW treatment, a detailed sensitivity analysis was performed. This analysis aimed to identify the key cost factors that exert the most significant influence on the system's economic viability, thereby providing crucial insights for decision-makers in selecting cost-effective and sustainable treatment solutions. To achieve this, ten scenarios were examined, each involving a ± 20% fluctuation in five critical factors impacting overall treatment costs: system's lifetime, interest rate, reagent prices (H_2O_2 and $FeSO_4 \cdot 7H_2O$), electricity price, and labor cost. For each scenario, the annualized capital cost, the annual operating cost, and the resultant total treatment cost were systematically recalculated. The subsequent cost variations due to changes in each factor were rigorously analyzed. The graphical representations of these sensitivity results for annualized capital, annual operating, and total treatment costs are presented in Figs. 5, 6, and 7, respectively. The baseline economic parameters used for these evaluations included a 25-year system lifetime, a 0.23 interest rate, a H_2O_2 cost of \$0.8 L^{-1}, a $FeSO_4 \cdot 7H_2O$ cost of \$0.6 kg^{-1}, an electricity cost of \$0.04 kWh^{-1}, and an annual labor cost of \$8500.

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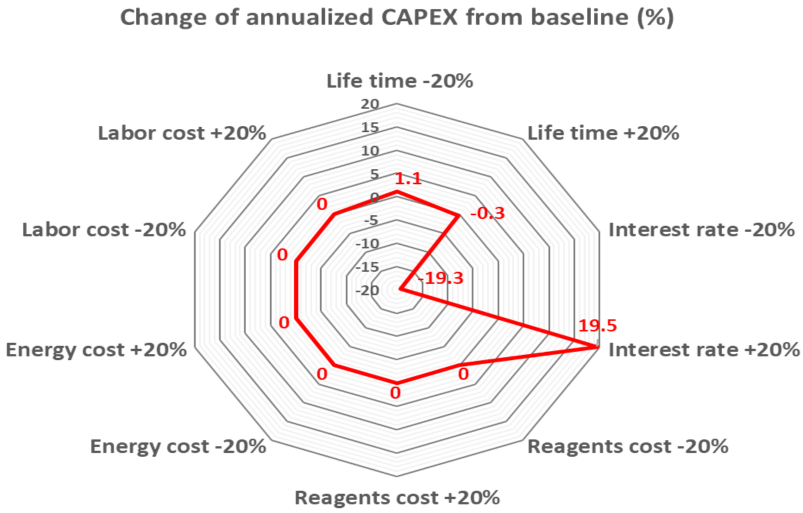

Fig. 5. Sensitivity analysis results of the annualized capital cost for the Fenton-based CPWW treatment plant, showing the impact of a ± 20% variation in five key factors that influence overall costs.

The sensitivity analysis results illustrated in Fig. 5 revealed that annualized CAPEX was primarily influenced by the interest rate. A ± 20% fluctuation in the interest rate led to an approximately ± 19.5% change in annualized CAPEX, ranging from \$0.121 m^{-3} to \$0.179 m^{-3}. Conversely, a ± 20% variation in the system lifetime resulted in a minimal change in annualized CAPEX, specifically ranging from +1.1% to -0.3% (equivalent to \$0.152/m^3 to \$0.149 m^{-3}). Despite these internal sensitivities, the overall impact of CAPEX fluctuations on the total treatment cost of \$7.563 m^{-3} remained negligible, as its proportion is inherently small. The baseline annualized CAPEX was \$0.150 m^{-3}, representing just 2% of the total treatment cost. This finding strongly supported the notion that the Fenton process, being chemical-intensive, requires minimal capital investment in specialized or expensive infrastructure. These results highlighted the sensitivity of CAPEX to financing conditions, particularly interest rates, which directly influenced the capital recovery factor. However, compared to operating expenditure, CAPEX's contribution remains minor, emphasizing that cost optimization efforts should prioritize operational parameters over infrastructure investments. Decision-makers are advised to focus on securing favorable financing terms to mitigate CAPEX impacts, as these adjustments could yield up to 20% reductions in annualized capital costs.

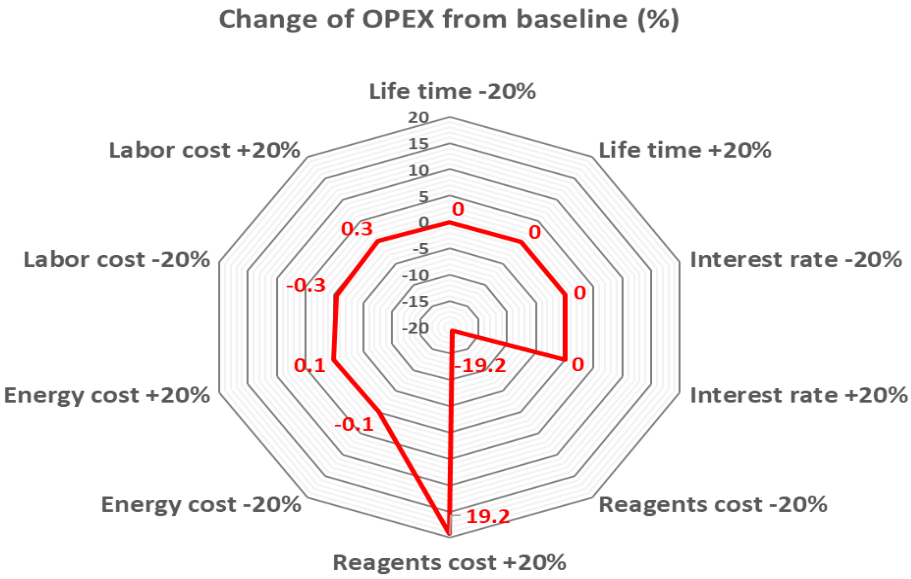

Fig. 6. Sensitivity analysis results of the annual operating cost for the Fenton-based CPWW treatment plant, showing the impact of a ±20% variation in five key factors that influence overall costs.

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Fig. 6 illustrates the sensitivity analysis results for the annual operating cost of the Fenton-based CPWW treatment plant. OPEX, with a baseline of \$7.413 m^{-3}, dominated the cost structure of the Fenton process, accounting for approximately 98% of total treatment costs. Reagent costs, particularly H_2O_2 and $FeSO_4$, were the primary contributors, representing 96% of OPEX. A 20% reduction in reagent prices decreased OPEX from \$7.413 m^{-3} to \$5.9892 m^{-3} (-19.2%), while a 20% increase raised it to \$8.8358 m^{-3} (+19.2%), highlighting their critical role in cost variability. Other operational factors, such as electricity and labor costs, exhibited negligible impacts. For instance, a 20% decrease in electricity price reduced OPEX by only 0.1% (\$7.4018 m^{-3}), and a 20% reduction in labor costs lowered OPEX by 0.3% (\$7.3867 m^{-3}). These findings aligned with literature (Ameta *et al.* 2018; Bello *et al.* 2019) emphasizing the chemical-intensive nature of the Fenton process, where reagent dependency drives the financial burden. To improve economic feasibility, decision-makers are encouraged to pursue strategies to optimize reagent usage, such as hybrid Fenton systems (e.g., photo-Fenton or electro-Fenton), which enhance radical utilization efficiency. Furthermore, pre-treatment to remove radical scavengers could reduce H_2O_2 demand, offering significant cost savings (Asghar *et al.* 2015; Wang *et al.* 2017).

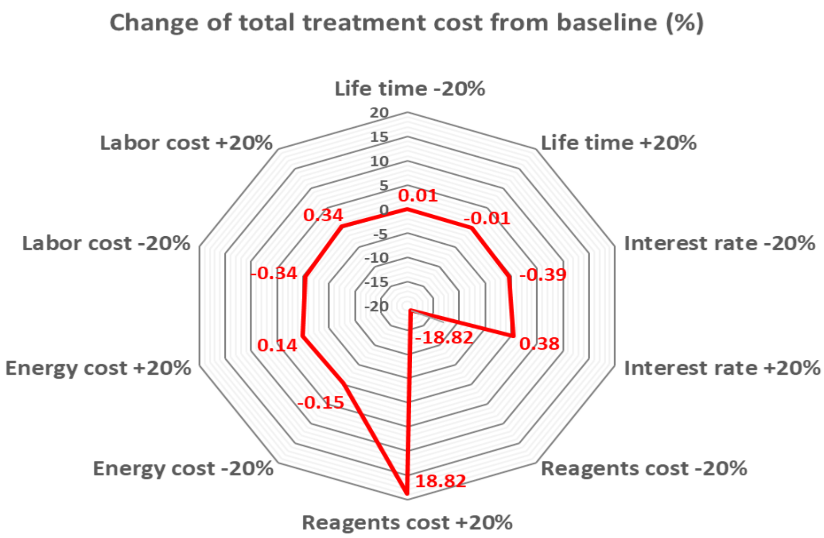

Figure 7. Sensitivity analysis results of the total treatment cost for the Fenton-based CPWW treatment plant, showing the impact of a ± 20% variation in five key factors that influence overall costs.

The total treatment cost integrating both CAPEX and OPEX, provides a comprehensive assessment of the economic viability. At baseline, the total cost was \$7.563 m^{-3}, with OPEX dominating at \$7.413 m^{-3}. The results of the sensitivity analysis for the total treatment cost, as depicted in Fig. 7, indicated that reagent prices exerted the most substantial influence on total costs. A 20% decrease in reagent costs reduced total treatment costs to \$6.139 m^{-3} (-18.8%), while a 20% increase raised them to \$8.986 m^{-3} (+18.8%). Interest rate fluctuations also played a notable role, with a 20% decrease lowering total costs to \$7.534 m^{-3} (-0.39%) and a 20% increase raising them to \$7.592 m^{-3} (+0.38%). In contrast, factors like energy and labor costs had minimal impacts, contributing less than ± 0.35% variation. This analysis highlighted the necessity of addressing reagent costs to achieve meaningful cost reductions. Furthermore, the stability of total costs under varying system lifetimes (+0.01% to -0.01%) suggests that extending system longevity offers limited economic benefits compared to optimizing operational efficiencies. Decision-makers are urged to prioritize reagent cost mitigation strategies, as these represent the most effective lever for enhancing economic resilience.

2.3.5. Decision-Making Guidelines and Recommendations

Based on the sensitivity analysis results, several actionable recommendations emerged to enhance the economic feasibility of the Fenton process for CPWW treatment. First, reagent cost optimization was prioritized, as it accounted for 89.9% of OPEX and exhibited the highest sensitivity (±18.8% impact on total costs). Strategies included negotiating bulk purchasing agreements, exploring alternative suppliers, or adopting hybrid Fenton systems to enhance oxidant efficiency. Second, managing financing conditions was critical, as interest rate

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fluctuations significantly impacted CAPEX (± 19.6%) and total costs (± 0.38%). Securing low-interest loans or government subsidies could mitigate these effects. Third, while system lifetime and energy/labor costs had minimal impacts, extending system longevity through durable materials and energy-efficient designs provided marginal cost savings. Finally, integrating pre-treatment steps to remove radical scavengers could further lower H_2O_2 demand, thereby reducing operational costs. By focusing on these high-impact areas, decision-makers can ensure the sustainable and cost-effective implementation of the Fenton process. This strategy effectively balances technical performance with economic constraints to meet both regulatory and environmental goals.

3. CONCLUSION

The present study comprehensively evaluated the technical and economic feasibility of utilizing the Fenton process for treating real CPWW, a challenging industrial effluent characterized by high organic load and complex matrix composition. Through a meticulously designed response surface methodology-central composite design (RSM-CCD) experiment, the optimal operating conditions for maximizing COD removal were determined as 2500 mg L^{-1} H_2O_2, 750 mg L^{-1} $FeSO_4$, pH 3, and a 90-minute reaction time, achieving a maximum COD removal efficiency of 38.2%. Although this efficiency represents a significant reduction in organic load, it is lower than the higher removal rates (e.g., 61–76.9%) reported for synthetic CPWW in previous studies (Guzmán *et al.* 2015; Guzmán *et al.* 2016). This disparity highlights the challenges posed by the complex matrix of real CPWW, which includes high initial COD levels (3850 ± 150 mg L^{-1}) and interfering compounds that limit hydroxyl radical availability. Statistical analyses, including ANOVA and Pareto charts, rigorously confirmed the significance and interactions of key process parameters, highlighting the dominant role of H_2O_2 concentration and the critical importance of pH control.

The economic analysis revealed that reagent costs, particularly H_2O_2, dominate OPEX, accounting for nearly 90% of the total treatment cost of \$7.563 m^{-3}. A ± 20% fluctuation in reagent prices resulted in a corresponding ±18.8% variation in total treatment costs, underscoring the critical need for strategies to optimize chemical usage. Interest rate fluctuations also significantly impacted annualized CAPEX, with a ± 20% change leading to approximately ± 19.6% variations in CAPEX. Despite these sensitivities, the overall contribution of CAPEX to total costs remained minimal (< 2%), highlighting the Fenton process's low infrastructure dependency compared to other treatment technologies, such as membrane filtration. This cost structure suggests that efforts to enhance the economic viability of the Fenton process should prioritize operational efficiencies over capital investments. Compared to solar photo-Fenton, which achieved 76.9% COD removal at 13.8 € m^{-3} (approximately \$16 m^{-3}; Guzmán *et al.* 2016), the conventional Fenton process offers a more cost-effective option for initial COD reduction, though its lower efficiency suggests it may serve best as a pre-treatment step. Overall, the estimated treatment cost falls between the relatively high costs reported for photo-Fenton processes and the lower costs generally associated with biological treatment technologies.

From a broader perspective, the findings highlight the Fenton process's potential as a sustainable solution for managing agro-industrial wastewater, particularly in regions like Iran where stringent environmental regulations and limited water resources necessitate effective treatment strategies. Nevertheless, the economic analysis reveals limitations in scalability due to the chemical-intensive nature of the process, suggesting that hybrid approaches—such as integrating the Fenton process with biological treatment or electrochemical oxidation—may offer enhanced cost-effectiveness and sustainability.

Several limitations of this study warrant acknowledgment. First, the reliance on laboratory-scale (batch) experiments may constrain the generalizability of the findings to full-scale applications, as operational effects such as continuous flow and equipment cleaning were not considered. Second, the analysis did not account for seasonal variations in CPWW composition, which could influence treatment performance and cost dynamics. Third, a lifecycle assessment (LCA) would further illuminate the process's environmental footprint, supporting its adoption as a sustainable solution. Future research should address these gaps by conducting pilot-scale studies, incorporating real-time monitoring of wastewater characteristics, and exploring integrated treatment systems that leverage synergies between different technologies.

In summary, this study offers three key implications:

- Decision support for industry and regulators: The transparent cost model establishes realistic benchmarks for evaluating the feasibility of Fenton oxidation in citrus wastewater treatment.

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- Identification of economic bottlenecks: Reagent consumption, particularly H_2O_2 (89.9% of OPEX) and $FeSO_4$ (6.1% of OPEX), accounts for the majority of costs, highlighting the need for chemical optimization or substitution.
- Pathways for innovation: The findings justify further exploration of integrated treatment strategies (e.g., coagulation–Fenton, biological–Fenton, or electro-Fenton) to reduce oxidant demand while enhancing pollutant removal.

Overall, this work extends beyond conventional process optimization by coupling laboratory performance with full-scale economic analysis, thereby providing both technical insights and cost-based decision support. The proposed bench-to-budget framework advances the application of advanced oxidation processes, positioning Fenton not as a standalone solution but as a strategic component within hybrid, cost-effective treatment trains for food-industry effluents. The proposed bench-to-budget framework advances the application of advanced oxidation processes, positioning Fenton not as a standalone solution but as a strategic component within hybrid, cost-effective treatment trains for food-industry effluents. This framework can be adapted for other agro-industrial wastewaters by adjusting reagent prices, energy tariffs, and plant capacity.

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Authors' Contributions

All authors read and approved the final manuscript. **Shahram Niazi**: Data curation, Writing – original draft, Visualization, Supervision, Methodology, Conceptualization. **Alireza Pendashteh**: Writing – review and editing, Visualization, Validation, Methodology, Investigation.

Ethical Approval

This is not applicable.

Consent to Participate

This is not applicable.

Consent to Publish

This is not applicable.

Clinical trial number

This is not applicable.

Competing Interests

The authors declare no competing interests.

Data Availability Statement

The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

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