New Design Electro-Catalytic Digital Baffle Batch Oxidation Reactor for Organic Removal from Refinery Wastewater

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Highlight:

- The reactor uses **electro-catalysis**, which involves applying an electric current to catalyze oxidation reactions.
- Electro-catalysis promotes more efficient breakdown of organic pollutants, improving overall treatment efficiency.
- The reactor design incorporates **digital baffles**, which are adjustable and can optimize the flow of wastewater through the system.
- The "digital" aspect suggests the possibility of **intelligent control** of the baffles, allowing for fine-tuned adjustments based on real-time conditions or process needs, improving overall system adaptability and efficiency

Abstract: A novel design Digital Baffle Electro Catalytic Batch Oxidation Reactor (DBECB) has been conceived to upsurge the specifications of refinery wastewater (RWW), through the removal of organic pollutants by electro catalytic oxidation treatment. The modern design that eliminates organic from RWW with improving its qualities has drawn a lot of attention. These days, one of the things that the world needs are the production of clean water with strict limitations on dangerous compounds. The degree of chemical oxidation that has been used to purify this wastewater is reflected in the electro- catalytic oxidation (ECO) process .The experimentations were intended by a Response surface method experimental design comprising the consequence of the time (10-40) min, magnetite Nano particles (0.1–0.5) gm, temperature (25-60) ^{o}C, and pH (3-9) under 0.5 Amps and 200 rpm then examining the results by Minitab software of response surface method with Box Behnken design (BBD) to find the best values of these conditions (40 min ,0.5 gm s of nano particle 3 pH, and 60 ^{o}C of temperature). The organic elimination efficiency of electro catalytic oxidation was 98.2%. The electro catalytic process was suitable for organic compounds removal from refinery wastewater.

Keywords: Crude oil, wastewater treatment, iron, electro-oxidation, DBBR, optimization, BBD.

1. Introduction

The environmental impact of oilfield plant industries is still often undesired; in the short term, they require a lot of water and generate a lot of oily waste water, which is a serious issue for oil manufacturing facilities and the environment [1]. The types of organic forms that are present, the physical method the water was bent, and the location all affect the refinery wastewater's characteristics [2]. Overall, more stringent environmental regulations require oil and gas companies to take a variety of RWW actions prior to reservoir inoculation and earlier notice in order to reduce created injury [3]. Water that has been properly maintained can now be recycled and used for water flooding. There are many problems with the conservative separation methods because of some contaminants are dangerous, non-biodegradable, and rowdy, these methods are less real at removing them from refinery wastewater[4]. However, none of these methods of treatment was adequate to contaminate water with adequate concentrations of the most persistent contaminants. Frequently, further therapy steps are required to achieve this goal .coagulation [5], flotation [6], and membrane treatment are just a few of the industrialized methods used to remove heavy metal from wastewater [7]. However, because the membrane is still contented to place and works finished scums with poor competence, the use of membrane separation procedures has limitations [8].The disadvantage of the precipitation process is its high cost, which consequences from the addition of numerous components [9]. As a result, more authors focus on honing their methods for removing organic complexes from refinery wastewater by hybrid methods of adsorption and oxidation [10]. Magnetic nanoparticles (MNPs) are an interesting class of nanomaterials that have been widely researched for usage in numerous industrial applications [11].MNPs have been applied in sensing technologies, memory storage devices, magnetic separation, magnetic labeling, and catalytic processes . MNPs have been employed in biological applications to give contrast effects for magnetic imaging, to remotely control targeted drug administration, and to produce warmth for the treatment of hyperthermia [12]. Iron oxide nanoparticles (NPs) are a potential class of magnetic materials because of their high biocompatibility. The primary motivation for significant research efforts to commercialize iron oxide nanoparticles for enhanced medical technology applications is their biocompatibility. While there are many other kinds of iron oxides, the term "iron oxides" usually refers to three types: Fe_3O_4 [13].

Advanced oxidation processes (AOPs) have been unable to overcome the disadvantages of conservative methods in previous decades [14] . Under sensible conditions, these methods can eliminate the pollutants into innocuous inorganic elements that are unconfined as CO_2 and H_2O and sludge [15]. In rural areas, AOPs are essentially physicochemical processes that generate a lot of oxidizing species, chiefly free radicals (•OH), which have the highest potential for oxidation [16] . The conduct of advanced oxidation processes, which are based on extremely reactive radicals, has remained relatively well defined at near ambient temperature [17].

Intractable pollutants in the wastewater environment can be wickedly soiled by AOPs [18] . This work was interested in organic elimination from refinery wastewater by new design of Digital Baffle oxidation of the electro-catalytic/oxidation treatment with working variables of the time, Nano particles, pH, and temperature using response surface method.

2. Experimental Work

2.1 Chemical and logical Examination

Fe_3O_4 nano particles supplied from ACS material, HCL (Scharlau, Spain 98 % purity) and NaOH (Scharlau, Spain ,97% purity). The refinery wastewater came from the oil station's clearing at Al-'Mathanna Oilfield in Iraq, which is home to the wet oil unit. It was kept in a polypropylene ampule on its own and maintained at 4 °C to keep the properties of RWW and shown in Table 1. At the finish of each experimentation, 40 mL of wastewater and 0.2 gram of NaCl were added to break up the organic emulsion. After adding 4 milliliters of CCL_4, it was vigorously shaken for two minutes. The lower (organic) layer was used for the absorbance test after the solution split into two separate coats after 20 minutes, and organic was identified using the calibration curve [19].

Table 1: Properties of refinery wastewater

Working	value	Working	value
Organic concentration	155.4 ppm	density	0.997
Turbidity	44.2 NTU	conductivity	72357 µs/cm
pH	6.85	TSS	17.2 (ppm)
Dissolved oxygen content	0.045 (ppm)	viscosity	1.05 m Pa. S^{-1}

2.2 Novel Design of Pilot Plant (DBEBR)

The Digital Baffle Electro Batch Reactor (DBEBR) was designed in the Faculty of Engineering, Chemical Engineering Department, University of Al Muthanna, and Muthanna, Iraq to produce water clean by removing organic compounds from the wastewater via electro-catalytic oxidation. In this work, we have designed the new reactor of different workings of the reactor. Assessment of the efficiency of the reactor in the context of RWW treatment via electro coagulation/ oxidation method is one of the aims of this study. The design of DBEBR enhanced distribution of iron in wastewater and attained high mass transfer rate in the electro oxidation process. The DBEBR design included digital mixer (range of Impeller speed up to 3000 RPM), which was linked to a rod, and the end of the rod was connected to the three-baffle impeller. Following that, the baffle represented a good equal distribution at the end of the rod with the (8 cm, 4cm, and 0.11cm) of aluminum and (8 cm, 3 cm, 0.12 cm) of iron as anode and cathode respectively. By increasing the amount of transfer reactants inside the reactor, these baffles improve the effectiveness of the electro-adsorption/oxidation technique. Additionally, three baffles were added to the reactor wall to prevent stagnant zones from developing and to encourage a chain reaction among the reactants. Equally, spaced baffles were placed inside the reactor's surface, with 36 cm between each pair. The reactor was also insulated using a 2.5 cm protrusion of woolen material (for high temperatures above 1000 °C). Stainless steel was used to build the reactor. Table 2 contains a list of the DBBBR's specifications, and Figure 1 shows the system's schematic.

Table 2. The specifications of the DBBBR

Description	Specification
Reactor dimension	Height = 13 cm, Diameter = 9 m
Rod length	30 cm
Kind of impeller (Stainless steel)	Four basket impeller
Basket height	1.15 cm
Basket length	1.2 cm

Basket width	1.2 cm
Diameter of Impeller	85 mm
Baffle height (Stainless steel)	8 cm
Baffle width	1.3–1.4 cm

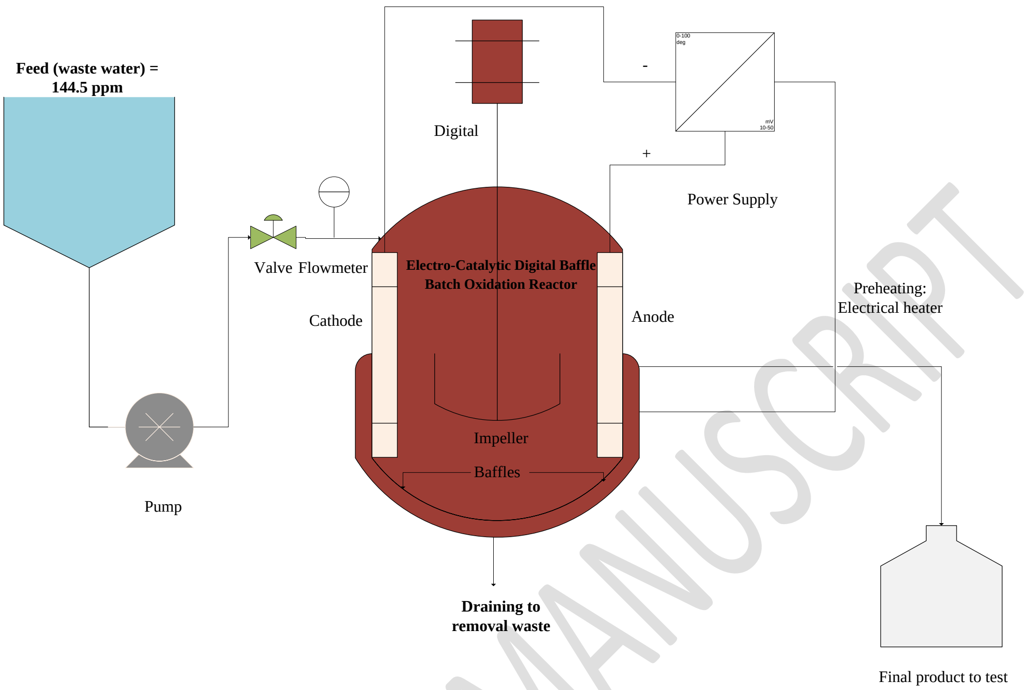

Figure 1: Hybrid adsorption and oxidation reactor

2.3 Hybrid treatment

A UV spectrophotometer (UV-1800 Shimadzu, Japan) was used to monitor the variation in the organic concentration in RWW during the adsorption and oxidation system procedure. The results were reconstructed into the conforming concentrations (C). Equation (1) was used to compute the organic efficiency.

$$\eta = \frac{C_o - C_t}{C_o} x100 \qquad (1)$$

Where η, percentage of organic removal; C_o and C_t, measured concentration before the and after treatment (ppm) respectively.

Response surface methodology, which is equivalent to statistical software (Minitab-17), was used to design the trials and forecast the outcomes of the working factors separately and in relation to one another. The primary factors influencing these problems, namely the electrolysis-oxidation time (X_1), pH (X_2), iron concentration

(X_3), and hydrogen peroxide (X_4), were purposefully rendered according to the ranges shown in Table 3.

Table 3: Working parameters

Working	Ranges
X_1: time (min)	10-40
X_2: pH	3-9
X_3: Iron dose (gm)	0.1-0.5
X_4: Temperature	25-60

3. Results and discussion

3. 1 Characterization of nano-Fe_2O_3

3.1.1 FESEM Result for Fe_2O_3-nanoparticles

FESEM determines the surface shape and active metal dispersion between them. The nano-particles (Fe_2O_3) surface mapping produced by FESEM is shown in Figure 2, which displays a superb distribution of active components. The precipitation technology is an effective way to create this kind of catalyst with good active metal dispersion [20]. The catalyst's surface morphology is depicted in Figure 5.7; the surface's shape is spherical.

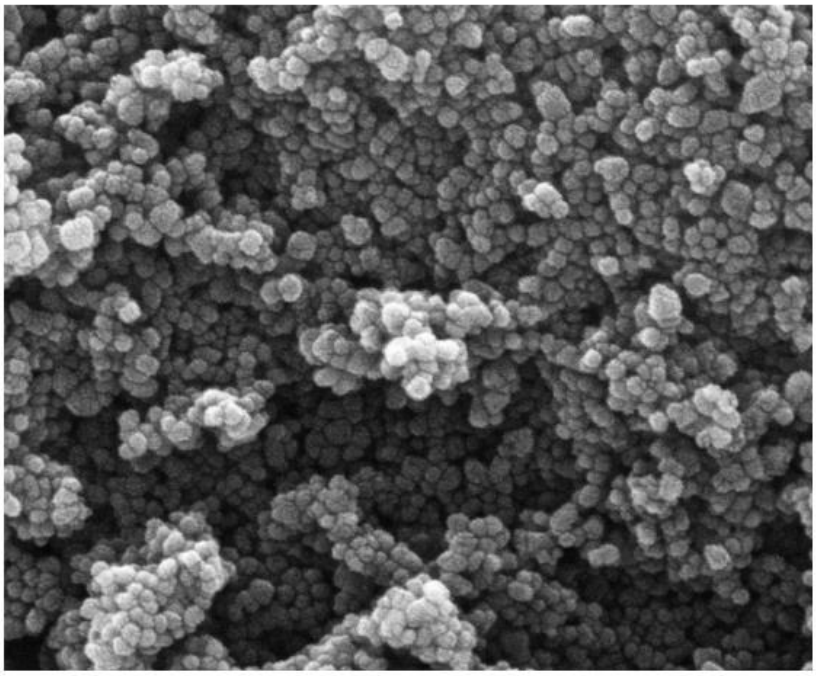

Figure 2: FESEM result for Fe_2O_3 nano-particles

3.1.2 FTIR results for Fe_2O_3-nanoparticles

Figure 3 displays the results of FTIR analysis for the synthetic nano-Fe_2O_3 in the 4000-500 cm^{-1} wave number range. It was determined that the O–H stretching vibration was responsible for the band at 3500-3000 cm^{-1}, whereas the H–O–H symmetric stretching vibration of adsorbed water molecules was responsible for the band at about 1645 cm^{-1}. As seen in Figure 3, the stretching vibrations of Fe-O-Fe, which are typical of Fe_2O_3, are responsible for the absorption bands centered at 440, 580, and 620 cm^{-1} [21].

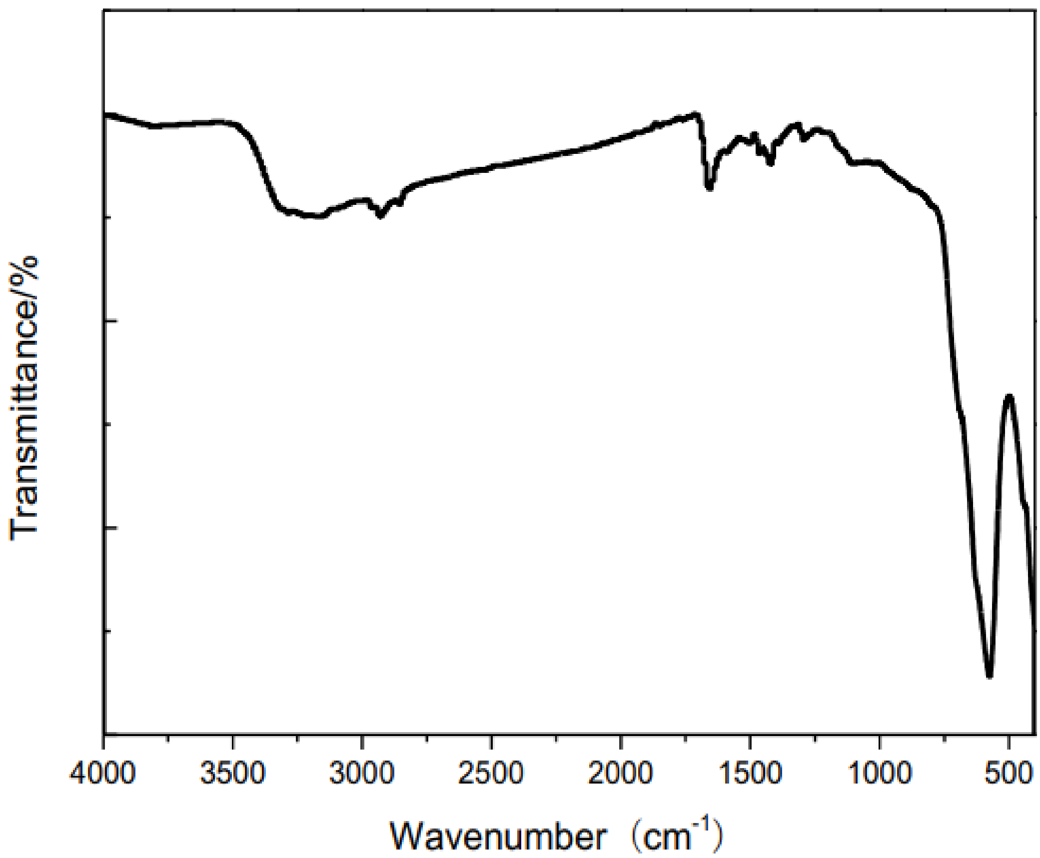

Figure 3: FTIR result for Fe_2O_3 nano-particles

3.1.3 XRD result for Fe_2O_3 nano-particles

The XRD pattern of the synthesized nano **Fe_2O_3 nano-particles** (commercial supplied) is displayed in Figure 4. Clearly visible as large peaks at 2θ angles approximately (31.234º, 38.02334º, 43.432º, 58,9283º and 62.3465º), respectively, are the many primary reflections of the nano **Fe_2O_3** phase. The pattern's peaks clearly show that **Fe_2O_3** crystallites of nanoscale size have formed. This behavior demonstrated a strong dispersion of molecules supported by Fe_2O_3 on the catalyst surface [22].

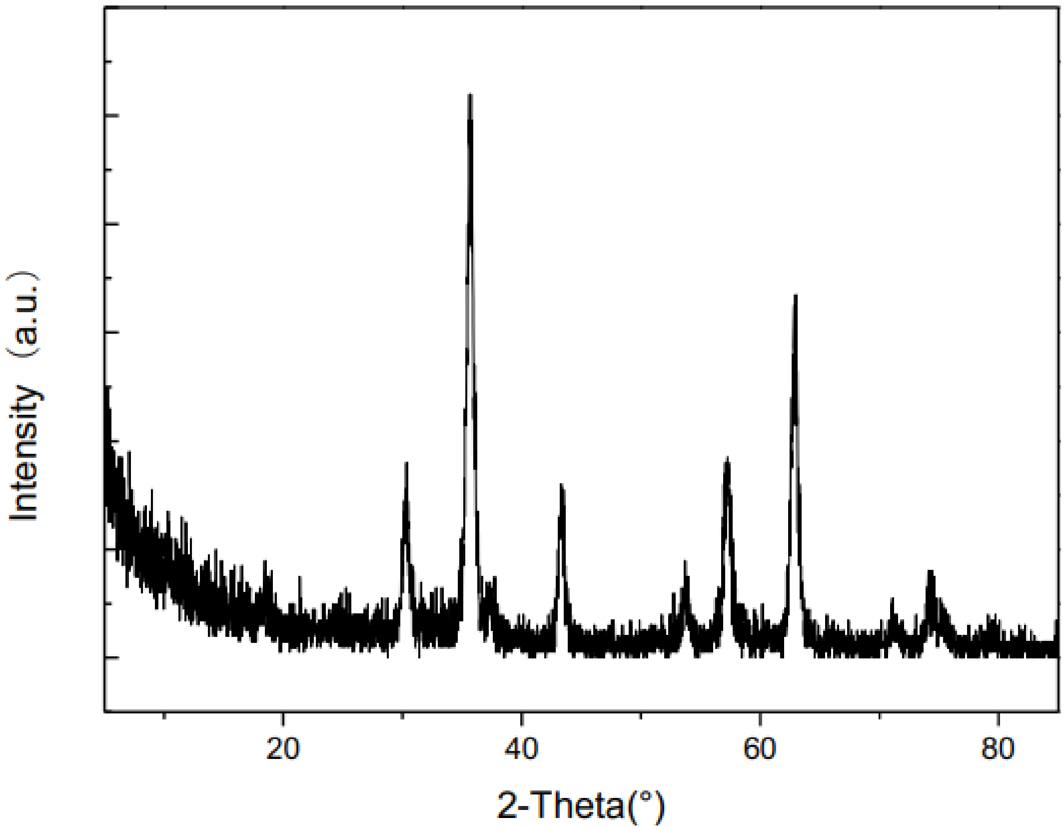

Figure 4: XRD result for Fe_2O_3 nano-particles

The working variable settings, the final organic concentration in wastewater (27 tests were conducted by BBD), and the elimination efficiency targeted at adsorption and oxidation treatment run are explained in Table 4. The mathematical equation (Eq. 2) was manufactured in terms of actual factors associating the copper removal response to the active variables, representative the interconnections between these variables, and was founded on investigative consequences:

Organic Removal = $16.2 + 2.45 X_1 + 0.773 X_2 + 1.33 X_3 + 3.73 X_4 - 0.0469 X_1^2 - 0.00447 X_2^2 - 0.0075 X_3^2 - 0.766 X_4^2 - 0.0022 X_1X_2 - 0.0089 X_1X_3 + 0.038 X_1X_4 + 0.004 X_2X_3 - 0.0182 X_2X_4 - 0.055 X_3X_4$ (2)

Table 4: Results of the BBD experiments

NO	Time (min) X_1	Dose (gm) X_2	Temperature X_3	pH X_4	Organic removal by ECO (%)
1	10	0.1	43	6	64.2
2	40	0.1	43	6	77.3
3	10	0.5	43	6	78.5
4	40	0.5	43	6	88.2
5	25	0.3	25	3	84.1
6	25	0.3	60	3	96.3
7	25	0.3	25	9	73.8
8	25	0.3	60	9	80.3
9	10	0.3	43	3	90.4
10	40	0.3	43	3	98.2
11	10	0.3	43	9	77.8
12	40	0.3	43	9	89.1
13	25	0.1	25	6	83.8
14	25	0.5	25	6	93.5
15	25	0.1	60	6	94.6
16	25	0.5	60	6	98.5
17	10	0.3	25	6	86.4
18	40	0.3	25	6	93.4
19	10	0.3	60	6	96.8
20	40	0.3	60	6	98.9
21	25	0.1	43	3	88.7
22	25	0.5	43	3	99.3
23	25	0.1	43	9	84.5
24	25	0.5	43	9	94.6
25	25	0.3	43	6	95.1
26	25	0.3	43	6	94.2
27	25	0.3	43	6	93.9

ANOVA is demonstrated in Table 5 with a focus on the electro catalytic oxidation response surface model. The results of the Fisher-value, P-test, adjusted sum of squares, adjusted mean of squares, degree of freedom, and sum of squares for all parameters are shown in Table 5. Just 4.33 percent of the total variants are not reinforced by schooling, according to the model's multiple correlation constant, which is 92.5% compatible with the statistical significance of the regression. Figure 5 shows that the adjusted manifold correlation coefficient (adj. R^2 = 91.3%) and R^2 matched well in this model [23].

Table 5: ANOVA for organic removal

Foundation	DOF	Seq. SS	Adj. MS	Fisher Value	P–test Value
1-Model	14	1253.56	89.54	1.34	0.309
Linear	4	994.9	248.725	3.72	0.034
X_1	1	218.79	218.787	3.27	0.096
X_2	1	297.89	297.889	4.45	0.056
X_3	1	211.68	211.68	3.17	0.101
X_4	1	266.55	266.545	3.99	0.069
Square	4	237.37	59.342	0.89	0.501
X_1^2	1	147.93	147.935	2.21	0.163
X_2^2	1	105.81	105.811	1.58	0.232
X_3^2	1	0	0	0	0.999
X_4^2	1	48.94	48.938	0.73	0.409
2-Way Interaction	6	26.9	4.484	0.07	0.998
X_1*X_2	1	2.89	2.89	0.04	0.839
X_1*X_3	1	5.46	5.459	0.08	0.78
X_1*X_4	1	3.06	3.063	0.05	0.834
X_2*X_3	1	7.9	7.897	0.12	0.737
X_2*X_4	1	0.06	0.062	0	0.976
X_3*X_4	1	7.53	7.532	0.11	0.743
Error	12	802.53	66.877		
Lack-of-Fit	10	801.75	80.175	205.58	0.005
Pure Error	2	0.78	0.39		
Total	26	2056.08			

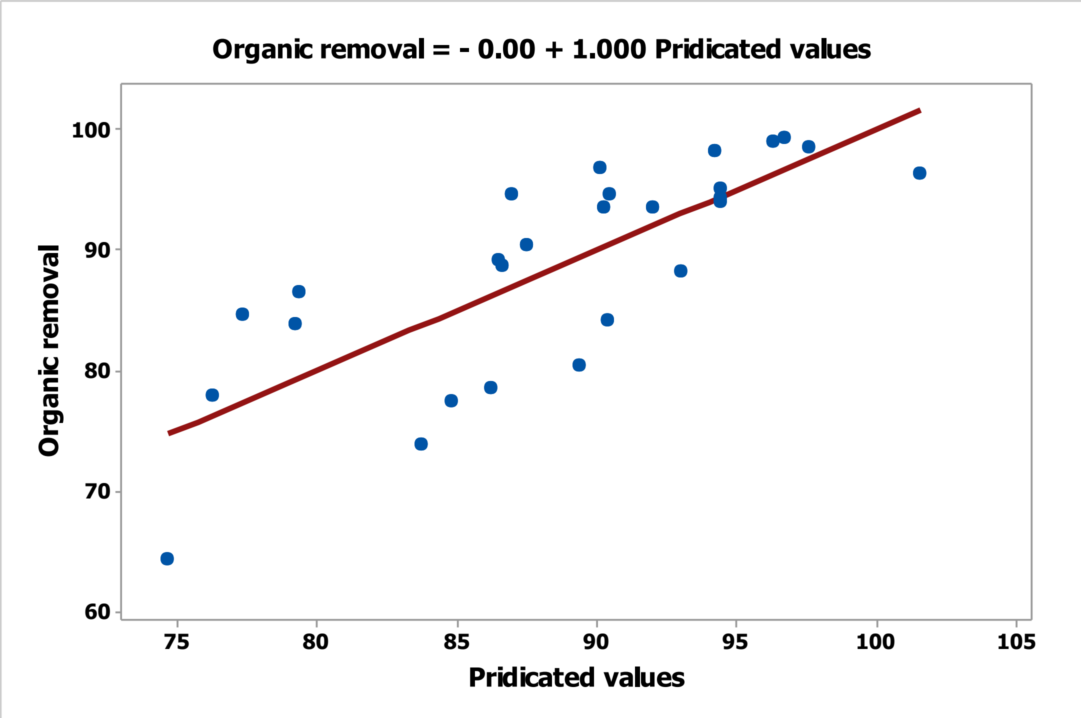

Figure 5: Observed vs predicated values of organic removal

The results in Figure 6 explain that the high competence response of elimination along the time of oxidation aimed at all values of the organic removal, the hybrid effects of dose and temperature, and time at acid solution had a strong interaction directed towards metal elimination and vice versa to pH on the organic removal when increasing the pH led to decrease the organic removal. This is because there are insufficient locations on the adsorption and oxidation of steal steel and iron as anode and cathode correspondingly, and the free radical from electro- oxidation is more compared to adsorption and oxidation method substantial surface to achieve a relatively high elimination ratio [24].

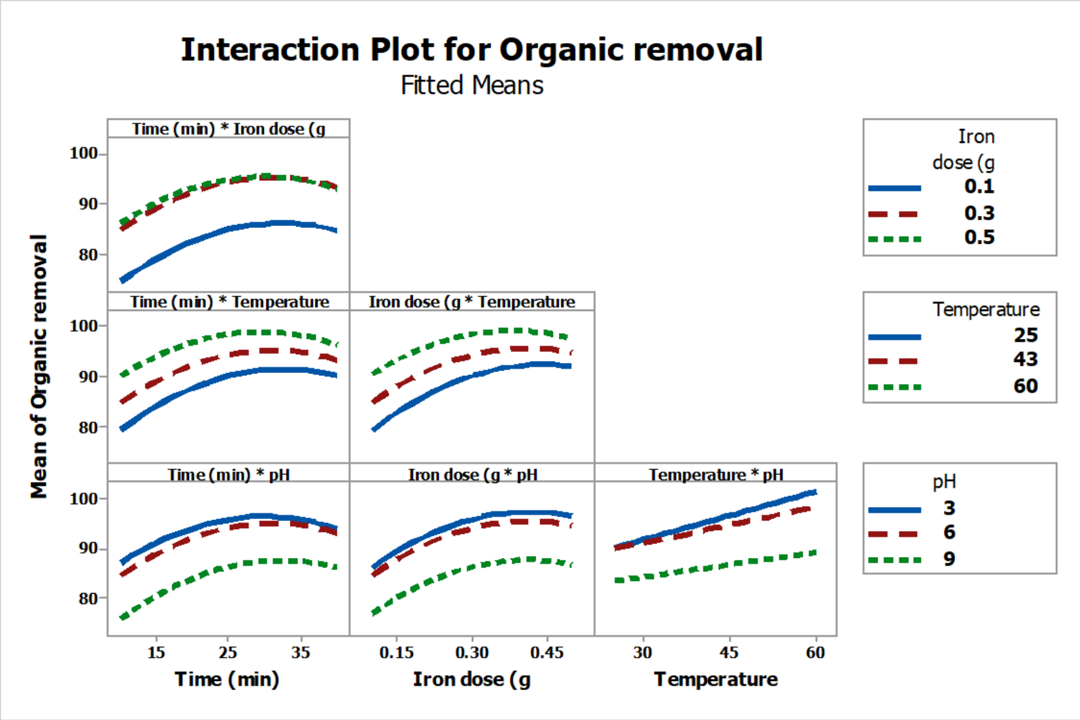

Figure 6: Interaction plot of variables of adsorption and oxidation

The data were analyzed using the "Minitab 17" application, and the main conclusions regarding the issues were consistent. The change in response resulting from a change in the level of an issue still determines its outcome. This is commonly referred to as the major effect because it tackles the primary issues of interest in the experiment [25]. Figure 7 demonstrate the key components of each restriction on the organic removal. The iron dose, temperature, pH, and duration had the biggest effects on the elimination of organic in RWW. In the range under study, this constant, which has a positive sign, indicates how rising concentrations of iron dose and temperature as well as time would increase organic removal and, on the other hand, how increasing pH would decrease organic removal [26].

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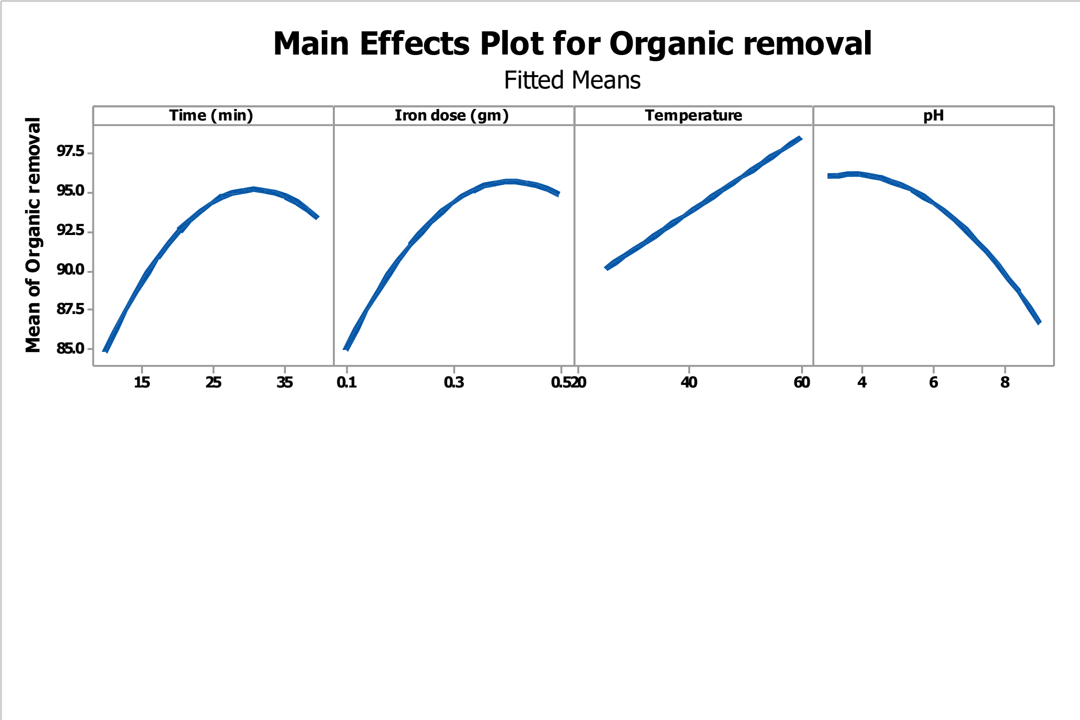

Figure 7: Impact maim effect of adsorption and oxidation

The ideal settings for best working variables including pH, temperature, iron dose, and time were still established. Figure 8 displays the measurement effects of the D-optimization for the electro catalytic oxidation of organic pollutants in refinery wastewater[27].

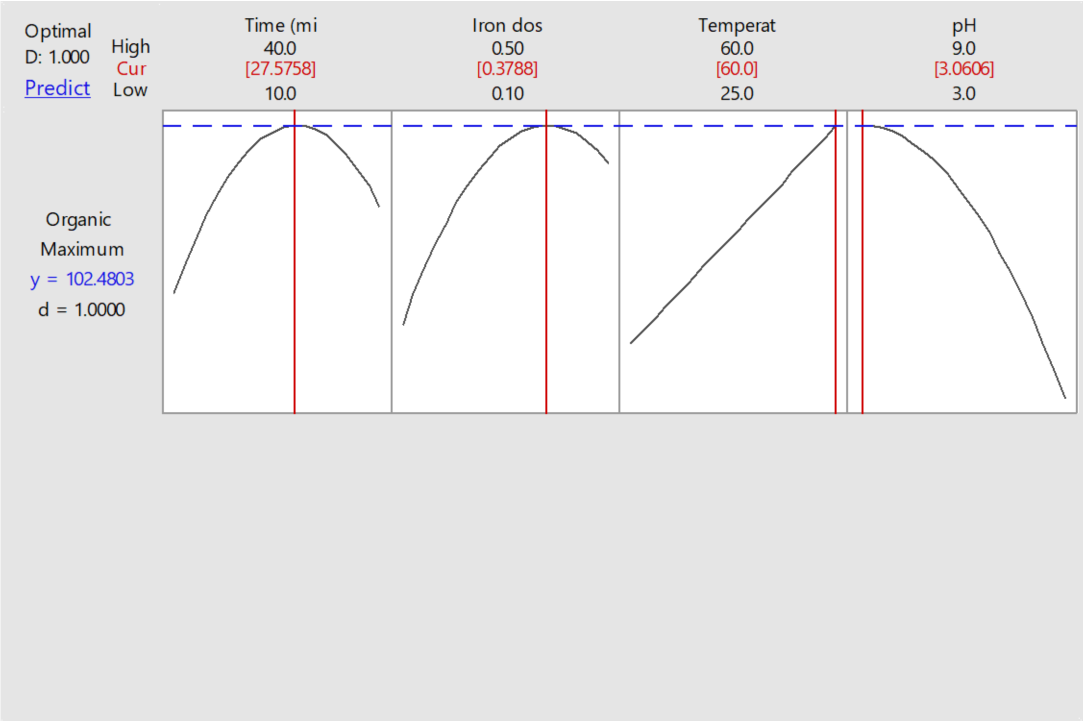

Figure 8: The optimum values of the operational variables

3.2 Effect of working variables on organic removal

3.2.1 Nano iron dose effect on the removal efficiency

The relationship between the dose and the organic elimination was shown in Figure 9. It revealed that the elimination competence increased as the dose increased from 0.1 to 0.5 gm, the all-out elimination competence was 92.3 % at 0.44 gm of Fe_3O_4, 40 minutes of irradiation time, pH 7, and 60 ^{o}C. Elimination competence increases with iron because continual amount additions produce high amount that remove organic pollutants in refinery wastewater [28].

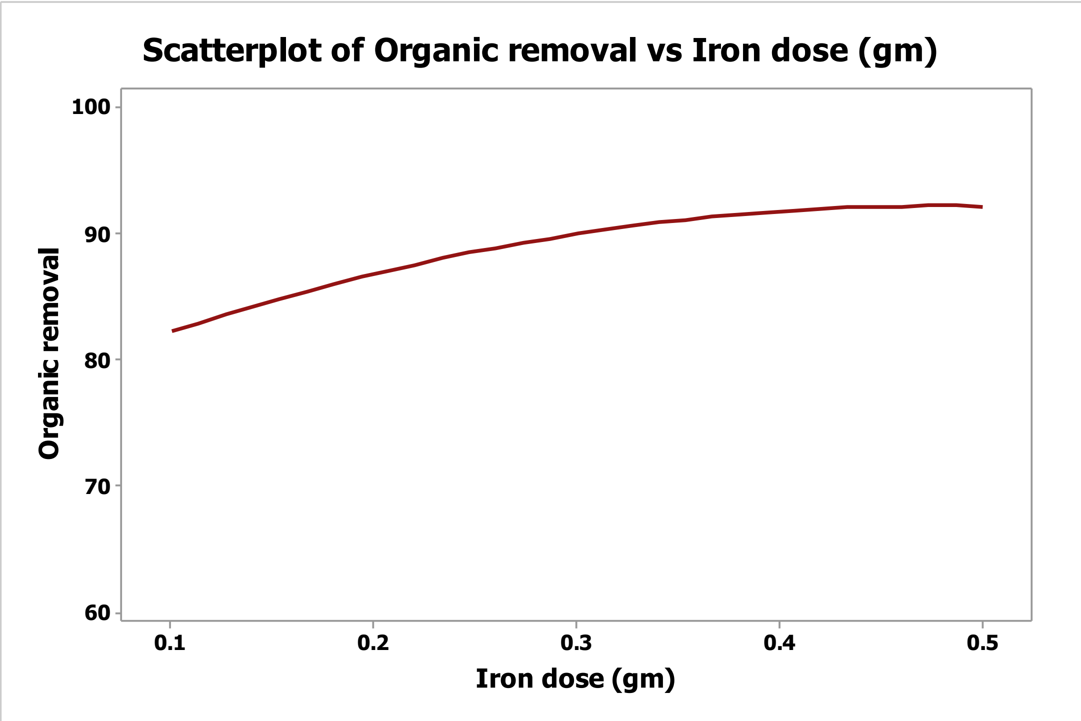

Figure 9: Effect of iron dose on the elimination process

3.2.2 The effect of pH on the removal efficiency

pH values were substantial impact on the adsorption and oxidation technique. Therefore, a chosen pH variety is required for the procedure to be obtainable in the greatest possible way. The effect of pH on the ability to eliminate organic was explained in Figure 10. The findings show that elimination competence increases as pH decreases, with 3.0 being the ideal pH. These theories align with author's findings [29] .which states that the pH value's impact on organic pollutants removal is still not particularly noteworthy., several experiments were still conducted with variations on the pH range from 3 to 9. The organic elimination efficiency was 93.2% has been preserved at pH=3 and gradually decreased to a pH=9 value just above this threshold. The combined methods had demonstrated a clear decrease in organic elimination of 82.5%. Given that the maximum amount of organic elimination was determined to be with a pH at an acidic solution [30].

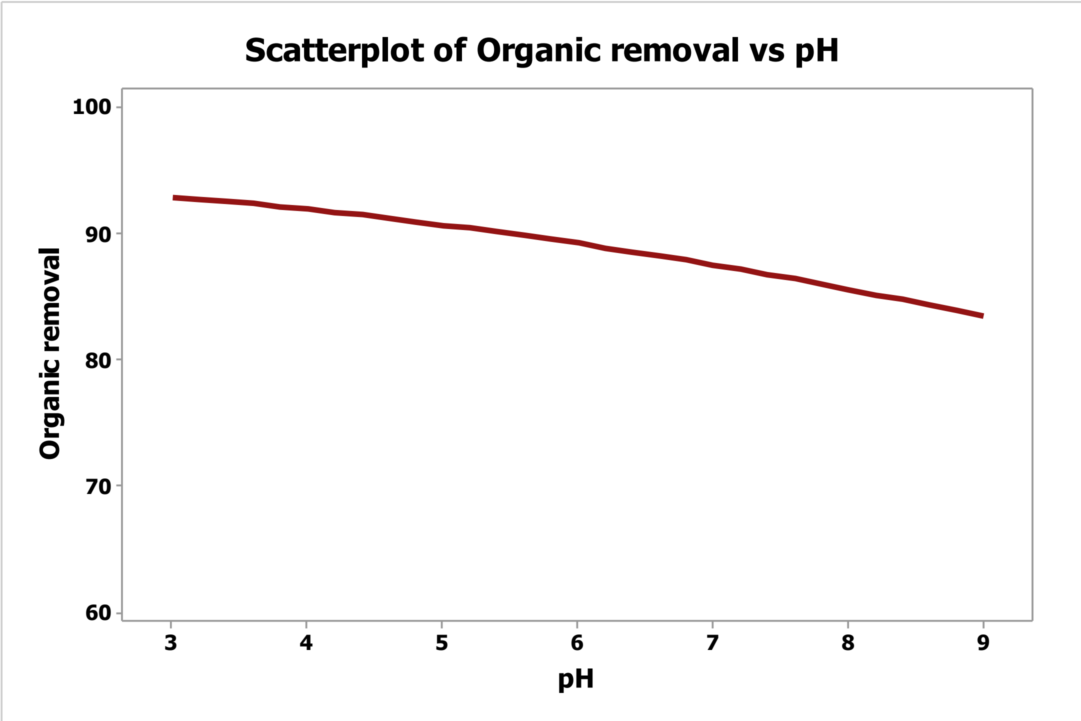

Figure 10: Effect of solution pH on elimination of organic compounds

3.2.3 The effect of oxidation time on the removal efficiency

It was purposeful to examine the adsorption and oxidation time variable in relation to the oxidation treatment and its impact on the ability to eliminate organic with the breakdown of ion constituents, the free radicals from anode that were created in the oxidation knowledge might quickly oxidize into hardness due to their strong oxidizing capacity [31]. The relationship between oxidation time and organic removal efficiency throughout the action phase is illustrated in Figure 11. The outcome was consistent with authors that demonstrated that an increase in oxidation time increases process efficiency, as demonstrated by the findings of Ali et al., 2020 [32].

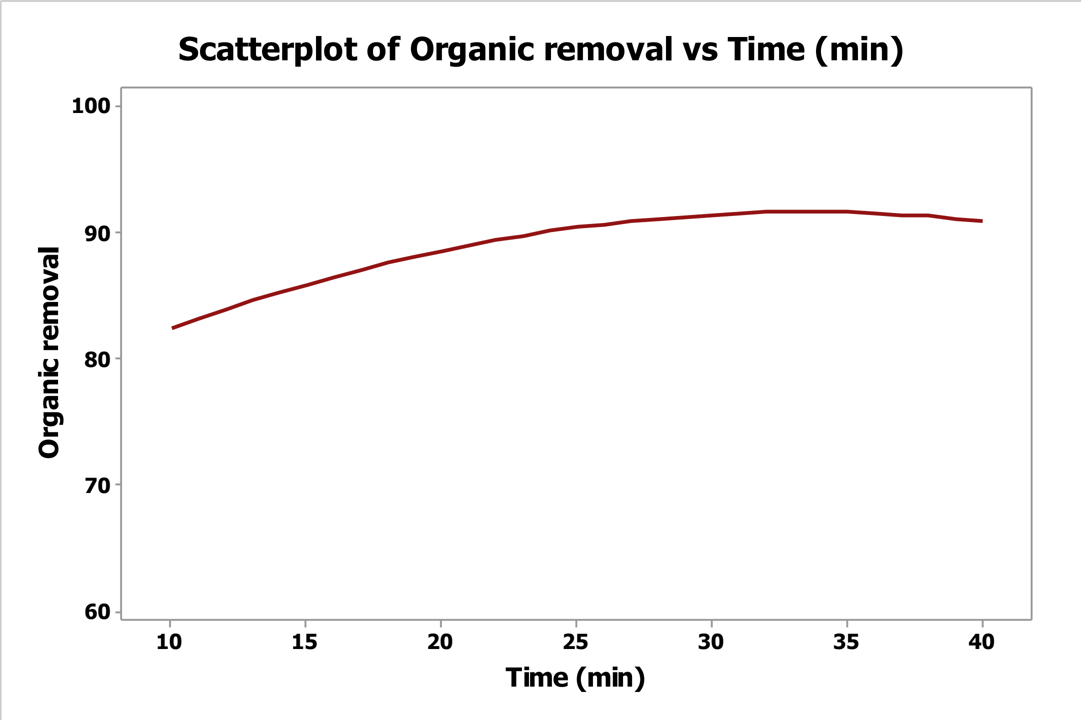

Figure 11: Effect of time in the elimination of organic compounds

3.2.4. Effect of temperature on the removal efficiency

The wastewater treatment was still impacted by the additional variable of temperature. The effect of varying temperature on the ability to remove organic compounds from refinery wastewater. The elimination competence increased from 84.2% to 93.5 % as soon as the temperature increased from 25 to 60 °C. Figure 12 illustrates that the all-out organic elimination increased with increased temperature [33] .

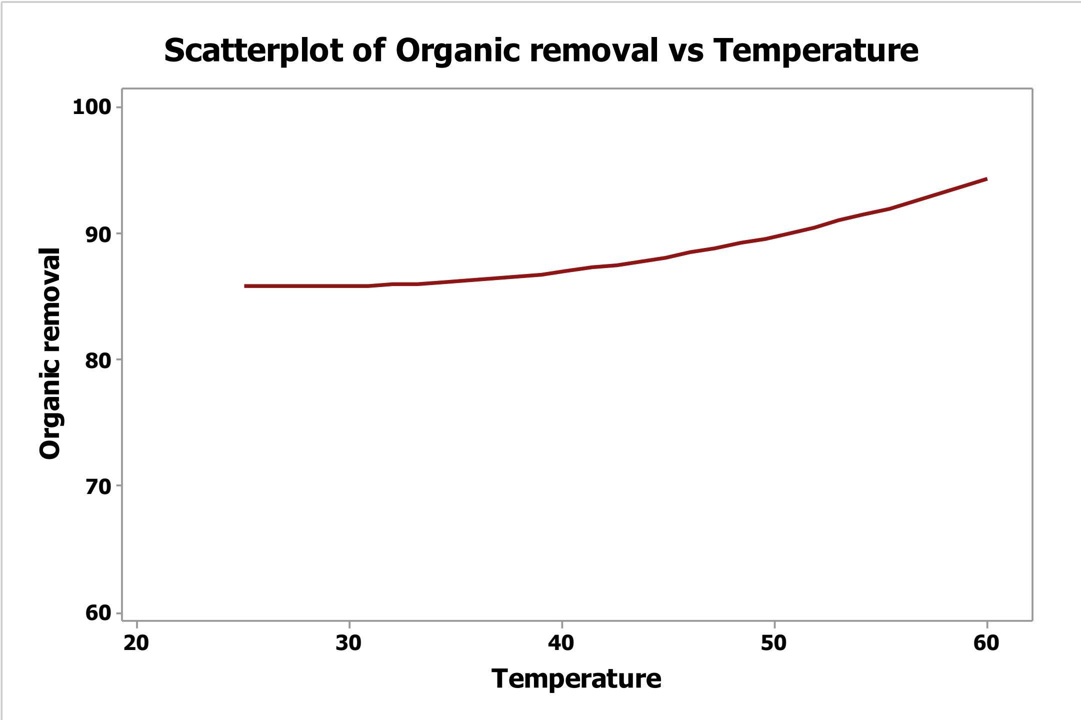

Figure 12: Effect of temperature of the organic removal

As shown in Figures 13, 14, and 15, respectively, the contour plot for organic removal shows how the x-axis responds to oxidation time and changes with pH, iron dose and temperature. The results of the Figure show that increasing the adsorption and oxidation at acid solution increases organic elimination when compared to the basic solution because free radicals are produced at lower pH, organic compounds in the refinery wastewater are oxidized, and the hybrid treatment efficiency is increased [34]. The results indicate that the oxidation reaction is comparatively faster than the adsorption and exhibits rapid elimination of organic molecules [35].

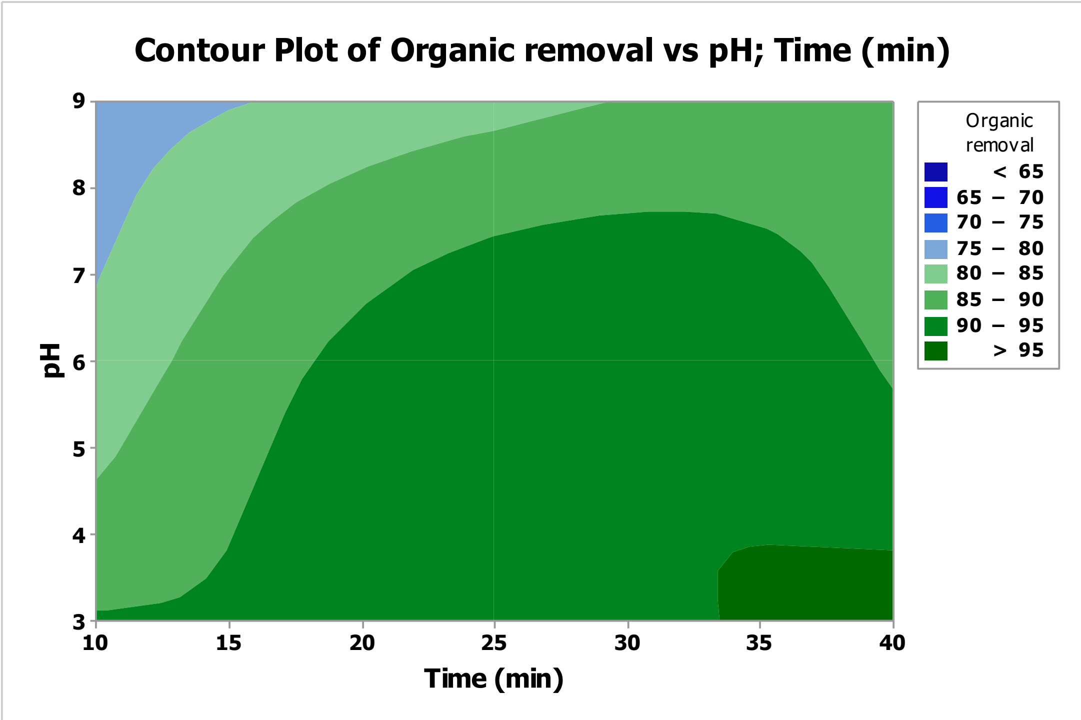

Figure 13: Contour plot of organic pollutants vs pH and time

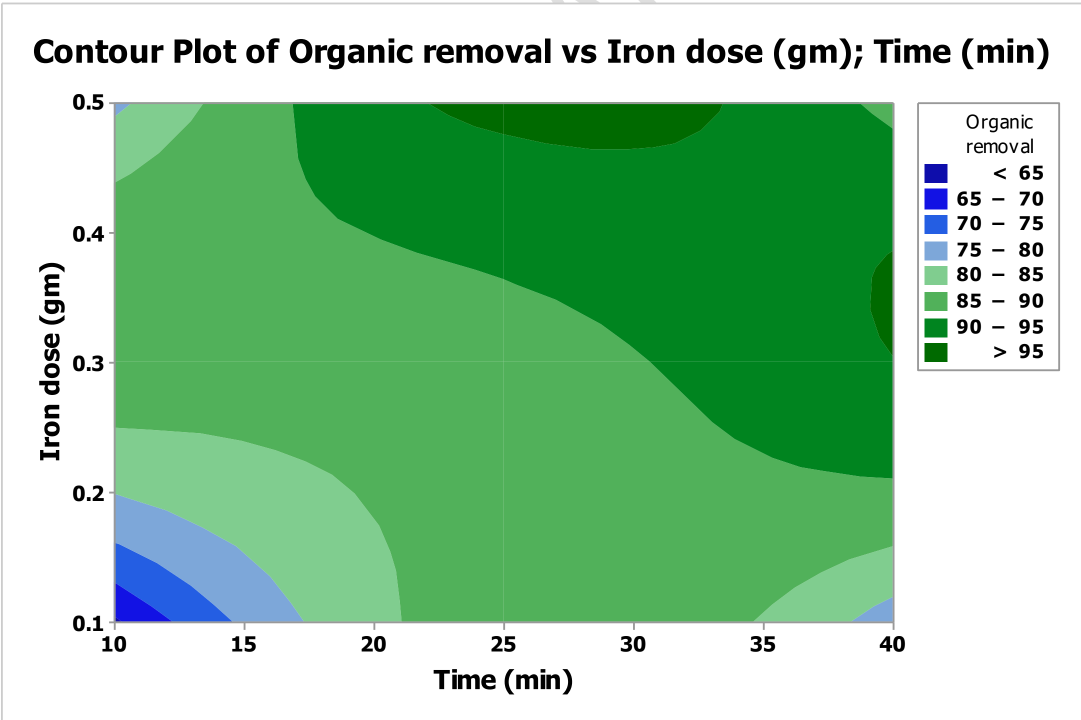

Figure 14: Contour plot of organic pollutants vs iron dose and time

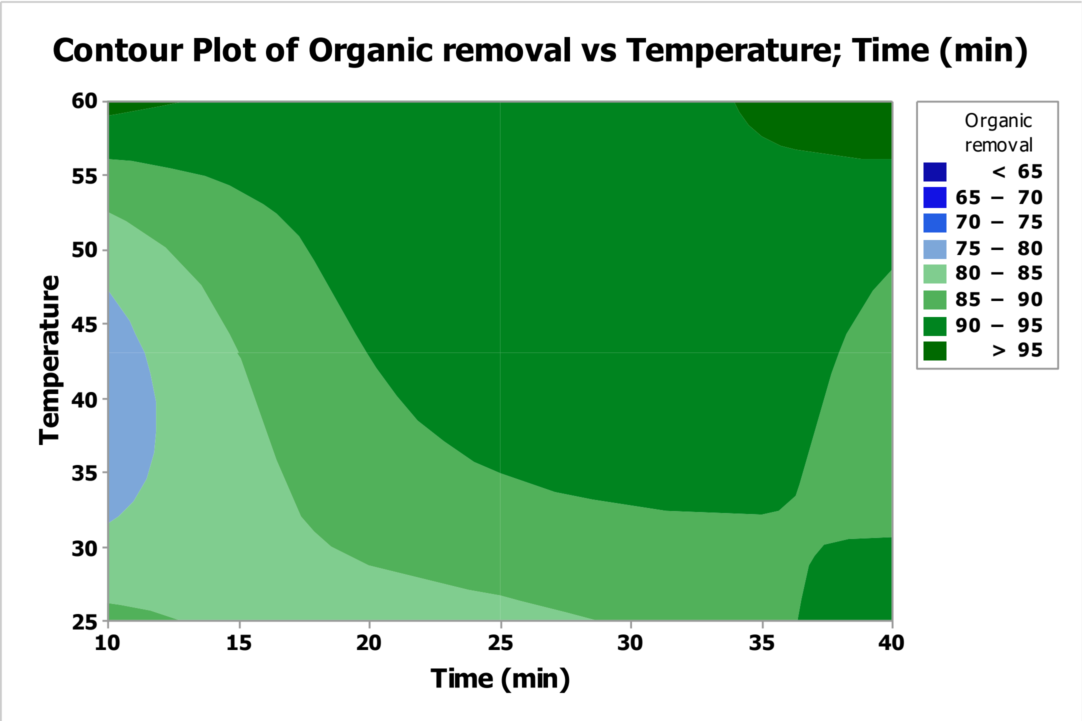

Figure 15: Contour plot of organic pollutants vs temperature and time

4. Conclusions

To advance the technology of electro-Fenton oxidation, a novel design of pilot plant Digital Baffle Electro Photo Fenton Batch Oxidation Reactor has been developed. Based on the findings, a new design that improves the mass transfer of copper reactants by Fenton, photo Fenton, and electro Fenton is presented, and their differences are contrasted. All of the intentional answers that show the satisfactory modification of the second order polynomial model must have high regression constants in the mathematical relationships that were discovered. An organized technique for cleaning up solutions with low copper concentrations is still the electro Fenton procedure. It was discovered that the novel DBEPBR design improved oxidant towards organic contaminants in wastewater, leading to 99.3% copper metal removal under mild operating circumstances (pH 3, ferrous Sulphate 18 ppm, electrolysis period 30 min, and hydrogen peroxide 75.8 ppm). This technique can be suggested as an unofficial means of producing water treatment due to the effectiveness of electro-Fenton operation.

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