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INTEGRATED DIGITAL BAFFLE ELECTRO-PHOTO CATALYTIC OXIDATION REACTOR FOR HEAVY METAL REMOVAL: PERFORMANCE AND STATISTICAL EVALUATION

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ABSTRACT

This study investigates the use of an Integrated Digital Baffle Electro-Photo Catalytic Oxidation Reactor (IDBEPCOR) for well-organised copper elimination from Produced Water (PW). The batch oxidation reactor employs aluminum anodes and iron cathodes, with a digital baffle to improve mixing of the PW with Titanium Dioxide (TiO_2) nanoparticles, optimising electrocoagulation and oxidation treatment. The high coefficient value (R^2 = 0.952) indicates a second-order evident association between copper elimination and the important parameters. Moreover, additional statistical analyses were used to evaluate the reliability and validity of the optional process. A regression model was developed based on unresolved values and demonstrated strong agreement with practical results. The IDBEPCOR produced the best equation for the empirical model to forecast metal removal via electrocatalytic oxidation using Minitab software with a Box-Behnken design. The metal removal ratio increased to 98.5% at 300 rpm, pH 9, 100 ppm catalyst concentration, and 50 minutes of oxidation time. Enhanced electrocatalytic knowledge may be a reliable means to reduce the environmental impacts of wastewater and support sustainable wastewater processes.

Introduction

Crude oil manufacturing requires large amounts of water and generates a high volume of wastewater containing organic and inorganic compounds (A. T. Nawaf *et al.*, 2021). Wastewater production is almost 1.6 times that of crude oil and contains hazardous compounds that can harm the environment such as copper, lead, as well as salts (Nsaif *et al.*, 2023). In particular, wastewater is produced from two primary sources: It may be naturally present in the hydrocarbon reservoir or a byproduct of injected water used during crude oil production (Kayan & Kayan, 2021). The metal compounds in wastewater pose significant environmental concerns (J. Humadi *et al.*, 2023). Hence, this water is frequently permitted for discharge to the environment (El Kaim Billah *et al.*, 2023). The effects of discharging wastewater into the sea are often described in terms of the water's toxicity (Jafer *et al.*, 2023).

Furthermore, conservative methods are inadequate to address metal compounds produced by human activity and eventually released into the environment, with impacts (Mousa Al-Zobai & Hassan, 2022). Using these methods such as adsorption, membrane filtration, and ion exchange, aqueous compounds are often readily separated (Abdulateef *et al.*,

2021). However, these methods were usually secondhand to luxury-Produced Water (PW) (Alakoul *et al.*, 2021). This restraint has spurred the development of more dynamic and environmentally friendly methods (Alturki *et al.*, 2021). On the other hand, the Advanced Oxidation Processes (AOPs) were increased in tandem with the quantity of free radicals produced from the catalyst agent or hydrogen peroxide. One likely process is the increased production of free radicals with a high potential to oxidise metals (Naeem *et al.*, 2018).

Chemical processes are also applicable to a broader range of metals such as copper, lead, and nickel, in PW (Hassan *et al.*, 2022). These processes can efficiently eliminate persistent compounds from the wastewater (J. I. Humadi *et al.*, 2024). Consequently, these occurrences have the potential to destroy the pollutants and turn them into harmless inorganic molecules (Hassan *et al.*, 2019).

In recent years, maintenance has focused on advanced treatment technologies such as electrooxidation, which generates free radicals (Atiyah *et al.*, 2020). The first paper on the use of electrooxidation treatment for wastewater in crude oil production was published in the late 1990s, highlighting many opportunities to fully apply these technologies (AlJaberi *et al.*, 2020). This study used a newly developed innovative electrocatalytic batch reactor for metal oxidation. It was specified that the Integrated Digital Baffle Electro-Photo Catalytic Oxidation Reactor (IDBEPCOR) would have a superior influence than a batch oxidation treatment. In this experiment, a specific amount of catalyst was used to enhance the oxidation of the copper compound in the oxidation reactor, followed by photooxidation.

Experimental

Analytical and Copper Measurement

For this work, a material oxidant purchased in Barcelona, Spain provided a 98% pure nanocatalyst [Titanium Dioxide (TiO_2)]. Aluminum and iron electrodes were among the most significant mutual capital used in the electrooxidation treatment, as depicted in Table 1. The wastewater containing copper metal was kindly provided. Notably, the wastewater used in these oxidation treatments is brought from an oil field that has been exposed to the environment.

The copper compounds in wastewater were measured using an Atomic Absorption Spectrometer (PerkinElmer, USA). Subsequently, a 100 mm slit, 6 mm high was heated and air-acetylene at a flow rate of 55 was used to measure the metal ion at 324.8 nm (Alamery *et al*., 2023).

Hence, the metal removal is calculated by equation (1):

$$\textbf{\textit{Copper removal}} = \frac{C_o - C_t}{C_o} X\ 100 \quad (1)$$

Anywhere: C_o & C_t metal amount before and after treatment (ppm).

Digital Baffle Catalytic Batch Reactor

This reactor was designed locally to exploit mass and heat transfer during combined photo- and electrooxidation treatment. Specifically, the digital baffled metal oxidation reactor features a digital mixer controller, three baffle accessories with a catalytic oxidation wall, an 18 cm impeller shift, and an impeller design with a 0.7 cm shift diameter (1.2 cm, 0.9 cm, and 2.5 mm of the impeller's dimensions). The

Table 1: Wastewater specification

Limit	Value	Limit	Value
Copper metal	3.1 (mg/L)	TDS	183215 (ppm)
pH	6.99	Density	0.998 (gm/cm^3)
TSS	17.3 (ppm)	COD	410.2 (ppm)
Turbidity	54.2 NTU	Viscosity	1.04 m Pa/S

photocatalytic reactor and power device were part of the full new plan. Figure 1 illustrates the electrocatalytic oxidation scheme in its form. The RXN-305D power supply, measuring 1.2 cm in diameter and 18 cm in length was still obtainable for this purpose. It was designed to reduce these unusable items while maintaining a 6 W UV disinfection power. In this trial, the electrocatalytic oxidation removed copper metal from wastewater at 1 Amp. The cathode electron measured 6 × 1.6 × 0.12 cm³ and the anode electrode measured 6 × 1.7 × 0.12 cm³.

Experimental Calculation

To calculate the metal oxidation and to regulate the impacts of the significant parameters, Minitab software was employed. The primary data for these parameters were process time (X_1), catalyst amount (X_2), agitation speed (X_3), and pH (X_4), as presented in Table 2.

Results and Discussion

The Characterisation of Nano Titanium Dioxide

Furthermore, the numerous functional groups present in the catalyst amount were identified by (Fourier Transform Infrared) FTIR spectroscopy (8400S, Shimadzu/Japan). Figure 2 illustrates the FTIR spectra of the commercial anatase catalyst agent in the 500-4,000 cm^{-1} range. The peaks in the spectra at 3,332.04 and 1,642.33 cm^{-1} are produced by the widening and bending of the -OH functional groups. Additionally, the spectra of antanse-TiO_2 demonstrate stretching vibrations of Ti-O and Ti-O-Ti at 591.11 cm^{-1} and 1,279.33 cm^{-1}, respectively (A. T. Nawaf & Abdulmajeed, 2024a). For each diffraction peak in the powder X-ray Diffraction (XRD) pattern of the TiO_2 nanocatalyst, Figure 3 depicts the crystal family of planes (Jabbarzare, 2022).

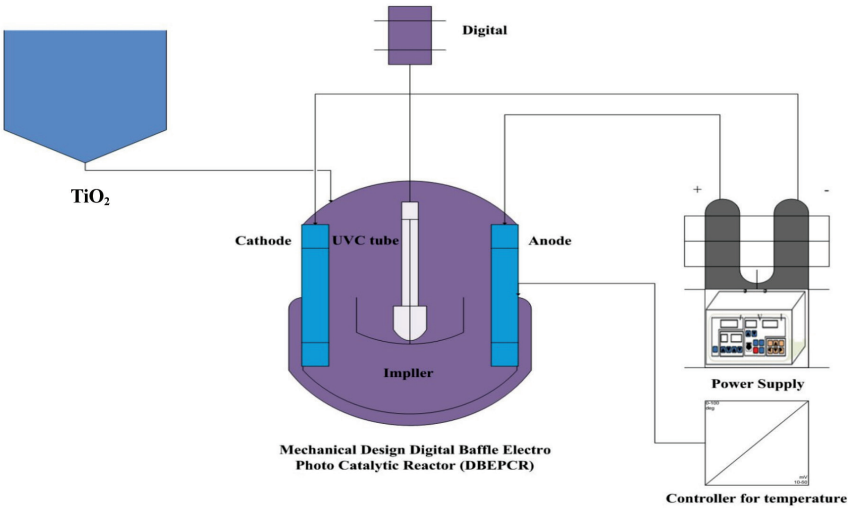

Figure 1: Schematic of photoelectrocatalytic oxidation batch reactor

Table 2: Oxidation variables

Parameters	Varies
X_1: Processes time (min)	10-50
X_2: Catalyst amount (ppm)	25-100
X_3: rpm	100-300
X_4: pH	3-9

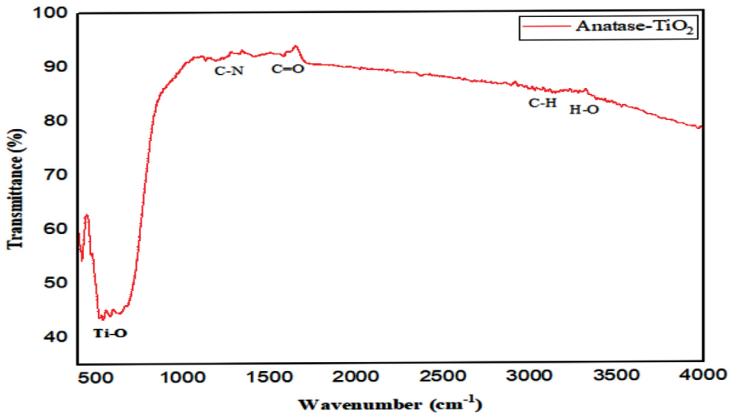

Figure 2: FTIR analysis of TiO_2

As a result, the preserved product exhibited numerous diffraction peaks that were readily identified as characteristic of the catalyst amount and could be precisely associated with TiO_2. The diffraction peaks from the (101), (004), (200), (204), and (215) orientations appear at 26.1, 36.3, 46.56, 61.66, and 76.55, correspondingly. The results designate that the characteristic crystallite size of TiO_2 is around 16.2 nm (Shihab *et al.*, 2020). However, a smaller crystallite might have a larger surface area, improving the material's photocatalytic activity. The Scanning Electron Micropscope (SEM) test, presented in Figure 4, specifies that the TiO_2 with a small size, the hydroxyl ions (OH-) in the wastewater have a lot of surface area to interact with the TiO_2 surface and produce free radicals (OH•), which are powerful oxidisers that oxidise a large number of copper metal (Yang *et al.*, 2022).

Statistical Analysis

It was investigated how several factors affected the digital baffled photocatalytic metal-oxidation reactor for wastewater treatment. A second-order metal removal provided a functional relationship between the important parameters and copper elimination via photoelectrocatalytic oxidation of PW, as depicted in Table 3.

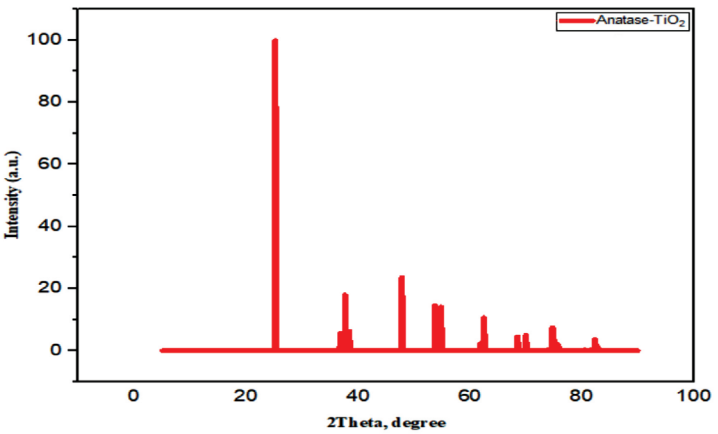

Figure 3: XRD analysis of anatase-TiO_2

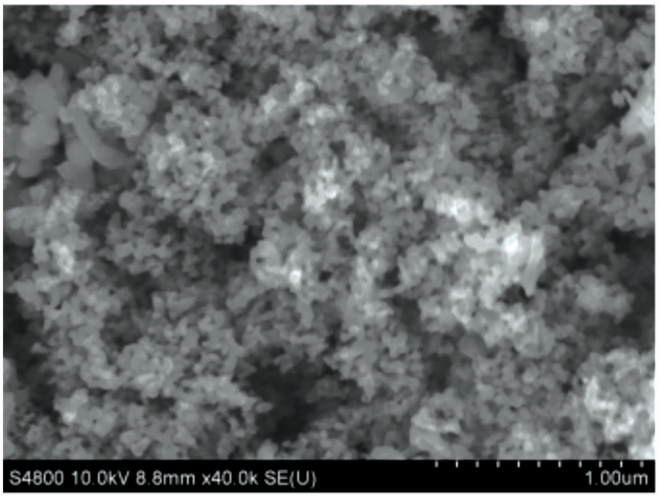

Figure 4: SEM analysis of anatase-TiO_2

Based on experimental data of photoelectrocatalytic oxidation treatment, equation (2) was identified in terms of real concerns concerning the elimination of metal to the parameter factors, representing the relation among these parameters:

$$\text{Metal elimination} = 56.2 + 0.5\,X_1 + 0.42\,X_2 + 0.0155\,X_3 - 2.480\,X_4 - 0.00175\,X_1^2 - 0.002373\,X_2^2 + 0.000019\,X_3^2 + 0.2917\,X_4^2 + 0.00023\,X_1X_2 - 0.000612\,X_1X_3 - 0.0154\,X_1X_4 + 0.00956\,X_2X_3 + 0.00158\,X_2X_4 - 0.0154\,X_3X_4 \quad (2)$$

The results for the Digital baffle metal oxidation batch reactor employing catalyst amount and electro-anode and cathode for free radical production, are illustrated in Figure 5. Thus, the standards of positive coefficients indicate that the rising issues related to these constants within the authenticated range improved the copper removal efficacy across all limitations used in the study (A. T. Nawaf & Abdulmajeed, 2024a).

The chief features of each metal-removal limit are demonstrated in Figure 6. The metal elimination in PW was affected by four factors: pH, agitation speed, catalyst agent concentration, and process time. The positive value of this constant indicates that each parameter was increased to restore the metal removal to the level observed for the difference under investigation. Moreover, as this limit shifts from low to high, copper removal increases (Ni *et al.*, 2007).

The best conditions were 50 minutes, pH 9, 300 rpm, 100 ppm catalyst agent, constant copper concentration, and 1 amp, the percentage of copper removed for photoelectrochemical metal was more than 97%. Notably, the best circumstances for conducting mechanical schemes design experiments are presented in Figure 7. The observed and predicted values of copper removal in PW are displayed in Figure 8 (Al-Zobai *et al.*, 2020).

Effect of Catalyst Amount

The amount of catalyst required to stain the copper while maintaining the other coefficients and amount constant was determined, the digital baffle oxidation treatment was revealed to benefit from catalyst concentrations in the range of 25 ppm to 100 ppm. Hence, the association between TiO_2 concentration and copper elimination efficiency is illustrated in Figure 9. The amount of copper decreased as the catalyst concentration increased after 50 minutes of photoelectrocatalytic oxidation. Conversely, the elimination efficiency reached an all-time high of 90.9% at a catalyst concentration of

Table 3: Photoelectrocatalytic oxidation

No.	Processes Time (min)	Catalyst Amount (ppm)	RPM	pH	Metal Elimination
1	50	25	200	6	76.4
2	50	62.5	200	3	85.3
3	10	25	200	6	72.1
4	50	62.5	100	6	88.8
5	30	62.5	300	3	90.4
6	50	100	200	6	96
7	30	25	100	6	75.1
8	30	62.5	100	9	91.1
9	50	62.5	200	9	97.2
10	30	100	100	6	84.9
11	50	62.5	300	6	90
12	30	25	200	3	74.8
13	30	62.5	100	3	84.1
14	30	100	200	3	89.2
15	30	100	200	9	96.7
16	30	62.5	300	9	97.3
17	30	62.5	200	6	85.3
18	30	25	300	6	80.8
19	30	62.5	200	6	87.2
20	10	62.5	200	9	93.2
21	30	100	300	6	94.1
22	10	100	200	6	87.5
23	30	62.5	200	6	86
24	10	62.5	100	6	87.4
25	10	62.5	200	3	80.3
26	10	62.5	300	6	85
27	30	25	200	9	81

100 ppm because free radical production was less reactive in the presence of TiO_2, lowering the total removal efficiency. Low nanocatalyst levels, though, will also lead to a shorter cohort of free radical production, which will lower the beneficial effectiveness. Thus, it was vital to maintain the ratio of high-to-low catalyst concentrations (Hassan & Al-Zobai, 2019).

Effect of pH

One of the main issues preventing the photoelectrocatalytic oxidation scheme from achieving competence has been pH. It meaningfully affects oxidant and substrate activities, catalyst stability, and copper removal. Specifically, the study examined the effect of pH on the efficiency of copper elimination in wastewater using a digital baffle

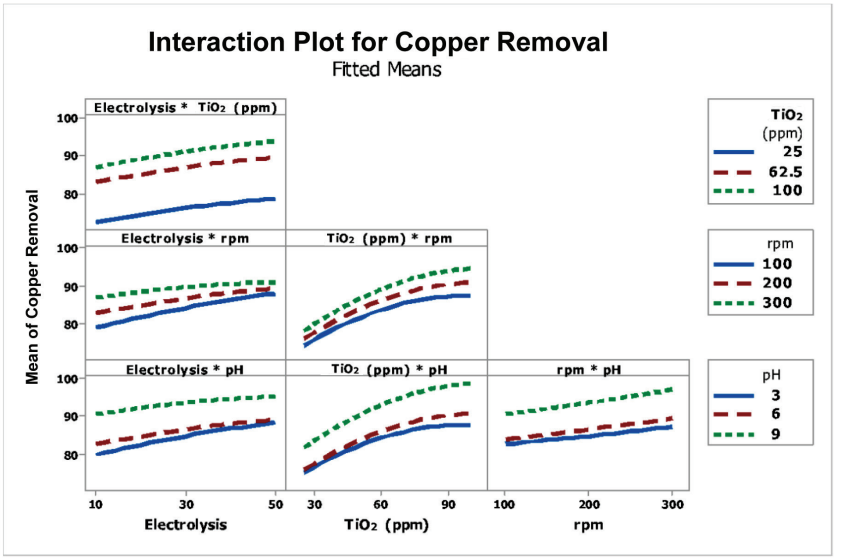

Figure 5: Interaction plot for copper elimination

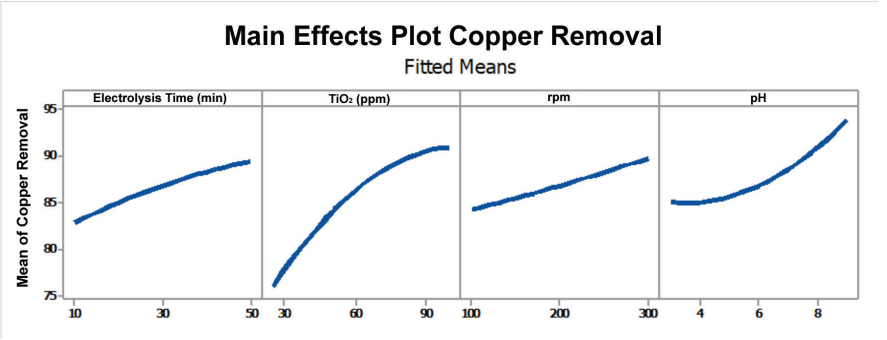

Figure 6: Effect of operating conditions

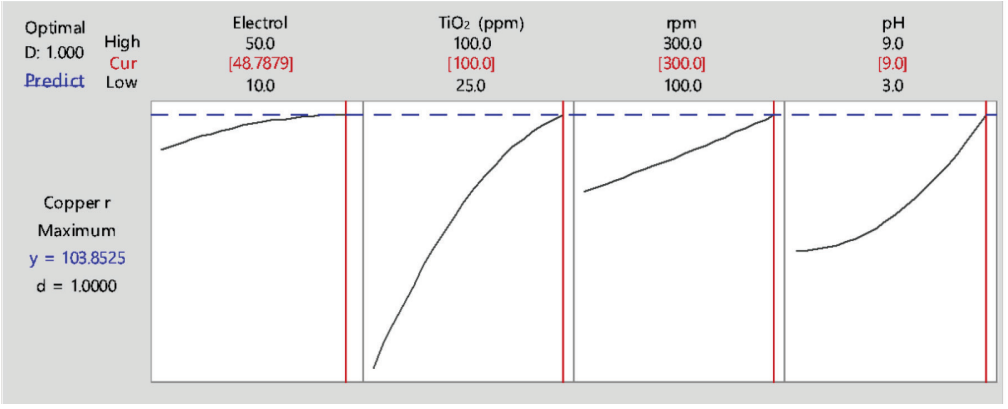

Figure 7: The best circumstances of copper removal

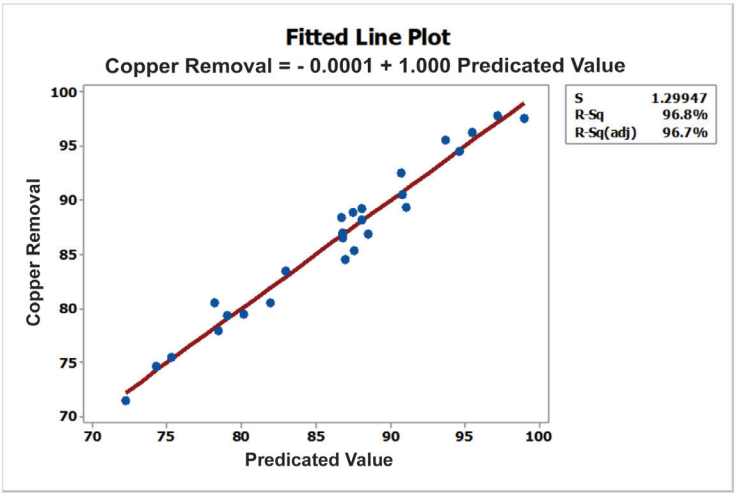

Figure 8: Metal elimination with predicated values

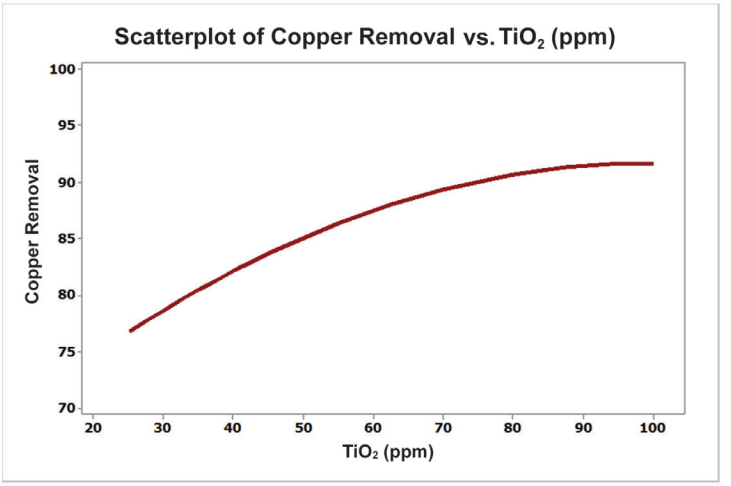

Figure 9: Effect of TiO_2 concentration on metal removal

photoelectrocatalytic batch reactor, following a series of experiments in which pH was varied from low to high while all other parameters remained constant (Cao *et al*., 2019). Figure 10 illustrates the results, in which, at the basic solution, a maximum copper removal competence of 93.9% was reached. It was reported that at pH 9, the copper metal has a high elimination rate. Moreover, a notable 84.8% reduction in copper elimination occurred directly above this value and continued to decrease until the acid solution. This could be due to the strong oxidising power of the nanocatalyst at pH 9. Note that the pH affects oxidation by regulating the production of free radicals and the ability of these radicals to oxidise increases with pH (Hassan & Shakir, 2024).

Effect of Process Time

The effect of the process time required for digital baffle catalytic oxidation to achieve the highest copper elimination capability was investigated (Alturki *et al.*, 2024). The initial pH and catalyst concentration were calculated. Additionally,

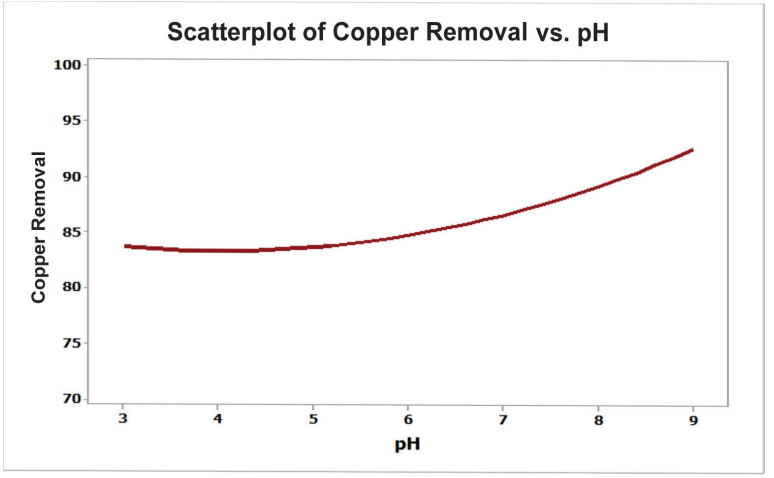

Figure 10: Effect of pH on metal removal

rapid oxidation by free radicals may lead to rigidity due to the oxidation scheme's rapid oxidation of copper compounds in wastewater. Thus, the association between the process time and the metal elimination in the investigation is illustrated in Figure 11 (Ahmed *et al.*, 2021).

Effect of Agitation Speed

The agitation speed, which affects the chemical reaction in low- and high-pH solutions was another factor affecting the oxidation process. Figure 12 depicts the percentage of copper ions extracted from the PW at different agitation speeds to determine the optimal agitation speed for mixing the nanocatalyst into wastewater and for the production of free radicals during electrophotooxidation treatment. In this context, the effects of agitation speed were noted. At 100 rpm and 300 rpm, the copper elimination efficiency rises from 82.9% to 90.9%, respectively. Notably, agitation speed-up surges cause the catalyst concentration to break down copper metal in PW, produce more free radicals, and boost oxidation efficiency (Alamery & Hassan, 2022).

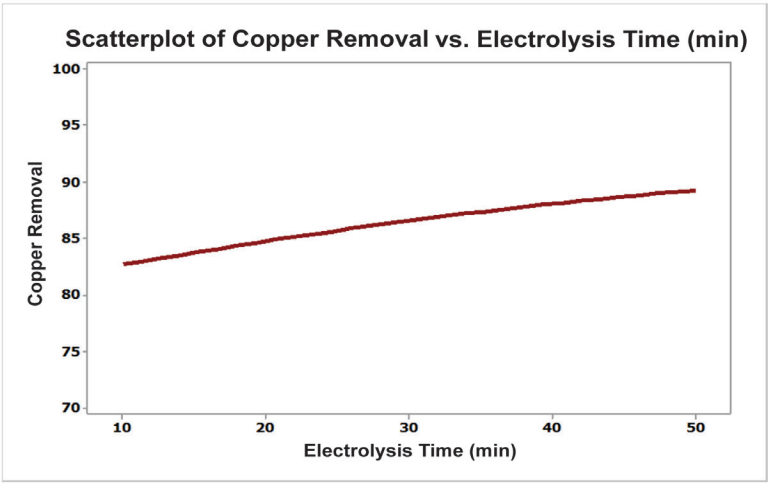

Figure 11: Effect of process time on metal removal

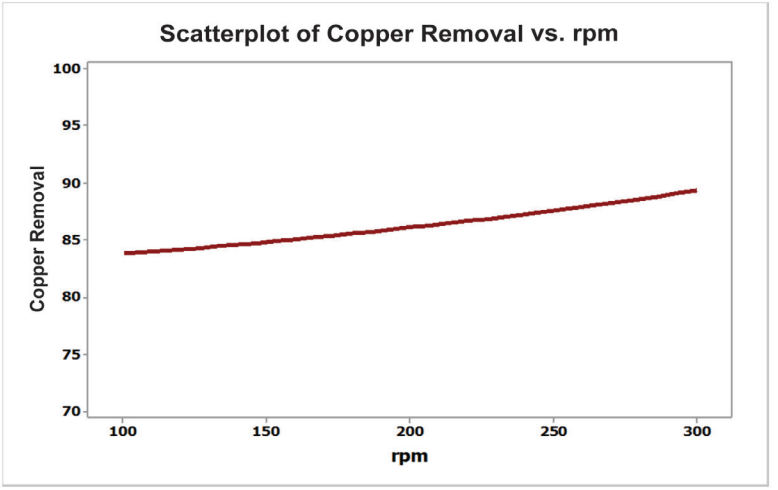

Figure 12: Effect of rpm on metal removal

As presented in Figures 13, 14, and 15, the contour plot for copper removal in wastewater demonstrates how process time (x-axis) and TiO_2, pH, and agitation speed (y-axis) affect copper removal. According to the figures, increasing the electrolysis time in basic solution and at high rpm increases copper elimination. Compared with low pH, photooxidation processes between wastewater and TiO_2 at pH 9 increase copper removal.

The Photo-electrooxidation Mechanism

The electrooxidation treatment is effective when used with this method (Figure 16). Gas bubbles (oxygen and catalyst amount) must be free from both the anode and cathode electrodes during the photoelectrocatalytic oxidation technique, which aids TiO_2-supported lightweight copper compounds in moving toward the surface of the treated wastewater (Jasim & Aljaberi, 2023). Therefore, based on the electrode's catalyst

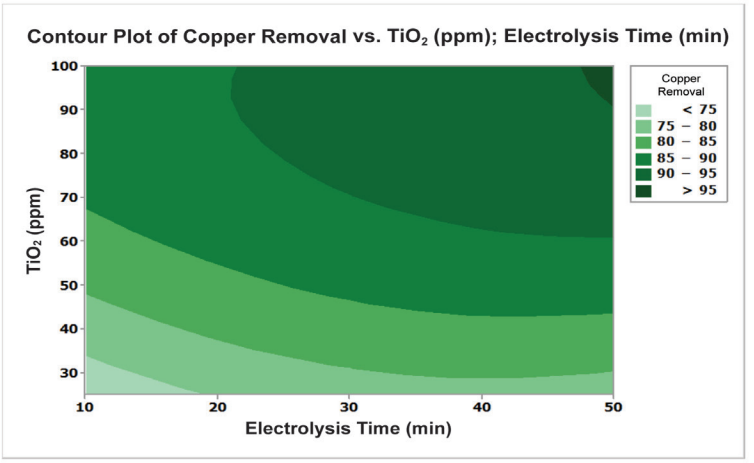

Figure 13: Contour plot of copper removal vs. catalyst and process time

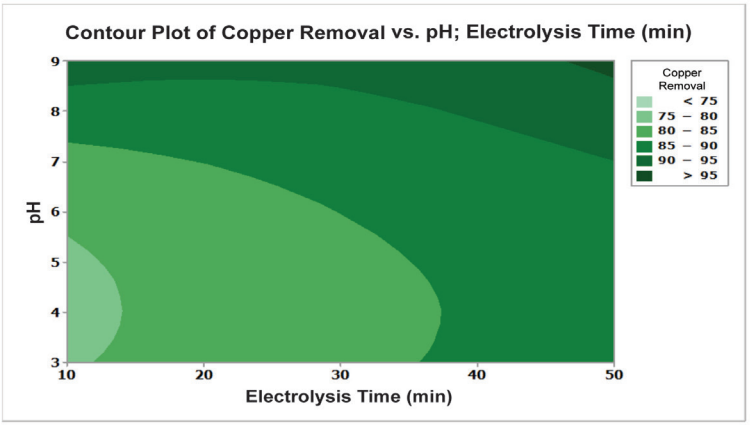

Figure 14: Contour plot of copper removal vs. pH and process time

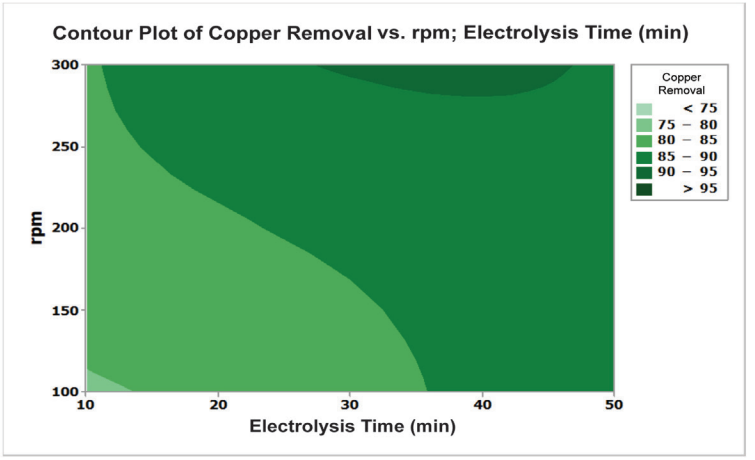

Figure 15: Contour plot of copper elimination vs. rpm and process time

amount for free radical production, this process proceeds according to the relationship between metal compounds and free radical production (Abdulateef *et al.*, 2018).

Over the past 40 years, extensive research has been conducted on photocatalysis and electrocatalysis, which use semiconductor metal oxides as catalysts. Numerous semiconductors have been proven to be photo-catalysts, including ZnO and TiO_2. However, because they tend to disintegrate and produce toxic byproducts via electrocatalysis, several other semiconductors such as TiO_2 cannot be employed for ecological cleaning when the energy (hv) applied to a semiconductor is equal to or greater than its band gap energy. Figure 17 illustrates how valence-band electrons combine with conduction-band electrons to form an electron-hole pair (Hassan & Shakir, 2024).

Furthermore, when the electron-generated hole in the valence band disperses to the surface of the TiO_2 and joins with the adsorbed water material, free radicals are produced. These oxidants, along with holes on the TiO_2 surface, may oxidise nearby copper metal. The electrons in the conduction band contribute to decreased activity when superoxide radical anions are produced in the presence of ambient molecular

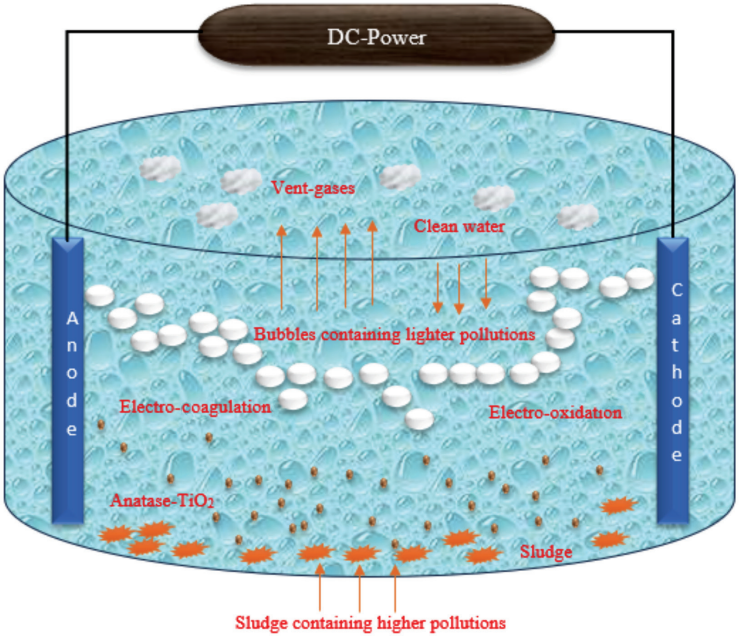

Figure 16: The mechanism of the hybrid treatment

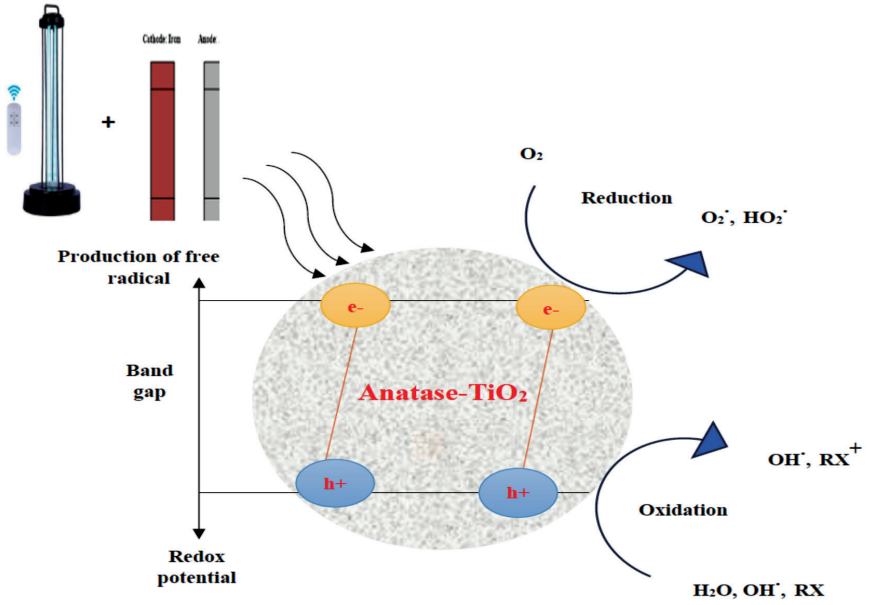

Figure 17: Photocatalysis mechanism

oxygen. To drive an electron to move from the valence band to the conduction band and reduce the band gap energy, an electron can provide the energy required. Hence, the fundamental reaction processes in the catalytic treatment were assumed using equations (3-6) (Al-Hassan & Shakir, 2024).

$$TiO_2 + EC \rightarrow E_{CB}^{-} + h_{VB}^{+}, \quad (3)$$

$$H_2O + h_{VB}^{+} \rightarrow \cdot OH + H^{+}, \quad (4)$$

$$O_2 + e_{CB}^{-} \rightarrow O_2^{\cdot -}, \quad (5)$$

$$O_2^{\cdot -} + H_2O \rightarrow \cdot OH + OH^{-} + O_2 + HO_2^{-}. \quad (6)$$

Conclusions

The results of this study go beyond technological novelty, demonstrating that electrocatalytic oxidation is a reliable and effective method for treating copper compounds in PW. Moreover, several important areas need to regain understanding and demand for mechanical design methods. The particle size is vital to assuring the heftiness and generalisability of the results. Notably, larger and more varied samples may demonstrate the hybrid of photo and electro for catalytic treatment, with scalability across the numerous types of PW examined in the work.

The second-order mathematical model provided a perfect fit to the experimental data. Copper removal in wastewater is 98.8% under the best conditions. Thus, the work established that photoelectrocatalytic oxidation in a batch reactor can be used to remove metal ions from wastewater. Additionally, it provides a basis for smearing the electrocatalytic oxidation method for metal removal from PW.

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Conflict of Interest Statement

The authors declare that they have no conflict of interest.

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