



# Efficient lead remediation with Fenton oxidation in produced water

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## Abstract

Wastewater contains multiple pollutants in different forms that may be biodegradable or non-degradable. Therefore, it was necessary to find different methods and processes to remove these pollutants, as Fenton oxidation (FO) processes were commonly used for commercial and residential operations. In a glass batch reactor, a Fenton oxidation process was performed for the purpose of purifying the produced water (PW) and removing the contaminated lead metal. Depending on the batch system used, the effect of operational process variables such as pH (3-10), oxidation time (20=120) min, concentration of hydrogen peroxide (25-100) ppm and ferrous sulphate (5-25) ppm was tested. Through the results obtained, it was demonstrated that the possibility of applying Fenton oxidation technology in removing lead metal contaminants from the produced water (PW) was successful. Lead elimination of 94.18% was achieved when using the Fenton oxidation technique, where a time of 120 minutes was taken at a pH of 6.5, 62.5 and 25 ppm for hydrogen and ferrous sulphate concentrations, respectively.

**Keywords:** Produced water, Heavy Metal, Wastewater treatment, Fenton Oxidation processes.

## 1. Introduction

Crude oil has gained great importance as a controller of the economy because the development taking place in all the various industrial processes has been accompanied by an increase in the rate of fuel consumption. From this perspective, oil is classified as being in the first ranks because of its wide spread in terms of use, as well as because it contains different oil derivatives that suit the range of a wide range of different industries, as well as in petrochemical laboratories and oil refineries [1]. The water accompanying the crude oil extracted from the reservoir, which is called the produced water, consists of the water found in the oil field in addition to the water that is injected during those extraction processes. [2],[3]. That produced water causes many problems, one of the most dangerous of which is that it leads to a shorter lifespan for the oil and gas shafts, as well as it is the main cause of corrosion problems in the various parts of the operating units, in addition to being the cause of the fines that are transferred to the loads [4]. The percentage of water produced from the volume of crude oil will range from about (0.4 - 1.6). The symbol PW was adopted as an indication of the waste water stream resulting from the productive processes of oil and gas [5]. The resulting oil-contaminated water that is discharged into the environment is considered one of the most important sources of water pollution due to the large quantities released, as its annual production ranges in billions of cubic meters that are polluted at a rate of (1 - 40,000) parts per million [6]. The presence of heavy metals is considered evidence of pollution, as these metals are characterized by their strong electrical negativity and relatively high density, which exceeds 5 grams per cubic centimeter. The most famous and environmentally relevant metals are mercury, cadmium, chromium, arsenic, nickel, and copper [4]. Some of the specifications that characterize some metals make them hazardous metals. For example, lead, cadmium, and mercury are characterized by their inability to decompose, so in 1978 the World Health Organization classified them as hazardous metals [7]. The sources of these pollutants vary according to the industrial processes used, such as electroplating and dye manufacturing plants, various chemical factories, and mining plants. These harmful pollutants affect organisms in general and human life in particular because of their continuous formation, which exposes these organisms to being affected continuously throughout almost their lives [8]. Therefore, due to the harmful nature of these polluting materials, it has become necessary to find multiple and effective ways to remove them or get rid of their harmful effects [9]. The external environment is preserved by adhering to the conditions of environmental licenses, as methods of treating contaminated

waste enable new dimensions in purifying water contaminated with oils and reducing the percentage of losses by increasing the percentage of recovered oil, through developing the technologies used as well as the possibility of recycling the used water again [10]. Pollutants have derived their dangerous character due to their toxic properties and volatile nature, as well as the complexity of their chemical composition, as they include various hydrocarbon groups such as alkanes, alkenes, alkynes, and single and multiple aromatics. Hydrocarbon groups may be linked to other molecules, such as oxygen, nitrogen, sulfur, and others, to form more complex molecules [11]. There are some processes that take place on the water before removing pollutants from it, which are considered to have a significant impact on increasing the efficiency of separation, recovery, or recycling, and thus achieving environmental conditions in a way that allows maintaining downstream facilities [12]. In removing pollutants from water, traditional methods are considered ineffective in removing toxic pollutants that are characterized as non-degradable compounds [13]. Therefore, it was necessary to find additional methods characterized by their high effectiveness in removing these dangerous pollutants and to reach a percentage of pollutants that is within the permissible ranges, as well as to transform these pollutants into a fixed and more stable composition [14]. Currently, as an additional stage, advanced oxidation technology (AOPs) has been adopted in the treatment of polluted wastewater [15]. The principle of using advanced oxidation technology is based on dismantling the chemical structure of contaminated organic compounds through oxidation and reduction, most of which occur naturally by liberated hydroxyl radicals (OH), which are classified as the second most powerful compound in its redox activity after fluorine, with E0 reaching 2.8 eV and 3.06 V. For hydroxyl and fluorine radicals, respectively [16]. These techniques used have a high ability to get rid of organic pollutants if used under correct operating conditions by converting them from their active organic formula to the inactive inorganic form such as water and carbon dioxide [17]. Therefore, it has become possible to adopt (AOPs) as a successful technology in getting rid of industrial liquid waste pollutants [18]. Photo-Fenton reagent is considered a precursor to free radicals, so it is increasingly used in various chemical oxidation processes [19]. The Photo-Fenton reaction has also been used in AOPs to increase sludge dewatering [20]. The reason for using the Fenton reaction is that a portion of the activated sludge will dissolve and thus lead to its reduction, and a portion of it will mineralize into carbon dioxide and water [21]. When using photo-Fenton processes, mineralization and solubility will play a major role in the disintegration processes of activated sludge [22]. Bacterial cell membranes can be broken down using the photo-Fenton technique and converted into soluble components such as lipids, proteins and polysaccharides. Therefore, this technique can be adopted to speed up the process and enhance the degradation of solid waste [23], [24]. In this work, attention was paid to studying the removal of lead metal from the resulting water using the photo-Fenton technique and finding the optimal operating conditions, represented by the concentration of the Fenton detector, as well as studying the effect of pH and oxidation time on Fenton oxidation.

## 2. Experimental work

### 2.1. Chemical and instrumentation test

In southern Iraq, specifically from the Al-Hadib refinery, samples were taken of the resulting polluted water containing heavy metals. A container made of polypropylene was chosen to be a container for the models, and in order for the oxidation techniques to work properly, it was chosen to store the containers at a temperature of 4 degrees Celsius. The specifications of the resulting polluted water are listed as shown in Table 1. The analytical grade was adopted in this study and was not expanded by further purifying it. Purchased from Scharlau, Spain, were ferrous sulfate, H<sub>2</sub>SO<sub>4</sub>, and hydrogen peroxide (Germin, 45 percent wt/wt), as well as NaOH (India 99 percent purity). To ensure the knowledge of the concentration of Pb<sup>2+</sup> in the resulting contaminated water, after each Fenton oxidation test, shadow atomic absorption spectroscopy was performed in sales mode. To determine the concentration of metal ions, a slot burner type (air-acetylene) with a flowrate of about 55 with a diameter of 100 mm and a height of 6 mm was used. It was discovered that the peak pressure was 283.3 nm, at which an interpretation and study was made about the metal Pb<sup>2+</sup>. The equation (1) was used for lead removal:

$$Y_{\text{OCRE}} = \frac{A_o - A_t}{A_o} \times 100\% \quad (1)$$

Anywhere: YOCRE is lead elimination; A<sub>o</sub>, lead concentration before the FO (mg lead/L), and A<sub>t</sub> is the lead concentration after FO (mg lead/L).

**Table 1:** Specification of produced water

Parameters	Values	Parameters	Values
Lead metal	3.42 (mg/l)	Conductivity	111321 µs/cm
Turbidity	51.2NTU	TDS	71245.44 (mg/l)
pH	6.72	Viscosity	1.0402 m Pa/S
Oil content	250.4 (mg/l)	Iron	0.42 (mg/l)
Specific gravity	0.992	Sulphate	58.7 (mg/l)

## 2.2. Batch FO reactor

In this study of Fenton methods on squalor, experiments were carried out using a 500 ml glass oxidation reactor in which 250 ml of generated water was placed and rotated at a constant speed equal to 250 rpm of equivalent stirring speed at different oxidation times. In order to guarantee that the PW would become a homogeneous mixture, including the heavy metal and the Fenton reagent in the beaker, a magnetic stirrer (LMS-1003; Korea; 60–1500 rpm, Labtech) was used. The SWW's pH was measured using a WTWpH-720 digital pH meter, and the reagents' pH was adjusted beforehand by adding diluted hydrochloric acid (0.1 N) or sodium hydroxide (0.1 N) to the beaker [25].

## 2.3. Batch FO reactor

To study the effects of variables controlling lead removal levels from generated water, a Box-Behnken design was adopted and the number of experiments was determined using the response surface methodology (RSM). The significance of these factors. Table 2 shows the examined versions of oxidation time (X1), pH (X2), H<sub>2</sub>O<sub>2</sub> (X3), and ferrous sulphate (X4) for each of these variables.

**Table 2:** Employed limits

Variables	Ranges
X <sub>1</sub> : Solar time (min)	20-120
X <sub>2</sub> : pH	3–10
X <sub>3</sub> : H <sub>2</sub> O <sub>2</sub> (ppm)	25–100
X <sub>4</sub> : Ferrous Sulphate (ppm)	5-25

## 3. Results and discussions

### 3.1. Statistical test for solar Fenton processes

The effect of operational variables on the removal efficiency responses was studied in each experiment, as shown in Table 3. Experiments were designed to research the Fenton process using the response surface methodology and those results were analyzed statistically based on the Box-Behnken model with four replicates at the central point. The results obtained in the practical experiments were statistically analyzed using the Design Expert statistical program [26].

**Table 3:** Results of experimental of BBD to remove lead

StdOrder	RunOrder	PtType	Blocks	Oxidation time (min)	pH	Hydrogen Peroxide (ppm)	Ferrous Sulphate (ppm)	Y <sub>OCRE</sub>
1	1	2	1	20	3	62.5	15	72.45
2	2	2	1	120	3	62.5	15	79.21
3	3	2	1	20	10	62.5	15	41.21
4	4	2	1	120	10	62.5	15	46.87
5	5	2	1	70	6.5	25	5	79.21
6	6	2	1	70	6.5	100	5	88.24
7	7	2	1	70	6.5	25	25	84.15
8	8	2	1	70	6.5	100	25	91.45
9	9	2	1	20	6.5	62.5	5	78.48
10	10	2	1	120	6.5	62.5	5	84.99
11	11	2	1	20	6.5	62.5	25	85.41
12	12	2	1	120	6.5	62.5	25	94.18
13	13	2	1	70	3	25	15	59.88
14	14	2	1	70	10	25	15	34.28
15	15	2	1	70	3	100	15	72.88
16	16	2	1	70	10	100	15	68.19
17	17	2	1	20	6.5	25	15	75.33
18	18	2	1	120	6.5	25	15	84.22
19	19	2	1	20	6.5	100	15	87.28
20	20	2	1	120	6.5	100	15	91.45
21	21	2	1	70	3	62.5	5	71.55
22	22	2	1	70	10	62.5	5	50.14
23	23	2	1	70	3	62.5	25	75.33
24	24	2	1	70	10	62.5	25	61.47
25	25	0	1	70	6.5	62.5	15	88.14
26	26	0	1	70	6.5	62.5	15	87.99
27	27	0	1	70	6.5	62.5	15	88.66

The outcome of tests to investigate a previously unexplored relationship between lead removal skills and a quadratic model was rummage-sale to acquire development variables and express in terms of real units [27],[10]. Regression Equation in Uncoded Units

$$\text{Lead Removal} = 6.0 + 0.226 X_1 + 21.00 X_2 + 0.235 X_3 - 0.24 X_4 - 0.000896 X_1^2 - 2.09 X_2^2 + 0.00199 X_3^2 + 0.007 X_4^2 - 0.016 X_1 X_2 - 0.00063 X_1 X_3 + 0.00113 X_1 X_4 + 0.0398 X_2 X_3 + 0.0539 X_2 X_4 - 0.00115 X_3 X_4 \quad (2)$$

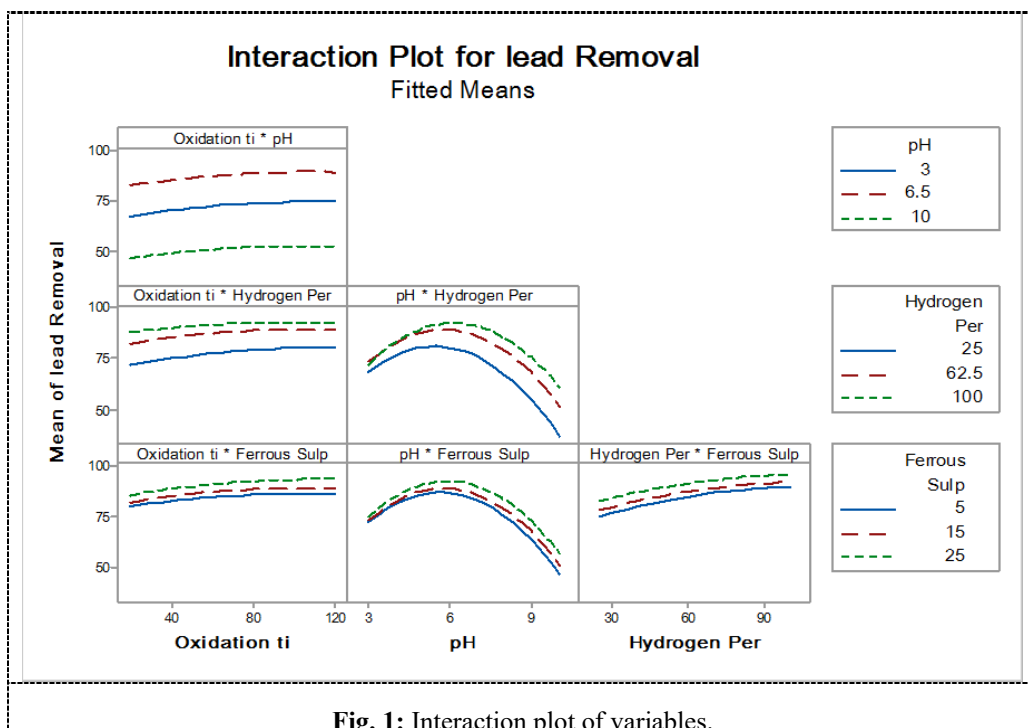
The effectiveness of lead removal with lead was demonstrated using Equation 2, in which separate linear and quadratic variables were used.

It has been shown that the removal process is positively affected by the cumulative effects of positive constant values and vice versa for negative constant values [28]. For multiple hypotheses, the Bonferroni correction was used with a one-way analysis of variance (ANOVA) test to compare the results values in all experiments, including the untreated models, that is, at time zero. At p 0.05, differences were deemed statistically significant [29]. The stability of the efficiency has been proven based on ANOVA analysis and according to the results of the F test and the P test related to lead removal, as shown in Table 4. The results showed that the change in responses increases in the regression equation as the Fisher value increases. BBD proficiency was verified. completed usage change components completed done certain sources of change [30].

**Table 4:** ANOVA for lead elimination

Foundation	DOF	Seq. SS	Adj. MS	Fisher Value	P-test Value
1-Model	14	6539.89	467.14	16.59	0.000
Linear	4	2223.53	555.88	19.74	0.000
X <sub>1</sub>	1	138.45	138.45	4.92	0.047
X <sub>2</sub>	1	1389.76	1389.76	49.36	0.000
X <sub>3</sub>	1	566.09	566.09	20.10	0.001
X <sub>4</sub>	1	129.23	129.23	4.59	0.053
Square	4	4184.91	4184.91	37.16	0.000
X <sub>1</sub> <sup>2</sup>	1	26.77	26.77	0.95	0.349
X <sub>2</sub> <sup>2</sup>	1	3520.67	3520.67	125.03	0.000
X <sub>3</sub> <sup>2</sup>	1	41.98	41.98	1.49	0.246
X <sub>4</sub> <sup>2</sup>	1	2.61	2.61	0.09	0.766
2-Way Interaction	6	131.45	131.45	0.78	0.603
X <sub>1</sub> *X <sub>2</sub>	1	0.30	0.30	0.01	0.919
X <sub>1</sub> *X <sub>3</sub>	1	5.57	5.57	0.20	0.664
X <sub>1</sub> *X <sub>4</sub>	1	1.28	1.28	0.05	0.835
X <sub>1</sub> *X <sub>5</sub>	1	109.31	109.31	3.88	0.072
X <sub>2</sub> *X <sub>3</sub>	1	14.25	14.25	0.51	0.490
X <sub>2</sub> *X <sub>4</sub>	1	0.75	0.75	0.03	0.873
X <sub>3</sub> *X <sub>4</sub>	12	337.90	28.16		
Error	10	337.65	33.76	273.11	0.004
Lack-of-Fit	2	0.25	0.12		
Pure Error	26	6877.79			
Total					

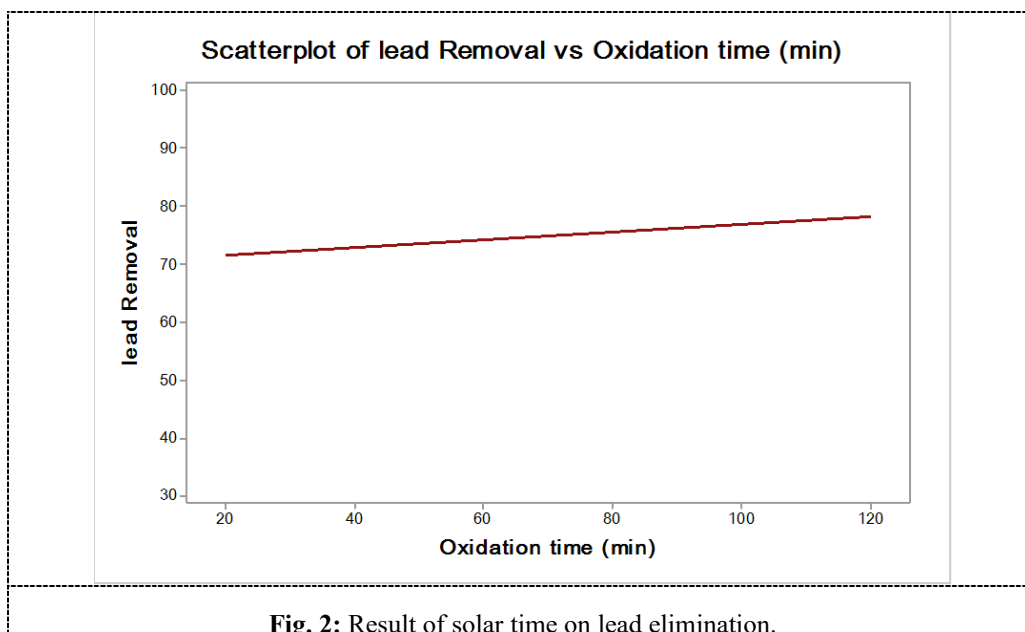
According to the results listed in Figure 1, it was observed that the responses over the oxidation period are directly proportional to the initial lead concentrations, in contrast to high concentrations, where the responses decrease and the removal rate decreases, because there is less availability of places on the surface for oxidation [31].



**Fig. 1:** Interaction plot of variables.

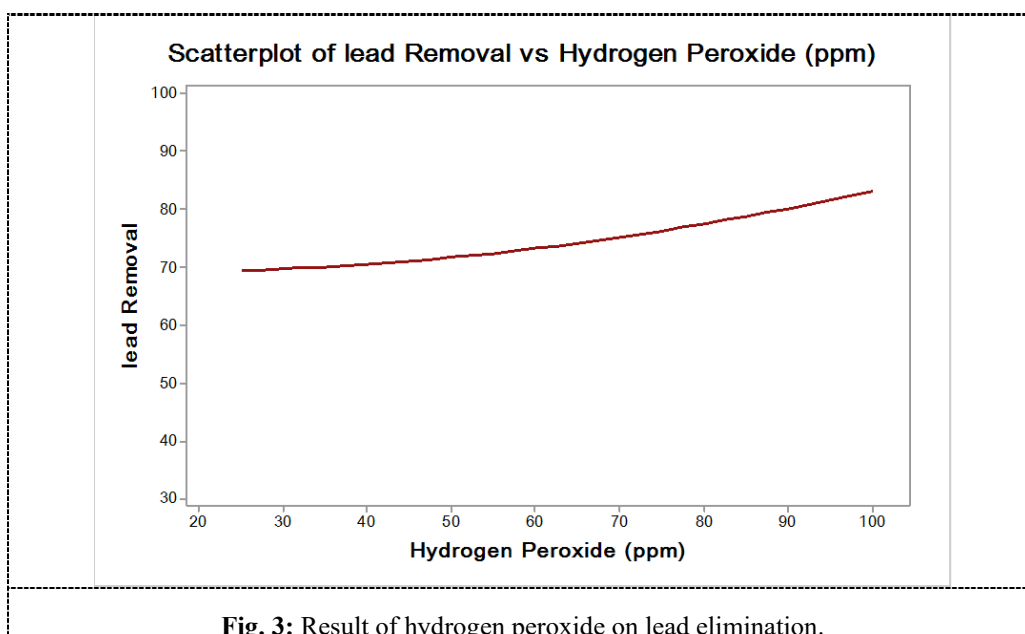
### 3.1.1. Oxidation time effect

The time required for the experiments was determined to be within the range (20 - 120) minutes in order to reach the effective oxidation time for the Fenton process. Naturally, the amount of lead removed increases with the passage of operation time until it reaches 78.4 percent after 120 minutes, which represents the upper limit of removal. The rate of free radicals formed for hydrogen peroxide and ferrous sulphate increases with increasing reaction time in practical experiments. For this reason, there will be rapid breakdown of lead in the bulk solution due to the accumulation of additional uncharged hydroxyl radicals. Previous research has supported the direct relationship between the efficiency of the removal process and the oxidation time, as was proven in the study [32].



### 3.1.2. Hydrogen peroxide effect

In this work, the removal of heavy metals from PW was studied using the Fenton oxidation technique using hydrogen peroxide at different concentrations. From the results of the practical experiments, as shown in Fig. 3, it was found that the relationship between lead removal and increasing agent concentration is a linear relationship, where the highest lead removal efficiency was recorded at a rate of up to 82.31 percentage points at the agent concentration, reaction time and pH of 100 ppm, 120 minutes and 6 respectively. The lowest percentage of lead removal was 68.1 percent, which occurred when using 25 ppm of hydrogen peroxide. Increasing the rate of the amount of H<sub>2</sub>O<sub>2</sub> added is of great importance, as it may lead to reducing the efficiency of lead removal due to an undesirable reaction. Instead of hydrogen peroxide reacting with pollutant ions, it reacts with free radicals [33], [21]. On the other hand, the efficiency and effectiveness of the removal will also decrease if the concentration of hydrogen peroxide is reduced due to the decrease in the number of free radicals formed, as was proven in the study [34].



### 3.1.3. pH effect

The pH greatly affects the Fenton oxidation process, the degradation of hydrogen peroxide, and the speciation of iron sulfate, so it is preferable to use different pH values to reach the highest removal value of pollutants [35]. The lead removal rate increases with increasing cumulative pH, as shown in Figure 4. These results contradict some studies such as those reported by Kansal et al., 2010 and agree with the results of Abbas et al., 2020 [36]. Wastewater is characterized by its pH having very little effect on the lead removal process. Many studies have focused on determining the effect of pH on lead removal efficiency based on the Fenton oxidation process at pH limits ranging from 3 to 10. The highest lead removal of 78 percent was recorded at pH = 6.5, while the removal percentages decreased to 72.41 percent and 51.45 percent at the highest and lowest pH limits, 3 and 10, respectively. For moderately polluted wastewater, the results of practical experiments have proven that the efficiency of lead removal depends on the amount of lead detected at a pH equal to 6.5, as stated in the study presented by [37].

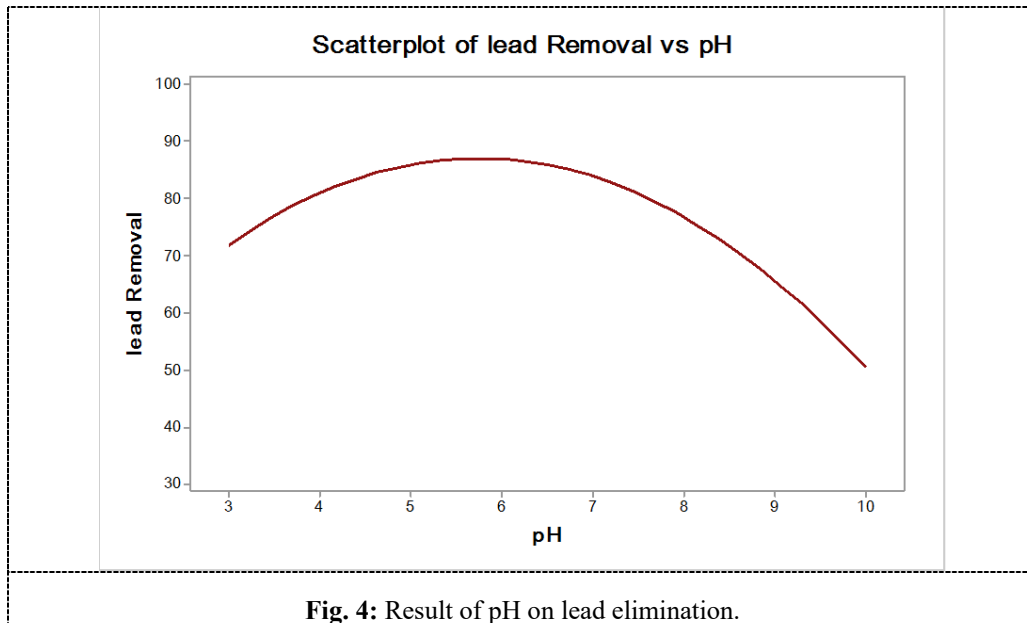


Fig. 4: Result of pH on lead elimination.

### 3.1.4. Ferrous sulphate effect

Practical experiments were conducted in one batch for the purpose of studying the effect of ferrous sulphate on the lead removal rate in the Fenton oxidation process, as shown in Figure 5. It is clear from the graph that the relationship is direct between the concentration of ferrous sulphate and the percentage of lead removal, as the removal percentage increased from 71.21 to 78.59% when ferrous sulphate was increased from 5 to 25 mg/l. Early response elimination continues to occur more quickly than late response elimination. The percentage of lead removal increases in the initial stage of the Fenton oxidation process due to the free radicals produced from the reagent used during the reaction. However, it is preferable to allow sufficient time for the reaction to completely eliminate pollutants in water with low concentrations of ferrous sulphate [25].

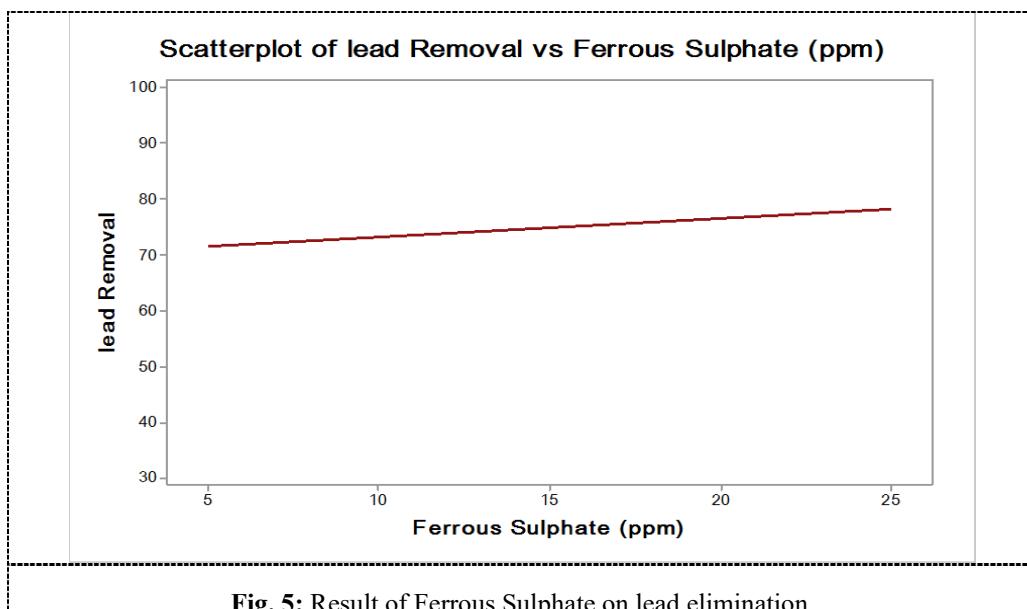


Fig. 5: Result of Ferrous Sulphate on lead elimination.

### 3.2. Optimization and main effect of the working variables

All The Minitab-17 statistical program was adopted to obtain optimal results by specifying appropriate operational variables such as pH, oxidation duration, ferrous sulfate, and lead content. Figure 6 shows the values for D-optimization.

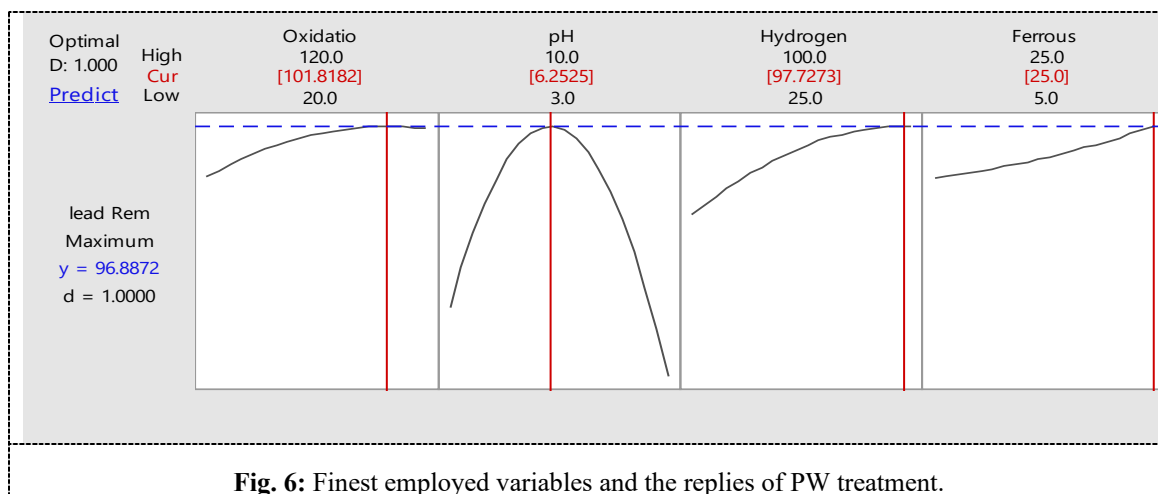


Fig. 6: Finest employed variables and the replies of PW treatment.

The required constraints are listed in their optimal form and arrangement in the master diagrams for the intended oxidation display as shown in Figure 7. The optimal arrangement of restrictions for every presenting education. According to several studies by previous researchers, lead removal has a direct relationship with the concentration of hydrogen peroxide, the concentration of ferrous sulfate, and the duration of oxidation, while the increase in pH is inversely proportional.

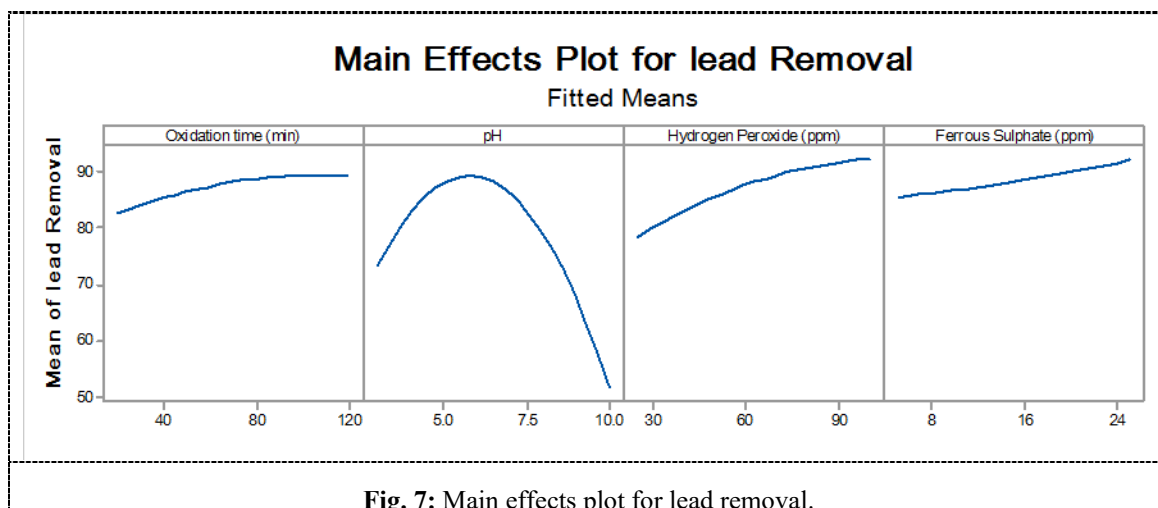


Fig. 7: Main effects plot for lead removal.

## 4. Conclusion

This study is of great importance in developing the design of Fenton oxidation reactors widely used to remove lead from generated water. The pH, Fenton reagent and oxidation period are the operational variables that most affect the efficiency of the removal process. The batch system was used in practical experiments to determine the effect of different operational variables on the decomposition performance to remove all contaminated heavy elements. The high regression coefficients obtained indicate the validity of the use of the second-order polynomial mathematical model. When using the optimal operational variables, the highest percentage of removal was obtained, equal to 94.18 percent. The Fenton oxidation process is a promising technique in removing pollutants due to the successes obtained through its use.

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