REMOVAL OF PHENOLIC COMPOUNDS FROM PULP AND PAPER INDUSTRIAL EFFLUENT USING THE $S_2O_8^{2-}$/UV PHOTOCATALYTIC PROCESS: OPTIMIZATION BY MULTIVARIATE ANALYSIS

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Advanced oxidation processes (AOPs) are presented as a viable alternative for treating wastewater generated by the cellulose production industry, as these waters contain high concentrations of phenolic compounds and elevated levels of color, which are difficult to remove by conventional methods. AOPs require short treatment times and can lead to the mineralization of organic matter present in the wastewater. The oxidation process used in this study employs persulfate as the oxidant, which, when activated by UV radiation, generates sulphate radicals over a broad pH range ($1 \leq pH \leq 10.5$). The sulphate radical is a highly reactive species due to its high oxidation potential (E^0 2.6-3.1 eV).

For process optimization, two experimental variables were studied: pH, ranging from 2.0 to 11.0, and persulfate concentration, ranging from 0.1 to 1.0 gL^{-1}. An experimental design was constructed using a full factorial design with three levels, resulting in 15 experiments. The experimental design revealed an optimal response at pH 6.0 and 0.55 gL^{-1} of oxidant, determining that within 1 hour of treatment, it is possible to achieve a reduction of 87.8% in phenolic compounds, with a constant rate of 0.022 min^{-1} (R^2=0.83) and EEO = 20 Kwh^{-1}. At the same time, the COD was reduced by 90.5%, the TOC by 79%, and the color was 100% eliminated, while increasing the effluent's bioavailability from 0.35 to 1.4 ($p \leq 0.001$, 95% confidence).

The high bioavailability of the organic matter obtained after the treatment is an excellent outcome for the subsequent management of the usage of the treated water, whether it is for reuse in the industrial process itself, for irrigation purposes, or for return to the aquifer without causing environmental harm.

Keywords: advanced oxidation process, phenolic compounds, sulphate radical, pulp and paper industry, wastewater treatment, multivariate analysis

INTRODUCTION

Historically, the pulp and paper industry has been recognized as one of the largest consumers of natural resources (wood and water) and energy (fossil fuels and electricity), while also standing out as a significant contributor to environmental pollution.[1-3]

Chemical pulp production is the most important wood conversion technique, with the Kraft process being the most widely used pulping method globally. In the Kraft pulping process, wood chips are digested using a $NaOH/Na_2S$ mixture to solubilize lignin and obtain cellulose. Lignin, a highly condensed aromatic polymer, constitutes between 20 and 30% of the wood's weight. The resulting cellulose is further oxygenated to continue delignification, resulting in 2 to 3% residual lignin that must be removed to obtain white cellulose, generating large volumes of water with high color, high chemical oxygen demand, and very low bioavailability.[4,5]

Commonly employed methods for removing toxic compounds from paper industry wastewater include biological treatments, primarily relying on microorganisms for degradation. The drawback of this method is the necessity to dilute the wastewater due to its high organic load and toxicity, leading to increased water consumption and achieving only around 35% removal over a 3 to 4-week treatment period.[6-8]

Despite advances in more efficient treatment systems, challenges persist in the final disposal of these effluents because the implemented

treatments have not been effective in eliminating most refractory compounds, preventing their reuse or discharge into the environment without ecological harm. Growing concerns about the potential impact of these pollutants on natural systems and, consequently, on human health, have prompted a continuous search for new, more efficient, and environmentally friendly treatments. Among these technologies, advanced oxidation processes (AOPs) have proven to be a promising alternative for degrading challenging compounds in a short time, transforming organic matter into less harmful molecules and allowing the reuse of treated water within the same processes, ultimately preserving the increasingly scarce water resource.[9-11]

Methods based on chemical or photochemical oxidation of contaminants constitute a group of chemical treatment technologies that offer advantages over commonly used techniques, such as primary treatment (physical treatments; coagulation and flocculation) and secondary treatment (biological treatments as activated sludge or aerated lagoons).[12,13] Particularly, secondary treatments fail to eliminate high color and toxicity because of the presence of polyphenolic structures. In contrast, biological treatments are highly sensitive to operating parameters, such as pH, temperature, and the presence of toxins, which may inhibit microbiological activity. These treatments also require long periods of residence in treatment lagoons, which demand significant space for disposal.[14-19]

AOPs are physicochemical processes capable of producing profound changes in the chemical structure of pollutants. The fundamental principle involves the *in-situ* production of highly reactive radicals through the combined power of ultraviolet light and chemical oxidants, allowing the definitive destruction of pollutants in water.[20-22] One of the advantages of applying these processes is the transformation of refractory contaminants into more biodegradable molecules, significantly reducing environmentally important parameters, such as biological oxygen demand, chemical oxygen demand, total organic carbon, and toxicity.[23-25]

Particularly, the oxidation-driven destruction of organic matter using persulfate ($S_2O_8^{2-}$) is gaining interest among AOP technologies. Despite being a strong oxidant (E^o 2.01 eV), it reacts slowly with organic compounds. However, in the presence of radiation, it decomposes to generate the sulfate radical anion ($SO_4^{\cdot-}$). This radical is a powerful oxidant capable of oxidizing a wide variety of organic and inorganic compounds. Similar to the hydroxyl radical, the sulfate radical can react with organic compounds through electron transfer, hydrogen abstraction, or addition mechanisms. It is generally accepted, however, that sulfate radicals react more selectively through electron transfer, especially in aromatic rings.[26-28]

The persulfate anion ($S_2O_8^{2-}$) enhances the speed of the photocatalytic reaction, because it generates additional hydroxyl radicals and, primarily, sulfate radicals ($SO_4^{\cdot-}$). The reduction of this anion produces two sulfate anions, according to Equation (1):

$$S_2O_8^{2-} + 2\ e^- \longrightarrow 2\ SO_4^{2-} \quad (E^\circ\ 2.01\ eV) \qquad (1)$$

When the sulfate free radical acts as an oxidant, it accepts a single electron, resulting in the production of sulfate anions (SO_4^{2-}), as described in Equation (2):

$$SO_4^{\cdot-} + e^- \longrightarrow SO_4^{2-} \quad (E^\circ\ 2.6\ eV) \qquad (2)$$

The sulfate radical is a stronger oxidant than the persulfate anion (E° 2.01 eV). Therefore, it is desirable to activate the persulphate anion and produce sulfate radicals, leading to increased oxidation of phenolic organic matter.[29]

When the persulfate anion undergoes thermal activation or photolysis, sulfate and hydroxyl radicals are generated *in situ* (Eqs. 3 and 4):[30,31]

$$S_2O_8^{2-} + h\upsilon \longrightarrow 2\ SO_4^{\cdot-} \quad (E^\circ\ 2.5\text{-}3.1\ V) \qquad (3)$$

$$SO_4^{\cdot-} + H_2O \longrightarrow HO^{\cdot}\ (E^0\ 2.8\ V) + HSO_4^- \qquad (4)$$

In this study, UVC radiation was used to activate the persulfate ion and generate *in situ* sulfate radicals and hydroxyl radicals, both of which are strongly oxidizing. The objective was to achieve, within a short treatment period, the maximum degradation of the real effluent, reducing pollution parameters, such as phenolic compounds, color, and COD. To optimize the process, the mathematical tool of multivariate analysis was used, which allows the prediction of the values of the variables that enable the greatest degradation of contaminants.

The results obtained indicate that the process can be efficiently used to treat wastewater from the cellulose industry, allowing this industry to produce without continuing to cause greater environmental damage.

EXPERIMENTAL

Experimental conditions

The effluent used for photocatalytic oxidation with the $S_2O_8^{2-}$/UV system was obtained from a local pulp company, corresponding to the first alkaline extraction (E_1) in an ECF kraft pulp bleaching sequence. To carry out the reaction, a 300 mL reactor equipped with a mercury lamp (HPL 120 W, $\lambda \geq 254$ nm) was used.

Optimization of experimental variables

The optimization of cellulose effluent oxidation was conducted through multivariate analysis using the MODDE 7.0 program. To determine the optimal operating conditions, the variables pH and oxidant dosage ($S_2O_8^{2-}$) in g L^{-1} were studied, while keeping the time constant at 60 min. The experimental design involved 15 experiments, and the response was determined as the percentage of removal of phenolic compounds. The ranges of the values associated with the studied experimental variables are shown in Table 1. The variable ranges have been determined on the basis of previous studies, in which it has been estimated that the sulphate radical can act over the entire pH range.

Since cellulose effluents have a pH of 10.8, pH was investigated to determine if working at high pH levels is feasible or if adjustment to more neutral or acidic levels would lead to a more effective process optimization. For optimization processes using multivariate analysis, ranges of variables, both minimum and maximum, were studied.

Table 1
Ranges of studied experimental variables

Variable	Range
Oxidant ($S_2O_8^{2-}$, g L^{-1})	0.1–1.0
pH	2–11

After each experiment, samples were analyzed to determine the removal of phenolic compounds (λ = 280 nm) derived from lignin (λ = 254 nm) and color removal (λ = 460 nm), respectively. The Spectroquant Pharo 300 Merck spectrophotometer was used for analysis.

Once the optimal experimental variables had been determined, the reaction kinetics study was conducted to verify the optimization of the time in effluent degradation.

Measurement of analytical parameters

Determination of chloride ions

The determination of chloride ions followed ISO 8466-1, DIN 38402 A51, EPA 325.1, and US Standard Methods 4500-Cl methods.

Chemical oxygen demand (COD)

A Spectroquant kit from Merck, with a measurement range of 25–1500 mg O_2 L^{-1}, was used for the determination of COD mg O_2L^{-1}. The procedure followed EPA 410.4, ISO 15705, and Standard Methods 5220 D.

Total phenols determination

For the measurement of total phenols, a Spectroquant kit from Merck was used with a measurement range of 0.02–5.00 mg L^{-1}; following ISO 8466-1, DIN 38402 A51 methods.

Total organic carbon (TOC)

The measurement of TOC in mg L^{-1} was carried out by carbon disintegration using sulfuric acid and persulfate. Samples underwent digestion in a thermostatic reactor at 120 °C for 2 hours, followed by spectrophotometric readings at 592 nm. The method followed ISO 84661-1 and DIN 38402 A51.

Biodegradability assay

A strain of *Pseudomonas aeruginosa* bacteria was grown on trypcase-soy agar and used for serial dilution tests, following the McFarland standardization method on day 0.[32] The plates were incubated inverted for 24 hours at 37 ºC. Bacterial colonies were counted every 24 hours for 120 hours, and the results were expressed in CFU*mL^{-1}.

RESULTS AND DISCUSSION

For the optimization of phenolic compound removal from the cellulose effluent, multivariate analysis was employed, constructing a full factorial experimental design with three levels. After conducting experiments following the experimental design order and determining the response for each experiment, such as the removal of phenolic compounds, the data were entered into the MODDE 7.0 program according to the experimental design (Table 2). The analysis determined that the model had a good fit, as the experimentally observed response (R^2 = 0.9983) and the model's expected response (Q^2 = 0.9983) are equal and exhibit a good fit, as shown in Figure 1.

Multivariate analysis

After obtaining the experimental responses and introducing them into the model, the experimental variables were combined to create a response

surface, allowing the determination of the variable values that achieve the highest removal of compounds from the effluent.

According to Figure 2, the response surface illustrates the interaction between the oxidant amount and pH and their influence on the removal of phenolic compounds. The red zone indicates the interaction of variables where the highest removal is achieved, specifically with a higher persulphate dosage and lower pH.

Table 2
Experimental design and experimentally obtained responses in the removal of phenolic compounds from cellulose bleaching effluent

Exp. Nº	Run order	$S_2O_8^{2-}$ (g L^{-1})	pH	Phenolic compounds removed (%)
1	13	0.1	2.0	79.9
2	2	1.0	2.0	76.2
3	7	0.1	11	72.3
4	12	1.0	11	82.0
5	6	0.1	6.5	67.2
6	14	1.0	6.5	69.5
7	15	0.1	6.5	68.8
8	4	1.0	6.5	73.5
9	10	0.55	2.0	74.0
10	1	0.55	11	69.0
11	9	0.55	2.0	73.5
12	11	0.55	11	76.8
13	5	0.55	6.5	88.0
14	3	0.55	6.5	88.7
15	8	0.55	6.5	87.9

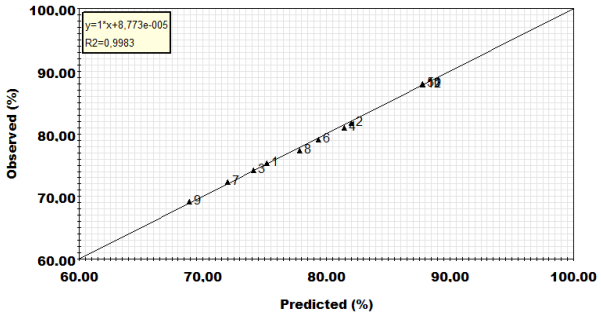

Figure 1: Experimentally observed response *vs*. model-predicted response for the removal of phenolic compounds from cellulose effluent (95% confidence, $p \leq 0.001$)

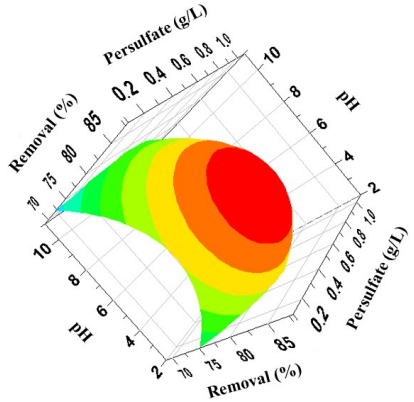

Figure 2: Response surface provided by the experimental design for optimal response in removal of phenolic compounds from the cellulose effluent

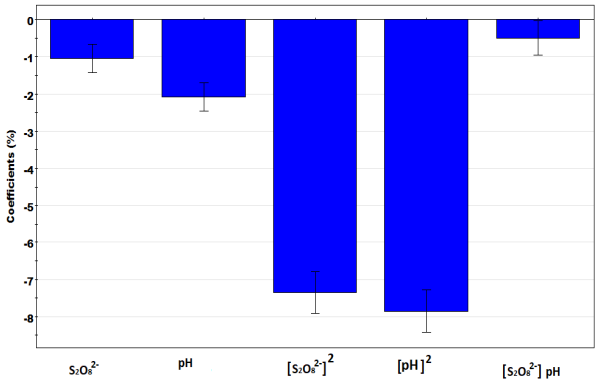

Figure 3: Influence of the studied experimental variables on the removal of phenolic compounds from the cellulose effluent

The peak zone indicates that the removal reaches 87.8% in 60 minutes of treatment at pH 6.0 and 0.5 gL^{-1} of persulphate.

This result is promising, as the amount of oxidant required for significant removal of phenolic compounds is low compared to other studies that have used concentrations ten times higher, without achieving the expected results, particularly in reducing COD.[33,34]

Based on the analysis of the experimental design, a polynomial equation was developed, representing a second-order equation involving both studied variables (Eq. 5). In Figure 3, the influence of each variable of the polynomial on the response is shown, confirming the observations from the response surface. Specifically, lower pH and lower oxidant concentration result in higher removal, reaching an average of 87.8%.

The quadratic equation of the model, where the percentage of removal of phenolic compounds is influenced by the variable's oxidant dosage in gL^{-1} (X_1) and pH (X_2), is as follows:

$$Y\ (\%) = 87.8\ (\pm 0.17) - 1.05\ (\pm 0.16)\ X_1 - 2.08\ (\pm 0.16)\ X_2 - 7.4\ (\pm 0.23)\ X_1^2 - 7.85\ (\pm 0.23)\ X_2^2 - 0.5\ (\pm 0.19)\ X_1X_2 \quad (5)$$

The statistical analysis determined that the quadratic effects of the variables were significant in the response. Although the pH range for maximum removal is between 2 and 8, the maximum removal is achieved at pH 6.0. As seen in the figure, the coefficient is negative, indicating that lower pH within the studied range (2 to 11) leads to higher removal. However, the quadratic term suggests that it has a maximum value, which coincides with pH 6.0. The same pattern is observed for the oxidant concentration.

Validation method

In the optimization of experimental variables, the model validation was analyzed based on the following hypothesis:

H_0: There is no difference between variables

H_1: There is difference between variables

$F_{exp} = 8.3$ and $F_{critical\ 6.8} = 5.60$ $\quad F_{exp} > F_{critical\ 6.8}$

$F_{error} = 1.5$ and $F_{critical\ 6.2} = 7.26$ $\quad F_{error} < F_{critical\ 6.2}$

Therefore, if there is a difference between the variables, but it is not significant because $F_{error} < F_{critical}$ 6.2, the alternative hypothesis H_1 is accepted.

The model is statistically evaluated as valid, given the values of R^2 (0.988) and Q^2 (0.988), both equal and very close to 1. This indicates that the regression model provides a good description of the relationship between observed responses and expected responses based on the design's variable values. R^2 represents the fraction of the response variation explained by the model, and Q^2 is the fraction of the response variation that can be predicted by the model. This results in the statistical validity of the quadratic model with $p<0.001$ and 95% confidence.[35,36]

Experimental design validation

The optimal conditions identified for phenolic compound removal with the $S_2O_8^{2-}$/UV system were experimentally validated following the reaction kinetics. In Figure 4, a clear trend is observed in the effect of photooxidation on the removal of phenolic and lignocellulosic compounds, reaching a maximum at 60 minutes. This aligns with the time observed in the experimental design analysis and the polynomial equation. There is no further removal with longer treatment times. Additionally, color is rapidly removed in the first 10 minutes of the reaction, reaching 94.07%, and achieving 100% removal at 60 minutes of treatment. Simultaneously, it is observed that only UV radiation does not lead to the degradation of organic matter in the effluent. This indicates that in these types of effluents, photolysis alone is not effective in promoting the degradation of phenolic compounds. It is the combination of the oxidant with UV radiation, which promotes the *in-situ* formation of sulphate and hydroxyl radicals, both responsible for the degradation of organic matter.[37]

Figure 5 shows the evolution of organic matter removal through the determination of COD, with a removal rate of 90.5% achieved during the first 60 minutes of the reaction. This timeframe corresponds to the duration used for all experiments in the experimental design, where the time was kept constant at 60 minutes for each experiment.

Figure 6 shows the kinetics of total phenol removal, which is observed achieving around 90%, with a constant rate of 0.022 min^{-1} (R^2 = 0.83) and 20 Kwh^{-1} EEO. This indicates that the treatment is efficient in breaking the benzene rings present in lignocellulosic compounds, as reflected in the significant removal of COD and a decrease in TOC, indicating the mineralization of organic matter.[38]

It is worth noting that this study worked with a real effluent, with a high content of organic

matter, achieving a high removal in a short treatment time, mineralizing the organic matter. In other studies, on phenol degradation, pure compounds have been used, yet the processes employed only managed to degrade the compound between 54% and 64%, in prolonged treatment times.[39,40]

In Figure 7, the increase in the concentration of chloride ions released during the photochemical process is observed. The release of Cl^- ions into the solution is a preliminary indicator of the mineralization of chlorinated organic matter present in the effluent due to the cellulose bleaching process with chlorine dioxide.[41]

Figure 8 shows the mineralization of organic matter expressed as TOC. As observed, a reduction of 67% is achieved after 60 minutes. To achieve a greater mineralization of organic compounds, more time is required. Thus, after 90 minutes, a 79% removal was obtained, indicating that sulphate radicals are effective in breaking down molecules, transforming them into less complex species or simple compounds such as CO_2 and H_2O.

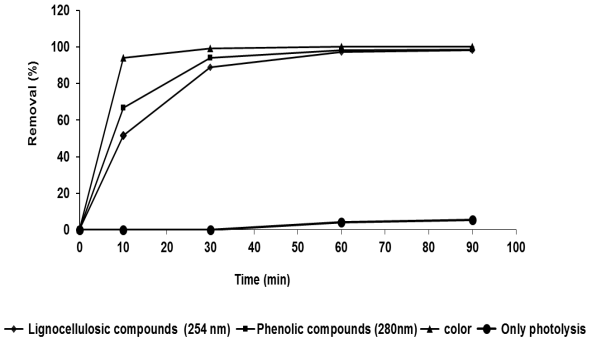

Figure 4: Kinetics of removal of phenolic compounds, lignocellulosic compounds and color at pH 6.0 and 0.55 gL^{-1} $S_2O_8^{2-}$

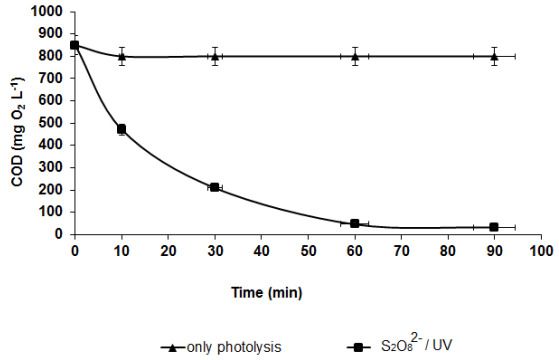

Figure 5: Removal of COD over time with optimal variables: pH 6.0 and 0.55 gL^{-1} $S_2O_8^{2-}$

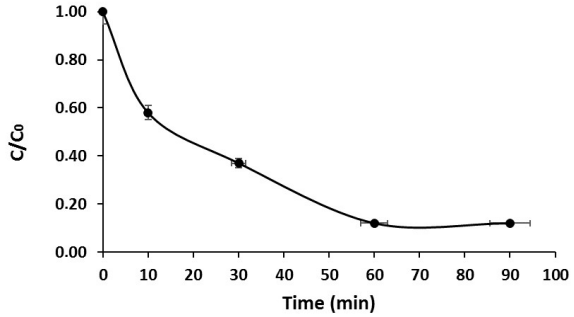

Figure 6: Kinetics of total phenol removal over time with optimal variables: pH 6.0 and 0.55 gL^{-1} of $S_2O_8^{2-}$

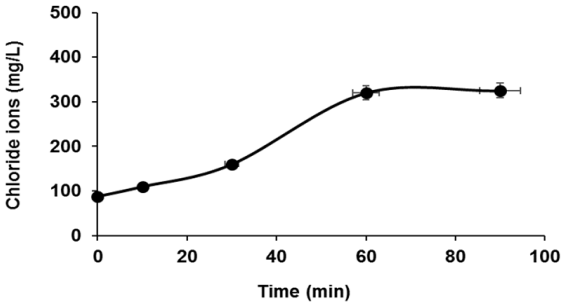

Figure 7: Increase in chloride ions as a product of compound mineralization during the treatment

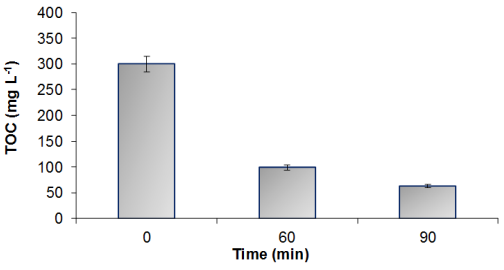

Figure 8: Removal of total organic carbon from the kraft cellulose bleaching effluent at pH 6.0 and 0.55 gL^{-1} of $S_2O_8^{2-}$

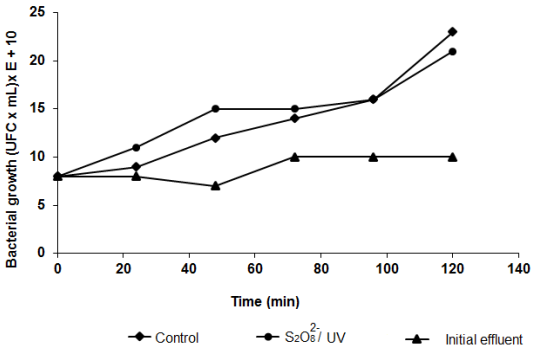

Figure 9: Bacterial growth with the optimized $S_2O_8^{2-}$/UV process and compared to growth in the initial effluent

The efficiency of the $S_2O_8^{2-}$/UV system is primarily based on the oxidation potential of the sulphate radical generated *in situ* during the reaction. The sulphate radical is a strong oxidant that attacks aromatic structures, such as the benzene ring, capturing the electrons from the ring and causing its breakage. This transforms aromatic rings into simpler structures, potentially achieving the transformation of organic matter into carbon dioxide and water, as evidenced by the reduction in TOC.[42,43]

Toxicity: *Pseudomonas aeruginosa* assay

Bacterial development is intricately linked to the environmental conditions in which bacteria thrive, and this is reflected in their ability to utilize substrates as a source of energy for growth. When microorganisms encounter foreign agents in their environment that inhibit their growth, the initial response is slow development, primarily due to the process of adapting to the new environment. If the agents introduced into a culture medium do not pose significant issues, the individuals will continue to develop normally. In this study, the focus was on examining the impact of the $S_2O_8^{2-}$/UV system on bacterial growth in the bleaching effluent of Kraft pulp, serving as a reliable indicator of the bioavailability of residual organic matter.

Figure 9 illustrates the growth of *Pseudomonas aeruginosa* bacteria following treatment with the $S_2O_8^{2-}$/UV system, alongside the control (distilled water) and the real Kraft pulp bleaching effluent. It is evident that, after the treatment with the $S_2O_8^{2-}$/UV system, bacteria exhibit faster growth compared to the control, suggesting that the residual organic matter is more bioavailable and serves as a nutrient for bacterial growth. Simultaneously, it is observed that in the untreated effluent, bacteria not only fail to grow in the first 60 hours of exposure, but also tend to decrease. Subsequently, their growth does not continue, indicating that the initial effluent is toxic to microorganisms since the organic matter is not bioavailable.[44-47]

Based on the obtained results, it can be stated that the photooxidative system improves the quality of the treated wastewater, reducing its toxicity and delivering positive outcomes for this link in the food chain. Bacterial presence is ecologically significant, as bacteria play a crucial role in the aerobic degradation of many compounds derived from the breakdown of animal and plant organic matter in soil and water. It is worth noting that the treatment significantly increases the availability of organic matter in the initially low-bioavailable effluent, raising the value from 0.35 to 1.4, indicating very high bioavailability. This suggests a radical change in the effluent, allowing the treated water to be discharged without consequences for the environment or reused in industrial processes.[48,49] For future work, it would be advisable to continue advancing in scaling up the process in order to assess its potential for industrial application. This work is at TRL3 level.

CONCLUSION

The advanced oxidation process used in this study achieved optimal conditions after 60 minutes of reaction with 0.55 gL^{-1} of persulfate and a pH of 6.0. The analysis determined that the model showed a good fit, as the experimentally observed response (R^2 = 0.9983) and the model's predicted response (Q^2 = 0.9983) were equal and exhibited a good correlation. In 60 minutes of reaction, the process successfully reduced phenolic compounds by 90%, COD by 90.5%, and completely removed the color from the effluent (100%). Additionally, it was determined that 79% of the organic matter was mineralized, meaning it was transformed into CO_2 and water, and the bioavailability of the remaining organic matter increased significantly, from 0.35 to 1.4.

AOP-based treatments represent an effective alternative for treating effluents from the pulp and paper industry, allowing the discharge of treated water into aquifers without causing environmental harm.

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