

Case Report

Treatment of wastewater produced during the hydrometallurgical extraction of silver from in-mold structural electronics

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ABSTRACT

Hydrometallurgical processes utilize aqueous solutions to extract metals from ores, concentrates, or waste, resulting in a substantial consumption of freshwater and wastewater generation. This study investigates the treatment of wastewater produced during the hydrometallurgical silver extraction from in-mold structural electronics (IMSEs), aiming at minimizing discharge and ensuring water reuse. IMSE is an emerging technology that offers a lighter, cost-effective alternative to traditional PCBs. The generated wastewater contains high levels of COD (14.48 g/L) and iron (8.69 g/L). It was treated using a Fenton process by adopting a 2²-full factorial design with center points to identify the best operative conditions. Following the analysis of variance (ANOVA) and model determination for COD abatement, Fisher's test was conducted to evaluate whether incorporating curvature into the model would enhance its fit to the experimental data. To further refine the process optimization, star points were added, resulting in a central composite design (CCD). The treatment resulted in a 93.6 % reduction in COD and a 99.9 % reduction in iron, with lime addition aiding in iron precipitation for water reuse. A quadratic model for COD removal indicated a high fit ($R^2 = 0.96$), demonstrating the process's efficiency in reducing pollutants and promoting water reuse. Preliminary cost analysis revealed a total expenditure of 81.60 €/m³, significantly lower than the typical disposal costs of hydrometallurgical wastewater.

1. Introduction

Water is a crucial natural resource that is essential for industrial production, as well as in the domain of hydrometallurgical processes. These processes rely heavily on water, with demand steadily rising year by year [1]. However, the extensive use of leaching solutions in certain hydrometallurgical operations generates substantial volumes of wastewater [2] characterized by hazardous constituents such as heavy metals [1,3], and acidic pH levels [4]. These components pose substantial environmental risks and require rigorous treatment before safe discharge. This situation, intensified by rapid industrial growth and increased productivity in diverse sectors, has significantly contributed to the pollution of groundwater sources and aggravated the worldwide issue of freshwater scarcity. Addressing these pressing issues requires a unified approach and concerted effort toward the sustainable wastewater management, recognizing its potential as a renewable resource that can be recovered and reused to alleviate water stress [5]. Consequently, extensive research has focused on exploring and developing various physicochemical methods, including sorption, ion exchange,

coagulation, and flotation, to treat hydrometallurgical wastewater and reuse the treated water in the same process [1,6,7]. Given the rising concerns about water scarcity and the global water goals set for 2030 [8], addressing the sustainability of wastewater treatment methods becomes necessary. Allayorov et al. [1] attempted to purify hydrometallurgical effluents via coagulation and precipitation, focusing on reducing total hardness. They optimized reagents such as sodium carbonate and utilized aluminium and iron sulphate sorption materials, producing effective coagulants for wastewater impurities. Sudiby et al. [4] utilized electrocoagulation to treat laterite hydrometallurgy wastewater, targeting nickel and metal reduction. Using a tubular aluminium electrode, a reduction in turbidity of 67.07 %, and a sludge nickel content of 0.62 % wt., have been obtained. Metal ions were effectively removed by deposition on the cathode.

In this context, the advanced oxidation Fenton process emerges as a promising technique for wastewater treatment. By utilizing the catalytic properties of iron salts in the presence of hydrogen peroxide, the Fenton process facilitates the efficient degradation of organic pollutants, thereby mitigating the environmental footprint of wastewater generated

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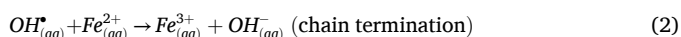
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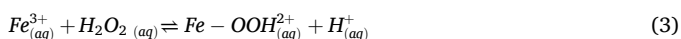
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by hydrometallurgical processes.

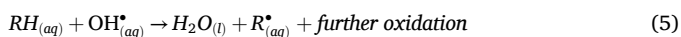
Fenton reaction allows the decomposition of hydrogen peroxide and the formation of reactive hydroxyl radicals, which degrade organic pollutants into harmless compounds like CO₂, water, and inorganic salts. The mechanism of the Fenton process is shown in Fig. 1 [9]. The key reagents in Fenton's reaction are hydrogen peroxide and ferrous ions [10]. They initiate a chain reaction that proceeds as follows:



As illustrated in Eqs. (1) and (2), ferrous iron (Fe²⁺) initiates the reaction and catalyses the decomposition of H₂O₂ into hydroxyl radicals [11–16]. However, the newly formed ferric ions (Fe³⁺) can decompose hydrogen peroxide into water and oxygen, regenerating ferrous ions and radicals:



The reactions described above are referred to as Fenton-like reactions. The organics (RH) undergo oxidation by hydroxyl radicals through proton abstraction, resulting in the production of organic radicals (R[•]). These final products are highly reactive and can undergo further oxidation:



The primary operating conditions affecting the performance of the Fenton processes are pH, oxidant and catalyst concentrations, and temperature [17,18]. In terms of pH, Fenton processes exhibit maximum catalytic activity at a pH of approximately 2.5–3.5 [19,20] ambient conditions can safely be used with good efficiency [21].

This paper aims to explore the treatment of wastewater produced from a hydrometallurgical process for the recovery of silver from IMSEs to significantly reduce water consumption since the treated water can be reused in the next cycles of the process. There are few works in the literature related to the development of hydrometallurgical processes for the recovery of metals that also address the treatment of generated wastewater, this is an area lacking significant research, thus presenting a novel avenue for investigation. Many studies do not even characterize the wastewater generated by hydrometallurgical processes, often overlooking the associated environmental impacts and disposal costs. This oversight becomes even more significant when scaling the process to an industrial level. Proper wastewater treatment and reuse of the treated water are crucial for reducing freshwater consumption and ensuring both the sustainability and economic viability of the process. This work addresses this gap, offering a pathway to more sustainable industrial practices.

Advanced oxidation processes are affected by many variables in

terms of pollutant removal. Response surface methodology can evaluate the influence of the variables on the performance of the process, also leading to the evaluation of interactions to identify the operative conditions that allow the optimization of the process. The central composite design is one of the most popular design methods that facilitate the determination of predictive quadratic models in removing contaminants from wastewater through the Fenton process [22]. By incorporating factorial design with multiscale optimization analysis, the process can be optimized at different scales [23]. In this study, a full factorial design was initially employed to determine the optimal dosage of Fenton reagents to maximize the COD abatement and to determine a theoretical model for its removal. Following this, Fisher's test suggested adding star points to transform the experimental design into a central composite design, as the quadratic model provided a better interpretation of the experimental data. The qualitative characteristics of the treated water confirmed its suitability for reuse in the hydrometallurgical process. This way, the hydrometallurgical process significantly reduced the water footprint and the discharge according to an MLD approach. This study offers insights into wastewater treatment efficacy, environmental implications, and applicability in achieving sustainable water management goals for 2030 [8].

2. Materials and methods

2.1. Materials

Experiments were carried out on the wastewater generated by the hydrometallurgical process for extracting silver from IMSEs of the automotive sector, conducted at a pilot scale in the ambit of the EU Treasure project (Horizon 2020). The wastewater is acidic (pH 1.39) and contains COD 14.48 g/L, Fe 8.69 g/L, and Cu 117 mg/L. All chemicals utilized in these experiments were of analytical grade, comprising H₂O₂ (30 % w/v), ferrous sulphate (II), and calcium hydroxide that was prepared at a concentration of 10 % (w/v). Lime was utilized to adjust the pH and precipitate the iron after the Fenton process, as well as to ensure that treated water had a pH level within the neutral range.

2.2. Methods

The initial and final concentrations of COD in the wastewater were measured using the LCK 014 Hach cuvette test (COD range: 1–10 g/L O₂). For Fe and Cu analysis before and after the Fenton process in each experiment, inductively coupled plasma with optical emission spectroscopy (ICP-OES) (Agilent technologies, 5100) was used. The initial pH of the wastewater was measured with a pH meter (Hanna). Fenton experiments were carried out using a conical flask with a capacity of 100 mL. The volume of wastewater taken for all the experiments was 40 mL. The initial pH was adjusted to approximately 3.5 ± 0.1 using a lime solution (10 % w/v). Then, the necessary quantity of powdered FeSO₄ × 7H₂O was introduced into the reaction mixture and agitated for 5 min,

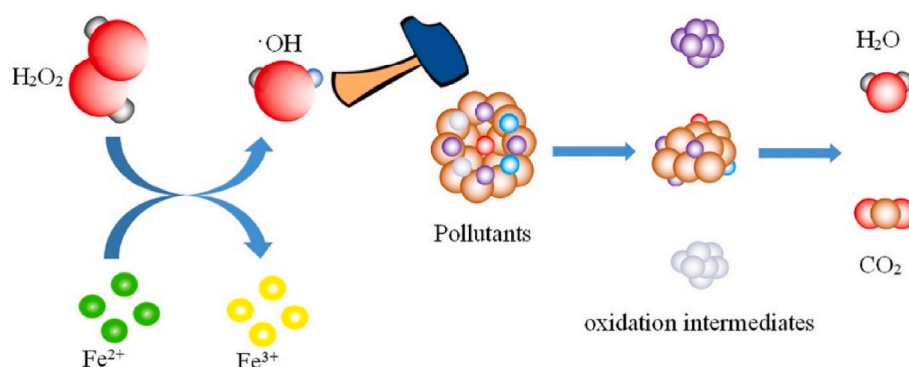


Fig. 1. Reaction mechanism for the Fenton process.

and the Fenton reaction was initiated by adding the necessary amount of H_2O_2 . The solutions were then placed on a shaker (SKI 4, shaking incubator) at 120 rpm for 90 min at room temperature and atmospheric pressure. After the experiment, the pH was readjusted to 8.5 ± 0.1 by adding lime solution (10 % w/v) to precipitate iron and copper as hydroxides. Afterward, the solutions were allowed to stand for half an hour and then filtered using a vacuum filtration system (STEROGLASS, Model ST510) with a nitrate cellulose filter with $0.45 \mu\text{m}$ pore size.

UV-VIS spectral analyses aimed at qualitatively assessing the variation of the spectra before and after Fenton treatment. Analyses were conducted on a UV-VIS spectrometer (Agilent Technologies, Cary 60).

Due to the high content of organic substances in the wastewater, a factorial design was adopted to study the Fenton process, with COD reduction selected as the target output. Experimental tests were initially designed according to a 2^2 -full factorial plan with three centre points. The plan helps to find the main effect of different variables (factors) and their interactions. A 2^2 -full factorial design encompasses every possible combination of two factors, each at two levels, resulting in 4 experimental runs. This setup enabled us to explore the factors' individual effects and their interaction effect on the response variable. Subsequently, the comparison between critical and experimental Fisher was performed to check if the determined linear model could be improved by adding quadratic terms. The comparison between the experimental F-value and the critical F-value is used to examine the statistical significance of the linear model. When the experimental F-value surpasses the critical F-value, it suggests that incorporating quadratic terms might improve the model's overall fit. Therefore, star points were added to enhance the efficiency and robustness of the experimental design. Star points are additional experimental runs performed at a distance α from the centre of the design, where α is a value that depends on the number of factors and the desired properties of the design. The inclusion of star points transforms the full factorial plan into a Central Composite Design (CCD), which allows for the estimation of quadratic effects.

The factors and their levels are given in Table 1. The investigated range for the factors were selected to maintain the H_2O_2/Fe^{2+} ratio ranges from 25:1 to 5:1 w/w [24].

2.3. Theory

The efficiency of the Fenton process depends on many factors including pH, H_2O_2 dosage, Fe (II) concentration, and temperature [25–27]. If all these factors were considered, many experiments would be needed to investigate the effects of the factors and their interaction. The oxidation activity of hydroxyl radicals ($\bullet\text{OH}$) is influenced by the pH of the solution. $\bullet\text{OH}$ oxidation potential increases with decreasing pH, enhancing oxidation capacity [28]. Conversely, Fenton reagent activity decreases with rising pH due to decreased active Fe^{2+} , forming inactive iron oxyhydroxides and ferric hydroxide precipitates. H_2O_2 auto-decomposes at high pH values [29]. At the lowest pH values, specific iron complex species reduce reactivity between Fe^{2+} and H_2O_2 [30, 31], diminishing the efficiency of the Fenton process for organic compound degradation across pH extremes.

Table 1
Factors and levels investigated with the CCD.

Factors	Coded factors			
	−1.414	−1	+1	+1.414
H_2O_2 (% v/v)	2.93	5	15.00	17.07
Fe^{2+} (g/L)	1.14	3	12.00	13.86

3. Results and discussion

3.1. Composition of wastewater

Wastewater composition from the hydrometallurgical process for the recycling of silver from IMSEs is given in Table 2.

The high levels of COD and Fe in the wastewater are attributable to the chemicals used in the hydrometallurgical process, primarily thiourea and ferric sulphate in acidic media.

3.2. Factorial experimentation and statistical analysis

Preliminary Fenton's experiments, as a function of initial pH correction, indicated that adjusting the pH before the Fenton process may not be necessary, as the pH of the wastewater was already found to be suitable for the initiation of Fenton reactions. Under decreased pH level, an elevation in the oxidation potential of hydroxyl radicals ($\bullet\text{OH}$) occurs, leading to an enhanced ability for oxidation reactions. Conversely, in elevated pH environments, hydrogen peroxide (H_2O_2) experiences expedited decomposition, influencing its stability and efficacy in oxidative processes. Therefore, the Fenton process was studied by working at constant pH (1.39), and room temperature, and only the effect of two main factors (Fe (II) and H_2O_2) was investigated by using 2^2 -full factorial plans with three centre points. The experimental conditions and the results of the full factorial plan are given in Table 3.

The analysis of variance (ANOVA) was conducted using Yates' algorithms, revealing factor A (hydrogen peroxide) as the sole significant factor exceeding the 95 % confidence level. Moreover, it's noteworthy that the wastewater already contains iron, with a concentration of 8.69 g/L; for this reason, factor B is not significant for COD removal. Anyway, also other studies found that Fe^{2+} concentration is not a significant factor [10,32].

Following this, the model equation was determined, incorporating this statistically significant factor. The ANOVA findings, alongside the derived equation, are presented below in Table 4. Moreover, it is noteworthy that the same dataset underwent analysis in Design Expert-13 software, which confirmed the obtained results, reaffirming the consistency of the derived equation.

Based on the results obtained by the plan the following relation was obtained:

$$COD_{\text{removal}}(\%) = 78.20 + 12.95 \times X_1 \quad (6)$$

where X_1 is the coded factor (hydrogen peroxide % v/v).

For the optimization purpose, based on the comparison of experimental and critical Fisher ($F_{\text{experimental}} > F_{\text{critical}}$) star points were added to enhance the goodness of the determined model for the experimental data. The results obtained from the inclusion of star points are provided in Table 5.

Based on the experimental results regarding COD removal, it can be noted that run 3 in Table 5, with less hydrogen peroxide, showed higher efficiency than run 2, suggesting excess iron and hydrogen peroxide inhibit mineralization. This imbalance leads to incomplete oxidation, reducing mineralization efficiency, as the literature indicates [33].

ANOVA was conducted with Yates' algorithm to evaluate the effect of the two investigated factors and their interaction on COD removal. The significance of the effects was assessed using the F-test at a 95 % confidence level. This method allowed for the evaluation of whether the

Table 2
Composition of wastewater generated by the hydrometallurgical process.

pH	1.39
COD (g/L)	14.48
Fe (g/L)	8.69
Cu (mg/L)	117.4

Table 3Experimental conditions and results of the 2²-full factorial plan.

Run	A	B	A: H ₂ O ₂ (30 %) (% v/v)	B: FeSO ₄ × 7H ₂ O (g/L)	COD removal (%)	Fe removal (%)	Cu removal (%)
1	-1	-1	5.0	3.0	66.7	98.6	99.7
2	1	-1	15.0	3.0	92.4	99.9	98.2
3	-1	1	5.0	12.0	63.8	97.0	99.9
4	1	1	15.0	12.0	89.9	98.9	95.2
I	0	0	10.0	7.5	84.4	98.8	99.4
II	0	0	10.0	7.5	86.2	96.5	99.7
III	0	0	10.0	7.5	83.7	96.5	99.7

Table 4Yate's algorithm for the 2²-factorial plan, confidence level at 95 %.

Terms	Effect	SS	F-value	P-value	1-p	Significance, (%)
intercept	78.20	–	–	–	–	–
a	25.90	670.88	413.02	0.03	0.97	96.87
b	-2.77	7.67	4.72	0.27	0.73	72.54
ab	0.19	0.04	0.02	0.90	0.10	9.64

effects were significant compared to the experimental error, as shown in Table 6.

The general quadratic model is given below:

$$Y = a_0 + a_1x_1 + a_2x_2 + a_{12}x_1x_2 + a_{11}x_1^2 + a_{22}x_2^2 \quad (7)$$

where y represent % COD removal, x_1 and x_2 are the coded factors for H₂O₂ and FeSO₄ × 7H₂O, respectively. The ANOVA showed that significant factors are H₂O₂ with a positive effect and its square with a negative effect. The negative coefficient for the squared term of H₂O₂ concentration shows that initially adding more H₂O₂ improves COD reduction, but there's a threshold beyond which adding more H₂O₂ worsens the abatement of COD. Based on the most significant factors (x_1 and x_1^2), a quadratic model for COD removal from wastewater was identified:

$$\text{COD}_{\text{removal}}(\%) = 84.74 + 12.21 x_1 - 5.63 x_1^2 \quad (8)$$

The goodness of the model is confirmed by the scatter diagram of predicted against actual values the experimental and theoretical data are well correlated ($R^2 = 0.96$). Based on the determined quadratic model, the optimal H₂O₂ concentration to maximize COD removal (91.38 %) has been calculated to be 15.45 % v/v (1.05 as coded value).

For testing the model, a validation experiment was conducted using this optimized H₂O₂ concentration, resulting in a measured COD removal efficiency of 93.6 %. The difference in terms of COD removal falls within the experimental error. This test confirmed the accuracy and reliability of the quadratic model in predicting the effectiveness of H₂O₂ concentration for COD removal.

The response surface plot in Fig. 2 illustrates how COD removal varies with different concentrations of hydrogen peroxide (H₂O₂) and Fe (II). It is evident that the effect of H₂O₂ is noticeable in influencing COD removal efficiency, while Fe(II) is negligible.

The graph in Fig. 3 illustrates an impressive alignment between predicted and actual responses, showcasing the precision of our model in

Table 5

Star points and their results in terms of COD, Fe, and Cu removal.

Run	A	B	A: H ₂ O ₂ (30 %) (% v/v)	B: FeSO ₄ × 7H ₂ O (g/L)	COD removal (%)	Fe removal (%)	Cu removal (%)
1	-1.414	0	2.93	7.50	59.9	100.0	99.8
2	1.414	0	17.07	7.50	92.3	100.0	97.9
3	0	-1.414	10.00	1.14	94.3	100.0	91.4
4	0	1.414	10.00	13.86	87.0	99.9	99.8

forecasting COD removal under different concentrations of H₂O₂ and Fe (II) for wastewater treatment of IMSEs.

3.3. Water quality characteristics after treatment

The main contribution to the wastewater COD depends on the thiourea since it was used as a reagent to complex silver in solution during the hydrometallurgical process. By UV-VIS analyses, the variation of the peak of thiourea with the Fenton treatment has been evaluated. In Fig. 4, the spectra of the samples before and after the Fenton treatment are shown. The blue curve, indicating the wastewater sample before the treatment, shows a maximum absorption in the range of 230–250 nm for the presence of thiourea, as confirmed by other research [34,35]. By assessing the red curve related to the sample after Fenton treatment, it appears evident that the peak of thiourea has disappeared since it was

Table 6

Yate's algorithm for the CCD, confidence level at 95 %.

Terms	Coefficients	Std. Error	t-value	P-value	1-p	Significance, %
Intercept	84.74	–	–	–	–	–
x_1	12.21	1.26	9.66	2.01E-04	0.9997	99.97
x_2	-2.00	1.26	-1.58	1.75E-01	0.8253	82.53
$x_1 x_2$	0.10	1.79	0.05	9.59E-01	0.0413	4.13
x_1^2	-5.63	1.50	-3.74	1.34E-02	0.9866	98.66
x_2^2	1.66	1.50	1.10	3.20E-01	0.6800	68.00

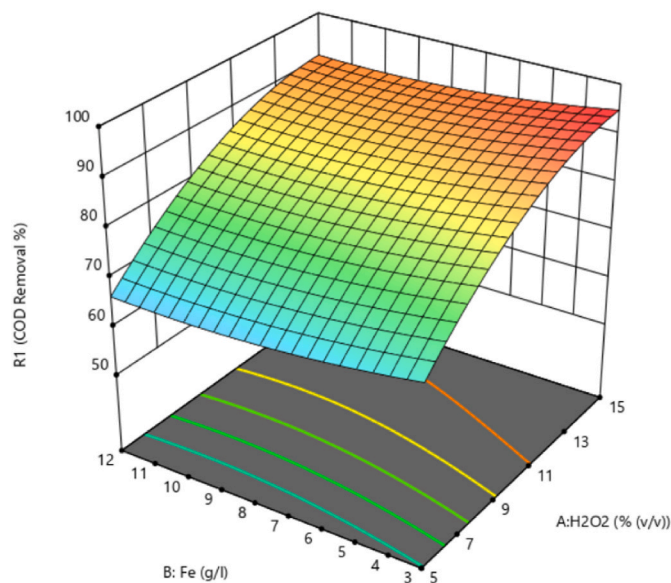


Fig. 2. Response surface-plot for COD removal as a function of H₂O₂ and Fe(II) at pH 3.5.

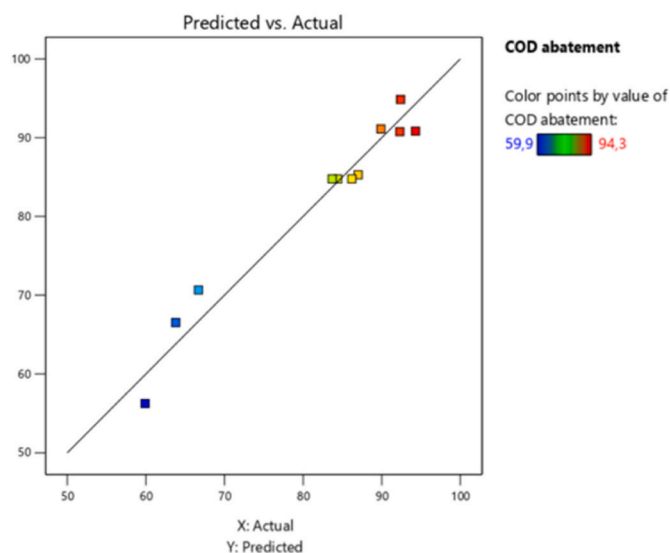


Fig. 3. Predicted response vs Actual response for COD abatement (%) based on the determined model.

decomposed by the radicals in simpler organic substances. To qualitatively evaluate the formation of other organic substances, another analysis of the sample after Fenton treatment was carried out with a lower dilution factor. This test showed an absorption peak at about 300 nm (red curve in the figure in the upper right), confirming that Fenton allows degradation of thiourea in other organic substances.

In Table 7, the water composition after the Fenton treatment and lime precipitation is reported. The quality of the treated water, which would be within the limits for a direct discharge into the sewer, demonstrates its suitability for direct reuse in hydrometallurgical processes, reinforcing operational efficiency and sustainability. This way, the process can be performed by reducing the water footprint using an MLD approach. More in detail, the introduction of the wastewater treatment section allowed to achieve almost a 90 % of reduction in water consumption, with the following water footprint: 42.7 L for 1 kg of silver.

It is crucial to properly treat wastewater from hydrometallurgical processes and reuse the treated water to prepare the leaching solution in the next cycles of the same process. The hydrometallurgical processes are mainly used to treat industrial waste and to recover secondary materials that would otherwise be lost. It is necessary to develop a process that does not generate additional hazardous waste when reclaiming these metals and reduces the use of fresh water to conserve this resource.

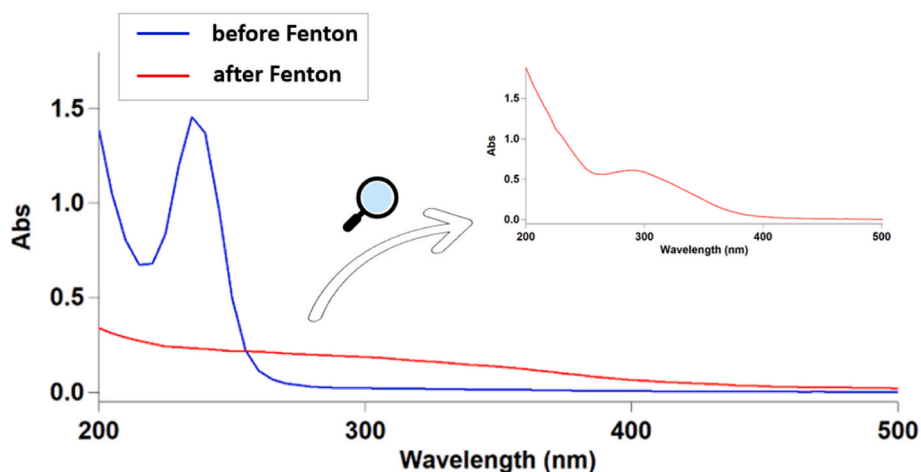


Fig. 4. UV spectra of wastewater before Fenton treatment (blue curve) and after Fenton treatment at different dilution factors (red curves). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

While there are numerous scientific articles on recovering metals from industrial waste by adopting hydrometallurgical processes, very few of them focus on wastewater treatment. Therefore, it is vital to embrace a sustainable approach to hydrometallurgy, known as 'circular hydrometallurgy' [36].

3.4. Mass balance and preliminary cost analysis

A flowsheet of the proposed treatment has been shown in Fig. 5 and a mass balance has been reported, according to the obtained results, for treating one cubic meter of hydrometallurgical wastewater. The hydrometallurgical wastewater is directed into a reactor where hydrogen peroxide is added to initiate the Fenton reaction, aimed at degrading complex organic substances. Subsequently, in the same reactor, lime is introduced to neutralize the wastewater and precipitate iron and other contaminants. The treated water is then separated from the sludge using a filter press. The treated water can be reused, while the sludge must be properly disposed of. This process requires specific quantities of reagents to effectively treat the wastewater: 171.5 kg of hydrogen peroxide (30 % w/v) and 42.5 kg of calcium hydroxide (10 % w/v). Calcium hydroxide serves a dual purpose in this treatment process. It is utilized to adjust the pH after Fenton around the neutrality and allows the precipitation of iron and other pollutants present in the wastewater, aiding in their removal from the solution. As a result of the treatment process, 190 kg of wet sludge is generated per cubic meter of treated wastewater. Different studies showed the possibility of the reuse of iron-containing sludge as an iron source in the oxidation part of the Fenton treatment [37] or the recovery of hazardous metals by thermal treatments [38] to minimize the production of hazardous ferric waste and reduce the overall cost of the treatment process.

Based on the model optimization, the Fenton process was performed at pH 1.39, without any further correction, by using a hydrogen peroxide (30 % w/v) concentration of 15.45 % (v/v) without any addition of ferrous sulphate. The results showed significant removal percentages: 93.6 % for COD and 99.9 % for Fe and Cu.

A preliminary cost analysis showcases a total expenditure of 81.60 €/m³, compared with a typical hydrometallurgical wastewater disposal cost of 150 €/m³. It encompasses the costs associated with hydrogen

Table 7
Composition of wastewater after treatment.

COD (g/L)	0.46
Fe (mg/L)	<0.10
Cu (mg/L)	<0.10

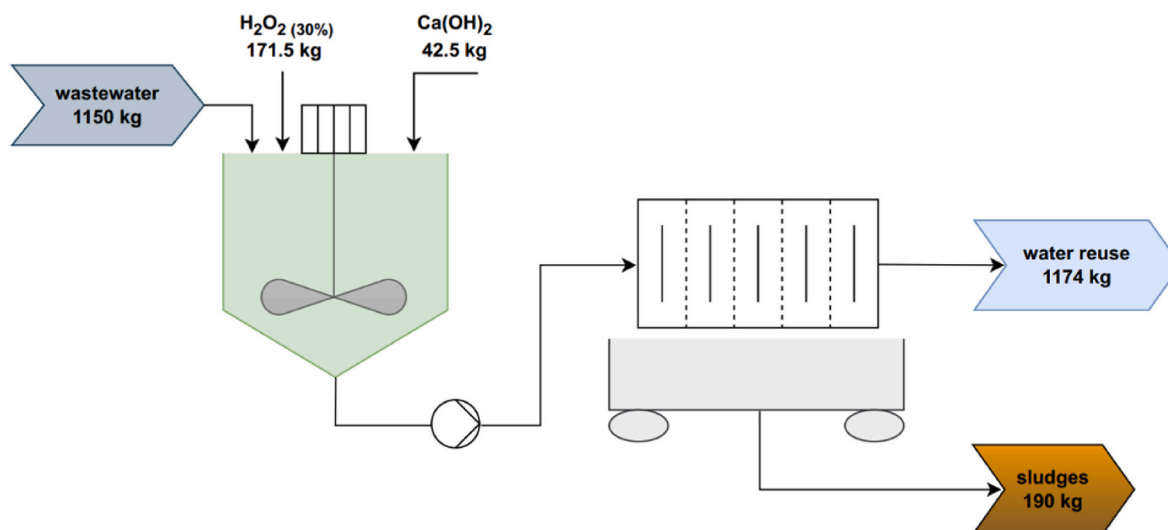


Fig. 5. Flowsheet and mass balance for IMSEs wastewater treatment.


peroxide at 60.02 €, sludge disposal at 15.20 €, and calcium hydroxide at 6.38 €. These findings underscore the financial considerations highlighting COD removal to ensure sustainable water use.

4. Conclusions

A study was conducted on removing Fe, Cu, and organic compounds (COD 14.48 g/L) in hydrometallurgical wastewater generated by a process for recovering silver from IMSEs in the automotive sector. The objective was to treat the wastewater for reuse in the same hydrometallurgical process to reduce wastewater production and water footprint. The Advanced Oxidation Process (Fenton) was employed at optimized operative conditions identified through a combination of 2²-full factorial and CCD experiments to achieve higher removal percentages of the targeted contaminants. The maximum COD removal of 93.62 % was achieved with a hydrogen peroxide (H₂O₂) concentration of 171.5 kg/m³ and a calcium hydroxide concentration of 42.5 kg/m³. Additionally, the removal percentages for Fe and Cu exceeded 99 %. UV-VIS analyses proved that with Fenton treatment, thiourea has been decomposed into other organic compounds. All experiments were conducted without any preliminary pH correction since the wastewater had a pH of 1.39. It was determined that the optimized Fenton condition, achieving a 93.62 % COD removal, required 81.60 € per m³.

The wastewater treatment section might be incorrectly considered as an additional cost for hydrometallurgical plants. However, it can be cost-effective when compared to the expenses of disposal. Additionally, reusing treated water can significantly reduce overall freshwater consumption in the hydrometallurgical process, in line with the MLD approach. It's crucial to emphasize that without proper wastewater management, scaling up the process to an industrial level would be extremely challenging. Moreover, wastewater treatment minimizes health risks for workers by preventing exposure to toxic substances and environmental impacts.

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CRediT authorship contribution statement

Misbah Ullah: Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Nicolò Maria Ippolito:** Writing – review & editing, Visualization, Validation, Supervision, Project administration, Methodology. **Loredana Spera:** Visualization, Validation, Software, Methodology. **Francesco Vegliò:** Resources, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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