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Research Article

Mechanical-Chemical Electro-catalytic Degradation of Heavy Metals in Produced Water: Statistical Analysis and Process Optimization

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Abstract:

To treat produced water (PW), this work uses the mechanical design of the Digital Baffle Electro Photo Catalytic Batch Reactor (DBEPCB) method as a viable supernumerary. Box Behnken Design (BBD) of Response Surface Methodology (RSM) in Digital Baffle Oxidation Reactor studied the electro reactor design consisting of anode and cathode electrodes made of aluminum and iron correspondingly, with a mechanical mixer for mixing the PW with TiO2 concentration to maximize presentation with optimization design methods. A second-order obvious relation between the metal removal and important factors were shown by the high coefficient value (R2 = 0.967). The validity and dependability of the proposed technique were evaluated through additional statistical analysis. Founded on unresolved values, a predicted regression model was created, and it showed excellent agreement with experimental values. Based on important parameters, this model produced the best equation for the experiential model to prediction copper removal (CR). Rendering to the BBD, at pH 9, 300 rpm, 50 min of electrolysis, and 100 ppm of TiO2 concentration, the ratio of CR augmented to 98.4%. According to the study's assumptions, the electro-catalytic technology that has been enhanced may be a dependable way to alleviate the ecological effects of PW and support maintainable wastewater treatment approaches.

1. Introduction

Crude oil production needed large volumes of water, including injecting water, process water, storm water, and sewage, are created during the production water with high organic and inorganic pollutants known as produced water [1]. Produced water volume is roughly 0.4–1.6 times that of treated crude oil. Its multifaceted composition consists of salts, free and emulsified oils, phenols, and organic acids making up the majority of its constituents addition to inorganic pollutants [2]. The PW generates as of two imaginable fundamentals, which can remain originate below/above or privileged the hydro-carbon area or after added liquids, used in the crude oil extraction. [3].The pollutants of organic and inorganic in produced water are of exact ecological concern [4]. This water often is allowable to be discharged to the environ-ment .Water's poisonousness and organic filling can usually describe the impact of discharging PW into the sea [5]. For the purpose that the wilds of oilfields reason ecological pollutions, treatment was energetic earlier removal .Convinced chemical pollutants created by human activities and finally released into the atmosphere cannot be sufficiently treated by conventional treatment of wastewater[6] .Aqueous contaminants are frequently uninvolved using conventional skills for example biological, flotation, adsorption, coagulation-flocculation, and flotation [7]. Although these treatment were widely used to treat real wastewater, they are non-destructive meanwhile they only move the pollutant from the PW to the solid matrix [8]. This constraint has sparked the formation of wastewater action approaches that are more active and environmentally benign [9]. The oxidation method as a post-treatment based on advance oxidation processes (AOPs) was one choice for cleansing produced water from organic and inorganic pollutants due to their ability to quickly break down stubborn and non-biodegradable materials in wastewater, AOPs have appeared by way of a viable another for waste water treatment [10]. As the quantity of free radicals produced rises,

consequently does the AOPs' presentation. Using high energy electromagnetic waves was one potential technique to boost the generation of free radicals ($\cdot_{OH}$).While oxidation means can result in nearly comprehensive mineralization of organic and inorganic pollutants and are efficient for a wider spectrum of metal in wastewater[11]. These method have ability of wicked squalor of obstinate contaminants in the water environment. These events can completely damage the contaminants into innocuous inorganic substances [12] . In original year's upkeep have were concentrating on advanced oxidation developments remains nameless by way of electro-oxidation [13]. Though the first investigation on the request of electrochemical procedures aimed at the action of wastewater in the oil manufacturing was available in the late 1990s, there are still numerous chances to implement these skills at full scale [14] . The significant drawbacks of chemical action approaches are low elimination competence and extended separation time. This process requirements a big quantity of oxidant and conductivity alteration, creation the action not cost-effective [15]. This work used a newly developed innovative mechanical digital baffle electro batch reactor for photo catalytic oxidation, of copper compounds in produced water. It was stated that the DBEBR would have a greater impact than a batch reactor. In this experiment, we used titanium dioxide to help oxidize metal molecules by utilizing the DBEBR. Subsequently to the Electro catalytic system modeling. In order to determine the ideal working for the electro metal oxidation process—the stage at which the elimination of metal matter was maximized—the optimization method using Minitab-17 was finally put into practice.

2.Experimental Work

2. 1. Analytical and metal test

In this work, 99% pure nano titanium dioxide was gotten from a chemical oxidant that was bought in Barcelona, Spain. By adding logical scores of NaOH, H_2SO_4 and HCL from Scharlau, Spain, the pH of the PW was measured. All of the wastewater were made with organic and inorganic pollutants. Unique of the most vital mutual resources utilized in the electro-catalytic scheme were aluminum and iron electrodes. A sample of PW tainted with metal ions was kindly conventional from the close Iraqi oilfield. Pending the treatment process was advantageous, the produced water utilized in these tests are transported from an oilfield that has been exposed to the atmosphere and then preserved in an oxygen-rich situation that was comparable to their natural habitat. The description of produced water was given in Table 1. The copper metal in PW was determined using an Atomic Absorption Spectrometer (America, Perkin Elmer) at the deduction of each chemical oxidation test. To measure the copper ion, heat a 100 mm slit that is 6 mm high and air-acetylene at a flow rate of 55. Explanations occur in the peak area mode targeted at metal at 324.8 nm.

The equation (1) was used for Copper Elimination :

$$\boldsymbol{Copper\ removal} = \frac{C_o - C_t}{C_o} X100 \quad (1)$$

Anywhere: C_o &C_t copper concentration before and after treatment (mg metal/L).

Table 1. *Properties of Produced water*

Parameter	**value**	**Parameter**	**value**
Copper metal	2.31(ppm)	conductivity	214526 µs/cm
Turbidity	55.3 NTU	TSS	18.2 (ppm)
pH	7.14	viscosity	1.05 m Pa/S
Solution oxygen c	0.056 (mg/l)	iron	0.41 (ppm)
Specific gravity	0.9981	COD	415.3 (mg/l)

2.2Technology of Photo Electro-Oxidation

By using the photo-catalytic oxidation treatment (PECO), the DBEPCB was locally built to provide the most heat and mass transfer possible. The Al Muthanna University, Chemical Engineering Department has built a new design of baffle digital reactor. Table 2 shows the pilot plant's stipulations. The digital baffle electro-photo-catalytic batch reactor has three baffle attachments with the reactor wall (at the high 5 cm, width 0.35 cm, and thickness 1.3 mm), a digital mixer controller, a 20 cm shift of the impeller, and an impeller design (three flat-blades turbine) with a shift diameter of 0.7 cm (the impeller's dimensions were 1.2 cm, 0.9 cm, and 2.5 mm). The entire experimental scheme involved of the photo chemical reactor and power device. The shape of the electro oxidation system is shown in Figure 1. The RXN-305D power supply, which had a diameter of 1.25 cm and a length of 21 cm (4-pin single-ended), was still obtainable for purchase. It was designed to minimize these unhelpful items while maintaining the light's 6 W overpowering disinfecting potential. Using a steady current of one amp, the electro chemical procedure eliminated metal contaminants from the PW in this experiment. The dimensions of the cathode electron were 5 × 1.5 × 0.15 cm3 of iron. The anode

electrode's dimensions are 5 × 1.5 × 0.15 cm³ of aluminum. Throughout the PECO technique, the internal electrode space was kept at 4 cm and the real electrode area was kept at 15 cm^2. The RXN-305D was supplemented with different substances after sweeping the iron ions in the glass reactor for six minutes. The electrodes were kept in distilled water once they were not in use. To get rid of any possible pollution, the 1M hydrochloric acids were allowable to use toward corroding electrodes after each usage.

Table 2. *Stipulations of the Pilot Plant Used in the PECO Technology*

No.	Description	Specification
	DBEPFB reactor	200 ml
	Diameter of cylinder (pyrex)	6 cm
	Depth of reactor	8 cm
	Shift length and diameter of shaft	25 cm and 1 cm
5.	Impeller design (stainless steel)	Length 1.1 cm, width 0.7 cm and thickness 1.5 mm
6.	Baffle (design from Pyrex)	Three distributions on the wall of the batch
7.	Baffle design	High 5 cm, width 0.3 cm, and thickness 1.5 mm
8.	Preheater	Electrical heater
9.	Insulated reactor	Glass wool

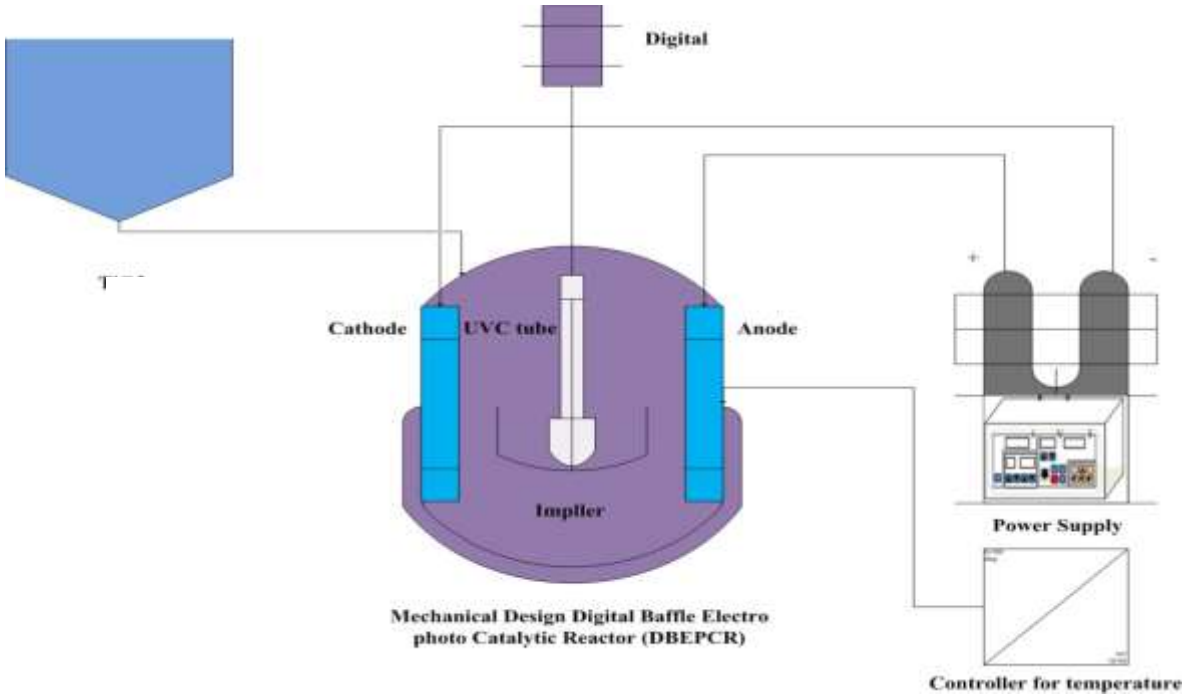

Figure 1. *Design of Digital Baffle Electro Photo Catalytic Batch Reactor (DBEPCB)*

2.3. Experimental Design

To design the treatment experimentations and determine the impacts of the important variables, a statistical method identified as the Response Surface Methodology-Box-Behnken method was working. The primary consequences of these variables were electrolysis time (X_1), and titanium dioxide (X_2), rpm (X_3) and pH (X_4), Table 2 shows the ranges of these variables.

Table 3*.Working parameters*

Variables	Ranges
X_1: Time (min)	10-50
X_2: TiO_2 concentration (ppm)	25–100
X_3: rpm	100–300
X_4: pH	3-9

3. Results and discussion

3.1 Statistical Analysis

It was inspected how several limits affected the photo electro -catalytic oxidation of copper metal in PW. In twenty-seven different studies, the batch oxidation experimentations were designed using the BBD method. At the foundation of the coded unit based on the untried results, a second-order model offered the helpful relationship between the important variables and removal. Based on the statistical analysis with Minitab software, the metal concentration was attained as anticipated by the equation. The operational variable values and the resulting copper removal responses for each run are explained in Table 4.

Table 4. *Results of BBD test*

NO	***time (min)***	***TiO_2 concentration (ppm)***	***rpm***	***pH***	***Copper removal***
1	10	25	200	6	71.4
2	50	25	200	6	77.9
3	10	100	200	6	88.4
4	50	100	200	6	95.6
5	30	62.5	100	3	83.5
6	30	62.5	300	3	88.9
7	30	62.5	100	9	90.5
8	30	62.5	300	9	97.8
9	10	62.5	200	3	79.5
10	50	62.5	200	3	86.9
11	10	62.5	200	9	92.6
12	50	62.5	200	9	96.3
13	30	25	100	6	74.6
14	30	100	100	6	85.4
15	30	25	300	6	80.5
16	30	100	300	6	94.5
17	10	62.5	100	6	79.4
18	50	62.5	100	6	89.2
19	10	62.5	300	6	84.5
20	50	62.5	300	6	89.4
21	30	25	200	3	75.4
22	30	100	200	3	88.2
23	30	25	200	9	80.5
24	30	100	200	9	97.6
25	30	62.5	200	6	86.5
26	30	62.5	200	6	87
27	30	62.5	200	6	86.9

The mathematical equation (2) was recognized in terms of real concerns related to the removal of copper to the working factors, indicating the relation among these variables, based on untried results:

$$\text{Copper removal} = 57.7 + 0.470\,X_1 + 0.388\,X_2 + 0.0155\,X_3 - 2.480\,X_4 - 0.00175\,X_1^2 - 0.002373\,X_2^2 + 0.000019\,X_3^2 + 0.2917\,X_4^2 + 0.00023\,X_1X_2 - 0.000612\,X_1X_3 - 0.0154\,X_1X_4 + 0.00956\,X_2X_3 + 0.00158\,X_2X_4 - 0.0154\,X_3X_4 \quad (2)$$

The ANOVA test produced the best model. Founded on the F and P tests for electro catalytic

oxidation with UV light, Table 5 for remove copper (CR). If the Fisher value is larger, a regression equation will exhibition additional variation in the removal. The BBD capability is documented after it has been used and improved by some sources. If the P-value was less than 0.05, the ratio of model variability could be accounted.

Table 5. *ANOVA for copper elimination*

Foundation	DOF	Seq. SS	Adj. MS	Fisher Value	P–test Value
1-Model	14	1284.07	91.719	26.07	0
Linear	4	1120	280	79.59	0
X_1	1	130.02	130.021	36.96	0
X_2	1	666.03	666.03	189.32	0
X_3	1	90.75	90.75	25.8	0
X_4	1	233.2	233.201	66.29	0
Square	4	146.44	36.609	10.41	0.001
X_1^2	1	2.61	2.613	0.74	0.406
X_2^2	1	59.41	59.408	16.89	0.001
X_3^2	1	0.19	0.187	0.05	0.821
X_4^2	1	36.75	36.75	10.45	0.007
2-Way Interaction	6	17.63	2.939	0.84	0.565
X_1*X_2	1	0.12	0.122	0.03	0.855
X_1*X_3	1	6	6.002	1.71	0.216
X_1*X_4	1	3.42	3.423	0.97	0.343
X_2*X_3	1	2.56	2.56	0.73	0.41
X_2*X_4	1	4.62	4.622	1.31	0.274
X_3*X_4	1	0.9	0.902	0.26	0.622
Error	12	42.22	3.518		
Lack-of-Fit	10	42.08	4.208	60.11	0.016
Pure Error	2	0.14	0.07		
Total	26	1326.29			

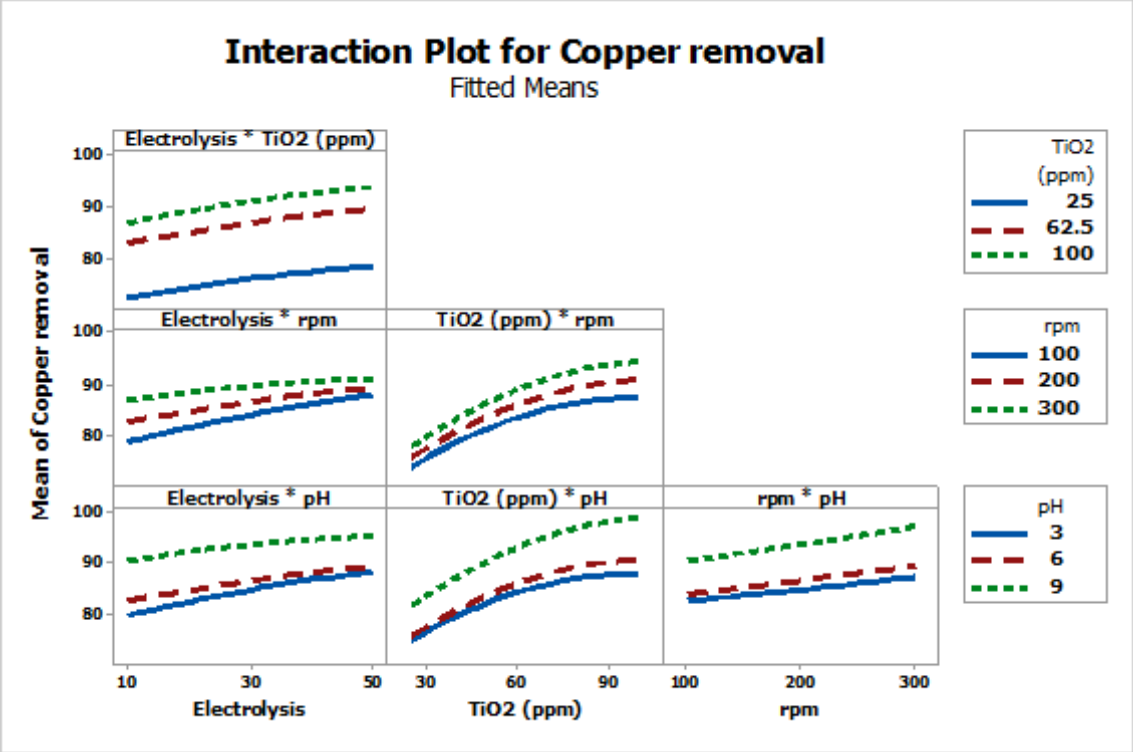

Figure 2. *Interaction plot for copper elimination*

Figure 2 presented the findings for metal oxidation using photo-electro catalytic treatment therapy. The increasing factors related with these coefficients within the validated range enhanced the elimination efficacy for all limits employed in the work, as shown by the standards of positive coefficients [16].

Figure 3 shows the key possessions of each limit on the CR. Three influences influenced copper removal in produced water: pH, rpm, TiO_2 concentration, and electrolysis time. This constant's positive value indicates that all of the variables were raised in order to increase the recovery of the CR for the variation under study. Furthermore, a rise in CR is observed as this limit moves from low to high [17].

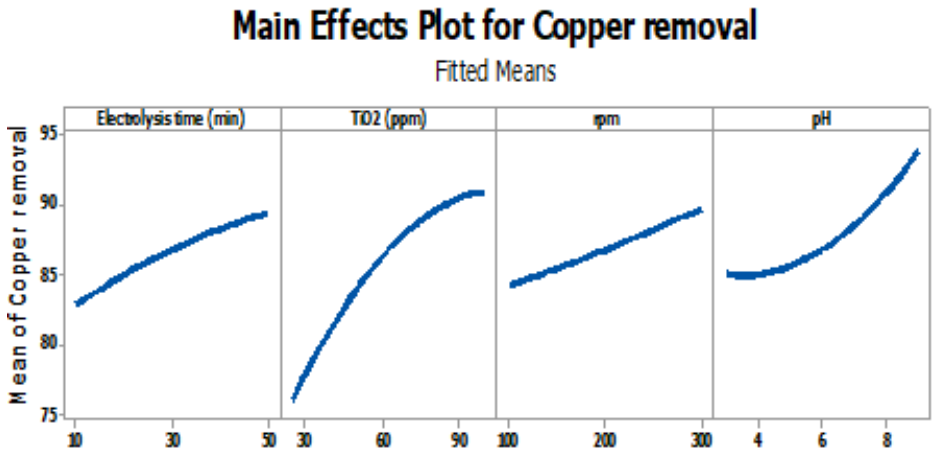

Figure 3. *Effect of operating conditions*

At 50 min, 9 pH, 300 rpm, 100 ppm of titanium dioxide concentration constant copper concentration, and 1 amp at room temperature, the percentage of copper removed for photo electro catalytic treatment was greater than 98.4%. The best circumstances for carrying out mechanical schemes design experiments, as established using Minitab software, are shown in Figure 4. The observed and predicted values of copper removal in produced water are displayed in Figure 5 [18].

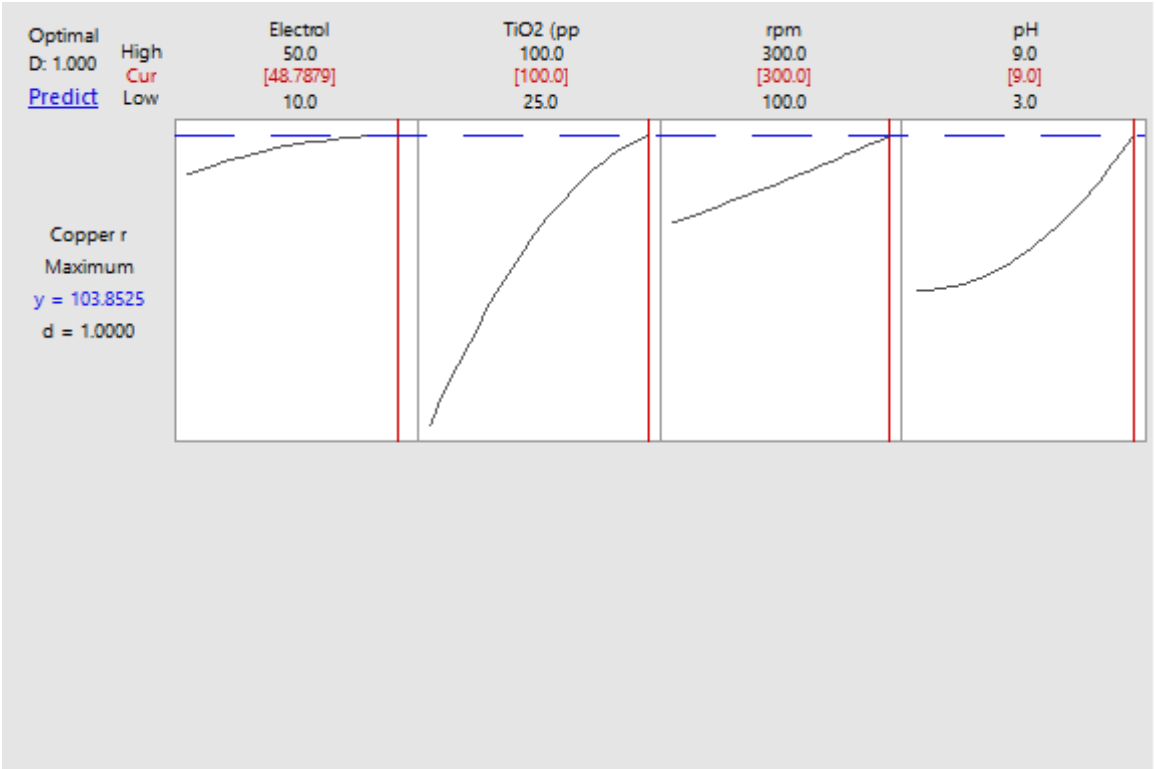

Figure 4: *The best conditions of oxidation*

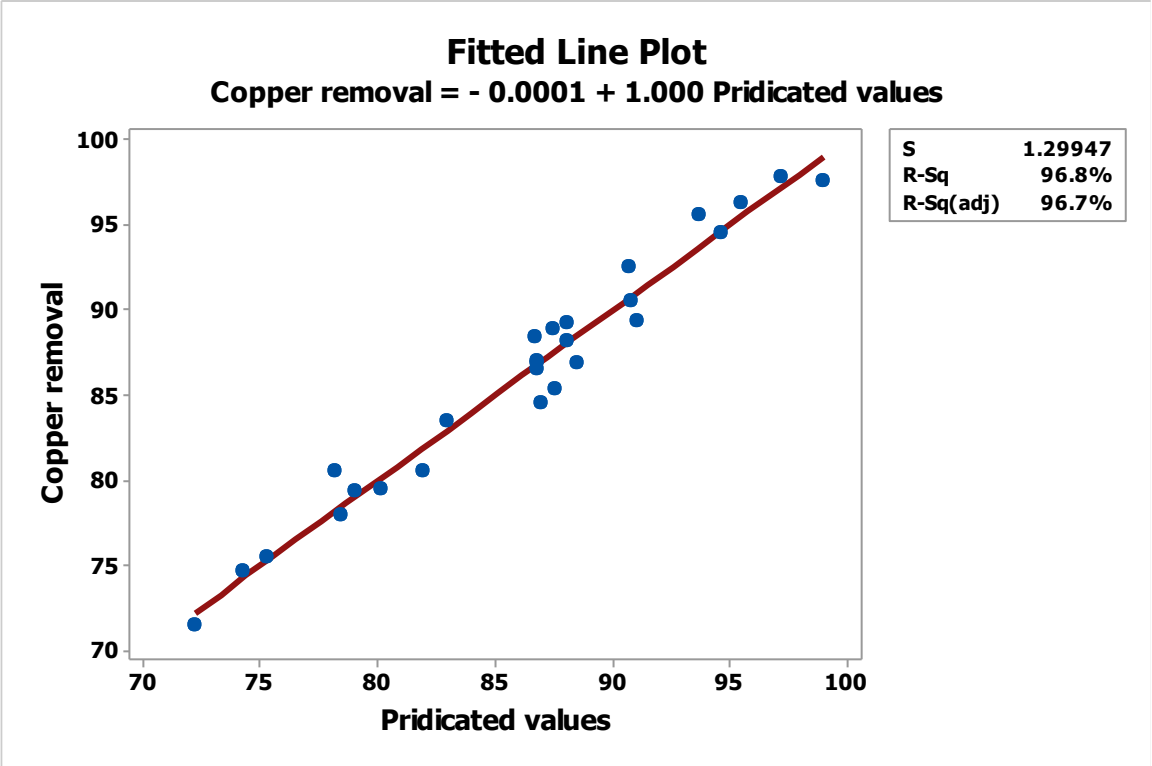

Figure 5. *Copper removal with predicated values*

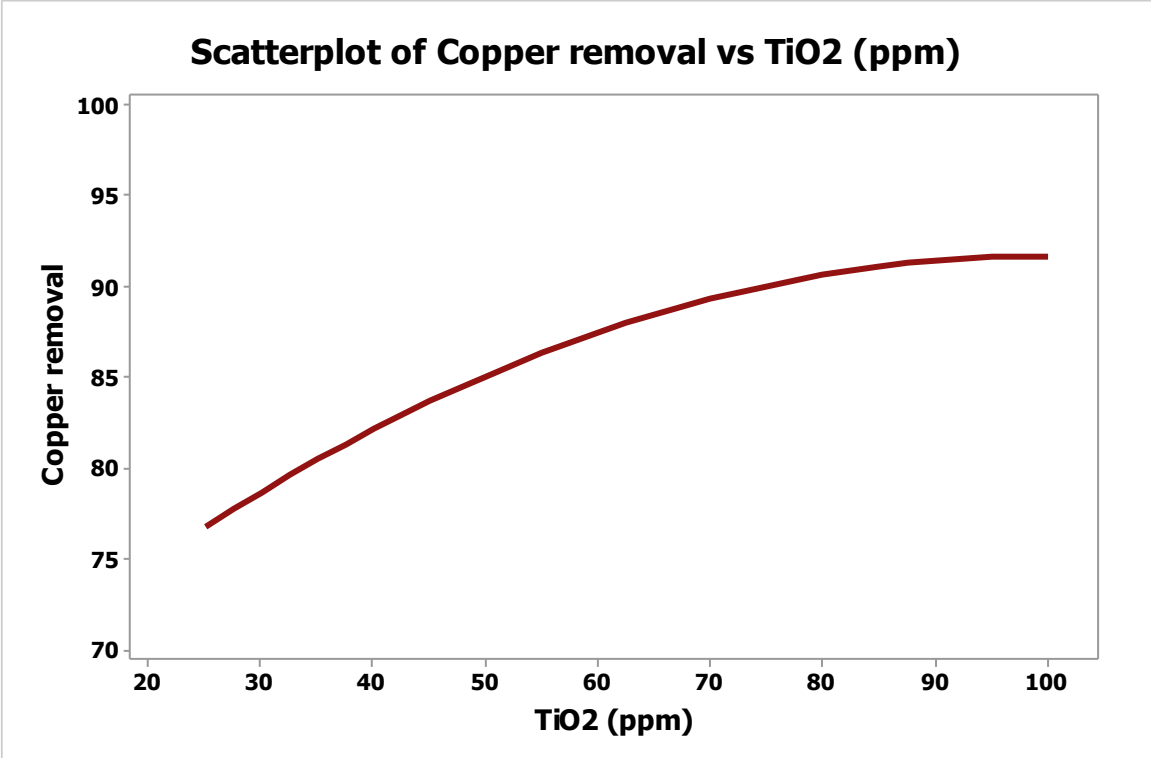

Figure 6. *Effect of TiO_2 concentration on CR*

3.2.1 Effect of TiO_2 concentration

The Electro catalytic oxidation procedure was qualified to profit from an initial concentration of titanium dioxide in the range of 25–100 ppm to upsurge the quantity of TiO_2 concentration required to stain the metal while keeping the other limits and amount constant. Figure 6 showed the correlation between the initial nano concentration and the metal removal efficiency. After 50 min of exposure

to room temperature oxidation, it is clear that the amount of metal that was degraded rose as the concentration of titanium dioxide grew. At 100 mg/L of TiO_2, the elimination effectiveness reached a all-out of 91.5%. Free radicals efficiently react with copper metal to generate clean water and oxides. An excess of nano concentration may result in a reduction in overall removal efficiency because radicals were less reactive. Though, low TiO_2 levels will also result in abridged cohort of free radicals, which will reduce the efficiency of treatment. So, maintaining the ratio of high to low titanium dioxide levels was crucial [19].

3.2.2 Effect of pH

pH has been found to be unique of the chief issues limiting the electro oxidation system's efficiency. It has a major impact on the stability of nano concentration, the control of metal speciation, and the activities of the oxidant and substrate. Finished a series of experimentations conducted in the pH range of 3 to 9 while keeping all other factors were constant, the work study investigated the effect of pH on the metal elimination efficiencies for produced water by mechanical design [20]. The results were shown in Figure 7 , a maximum elimination efficiency of 94.5% was touched at pH=9. It was observed that a high elimination ratio in the metal occurs when the solution is basic. Directly above this point, there was a noteworthy decline in metal elimination of 84.8%, which reduced gradually until pH=3. A pH of 9 yields the highest inorganic removal, while pH values below 9 result in a drop in copper removal. This might be as a result of nano concentration potent oxidizing power at high pH levels. Though, a high pH resulted in an important production of free radicals, which in turn caused the metal pollutants in the PW to breakdown. Since pH controls the generation of free radicals, it has an impact on oxidation. Moreover, as pH increased, so did radicals' capacity for oxidation [21].

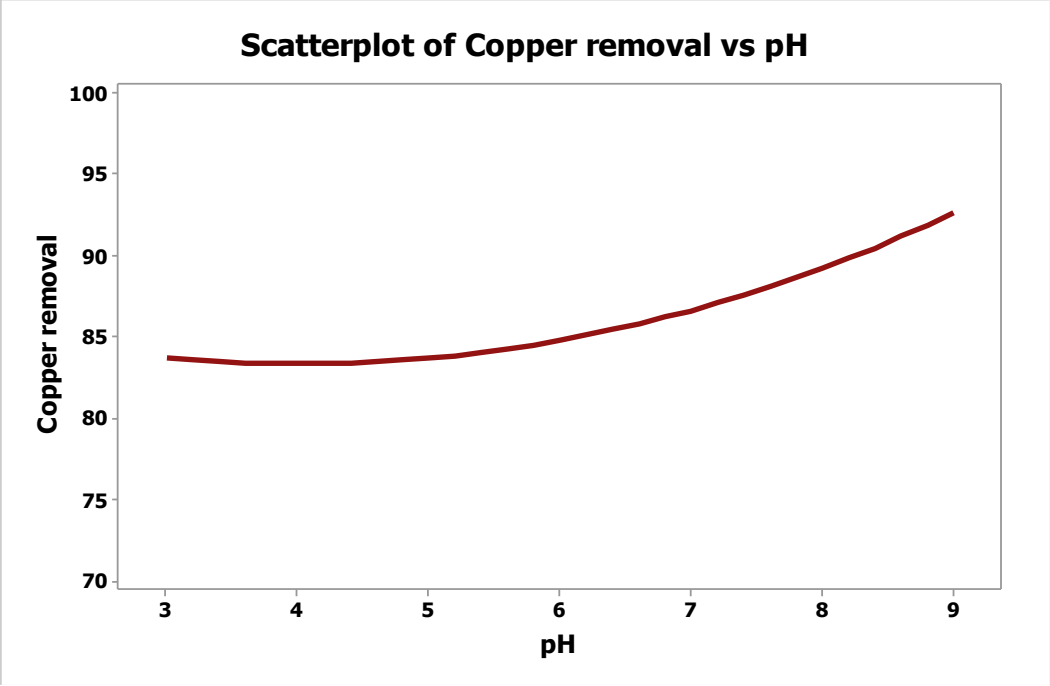

Figure 7. *Effect of pH on CR*

3.2.3 Effect of electrolysis time

A work was conducted to inspect the impact of the electrolysis time needed for the photo-chemical treatment to attain the highest metal removal competence. At room temperature, the initial pH and nano concentration values were established. The oxidation system's rapid oxidation of metal ions may cause the rapidly oxidizing free radicals to oxidize into hardness . Figure 8 shows the correlation between the electrolysis time and the copper removal in the experimental study. It could take many hours to finish the process that removes more complex or concentrated wastes [22]. Experimentations were carried out to regulate the best electro catalytic chemical efficiency time for copper elimination. It is clear from Figure 8 that the efficiency of copper elimination increased gradually and proportionately after then. An upsurge in the efficiency of copper elimination may be related to the chemical oxidation of copper via free radicals [23].

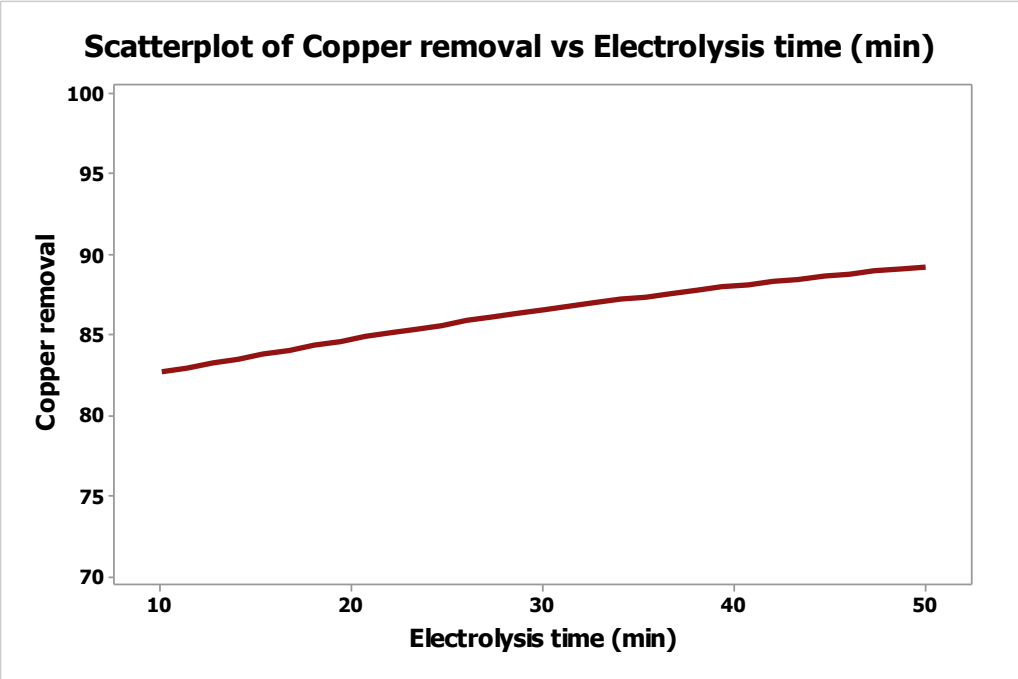

Figure 8. *Effect of electrolysis time on CR*

3.2.4 Effect of agitation concentration

The agitation speed, which affects the chemical relation in the acid and basic solution, was another element affecting the oxidation process. Figure 9 showed the percentage of copper ions extracted from the produced water at different concentrations. The effects of speed are observed. At 100 and 300 rpm, the copper elimination efficiency rises from 83.4% to 91.8% correspondingly. Speed increases cause titanium dioxide to break down more quickly, produce more free radicals, and boost oxidation efficiency [24]. As shown in Figures 10, 11, and 12, the contour plot for metal elimination responds to the electrolysis time on the y axis and changes with nano concentration, pH, and rpm, correspondingly. According to the results of the Figures, increasing the electrolysis time at basic solution and high rpm

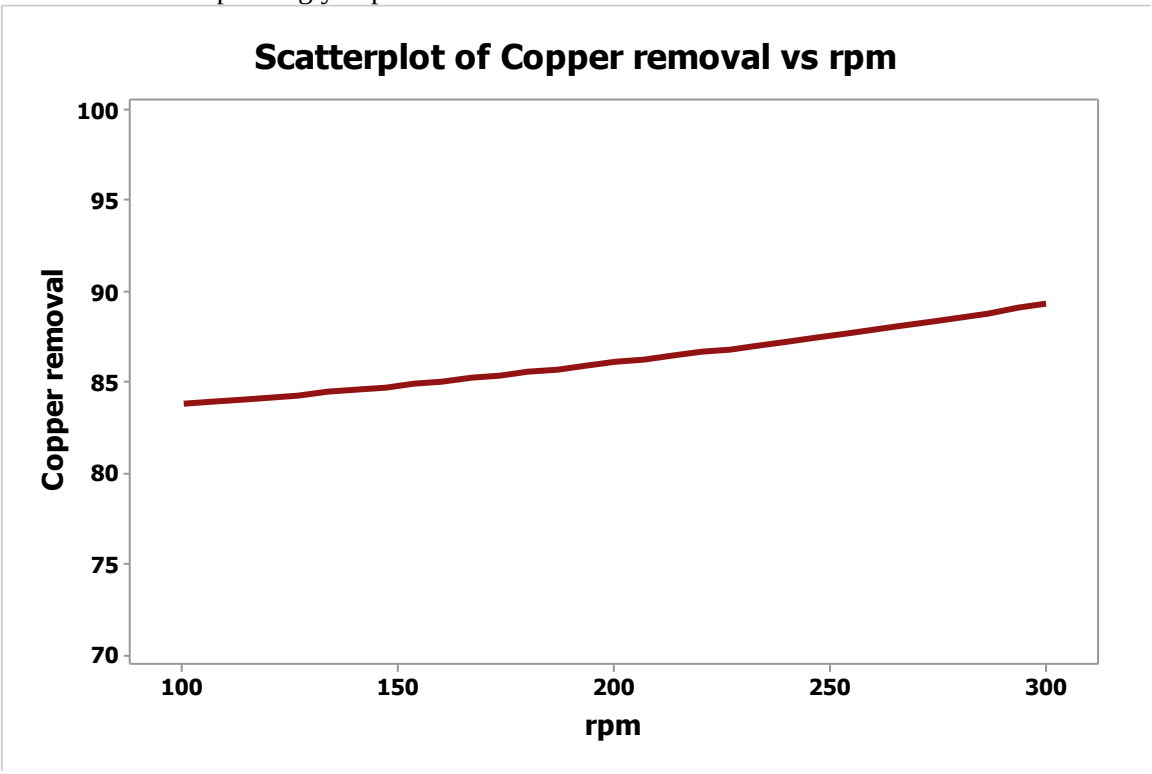

Figure 9. *Effect of Agitation speed on CR*

upsurges the removal of metal, while comparing the acid solution, the electro chemical treatment between PW and nano concentration decreases at high pH and upsurges the oxidation of metal in the produced water, cumulative the oxidation scheme's competence.

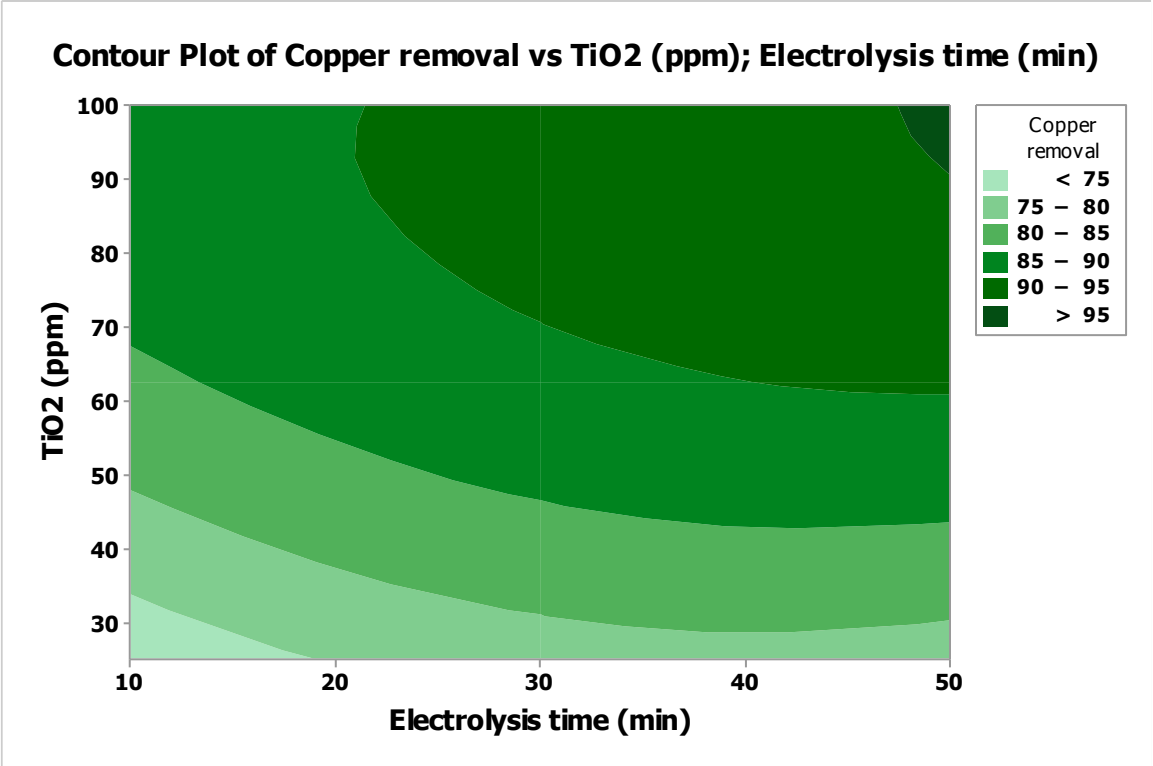

Figure 10. *Contour plot of CR vs TiO_2 and time*

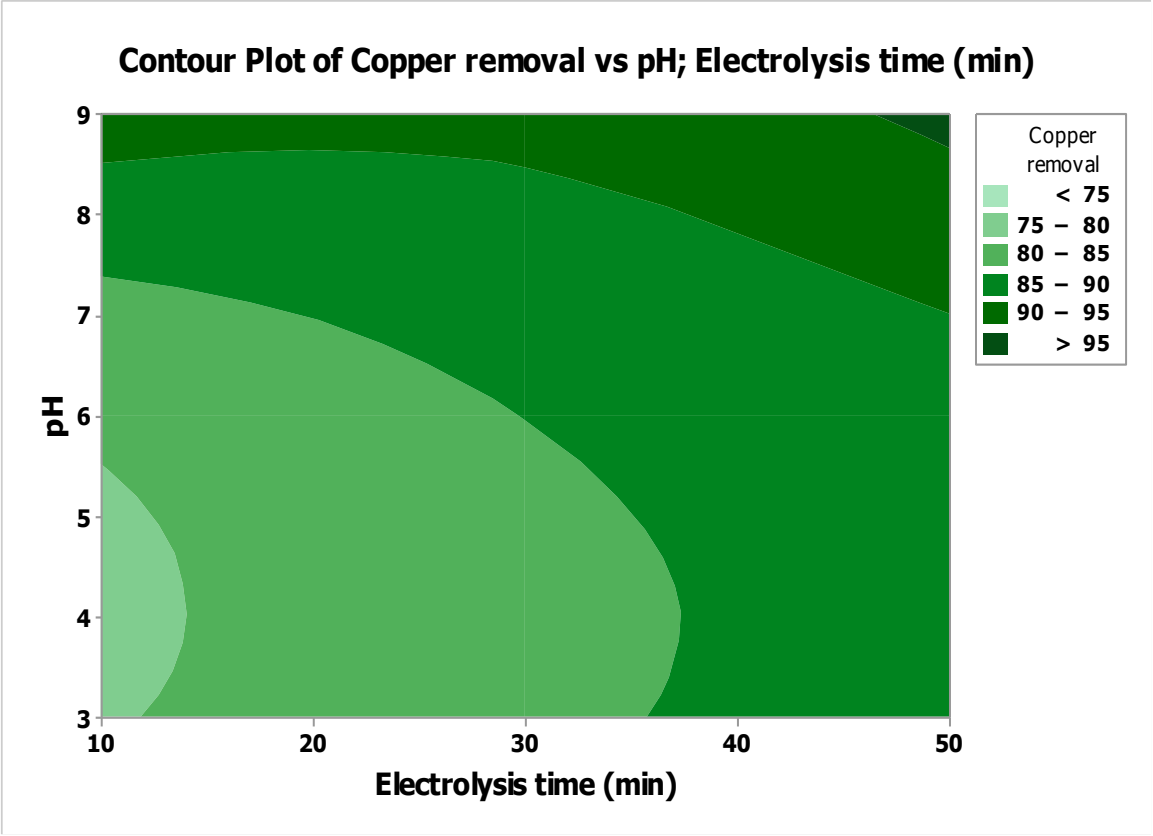

Figure 11. *Contour plot of CR vs pH and time*

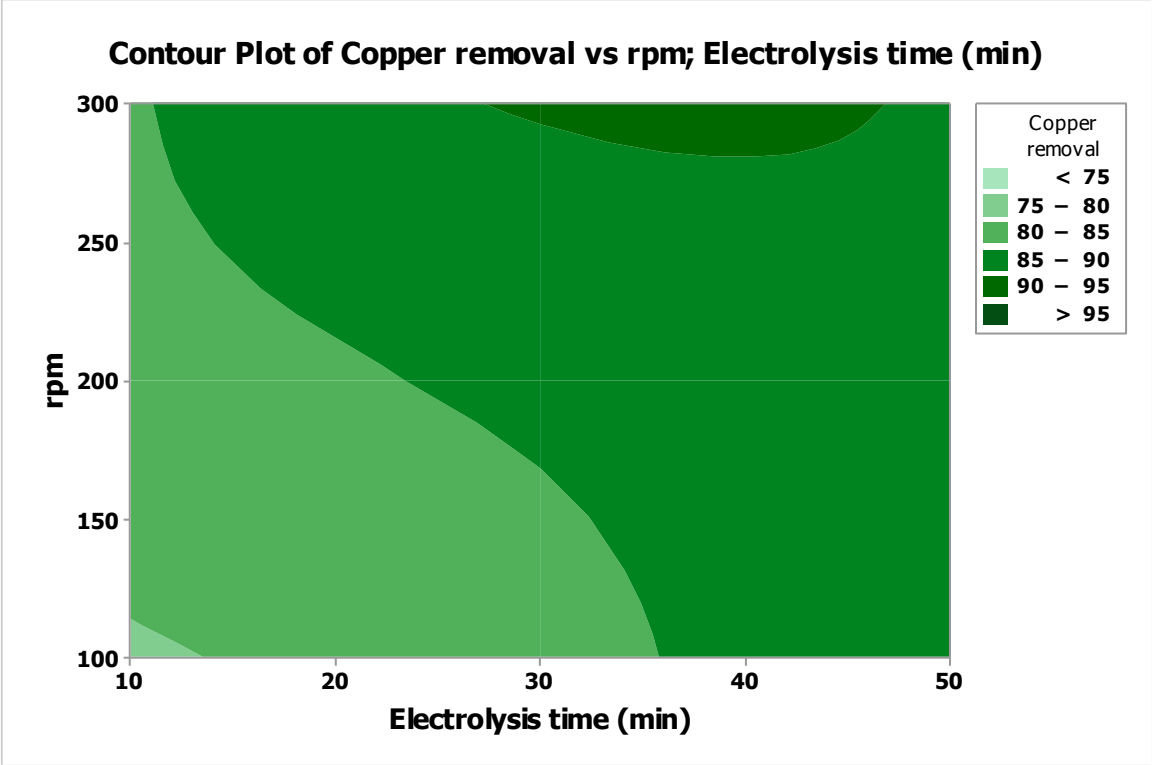

Figure 12. *Contour plot of CR vs agitation and time*

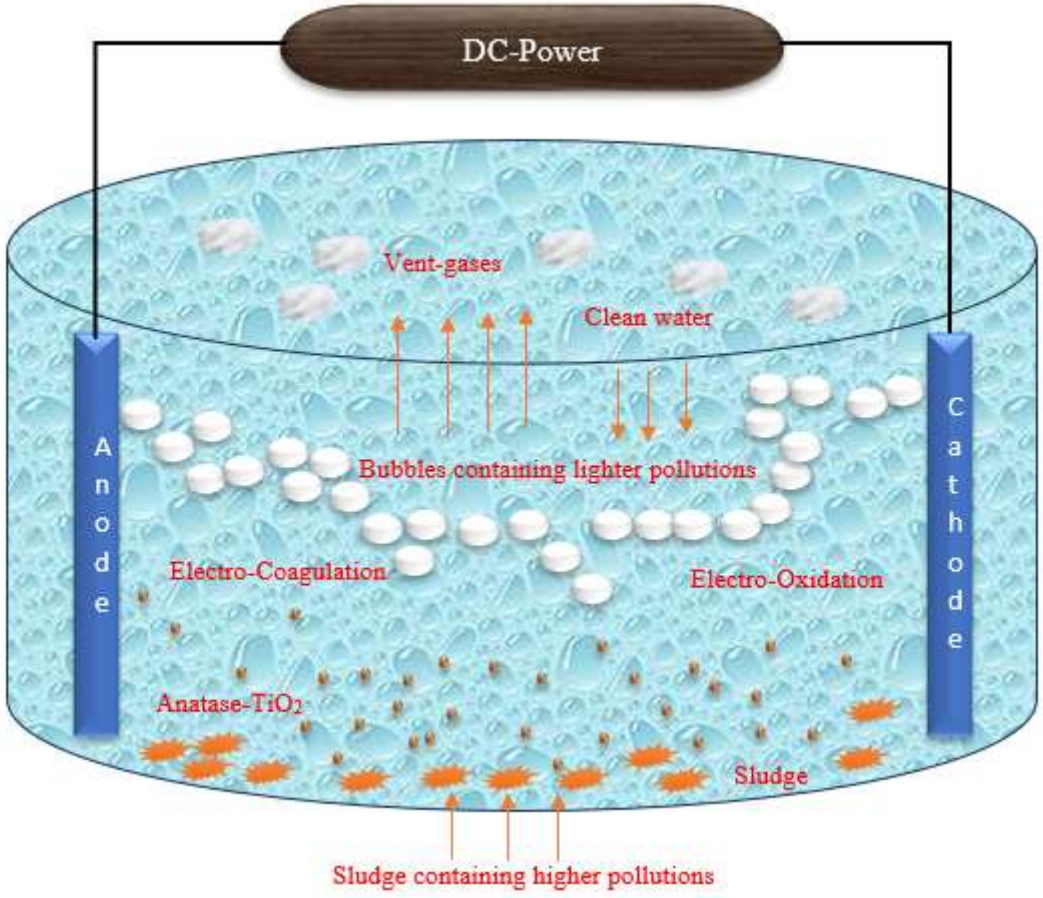

Figure 13. *Schematic of the hybrid process*

3.2 The Mechanism Suggested of Electro Oxidation

With this method, the electro-oxidation and electro-coagulation processes work together (Figure 13). The electro-oxidation process, which helps the nano catalyst of lightweight contaminants move toward the surface of the treated produced water, requires the release of gas bubbles (oxygen and titanium dioxide) from both electrodes. The relation between copper ions produced from the anode and hydroxyl ions emitted from the cathode causes the electrocoagulation process to proceed based on the electrode itself of the electrocoagulants [25] . Equations (3) to (5) explain the chemical reactions occurring in an electrocoagulation reactor in case of using (M) metal for both electrodes [26]:

1- The redox reactions on both electrodes resulted in the generation of electro-coagulants.

- At the anode with metal M:

$$M_{(S)} \rightarrow M^{+n}_{(aq)} + ne^- \quad (3)$$

$$2H_2O \rightarrow O_2 + 4H^+ + 4e^- \quad (4)$$

- At the cathode:

$$2H_2O + 2e^- \rightarrow H_{2(g)} + 2OH^-_{(aq)} \quad (5)$$

2- Emulsion breakdown, particulate suspension, and destabilization of the pollutants:

$$M^{+n}_{(aq)} + ne^- \rightarrow M_{(S)} \quad (6)$$

3- Aggregation of the destabilized phases to form flocs.

Thereby, the electrocoagulation method involves two cooperative procedures; the dissolution of anode metal to its ions then reacted with hydroxyl ions produced at the cathode. For aluminum electrode as an example (Eq. 7 and Eq. 8) [27]:

$$2Al \rightarrow 2Al^{+3} + 6e^- \quad (7)$$

$$Al^{3+} + 3OH^- \Leftrightarrow Al(OH)_3 \quad (8)$$

For iron electrode, two oxidation states of iron, Fe^{+2} (Ferrous) and Fe^{+3} (Ferric) are formed as shown in (Eq. 9 to Eq. 11) [14]:

$$Fe \rightarrow Fe^{+2} + 2e^- \quad (9)$$

$$4Fe^{+2} + O_2 + 4H^+ \rightarrow 4Fe^{+3} + 2H_2O \quad (10)$$

$$Fe^{+3} + 3H_2O \rightarrow Fe(OH)_3 + 3H^+ \quad (11)$$

4. Conclusions

The results of this work extend beyond technological novelty. Electrocatalytic oxidation of copper metal in PW has been established to be a reliable treatment solution for copper oxidation compounds in industrial wastewater. a number of crucial areas to recover mechanical design method comprehension and request. Enhancing the sample size is a crucial step in ensuring the results' generalizability and robustness. The scalability of the catalytic process across the several types of produced water that were studied in the work may be shown by larger and more varied samples. Our untried data was perfectly fitted through the second-order polynomial. Associated to best working conditions, we achieved a higher removal of 98.4% of CR. The work showed that copper metal might be mineralized through electrocatalytic development. Additionally, it delivers a basis for using the electrochemical oxidation method in applied demands to solve the inorganic chemical pollution in PW**.**

Author Statements:

- **Ethical approval:** The conducted research is not related to either human or animal use.
- **Conflict of 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
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- **Author contributions:** Salwan F. Alturki: Original draft, review, and editing of writing, software, methodology, and conceptualization. Formal analysis, methodology, writing (original draft), writing (review & editing), Mahmood Sh. Suwaed. Ahmed Ghareeb: Original draft writing, review and editing, and validation.
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- **Data availability statement:** The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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