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Sequential electrocoagulation-ozonation treatment: Effect of suspended solids removal on sulfide elimination in tannery wastewater

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ABSTRACT

The treatment of tannery wastewater is a complex environmental challenge. These effluents have high loads of total suspended solids (TSS) and, especially, sulfides, pollutants that require advanced technologies due to their toxicity and difficulty in removal. This study presents and evaluates an integrated electrocoagulation (EC) and ozonation (O_3) system as an alternative for the comprehensive treatment of this effluent. In the EC stage, it was optimized using the response surface methodology (RSM) with a 3^k factorial design, using current intensity (I) and treatment time (t) as factors to maximize TSS removal. The EC results demonstrated high TSS removal efficiency, reaching 97.91% under optimal conditions ($p < 0.05$), which significantly facilitates subsequent treatment. The most critical finding of the study lies in the ineffectiveness of EC for dissolved contaminants: the removal of sulfides was extremely limited, with an efficiency of only 4.2%. In contrast, the subsequent ozonation stage achieved more than 99% elimination of sulfides using a dose of 12 g h^{-1} of ozone after 20 minutes of reaction. This result justifies the need for an integrated system, where EC handles the load of solids (TSS) and other contaminants and ozonation performs the essential chemical oxidation of sulfides. The operating cost of the combined EC–O_3 treatment was determined to be US$ 0.707 m^{-3}, demonstrating the economic viability of the technology. The results confirm that the EC–O_3 system is a synergistic solution for the environmental management of tannery wastewater, enabling compliance with maximum permissible limits.

Keywords: tannery wastewater, electrocoagulation, ozonation, response surface methodology, sulfide removal, advanced oxidation processes.

INTRODUCTION

The tanning industry generates large volumes of wastewater characterized by the presence of toxic and recalcitrant contaminants (Hira et al., 2022). During the transformation of hides into leather, various compounds are used, such as acids, alkalis, chromium salts, tannins, solvents, sulfides, dyes, and auxiliary agents, which results in effluents with highly variable composition (Lofrano et al., 2013).

The tanning process is generally divided into three main stages: beamhouse, tanning, and finishing. In the beamhouse stage, operations such as soaking, liming, and unhairing are carried out to prepare the hides for tanning (Ngobeni et al., 2024). During the liming and unhairing stages, sodium sulfide is used to remove hair from the hides, which generates the release of hydrogen sulfide (Uez et al., 2023). This process produces approximately 5 m^3 of effluent per ton of processed hide (Prabhakaran et al., 2018), which, when stored in lagoons, may undergo anaerobic decomposition, generating compounds such as H_2S and ammonia, which has led to the implementation of environmental regulations to reduce sulfur levels (Shivasankaran et al., 2020; Alheety et al., 2019).

Hydrogen sulfide (H_2S) is highly soluble in water and has a characteristic rotten egg odor, detectable at concentrations between 0.02 and 10 mg L^{-1}, while concentrations above 500 mg L^{-1} can be lethal to humans (Uez et al., 2023; Aroca et al., 2020; Prabhakaran et al., 2018). Likewise, it has been reported that high sulfide concentrations in wastewater generate significant environmental problems, such as odor nuisance, anaerobic conditions, and corrosion (Sameh et al., 2020; Prabhakaran et al., 2018).

Various methods have been employed for the removal of hydrogen sulfide from wastewater, including biological and chemical treatments (Lin et al., 2018; Cen et al., 2023). Although biological processes present economic and environmental advantages, their performance is highly sensitive to variations in temperature and pH, which limits their operational stability (Nielsen et al., 2006). On the other hand, chemical treatments offer greater stability, although they generally involve higher operational costs (Tamersit and Bouhidel, 2020; Cen et al., 2023). In addition, conventional physicochemical treatments, such as precipitation, air stripping, and chemical oxidation, may require high reagent consumption and generate secondary pollutants or significant amounts of sludge, increasing costs and environmental impact (Salazar-Sogamoso et al., 2025; Oladimeji et al., 2024).

Among advanced technologies, electrocoagulation (EC) has demonstrated to be effective in the removal of various contaminants present in tannery effluents, such as chromium, COD, and turbidity (Aguilar et al., 2019; Villalobos Lara et al., 2020). This electrochemical process is based on the in situ generation of coagulants from metallic electrodes, generally iron or aluminum, which allow the destabilization and agglomeration of suspended and dissolved contaminants (Perren et al., 2018; Sarinho et al., 2023). In addition, it integrates coagulation, flocculation, and flotation processes through the formation of hydrogen bubbles at the cathode, facilitating the separation of flocs (Naje et al., 2016). Among its advantages are the in situ generation of the coagulant, lower consumption of chemical reagents, operational simplicity, and lower sludge production compared to conventional coagulation (Tegladza et al., 2021; Huang et al., 2020).

However, several studies have reported limitations of electrocoagulation in the removal of dissolved sulfides (Feng et al., 2007; Şengil et al., 2009). Feng et al. (2007) observed that in tannery wastewater the removal was lower than 12% with aluminum electrodes, while the use of iron electrodes allowed efficiencies higher than 90% to be achieved, due to the formation of black iron sulfide precipitates. Similar results were reported by Şengil et al. (2009), who obtained approximately 90% removal in liming wastewater using iron electrodes under optimal conditions.

Ozone (O_3) is a powerful oxidizing agent used in wastewater treatment for disinfection and the degradation of organic contaminants (Liu et al., 2022). One of its main advantages is that it does not generate secondary sludge and the residual ozone decomposes easily, making it a clean and sustainable process (Joseph et al., 2021). In addition, it can react directly with recalcitrant compounds or indirectly through hydroxyl radicals generated during its decomposition, improving color removal and reducing the organic load (Preethi et al., 2009; Hernández-Ortega et al., 2010; Asaithambi et al., 2012). Likewise, ozonation can effectively oxidize sulfides into less harmful species, such as sulfates, suggesting its potential as a complementary technology to electrocoagulation (Aziz et al., 2016; Pranjal, 2020).

To overcome these limitations, hybrid processes have emerged as a promising strategy. In particular, the integration of electrocoagulation and ozonation (EC–O_3) has been demonstrated to be effective in the removal of recalcitrant compounds in industrial wastewater (Asaithambi et al., 2016; Barzegara et al., 2019), due to the high oxidizing capacity of ozone (von Gunten, 2003; Rekhate and Srivastava, 2020).

Despite these advances, most studies have mainly focused on the removal of organic matter, color, turbidity, or chromium, while the specific behavior of sulfides in integrated EC–O_3 systems has received less attention. Likewise, the differential contribution of each stage (electrocoagulation versus ozonation) in the removal of suspended solids and dissolved sulfide species has not been clearly quantified. Therefore, a research gap persists related to the synergistic evaluation of both processes specifically aimed at optimizing sulfide removal in tannery effluents under controlled operating conditions (Asaithambi et al., 2016; Barzegara et al., 2019).

In this context, the present study aims to evaluate the integration of electrocoagulation and ozonation for the removal of sulfides in tannery effluents, in order to reduce their concentration to comply with the limits established by environmental regulations. For this purpose, Response Surface Methodology (RSM) is employed as an optimization tool, allowing the analysis of the individual and combined performance of both processes and providing technical and economic evidence on the feasibility of an integrated treatment system for effluents with high sulfide concentrations.

MATERIALS AND METHODS

Collection, preparation, and characterization of wastewater

The wastewater used in this study was generated by the Center for Productive Innovation and Technology Transfer of Leather, Footwear and Related Industries (CITEccal) in Peru. Approximately 90 L of effluent were collected from different stages of bovine leather processing during a complete production cycle at the pilot plant. The physicochemical characterization included TSS, COD, BOD, turbidity, pH, conductivity, oils and grease, chromium species, ammoniacal nitrogen, and sulfides. The analyses were performed following Standard Methods for the Examination of Water and Wastewater (23rd edition). The results are presented in Table 1.

Table 1. Physicochemical analysis of the effluent

Parameters	Value	MAV*
Total suspended solids (mg L^{-1})	1820	500
Chemical oxygen demand (mg L^{-1})	5608.3 ± 280.40	1000
Biochemical oxygen demand (mg L^{-1})	1526.3 ± 320.50	500
Ammoniacal nitrogen (mg L^{-1})	188.40 ± 16.96	80
Turbidity (UNT)	1675	-
pH	8.62	6–9
Conductivity (mS)	22.50	-
Chromium (mg L^{-1})	107.87 ± 26.97	10
Hexavalent chromium (mg L^{-1})	<0.005	0.5
Oils and fats (mg L^{-1})	72.1 ± 15.90	100
Sulfides (mg L^{-1})	110.81 ± 11.69	5

Note: *Decreto supremo Nº 010-2019-vivienda.

Integrated treatment system

Figure 1 shows the integrated treatment system: (a) a photograph of the experimental setup and (b) a schematic diagram at laboratory scale. The system consisted of two units operated in series: an electrocoagulation (EC) reactor followed by an ozonation (O_3) contact tank. The effluent treated in the EC reactor was transferred by gravity to the ozonation tank, without recirculation.

Electrocoagulation reactor

A batch-type electrocoagulation reactor made of acrylic with dimensions of 15 × 15 × 20 cm and a working volume of 4.5 L was used. In each experiment, 4.0 L were treated, leaving a free volume for sludge accumulation. The system included eight aluminum electrodes 99.7% purity; (10 × 10 × 0.1 cm), arranged alternately (four anodes and four cathodes) with a spacing of 1 cm, fixed to a slotted support to ensure their stability. The current was supplied by a DC power supply (BK PRECISION 9115, 0–60 A, 0–80 V).

Ozonation reactor and process (O_3)

The effluent treated in the EC process was transferred by gravity to a laboratory-scale ozonation reactor, consisting of a transparent acrylic tank (15 × 15 × 20 cm; working volume: 4.5 L). A working volume of 2 L was used to promote ozone dispersion. The ozone–air mixture was introduced by bubbling at the bottom of the reactor, using three diffusers that generated fine bubbles, promoting gas dispersion and gas–liquid contact during the oxidation process. Although the ozone transfer efficiency was not experimentally determined, this configuration allowed adequate interaction between ozone and the treated effluent. Ozone was generated in situ from ambient air using a generator (PINUS LONGAEVA, model PL-10QBAO; maximum capacity: 12 g h^{-1} O_3). The gas flow was set at 1.0 L min^{-1} using a calibrated rotameter (IBUW®, model WH5). The ozone dose was regulated using a potentiometer with a graduated scale that allows adjustment of the generator output in the range of 0–12 g h^{-1} O_3. The ozone–air gas was introduced into the reactor through three diffusers located at the bottom of the tank, promoting ozone transfer to the effluent.

Experimental methodology

Optimization of the electrocoagulation process

Electrocoagulation (EC) was optimized to maximize the removal of TSS and to condition the effluent for the ozonation stage. Response surface methodology (RSM) was applied using a full

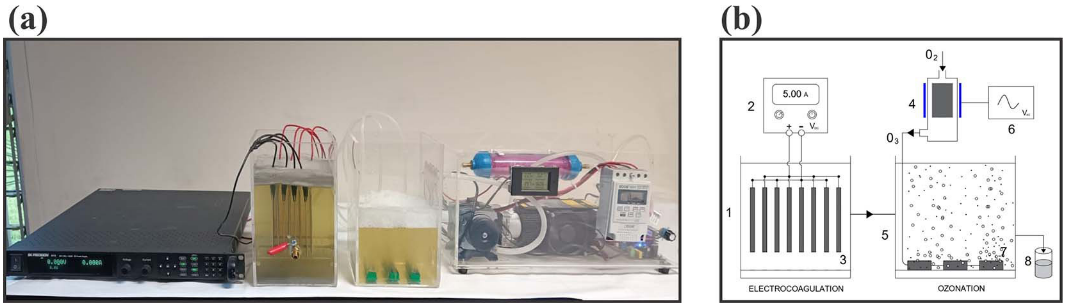

Figure 1. Integrated EC–O_3 treatment system: (a) photograph of the experimental setup, (b) schematic diagram of the system: (1) electrocoagulation reactor, (2) direct current power supply, (3) aluminum electrodes, (4) ozone generator, (5) ozonation tank, (6) AC power supply, (7) diffusers, (8) collector tank

factorial 3^2 design, considering current intensity (4, 6, and 8 A) and treatment time (10, 20, and 30 min) as independent variables. The design included nine experimental combinations performed in duplicate, with the addition of two center points to evaluate reproducibility and experimental error, totaling 20 experiments. Each experiment was conducted with 4 L of real effluent. At the end of the treatment, the samples were allowed to settle for 15 min, and pH and temperature were recorded at the beginning and at the end of each experiment. The operating ranges were defined based on previous studies (Aguilar et al., 2024) and preliminary exploratory tests.

Ozonation for sulfide removal

a) Preliminary tests with synthetic sulfide solution

Ozonation experiments were carried out using a synthetic sulfide solution, prepared by dissolving sodium sulfide nonahydrate ($Na_2S·9H_2O$) in deionized distilled water to obtain an initial concentration of 110 mg L^{-1} of S^{2-}. A volume of 2 L of solution was treated by ozonation at a fixed dose of 8 g h^{-1} O_3. Samples were collected at intervals of 5, 10, 20, 30, and 60 min to evaluate the sulfide oxidation kinetics under controlled conditions.

b) Ozonation of real effluent treated by EC

The real tannery effluent, previously treated by EC under optimal conditions determined by RSM, was subjected to ozonation to improve the quality of the treated water. In the reactor, 2 L of the supernatant were loaded, and ozone doses of 7 and 12 g h^{-1} O_3 were applied with contact times of 5, 10, 20, and 30 min. The ozone generator was stabilized at the selected dose, which was verified by iodometry, after which the bubbling of the ozone–gas mixture was initiated (time zero). Samples were collected at the established times using a syringe through a sealed port and were immediately analyzed for sulfide determination, avoiding subsequent oxidation reactions. The evaluated doses were defined considering the nominal capacity of the generator and were regulated using the equipment potentiometer, in accordance with values reported in the literature for tannery effluents (Aguilar et al., 2024). The sampling intervals were established based on the synthetic solution experiments in order to follow the evolution of sulfide oxidation.

Analytical methods

TSS were quantified in duplicate using a portable Hach DR900 colorimeter, applying the Hach 8006 photometric method pH, conductivity, and temperature were measured in situ using a calibrated multiparameter instrument (Oakton PCS 35). The sulfide concentration was determined according to Standard Methods (APHA, 2017), method 4500-S^{2-} (distillation and flow injection analysis with methylene blue).

The removal percentage of TSS and sulfides was calculated using Equation 1:

$$Y_n = \%R = \left(\frac{C_i - C_f}{C_i}\right) \times 100 \qquad (1)$$

where: C_i, C_f correspond to the initial and final concentrations (mg L^{-1}), respectively.

When the final sulfide concentration was below the analytical detection limit, the removal efficiency was conservatively estimated using the detection limit as C_f, and the result was therefore expressed as a minimum removal efficiency (≥ %).

Experimental design and statistical analysis

Experimental design for the electrocoagulation process

The optimization of the electrocoagulation process was carried out using response surface methodology (RSM) with a full factorial 3^2 design with two factors: current intensity (x_1) and treatment time (x_2). Nine randomized experiments were performed with one replicate and two center points (20 experiments in total) to evaluate experimental variability and model adequacy (Table 2). The relationship between the independent variables and the response (Y, % TSS removal) was modeled using a second-order polynomial equation, Equation 2:

$$Y = \alpha_0 + \sum_{i=1}^{n} \alpha_i X_i + \sum_{i=1}^{n} \alpha_{ii} X_i^2 + \\ + \sum_{i=1}^{n} \sum_{j=i+1}^{n} \alpha_{ij} X_i X_j \quad (2)$$

where: X_1, X_2 are the independent variables, and α represents the regression coefficients. The validity of the model was evaluated by analysis of variance (ANOVA) with a significance level of $p < 0.05$ and its fit was assessed using the coefficients R^2 and R^2_{adj}. Statistical analysis and optimization were performed using Statgraphics Centurion XVIII, and graphs were generated using MATLAB (R2024a).

Experimental design for the removal of sulfides by ozonation

The kinetic behavior of sulfide removal during ozonation was evaluated using a pseudo-first-order exponential model, $C(t) = a\, e^{-kt} + c$ where $C(t)$ is the concentration as a function of time, a is the adjusted initial concentration, k is the apparent rate constant, and c is the residual concentration. When final concentrations were below the analytical detection limit (0.019 mg L^{-1}), this value was adopted as the residual concentration (c = 0.019mg L^{-1}) for a conservative estimation of the process. Data fitting was performed using nonlinear least squares in MATLAB, obtaining the kinetic parameters and the coefficient of determination (R^2). Additionally, the half-removal time $t_{1/2}$ = ln (2)/k was calculated as a comparative parameter for the evaluated ozone doses (7 and 12 g h^{-1} O_3). The pseudo-first-order model has been widely applied in the oxidation of reduced compounds by ozone in aqueous phase (Umbarila-Ortega et al., 2019; von Gunten, 2003).

RESULTS AND DISCUSSION

Characteristics of tannery effluent and their implications for treatment

As shown in Table 1, the tannery effluent presents high concentrations of COD (5608.3 mg L^{-1}), BOD (1526.3 mg L^{-1}), and TSS (1820 mg L^{-1}), as well as elevated levels of sulfides (110.81 mg L^{-1}) and total chromium (107.87 mg L^{-1}), exceeding permissible limits and limiting the use of individual treatments. The high TSS concentration reduces ozonation efficiency by restricting mass transfer and promoting the non-productive consumption of the oxidant. In contrast, in electrocoagulation, TSS promote coagulation–flocculation by increasing collisions and floc formation (Gągol et al., 2019; Wang et al., 2020). However, this process shows low efficiency in the removal of dissolved species such as sulfides in complex matrices (Karlsen et al., 2021). Therefore, an integrated system is justified, in which electrocoagulation acts as a conditioning stage, while ozonation enables efficient oxidation of dissolved sulfides.

Optimization of the electrocoagulation process

The response surface methodology (RSM) experimental 3^2 design was used to optimize the removal of TSS, considering the factors Current Intensity (x_1), evaluated at 4, 6, and 8 amperes, and Treatment Time (x_2), evaluated at 10, 20, and 30 minutes. The experimental results (Table 2) showed that TSS removal efficiency ranged from 72% to 98%, demonstrating a strong dependence on these operational parameters. Analysis of variance (Table 3) indicated that current intensity (x_1), and treatment time (x_2), have a significant effect on TSS removal (p < 0.05), while the overall model was highly significant (F = 23.72; p < 0.0001). The high model fit (R^2 = 97.12% and adjusted R^2 = 95.79%) indicates a high level of reliability. The obtained quadratic model is presented in Table 3.

Figure 2 shows the 3D and 2D response surfaces, which allow visualization of the main

Table 2. Experimental design

Run	Blk	Current intensity (A)	Time (min)	TSS removal (%)		Run	Blk	TSS removal (%)	
		x_1	x_2	Exp	Pred			Exp	Pred
1	1	4	10	72	73.47	11	2	73	73.17
2	1	4	20	91	89.06	12	2	90	88.61
3	1	4	30	94	95.56	13	2	95	94.26
4	1	6	10	81	81.91	14	2	82	81.61
5	1	6	20	94	93.72	15	2	93	93.42
6	1	6	30	97	96.74	16	2	96	96.44
7	1	8	10	91	88.91	17	2	89	88.76
8	1	8	20	96	97.24	18	2	94	96.94
9	1	8	30	98	96.64	19	2	97	96.34
10	1	6	20	93	93.72	20	2	95	93.42

Table 3. Analysis of variance (ANOVA) and summary of the quadratic model for total suspended solids (TSS) removal

Source	Sum of Square	df	F-value	p-value	Significance
Model	272.97	5	23.72	<0.0001	Significant
x_1 : Current Intensity (A)	35.5008	1	402.64	0.0000	Significant
x_2 : Time (min)	43.5102	1	493.48	0.0000	Significant
x_1x_2	9.79031	1	111.04	0.0000	Significant
x_1^2	0.133172	1	1.51	0.3909	Not significant
x_2^2	0.165943	1	1.88	0.0000	Significant
Residual	0.09248	13			
Model fit	$R^2 = 97.12\%\,; Adj - R^2 = 95.79\ \%\,; p < 0.0001$				
Quadratic model	$y = 35.7619 + 6.99405x_1 + 3.22381x_2 – 0.18125x_1x_2 - 0.160714x_1^2 – 0.0439286x_2^2$				

effects and interactions between the variables (Dil et al., 2019), and presents the correlation between the experimental and predicted values for TSS removal, showing good agreement around the regression line. The positive linear coefficients confirm that TSS removal increases with current intensity and treatment time. The optimal conditions predicted by the model were 6 A and 24.3 min, with a maximum TSS removal of 97.91%.

Individual effect of operating parameters on EC

Effect of current intensity

Current intensity is a critical parameter in electrocoagulation, as it determines the amount of coagulant (Al^{3+} ions) generated according to Faraday's law. The increase in TSS removal efficiency when increasing the current from 4 A to 6 A (from 81% to 98%) is directly attributed to the greater release of Al^{3+} ions at the anode and the consequent formation of $Al(OH)_3$, which agglomerates suspended solids (Sandeep Kumar Patel et al., 2025; Mousazadeh et al., 2021; Deveci et al., 2019). This finding is consistent with that reported by (Villalobos-Lara, 2020), who obtained TSS removal efficiencies greater than 90% using electrocoagulation. On the other hand, (Kundu et al., 2024) achieved an SST removal efficiency of 73.12%, while (Varant et al., 2014) reported an efficiency of 85%.

Effect of treatment time

Similarly, treatment time showed a significant effect. Table 3 and Figure 2 show that TSS removal increased as the duration was extended from 10 to 30 minutes, reaching values up to 98%. In this case, a treatment time of 20 to 30 minutes appears adequate to achieve removal efficiencies above 90%. This behavior is explained by the fact that longer electrolysis times allow greater electrode dissolution and a more prolonged interaction between flocs and contaminants (Boinpally et al., 2023; Amitesh, 2022).

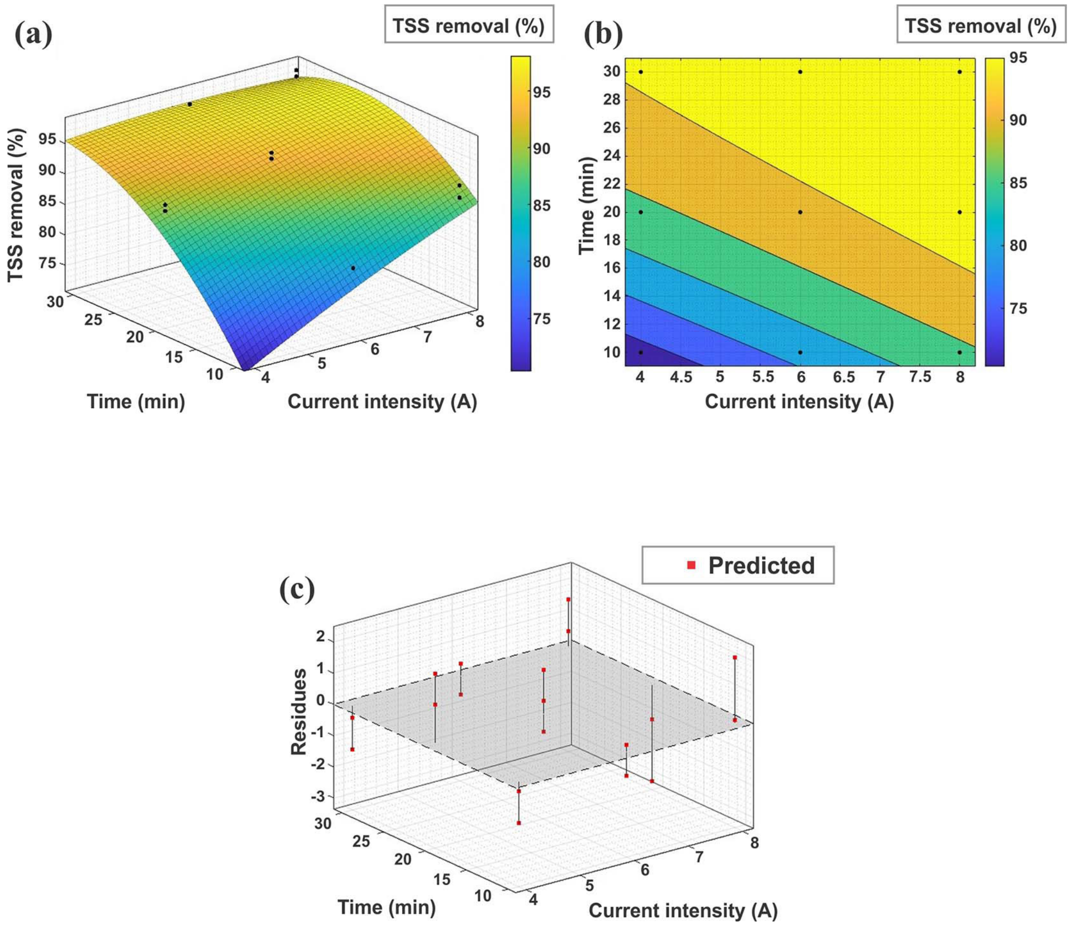

Figure 2. (a) 3D and (b) 2D response surfaces showing the effects and interactions of current intensity and treatment time on TSS removal; (c) correlation between experimental and RSM-predicted values

However, as noted by (Sriram et al., 2023), once the optimal time is reached, efficiency tends to stabilize due to saturation of the floc adsorption capacity. This agrees with the observations in this study, where after 24 minutes no further increase in efficiency was observed.

Ozonation for sulfide removal

Preliminary tests with synthetic sulfide solution

The ozonation of a synthetic sulfide solution (110.4 mg L^{-1}) reduced the concentration to 89 mg L^{-1} at 10 min and to 27 mg L^{-1} at 20 min, using an ozone dose of 8 g h^{-1} O_3 (Figure 3). From 30 min onward, sulfides were below the detection limit (< 0.0019 mg L^{-1}), indicating their complete removal within the analytical sensitivity of the method. These results confirm the high efficiency of ozone in simple matrices, where the absence of interfering compounds favors oxidation.

a) Kinetic analysis and fitting of the pseudo-first-order model (synthetic solution)

The model $C(t) = a\,e^{-kt} + c$ fitted for the experiment with synthetic water (8 g h^{-1} O_3) and fixing c = 0.0019 mg L^{-1}, allowed obtaining a kinetic constant $k = 0.060$ min^{-1} ($t_{1/2} \approx 11.4$ min). The parameter a (≈ 125.3 mg L^{-1}) represents the adjusted initial concentration, while $R^2 = 0.90$ indicates an acceptable fit. A minimum sulfide removal of ≥ 99.2% was achieved in 30 min (Figure 3).

Ozonation of actual effluent treated with EC

The ozonation of real tannery effluent (106.5 mg L^{-1}) showed a sulfide decrease dependent on the ozone dose (Figure 4). With 7 g h^{-1} O_3, the concentration was reduced to values below the detection limit (< 0.019 mg L^{-1}) at 20 min, remaining stable up to 30 min. When the dose was increased to 12 g h^{-1} O_3, the removal was faster, reaching 40.5 mg L^{-1} at 10 min and non-detectable levels at 20 min, evidencing the positive effect of the dose on oxidation kinetics. In both cases, the final concentrations widely complied with the permissible limit of 5 mg L^{-1} for

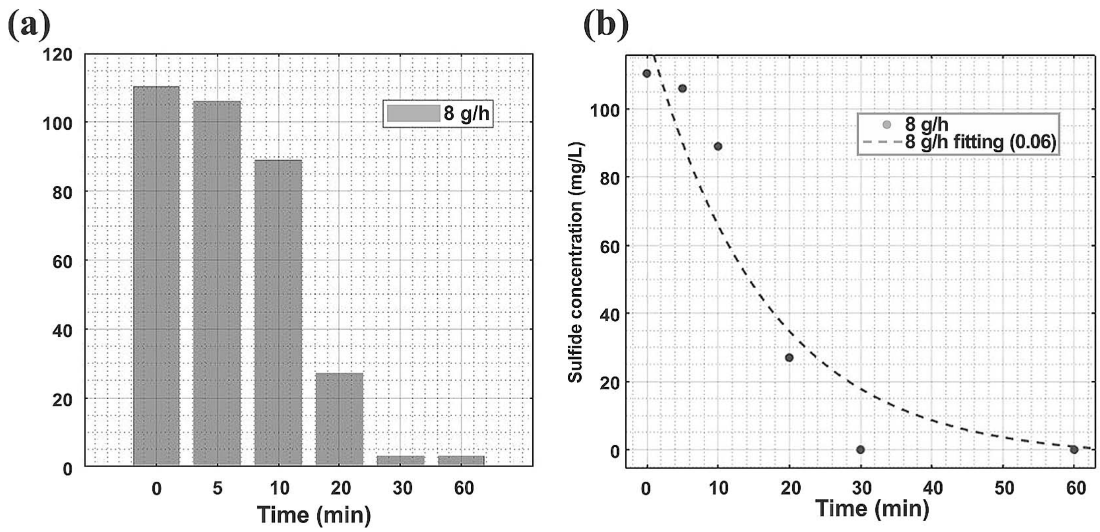

Figure 3. (a) Sulfide removal in synthetic solution at an ozone dose of 8 g h^{-1} O_3; (b) Fitting of the pseudo-first-order kinetic model for sulfide degradation

industrial discharges, and the removal efficiency can be conservatively expressed as ≥ 99%, considering the detection limit of the method. These results confirm the high oxidizing capacity of ozone in real tannery wastewater, in agreement with previous studies reporting higher efficiency at higher ozone doses in complex effluents (Umbarila-Ortega, 2019; Rekhate and Srivastava, 2020).

a) Kinetic analysis and adjustment of the pseudo-first-order model (actual post-EC effluent)

The pseudo-first-order kinetic model showed an exponential decrease of sulfides for both evaluated doses (7 and 12 g h^{-1} O_3), reaching values close to the residual concentration (0.019 mg L^{-1}) at 20 min (Figure 5). The obtained kinetic parameters show that increasing the ozone dose significantly increases the kinetic constant k (from 0.03860 to 0.09671 min^{-1}) and reduces the half-removal time from 17.96 to 7.17 min, approximately 2.5 times. These results indicate higher kinetic efficiency at higher ozone doses, in agreement with previous studies reporting an increase in the availability of oxidizing species and in the degradation rate of sulfur compounds in aqueous solution.

Effectiveness of the integrated EC–ozonation system for sulfide removal

Electrocoagulation showed a limited capacity to remove dissolved sulfides, with an efficiency

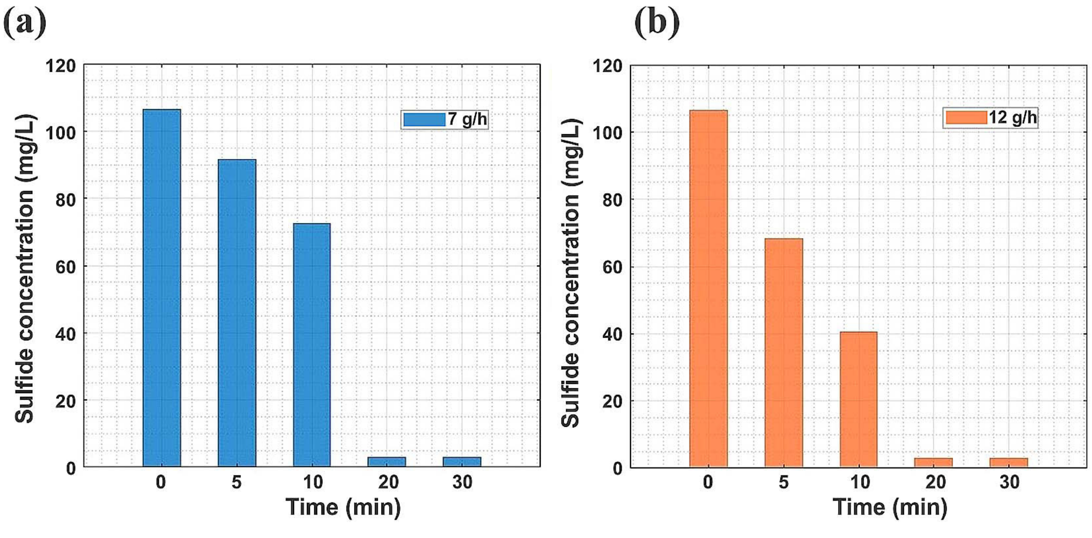

Figure 4. Removal of sulfides from tannery effluent pretreated with EC at dosis: (a) 7g h^{-1} O_3, (b) 12 g h^{-1} O_3

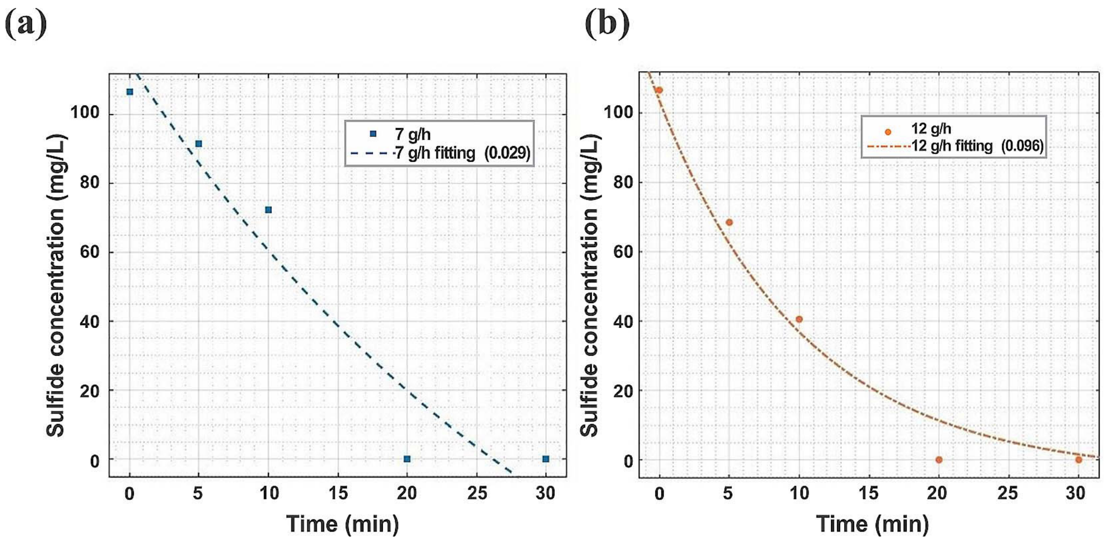

Figure 5. First-order exponential model fit for the ozonation of sulfides in real effluent: (a) 7 g h^{-1} O_3, (b) 12 g h^{-1} O_3

of only 4.2% (110.8 to 106.09 mg·L^{-1}), in contrast to its high effectiveness in TSS removal (97.5%). This behavior is explained by the process mechanisms – coagulation–flocculation and sweep flocculation by metal hydroxides – which are effective for particulate species but less efficient for dissolved contaminants such as sulfides (Chen, 2004; Jing et al., 2021). From a process design perspective, electrocoagulation acts as a pretreatment that improves effluent conditions for subsequent oxidation processes, since TSS removal reduces turbidity and enhances ozone mass transfer. In the real effluent pretreated with EC, ozonation achieved sulfide removals ≥ 99% within 20 min for both doses (7 and 12 g h^{-1} O_3). This high efficiency is attributed to the strong oxidizing potential of ozone (E° = 2.07 V) and the combined action of direct oxidation (O_3) and indirect oxidation via hydroxyl radicals (•OH). The alkaline pH of the effluent (≈ 8.6) favored ozone decomposition and radical formation, with the indirect pathway predominating, in agreement with the literature (Bracamontes-Ruelas et al., 2022; Wang et al., 2020).

From a mechanistic point of view, sulfide ozonation is controlled by its speciation. The reaction of the HS^- ion with ozone is extremely fast ($k \approx 3 \times 10^9$ M^{-1} s^{-1}), whereas H_2S reacts several orders of magnitude more slowly ($k \approx 3 \times 10^4$ M^{-1} s^{-1}). Since the pKa of H_2S is close to 6.9, the HS^- species predominates at alkaline pH values typical of wastewater, thus controlling the overall kinetics of the process (Mark et al., 2011). Under conditions dominated by HS^-, complete oxidation to sulfate can be represented in a simplified form as Equation 3:

$$HS^- + 4\,O_3 \rightarrow SO_4^{2-} + 4\,O_2 + H^+ \quad (3)$$

Although a simplified approach based on oxygen transfer suggests a demand of 4 mol of O_3 per mol of sulfate formed, experimental observations indicate an apparent consumption closer to 2.4 mol of O_3 per mol of SO_4^{2-} generated, evidencing that the actual ozone demand may deviate from the ideal stoichiometry depending on the reaction pathways (Mark et al., 2011). Assuming complete conversion of sulfide (mg L^{-1}) to sulfate, the maximum theoretical increase of SO_4^{2-} would be approximately three times the initial sulfide concentration, according to the molar mass ratio (96.06/32.06). This value establishes an upper limit and highlights the importance of evaluating sulfur speciation and mass balance in future studies. In comparison with previous studies, the integrated EC–O_3 system showed competitive performance, achieving sulfide removals ≥ 99% at shorter contact times and without the use of additional adsorbent materials, surpassing efficiencies reported for ozonation 97.4% and ozone–activated carbon systems 98% (Kadhim et al., 2021; Lagha et al., 2014). The integration of both technologies allowed electrocoagulation to act as a pretreatment through TSS removal, enhancing ozone mass transfer, while ozonation ensured high efficiency in sulfide removal. This behavior is consistent with previous studies highlighting the superiority of hybrid processes in the treatment of tannery effluents (Asaithambi et al., 2016; Aguilar et al., 2024), which indicate that hybrid processes outperform individual technologies in tannery wastewater treatment. From an industrial application perspective, these results reinforce the potential of hybrid EC–O_3 systems as viable alternatives for the advanced treatment of tannery effluents, especially in scenarios that require high removal efficiencies and reduced hydraulic residence times. From an environmental perspective, the possible formation of oxidized sulfur species during ozonation should be considered, since sulfate can also originate from inputs used in the tanning process (Galiana-Aleixandre et al., 2005; 2011). In the Peruvian context, organic acids were used as an alternative to regulated sulfuric acid (IQBF). Likewise, it is recommended to complement the evaluation of the process with sulfur speciation and ecotoxicity analyses for a comprehensive assessment of the effluent. Additionally, the integrated system showed high efficiency in total chromium removal, reducing its concentration from 107.88 to 0.24 mg L^{-1} (99.78%), mainly during electrocoagulation through precipitation and adsorption onto metal hydroxides generated in situ (Mollah et al., 2001; Jo et al., 2024). In contrast, COD (37.9%) and BOD (31.3%) reductions were moderate, which is consistent with physicochemical processes where electrocoagulation mainly removes the suspended fraction, while ozonation favors the oxidation of specific species such as sulfides rather than the complete mineralization of organic matter (Asaithambi et al., 2024). Overall, these results indicate that the EC–O_3 system is highly efficient for metals, solids, and sulfides, with a partial contribution to the reduction of the organic load.

Operating costs of the electrocoagulation-ozonation system

The operating costs of the integrated electrocoagulation–ozonation (EC–O_3) process were estimated considering electrical energy consumption, electrode consumption, and the energy demand of the ozone generator. The total operating cost was calculated as the sum of the contributions of each treatment stage, Equation 4:

$$C_{T(EC\text{-}O_3)} = C_{T(EC)} + C_{T(O_3)} \qquad (4)$$

Operating cost of electrocoagulation

The operating cost of EC included aluminum electrode consumption and electrical energy consumption, according to Equation 5:

$$C_{T(EC)} = \rho_{electrodes}\, \omega_{electrodes} + + \rho_{energy}\, \beta_{energy} \qquad (5)$$

where: $\rho_{electrodes}$ = aluminum price (US\$ kg^{-1}), $\omega_{electrodes}$ = electrode consumption (kg m^{-3}), ρ_{energy} = cost of electricity (US\$ kWh^{-1}) y $\beta_{electrodes}$ = specific energy consumption (kWh m^{-3}).

The specific energy consumption was calculated using Equation 6, considering the applied voltage, current intensity, treatment time, and the volume of treated water. Under the optimal conditions obtained by response surface methodology (6 A and 24.3 min), the energy consumption per treated volume was minimized while simultaneously maintaining high TSS removal efficiency.

$$\beta_{energy} = \frac{U \times I \times t}{V \times 1000} \qquad (6)$$

Operating cost of ozonation

The cost of ozonation was estimated based on the electrical consumption of the ozone generator and was expressed relative to the energy consumption of electrocoagulation, Equation 7. The results showed that ozonation represented approximately 17% of the energy consumption of electrocoagulation.

$$C_{T(O_3)} = \alpha\, C_{T(EC)} \qquad (7)$$

Considering local prices in Peru (October 2025), an electricity cost of 0.201 US\$ kW h^{-1} and an aluminum price of 2.615 US\$ kg^{-1} were used. The total operating cost of the integrated EC–O_3 system was estimated at 0.707 US\$ m^{-3}, distributed as follows: EC energy consumption (84%), electrode consumption (2%), and ozonation system energy consumption (14%).

The operating cost obtained in this study (0.707 US\$ m^{-3}) falls within the range reported for electrocoagulation systems in the literature (0.21–0.88 US\$ m^{-3}), depending on the electrode material, wastewater characteristics, and operating conditions (Ahangarnokolaei et al., 2021; Débora et al., 2022; Villalobos et al., 2020; Bingul et al., 2023).

CONCLUSIONS

This study confirms that an integrated electrocoagulation–ozonation system is effective for removing TSS and sulfides present in tannery wastewater. The RSM model applied to the electrocoagulation stage showed a good fit ($R^2 = 0.9712$) and allowed the prediction of optimal conditions of 6 A and 24.3 min. These conditions were experimentally validated, achieving a TSS removal efficiency of 97.5%. Electrocoagulation proved to be highly effective for TSS removal but exhibited very poor performance in sulfide removal (less than 5%), confirming its primary role as a physicochemical pretreatment step. Ozonation was demonstrated to be a highly effective complementary process, achieving sulfide removal efficiencies exceeding 99% in less than 20 min at ozone doses of 7 g h^{-1} and 12 g h^{-1}. The integrated electrocoagulation–ozonation treatment enhances sulfide removal and therefore optimizes the ozonation process. Additionally, it was found to be economically viable, with an estimated operating cost of 0.707 US\$ m^{-3}.These results not only demonstrate the effectiveness of the process but also confirm compliance with the maximum allowable values (MAV) for sulfides (< 5 mg L^{-1}). Additionally, the raw tannery wastewater exhibited high initial pollutant concentrations, including BOD (1526.30 mg L^{-1}), COD (5608.30 mg L^{-1}), and chromium (107.88 mg L^{-1}), confirming the severity of contamination. The integrated treatment achieved overall removal efficiencies of 31.3% for BOD, 37.9% for COD, and 99.78% for chromium, highlighting the strong performance of electrocoagulation for heavy metal removal and the complementary contribution of ozonation in improving overall effluent quality. The results indicate that the sequential integration of electrocoagulation and ozonation constitutes a technically viable alternative for the advanced treatment of tannery effluents. This study was conducted at laboratory scale under controlled operating conditions; therefore, long-term operational stability and large-scale implementation were not evaluated. Future studies should address continuous operation, electrode durability, and system scalability.

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