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Oxidation, coagulation and filtration processes for the removal of sulfides and organic matter from the effluents of a tannery

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ABSTRACT

Currently, the tanneries in the Río Seco Industrial Park discharge their liquid effluents into the sewer system, which then flows into the Añashuayco Stream, passing through nearby settlements, the Sillar tourist route, and agricultural areas. Finally, it enters the Chili River via the San Jacinto Stream, impacting agriculture in the lower Chili River Basin. In response to this problem, an alternative study is underway to achieve effluent treatment through oxidation, coagulation, and filtration processes. Two wastewater treatments were carried out. The first involved varying pH values and doses of H_2O_2 and $FeCl_3$, with a 24-hour sedimentation period followed by filtration. The treatment resulted in filtration, achieving 97% and 99% removal of COD and sulfides, respectively. The pH was not significant, but the H_2O_2 dose did influence the removal of sulfides and COD. In a second treatment, a combination of peroxone and H_2O_2/O_3 was applied, varying the sample concentration and H_2O_2 dosage while maintaining a constant pH of 8.5, flow rate, and O_3 concentration of 1000 mg/L. Oxidation was carried out for one hour, achieving COD removals of 81.7% and sulfide removals of 93.8%. Both treatments proved efficient for sulfide removal, but the peroxone treatment did not efficiently remove COD. It was concluded that both treatments remove sulfides below permissible levels, but the peroxone treatment uses less H_2O_2. The results obtained warrant further studies using oxygen for the simultaneous removal of sulfides and COD.

Keywords: chemical treatment, environmental, tanneries.

Processos de oxidação, coagulação e filtração para remoção de sulfetos e matéria orgânica dos efluentes de um curtume

RESUMO

Atualmente, os curtumes do Parque Industrial Río Seco vertem seus efluentes líquidos na rede de esgoto, que, por sua vez, deságua no córrego Añashuayco, atravessando assentamentos próximos, a rota turística de Sillar e áreas agrícolas. Finalmente, chega ao Rio Chili pelo córrego San Jacinto, impactando a agricultura na bacia do baixo Rio Chili. Em resposta a esse problema, está em andamento um estudo alternativo para o tratamento de efluentes por meio de processos de oxidação, coagulação e filtração. Dois tratamentos de águas residuais foram realizados. O primeiro envolveu a variação dos valores de pH e das doses de H_2O_2 e $FeCl_3$, com um período de sedimentação de 24 horas seguido de filtração. O tratamento por filtração resultou em remoção de 97% e 99% de DQO e sulfetos, respectivamente. O pH não foi significativo, mas a dose de H_2O_2 influenciou a remoção de sulfetos e DQO. Em um segundo tratamento, aplicou-se uma combinação de peroxona e H_2O_2/O_3, variando-se a concentração da amostra e a dosagem de H_2O_2, mantendo-se constantes o pH em 8,5, a vazão e a concentração de O_3 em 1000 mg/L. A oxidação foi realizada durante uma hora, atingindo remoções de DQO de 81,7% e de sulfeto de 93,8%. Ambos os tratamentos se mostraram eficientes na remoção de sulfeto, mas o tratamento com peroxona não removeu a DQO de forma eficiente. Concluiu-se que ambos os tratamentos removem os sulfetos abaixo dos níveis permitidos, mas o tratamento com peroxona utiliza menos H_2O_2. Os resultados obtidos justificam a realização dos estudos adicionais utilizando oxigênio para a remoção simultânea de sulfetos e DQO

Palavras-chave: ambiental, curtumes, tratamento químico.

1. INTRODUCTION

In Europe, leather exports are on the rise, with Italy accounting for 23.9% of tanned products (Fernández *et al.*, 2022). In Latin America, the production of tanned hides and skins for export has increased, particularly in Brazil and Argentina. Demand for tanned hides is increasing the use of chemicals such as sulfides, chromium, and anilines to obtain a pickling and fine texture in the leather (Almirón *et al.*, 2023).

In Peru, the cities of Trujillo, Lima, and Arequipa are the most developed in the tanning industry (Monge *et al.*, 2023). Currently, the Río Seco Industrial Park (PIRS), Arequipa, Peru, has a large number of tanneries that generate effluents with toxic waste that contain sulfides that give rise to unpleasant odors, ammonium salts, formaldehyde, among other chemicals (De Vettori Dorador *et al.*, 2022). It also has a high organic load composed of hair, epidermis, non-collagenous proteins, and fat extracted from hides and raw hides, generated mainly at the riverside stage (Andriamanohiarisoamanana *et al.*, 2024).

PIRS effluents carry sludge, suspended solids, putrescible organic matter and organic polymers that exceed the maximum contaminant level (MCL) (Peru, 2023). They move forming a stream that flows into the Añashuayco Stream, passing through the surrounding human settlements, the sillar tourist route and agricultural areas, finally entering the Chili River through the San Jacinto ravine, affecting humanity's cultural heritage, the working environment of ashlar quarry workers, and agriculture in the lower Chili River Basin (Almirón *et al.*, 2023).

Based on the above, this research aimed to evaluate the oxidation, coagulation and filtration processes for the removal of sulfides and organic matter and the peroxone technique and to propose an alternative treatment after evaluating which is the most efficient from a quantitative point of view of removal, analyzing the economic viability of the process for its application in tanneries, seeking to reduce the emission of pollutants into the environment.

The wastewater from the riverbank stage contains 60-70% of the pollutants generated in leather tanning, containing COD, BOD, soluble sulfide and total suspended solids. In a typical liming process, for every 1000 kg of raw leather, between 5000 and 7000 cubic meters of total effluents are produced (Anik Hasan *et al.*, 2022).

In the quantitative analysis of research and trends in tannery wastewater treatment technologies, they presented information from the studies with the greatest trend in tannery wastewater treatment, highlighting the biological treatment processes: Phytoremediation, artificial wetlands (Karam *et al.*, 2021), anaerobic treatment, chemical treatment, coagulation, flocculation, and advanced oxidation processes, using powerful oxidizing agents to treat wastewater, especially recalcitrant organic pollutants, including methods such as ozone and Fenton (Li *et al.*, 2022), UV radiation, and nanofiltration (Cao *et al.*, 2021). Conventional biological processes do not remove refractory organic substances (Pasciucco *et al.*, 2025).

For the oxidation of sulfides in wastewater with hydrogen peroxide, they used hydrated ferric oxide as a catalyst, and high sulfide oxidation efficiency was demonstrated by increasing the peroxide concentration, catalyst, and temperature (Ahmad *et al.*, 2009). Hashem *et al.* (2016) treated liming wastewater with oxidation, coagulation, and filtration techniques using hydrogen peroxide to remove hydrogen sulfide and aluminum sulfate as coagulant. They started from untreated effluents with sulfide concentrations of 7285.2 mgL^{-1}, pH 12.5, and EC 40.5 mS. After treatment, the sulfide content was reduced to 81.9 mgL^{-1}, pH 6.9, and EC 5.8 mS. The sulfide and EC removal percentages were 99% and 87%, respectively. In the Fenton reaction, the hydroxyl radical is a non-selective chemical oxidant capable of almost completely oxidizing non-biodegradable organic compounds (Cruz *et al.*, 2021). In the sulfide removal process, the redox reaction (Pudi *et al.,* 2022) let Fe^{3+} ion reduce to Fe^{2+} by the action of the hydroxyl anion (Wang *et al.,* 2019; Tian *et al.,* 2025).

$$H_2S + 2Fe^{3+} + 2OH^- \leftrightarrow S + 2Fe^{2+} + 2H_2O$$

In reactions in alkaline media, oxidation occurs according to the following Equation 1.

$$S^{2-} + 4H_2O_2 \rightarrow SO_4^{2-} + 4H_2O \quad (1)$$

In an acidic medium, Fe^{2+} acts as catalysts, breaking the H_2O_2 bonds, as occurs in the following reactions (Arka *et al.*, 2022).

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + \bullet OH + OH^-$$

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + HO_2\bullet + H^+$$

$$H_2O_2 + \bullet OH \longrightarrow HO_2\bullet + H_2O$$

$$O_2 + 2H^+ + 2e^- \longrightarrow H_2O_2$$

$FeCl_3$ and H_2O_2 are effective components to remove organic matter, turbidity, heavy metals and odors in an efficient and functional manner (Pastrana-Pacho *et al.*, 2023), they act in a wide pH range and do not generate hazardous residues found in homogeneous processes (Cruz *et al.*, 2021; Zeng *et al.*, 2017).

The Pourbaix diagram presents the sulfur species in an aqueous medium at 25 C° considering the electrochemical balance and stability with respect to pH (Patel *et al.*, 2019). Eh-pH diagram of the S-H_2O system at 25°C according to Figure 1.

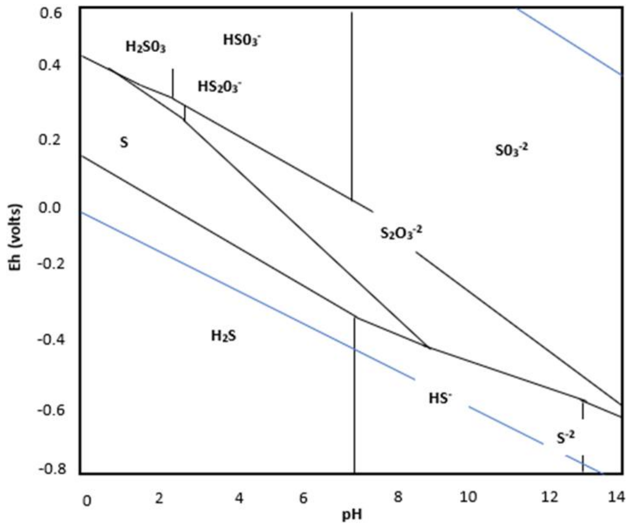

Figure 1. Applied research in mineral processing and hydrometallurgy (Carrillo Pedroza *et al.*, 2017).

2. MATERIAL AND METHODS

2.1. Methodology

Wastewater was sampled from the riverbank process of a tannery located in the PIRS in the city of Arequipa. The sampling and analysis of COD and sulfides was carried out according to the standard methodology (APHA *et al.*, 2017). The analyses were carried out in the Certificaciones del Perú laboratory. Sulfides were analyzed using a titration based on the reaction of iodine with sulfide in acid solution (El Brahmi and Abderafi, 2021). COD is defined as the amount of a specific oxidant that reacts with a sample under controlled conditions.

The amount of oxidant consumed was expressed in terms of its oxygen equivalence, mg O_2 L^{-1} (Mahmoud *et al.*, 2020). The colorimetric method was based on the oxidation of organic matter by a strong oxidant such as dichromate. The orange Cr^{+6} present in the analysis solution is reduced to green Cr^{+3}, and the absorbance was measured at 600 nm (Naguib *et al.,* 2024). For sulfide and COD analysis, pH, EC, and turbidity were determined in the field. The procedure was carried out according to Figure 2 COD, pH, EC, and turbidity were determined in the field.

To determine the effect of pH and oxidant dose, experimental design 2^2 was used, in which there are 2 factors and 2 levels for each factor.

2.2. Characterization and treatment

The effluent was characterized by measuring pH, turbidity, COD and sulfides (García Juárez *et al.*, 2022), then the pH value was regulated to 7.5 and 9.5 with HCl 6 M and 0.5 L of effluent from the riverbank stage was deposited in the six jars of the Jar Test equipment, doses of 50-75 mL of H_2O_2 3.0 M were applied (Song *et al.*, 2022) and stirred for one minute at 300 revolutions per minute (rpm) then $FeCl_3$ 0.007 M was applied in doses of 75 mL, then stirred for five minutes at 230 rpm, then allowed to settle for 24 hours. The supernatant obtained from 0.5 L of effluent was filtered with 0.2 g of sheep or camelid wool, 5 g of sand with a size of 1-3 mm and 1 g of activated carbon with a size of 0.5-4.0 mm. Finally, the pH, conductivity, turbidity, and sulfide parameters were characterized and COD. The results obtained are evaluated based on the MCL values of the Peruvian standards (Table 1).

For the treatment with a combination of H_2O_2/O_3 oxidants (Peroxone), 1 L of residual water from unhairing was used, pH 8.5, H_2O_2 concentration 3.02 M, doses of 0.005L, 0.01L and 0.02L, O_3 concentration 1000mgL^{-1} and flow 467Lmin^{-1} and the concentrations of COD and sulfides were determined.

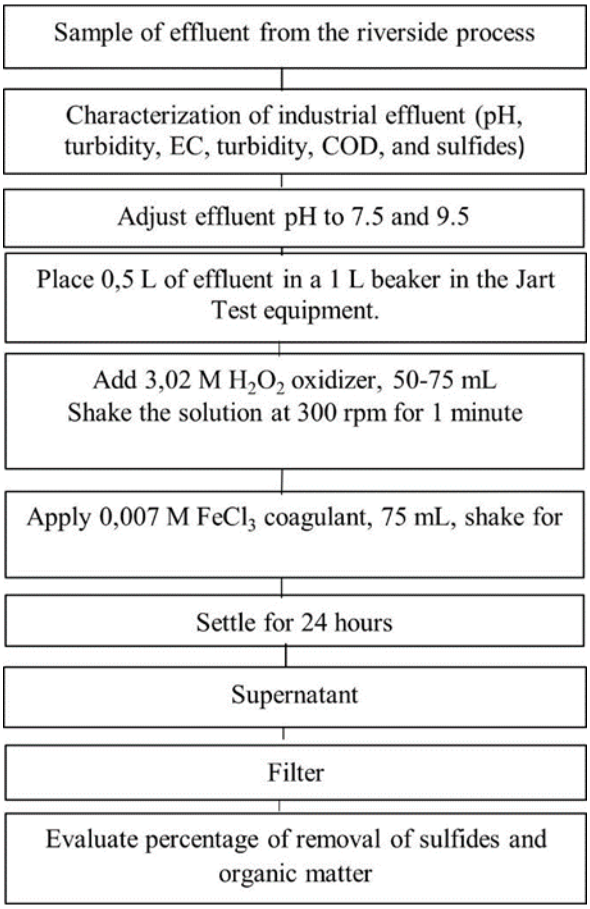

Figure 2. Diagram of the effluent treatment processes from the riverside stage of the tanning process. EC= Electrical conductivity.

Table 1. Maximum contaminant level for the tanning sector.

PARAMETERS	UNIT OF MEASUREMENT	MCL
pH		6-9
Temperature	C°	<35
COD	g L^{-1}	1.5
Sulfides	g L^{-1}	0.03

Source: D.S. No. 010-2023 (Peru, 2023).

3. RESULTS AND DISCUSSIONS

3.1. Results of wastewater characterization

The pH, EC and turbidity results carried out at the final of the liming process of the "La Unión" tannery, the pH value is due to the fact that sodium hydroxide and calcium oxide are used in the riverbank process to facilitate the splitting of the skin and hair fibers (Zhao *et al.*, 2024; Nur-E-Alam *et al.*, 2020) to accelerate the liming of the skins. The pH, EC and turbidity results were carried out at the final of the liming process of the "La Unión" tannery, according to Table 2.

Sulfur pollution (Rana *et al.*, 2020) is caused by Na_2S, it acts as a hair remover that removes and dissolves hair, and sulfur is often part of several redox reactions (Liu *et al.*, 2025; Chen *et al.*, 2022) in water it is found in its dissolved form (Yuan *et al.*, 2022). Turbidity and EC have high values due to the use of NaCl to prevent putrefaction. The added chemicals increase the inorganic load and organic matter formed by skin fibers, hair, keratin and unstructured proteins and micro proteins found in blood and hydrolyzed lymphatic fluid (Fernández *et al.*, 2022), which is why COD has high concentrations (Agudelo Valencia *et al.*, 2020).

Table 2. pH, EC, turbidity, temperature, sulfides and COD in the riverbank effluent.

Parameters studied	pH	EC (µS cm^{-1})	Turbidity (NTU)	Sulfides (mg L^{-1})	COD (mg L^{-1})
Triplicate assays	13.0	17700	5085	964	16898
	13.5	17600	5083	954	16889
	13.3	17700	5082	964	16906
Average	13.3	17667	5083	961	16898
Standard deviation	0.25	57.7	1.53	5.72	8.51

3.2. Characterization of treated wastewater

The results of turbidity, pH, EC, sulfides and COD obtained at pH 7.5, 9.5 after performing the oxidation, coagulation and filtration treatments according to Table 3.

Table 3. pH, EC, turbidity, sulfides and COD, after 24 hours after filtering.

24-hour treatment results after filtering						
Factors		Parameters studied				
pH	H_2O_2 (mL)	pH	EC (µS cm^{-1})	Turbidity (NTU)	Sulfides (mg L^{-1})	COD (mg L^{-1})
7.5	**50**	6.34	161.4	32.9	4.622	819.5
7.5	**50**	6.28	162.1	33.2	4.901	819.2
7.5	**50**	6.38	159.7	33.7	4.674	819.1
7.5	**75**	6.18	147.0	29.8	4.917	755.7
7.5	**75**	6.07	148.5	30.9	4.836	758.2
7.5	**75**	6.09	149.9	30.5	4.490	757.2
9.5	**50**	7.06	111.0	9.94	3.503	668.6
9.5	**50**	7.07	112.0	10.1	2.576	668.5
9.5	**50**	6.95	110.0	10.1	2.708	668.5
9.5	**75**	7.00	131.0	11.62	2.738	590.7
9.5	**75**	6.99	132.0	11.79	2.871	590.8
9.5	**75**	6.93	133.0	11.81	2.525	591.1
8.5	**50**	5.56	155.0	24.8	3.055	459.2
8.5	**50**	5.61	156.0	25.2	2.871	458.8
8.5	**50**	5.51	154.0	25.1	2.973	459.1
8.5	**75**	5.83	141.0	21.9	2.922	449.0
8.5	**75**	5.83	139.0	22.3	3.136	448.3
8.5	**75**	5.93	141.0	22.1	2.790	449.1

The results of turbidity, pH, EC, COD and sulfides obtained at pH 8.5 after performing the oxidation treatment with H_2O_2/O_3 in the Table 4.

Table 4. Physicochemical parameters after treatment with peroxone.

Dose H_2O_2 (mL)	pH	EC (µS cm^{-1})	% Removal	COD (mg L^{-1})	% Removal	Sulfide (mgL^{-1})	% Removal	Turbidity (NTU)	% Removal
-	8.5	35200	-	36688.7	-	5850.6	-	6300	-
5	8.4	27600	21.6	8479	76.9	510.4	91.3	795	87.4
5	8.4	27700	21.3	8463	76.9	506.4	91.3	795	87.4
5	8.3	27700	21.3	8495	76.8	506.4	91.3	795	87.4
10	8.1	27300	22.4	6785	81.5	376.0	93.6	670	89.4
10	8.2	27300	22.4	6769	81.6	378.0	93.5	670	89.4
10	8.1	27400	22.2	6802	81.5	380.2	93.5	670	89.4
20	8.1	27100	23.0	6621	82.0	363.9	93.8	260	95.9
20	8.1	27100	23.0	6637	81.9	359.9	93.8	260	95.9
20	8.1	17100	51.4	6654	81.1	359.9	93.8	260	95.9

3.3. Effect of pH and dosage on the removal of sulfides and COD

The test was carried out at pH 7.5, because at values lower than pH 7 toxic gases such as H_2S gas are released (Liu *et al.*, 2022; Zhao *et al.*, 2022), from the aqueous state it passes to the gaseous phase causing strong and unpleasant odors (Agudelo Valencia *et al.*, 2019) and they are also extremely toxic and very harmful to human health (Paul *et al.*, 2021; Zhang *et al.*, 2022), at pH values 7-9 the effluent has in its composition sulfides and bisulfides (Edathil *et al.*, 2017). At pH 9.5 level the reaction is faster than in acidic medium (Wang *et al.*, 2022; Marais *et al.*, 2020), when the pH of the solution is greater than 10, the sulfur species present in the solution are the reduced sulfur species, S^{2-} ions according to Figure 1 (Edathil *et al.*, 2020). It is shown that the pH has statistically positive influence, greater than dose in the removal of sulfides and COD, in alkaline medium the oxidizing property of H_2O_2 is favored (Hashem *et al.*, 2016). according to Figure 3.

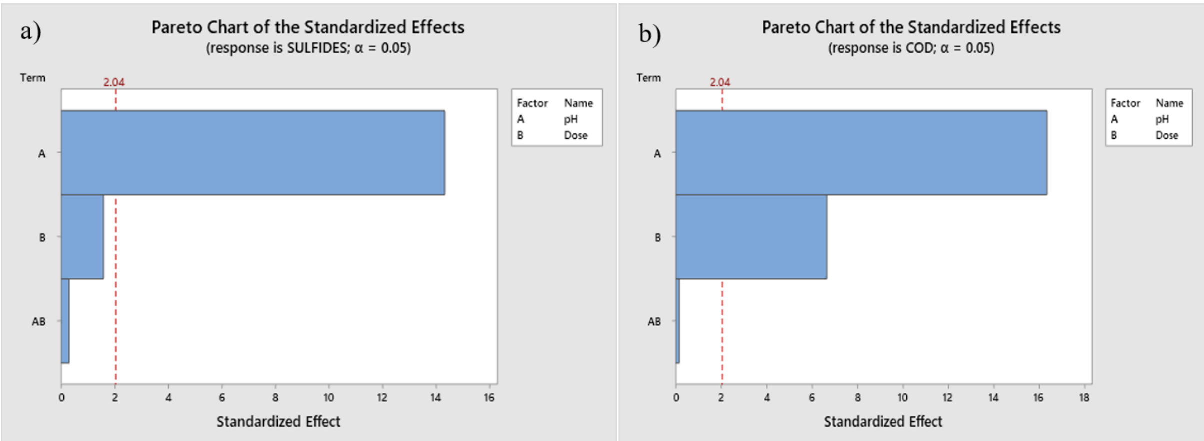

Figure 3. Pareto diagrams of the pH and dose factors in a) sulfides and b) COD.

We perceive that, at a higher volume of oxidant 75 mL and slightly basic pH 9.5, greater removal of sulfides 2.71 mg L^{-1}, at a lower volume of oxidant 50 mL and pH 7.5, lower removal of sulfides 4.73 mg L^{-1}, in the case of COD we perceive that when the dose of H_2O_2 decreases the elimination percentage decreases (Boczkaj and Fernandes, 2017; Barndõk *et al.*, 2016) due to the competitive consumption of hydroxyl radicals (Arka *et al.*, 2022), the results obtained with 50 mL of oxidant and pH 7.5 give COD 800 mg L^{-1} and with 75 mL of oxidant and pH 9.5 the COD result is 591 mg L^{-1} and it is shown in the Figure 4.

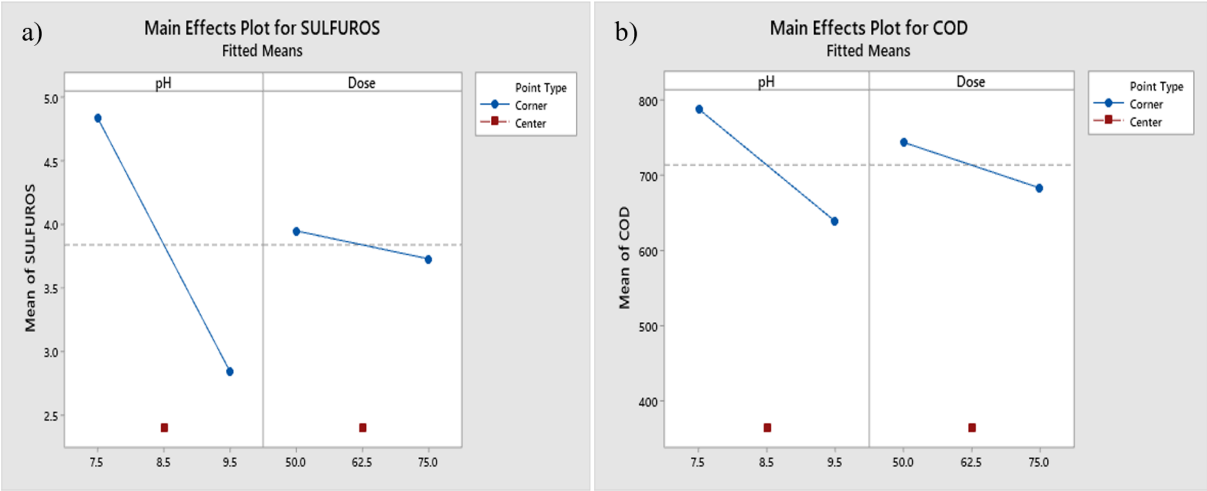

Figure 4. Main effects of pH and dose for a) sulfides and b) COD.

COD removal is higher as the oxidant dose increases (Barndõk *et al.*, 2016; Sawalha *et al.*, 2020). Its efficiency is due to the highly reactive hydroxyl radicals (Lee *et al.*, 2022) that are capable of oxidizing organic compounds, benefiting from the presence of humic acids in the effluent (Del M. Chaile *et al.*, 2024). At pH greater than 10, the production of the Fe^{2+} catalyst from Fe^{3+} decreases, therefore, the production of the hydroxyl radical to oxidize organic matter decreases (Gomes Júnior *et al.*, 2020). The influence of the H_2O_2 oxidant dose factor is positive on the COD results.

3.4. Statement and estimation of the mathematical model using the factorial design of experiments

To determine the effect of pH and oxidant dose, experimental design 2^2 was used, in which there are 2 factors and 2 levels for each factor according to Table 5.

Table 5. Levels of independent variable.

FACTOR	LETTER	LEVELS	
pH	A	7.5	9.5
Dose	B	50	75

Experimental design 2^2 presents the respective notation for each of the levels considered in the experimental design. The notation with the signs indicates the low and high values of each factor according to Table 6.

Table 6. Numerical notation for each value.

A	B	%	A	B
7.5	50		-1	-1
9.5	50		+1	-1
7.5	75		-1	+1
9.5	75		+1	+1

* Low (-1), high (+1).

The mathematical models obtained after developing the factorial method that will allow us to evaluate the behavior of sulfides and COD in the study system are:

$$Sulfides = 11.97 - 0.892pH + 0.0055Dose - 0.0016pH * Dose - 1.446central\ point$$

$$COD = 1531 - 78.3pH - 2.92Dose + 0.058pH * Dose - 349.7central\ point$$

There will be no significant difference, because the values produced by the pH and dose factor levels are produced by independent reactions; there is no interaction between the two factors, so the mathematical model obtained is valid in the vicinity of the experimental region.

3.5. Percentage of removal of sulfides and organic matter

The removal percentage for the combined processes was 99.5% for sulfides and 96.5% for COD, as shown in Tables 7 and 8. The removal percentages for sulfides and COD were calculated using the following formulas (Valderrama *et al.*, 2017):

$$Sulfide\ removal(\%) = \frac{Initial\ sulfide - Remaining\ sulfide}{Initial\ sulfide} x100$$

$$COD\ removal(\%) = \frac{Initial\ COD - Remaining\ COD}{Initial\ COD} X100$$

Table 7. Percentage of sulfide removal at pH 7.5 - 9.5.

pH	**7.5**	**8.5**	**9.5**
Results	**Sulfides (mg L^{-1})**	**Sulfides (mg L^{-1})**	**Sulfides (mg L^{-1})**
Initial	961	9601	9601
Final	4.73	2.97	2.93
Removal percentage (%)	99.5	99.5	99.7

Table 8. Percentage of COD removal at pH 7.5 - 9.5.

pH	**7.5**	**8.5**	**9.5**
Results	**COD (mg L^{-1})**	**COD (mg L^{-1})**	**COD (mg L^{-1})**
Initial		16898	
Final	818	459	664
Removal percentage (%)	95.5	97.3	96.5

The removal results at pH 9.5 are acceptable, but we observed a slight variation, because at values greater than pH 9 the oxidative power of H_2O_2 is affected, leading to the formation of hydroxides, which is not favorable for the removal process.

3.6. Treatment cost per cubic meter of effluent

The cost is expressed in terms of reagents and supplies needed for the treatment. Table 9 shows the cost per cubic meter of effluent for treatment with hydrogen peroxide and ferric chloride, and Table 10 shows the cost per cubic meter of effluent for treatment with peroxone.

Table 9. Evaluation of the treatment cost per cubic meter of effluent from the riverside stage.

Treatment system	Reagent concentration (M)	Reagent per m^3 of effluent (L)	Treatment cost per m^3 of effluent (USD)
Oxidation H_2O_2	3	30	72.3
Coagulation $FeCl_3$	0.007	3	24.1
pH Regulation HCl	6	10	19.3
Total Cost			**115.7**

Dollars per cubic meter ($ m^{-3}).

Table 10. Evaluation of the cost of peroxone treatment per cubic meter of effluent from the hair-dressing stage.

Treatment system	Reagent concentration (M)	Reagent per m^3 of effluent (L)	Treatment cost per m^3 of effluent (USD)
Oxidation H_2O_2	3	20	35.0
pH regulation HCl	6	8	15. 4
kWh cost			125.8
Total cost			**176.2**

4. CONCLUSION

The amount of organic and inorganic matter in the effluent, expressed as COD, was determined, yielding 17 g L^{-1} in the untreated effluent, while after treatment, it was 457 mg L^{-1}. The COD determined complies with the maximum contaminant level 1.5 g L^{-1} MCL for tannery wastewater, demonstrating the efficiency of the oxidant H_2O_2 and the coagulant $FeCl_3$.

The sulfide concentration in the initial effluent was 961 mg L^{-1}. It was reduced through oxidation, flocculation, and filtration processes, yielding 0.03 g L^{-1} of sulfides that comply with the maximum contaminant level 0.03 g L^{-1} for tannery wastewater. The unhairing stage is one of the problems faced by tanneries due to sulfide emissions. pH and dosage correction are highly relevant for process development; The highest removal was obtained at pH 8.5 with 75 mL of H_2O_2 3 M and $FeCl_3$ 0.007 M.

In the peroxone treatment, a removal of 81.7% and 93.8% for COD and sulfides respectively was obtained with a dose of 0.02 L of H_2O_2 with a concentration of 3.02 M and 1000 mg L^{-1} of O_3, and it presents an increase in the cost of the treatment of one cubic meter of river effluent, from 115.7 USD to 176.2USD.

The oxidation, flocculation, and filtration processes were efficient at 99% sulfide removal and 97% COD removal.

5. DATA AVAILABILITY STATEMENT

Data availability not informed.

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