



## Quality index for evaluating real effluent by Fenton process using desirability function

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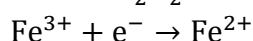
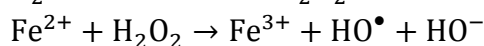
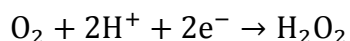
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### Abstract

This study deals with an evaluation of Fenton process applied for industry wastewater. Hence, different values are compared and discussed using statistical, mathematical and geometrical methods. These results are based on the generation of very reactive and very oxidizing species, hydroxyl radicals OH• which are able to oxidize any organic molecule until the ultimate oxidation stage. The experimental design methodology was applied in this work in order to investigate the influence of experimental parameters such as pH, concentration of FeSO<sub>4</sub> and concentration of H<sub>2</sub>O<sub>2</sub>. The application of Fenton reaction has been proved to be highly effective for the treatment of such type of wastewater, and several advantages for the technique application arise from the study. Under the optimal conditions pH about 3, concentration of FeSO<sub>4</sub> equal to 0,8 mM, concentration of H<sub>2</sub>O<sub>2</sub> equal to 4 mM and the ratio (H<sub>2</sub>O<sub>2</sub> concentration)/(FeSO<sub>4</sub> concentration) about 51, Fenton process allowed us to achieve a rate of 95% of discoloration and a 60% of reduction. A desirability function by Derringer and Suich confirms the results obtained by Fenton.

### 1. Introduction

Water is a natural resource, necessary for life and the human and aquatic systems, as well as for economic and social development. This indispensable natural resource is exhaustible. She is also faced with many problems mainly pollution [1]. Environmental pollution is certainly a global problem that affects different sectors. In this context, the textile industry specially dyeing process remains an important source of pollution producing considerable concentrations of organic matter which are hardly to remove [2, 3]. The elimination of contaminants from wastewaters is an essential step required for water pollution [4]. Among the different techniques for removing contaminants [5], Fenton process is an advanced, useful and effective process. This process is one of the most known methods for advanced treatment of organic pollutants in water. Fenton oxidation offers low energy consumption and strong adaptability to pollutants [6, 7]. It is based on the production of hydroxyl radicals (OH•). OH• is considered the most important free radicals in chemistry and biology because of their multiple implications [8, 9]. Hydroxyl radicals are produced from decomposition of H<sub>2</sub>O<sub>2</sub> catalyzed by ferrous or ferric salts:



The reagent ( $\text{Fe}^{2+}/\text{H}_2\text{O}_2$ ) has been carried out in mildly acidic solutions. This was produced by addition of  $\text{Fe}^{2+}$  ions to the solution in the reactor and the simultaneous electrochemical production of  $\text{H}_2\text{O}_2$  upon reduction of the dissolved oxygen. The efficiency of degradation of organic pollutants in Fenton reaction process depends on several parameters such as concentration of Fenton reagent, wastewater pH and initial organic pollutants concentration [4].

A goal of the wastewater purification by Fenton method is the reduction of the chemical contaminants and their toxicity due to the high oxidative power of the OH radical. Fenton process quality refers to the achievement of certain quality of textile effluent with predetermined and desired specifications. Therefore, a very useful part of the process quality is the understanding of different factors and their interaction effects by a set of experiments. To understand these interactions, many statistical and mathematical designs have been recognized as useful techniques [10]. Desirability function has been proved to be efficient for improving, developing and optimizing process. It was initially developed by Box and Wilson Harrington [11]. The fundamentals of desirability and its principal philosophy are studied in many scientific papers and a number of books. The most famous study is that given by [12]. The work of [11] has been used by a large number of authors [13, 14, 15, 16]. Other papers have also appeared [17, 18, 19, 20, 21, 22]. Based on many studies, there is a lack of works that treating water quality or water pollution assessment [2, 23].

In the present study, we have undertaken the study of the oxidation of the organic compounds present in a textile effluent by Fenton reaction. The role that several experimental parameters like temperature, light intensity, and reagent concentration have on the reaction yields has been examined. For that the desirability function is highly effective defining a compromise between these parameters. It proposes an interactive method to predict the quality of waste water treated by Fenton process.

## 2. Material and Methods

### 2.1. Chemical and reagents

The experiments were carried out mainly in the laboratory of the CHIMITEX Company but also in the Chemistry laboratory of the Faculty of Sciences in Monastir. All chemical reagents were purchased from CHIMITEX PLUS and used as laboratory grade. Sodiumhydroxide ( $\text{NaOH}$ ) and sulfuric acid ( $\text{H}_2\text{SO}_4$ ) were of pure grade and used for pH adjustment. Distilled water was used to prepare all wastewater solutions. Reagent grade Hydrogen peroxide ( $\text{H}_2\text{O}_2$  35%) was standardized using iodometric titration. Iron sulfate  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  was standardized before Fenton's experiments. Azoic structure ( $\text{N}=\text{N}-$ ) and vinylsulfonic structure ( $\text{CH}_2=\text{CHSO}_3\text{H}$ ) were used without any purification in order to make similar preparation conditions of synthetic and real wastewaters.

### 2.2. Oxidation with Fenton's reagent

The chemical oxidation with Fenton's Reagent was established in a closed cylindrical reactor to degrade the color of industrial effluent. This process uses hydrogen peroxide and iron compounds to produce non-selective and highly oxidizing hydroxyl radicals.

Several parameters influencing the discoloration of industrial wastewater were studied. The effect of the amount of ferrous sulfate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ) and hydrogen peroxide ( $\text{H}_2\text{O}_2$ , 33%) was established. All the tests were carried out by varying the pH from 2 to 5 (adjustment with 98% sulfuric acid), the concentration of ferrous sulphate from 0.8 mM to 2.2 mM and that of peroxide d from 36 mM to 44 mM. All experiments were performed at room temperature, in the dark, and using hydrogen peroxide as the oxidant and hydrated sulfate as the catalyst. The efficiency of the process will be judged in terms of reduction of the parameters conductivity, MES, color, chloride content and COD.

### 2.3. Experimental design

When talking about industrial production we often have to evaluate quality of products. It proves that frequently more than one measured property has to be considered to define quality. The most famous approach that suggested by literature is the concept [12]. It has been used to attempt multiple-response optimization problems in wastewater treatment. The desirability function converts multiple responses into a single dimension. This single objective is maximized to obtain a compromised solution. The reputation of desirability functions in industrial applications has been noticed and the use of such functions is beginning to include different fields [14].

#### **Proposed Method: Desirability function**

In 1965, Harrington proposed the first Desirability Function concept for determining compromise between different properties. This concept has been refined by the researchers Derringer and Suich. Desirability function transforms a number of properties into a single objective noted  $D$  ( $Y_i, i=1, \dots, m$ ) with range from zero to one. A zero value indicates an undesirable response. Meanwhile, a desirability of one reflects a desirable response. Harrington [11] selected the exponential forms to calculate the desirabilities related to individual criteria  $Y_i$  using the geometric mean for weighting all criteria together:

$$D = \sqrt[m]{\prod_{i=1}^m d_i} = (d_1 d_2 \dots d_m)^{1/m} \quad \text{Equation 4}$$

Where  $d_i$  is an individual desirability function,  $m$  is a number of indicators.

In this case there is possible to identify a compromise between properties affecting the water assessment, we allow to each property a coefficient representing the importance of this property in the Fenton process evaluation. Based on the Derringen and Suich desirability procedure and according to relative importance for each individual desirability we calculated the overall desirability function as follows [16]:

$$D = \sqrt[p]{d_1^{p_1} \times d_2^{p_2} \times \dots \times d_m^{p_m}} \quad \text{Equation 5}$$

Each response is divided into two types: ‘one-sided’ criteria whose acceptable values were bounded by a single specification limit and the ‘two-sided’ criteria whose acceptable values were bounded by a lower specification limit and an upper specification limit. Derringer and Suich recommended a family of functions that allowed the target value to be placed anywhere in the region of product specifications [9].

#### **One-sided transformation**

In the case of desirability function for one sided transformation, the response  $y_i$  must be lower than some maximum value ( $y_{max}$ ) and greater than a minimum value ( $y_{min}$ ), the transformation acquires the following form:

$$d_i(y_i) = \begin{cases} 0 & \text{if } y_i \leq y_{min} \\ \left(\frac{y_i - y_{min}}{y_{max} - y_{min}}\right)^a & \text{if } y_{min} \leq y_i \leq y_{max} \\ 1 & \text{if } y_i \geq y_{max} \end{cases} \quad \text{Equation 6}$$

The ‘ $y_{min}$ ’ parameter specifies the value of ‘ $y_i$ ’ below which the property is undesirable; similarly, the  $y_{max}$  parameter indicates the value above which ‘ $y_i$ ’ is completely desirable.

Finally, the parameter ‘ $a$ ’ is an arbitrary positive constant which controls the curvature of the function. It reflects the requirement of each property and makes subjective judgment on the importance of each

response. The choice of ‘a’ value is critical for an appropriate study and an optimum condition [24, 15, 17].

### Two-sided transformation

When the response  $y_i$  has a maximum and a minimum acceptable values, the individual desirability function is defined by the equation 7:

$$d_i(y_i) = \begin{cases} \left(\frac{y_i - y_{min}}{y_t - y_{min}}\right)^f & \text{if } y_{min} \leq y_i \leq y_t \\ \left(\frac{y_i - y_{max}}{y_t - y_{max}}\right)^s & \text{if } y_t < y_i \leq y_{max} \\ 0 & \text{if } y_i < y_{min} \text{ or } y_{max} < y_i \end{cases} \quad \text{Equation 7}$$

Two-sided desirability function is based on a principle identical to Derringer’s One-sided function Where ‘ $y_t$ ’ is the target value for the  $i^{\text{th}}$  response, and ‘ $f$ ’ and ‘ $s$ ’ are arbitrary positive constants. In this case, ‘ $y_{min}$ ’ is the minimum acceptable value of ‘ $y_i$ ’ and ‘ $y_{max}$ ’ is the maximum acceptable value. The values of ‘ $f$ ’ and the ‘ $t$ ’ in the two-sided transformation take the same position as that of ‘ $a$ ’ does in the one-sided transformation [25, 26].

### 2.4. Computer software

Statistical analysis and plots of the response’s desirability were performed using the software Nemrowd 2000D.

## 3. Results and discussion

The seed oil had an amber yellow colour and a very characteristic nutty flavour. The oil has also liquid state at ambient temperature with a yield (17.85%). The fatty acid compositions of the seed oils are given in Table 1.

### Characterization of the used effluent

The effluents used in the present paper were obtained from the dyeing and finishing of cotton fabrics. The wastewater was supplied by a Tunisian textile manufacturer. The samples of wastewater were collected just before treatment and stored at 4 ° C until their use according to the standard methods for the examination of Water and Wastewater. The characteristics of these wastewater solutions are showed in Table 1.

**Table 1.** Physico-chemical characteristics of real effluent

Parameter	pH	Chloride content (mg/L)	MES (mg/L)	COD (mg O2/L)	Conductivity (mS/cm)	Absorbance $\lambda_{max}=657\text{nm}$
Real wastewater	6.73	4828	896	640	13,9	2,34

From the table 1, we can notice that the COD, the chloride content and the MES exceed the requirements of the Tunisian standard NT 106. In order to reduce these parameters by Fenton process, a preliminary study is necessary. The effectiveness of Fenton's reagent depends on several factors: pH, concentration of catalyst, concentration of  $\text{H}_2\text{O}_2$  and the ratio  $[\text{H}_2\text{O}_2] / [\text{FeSO}_4]$  value. These are the parameters that most influence the catalytic process. We have studied the effect of all these parameters on discoloration; we have carried out tests for durations ranging from 0 to 40 minutes (with a step of 5 minutes).

## Experimental design

Three factors, three levels Box–Behnken design was applied for the optimization procedure using Nemrowd 2000D software (LPRAI-Marseille-France). The independent factors and the responses used in this design are listed in Table 2. The values of pH, concentration of ferrous sulphate [FeSO<sub>4</sub>] and hydrogen peroxide [H<sub>2</sub>O<sub>2</sub>] used to apply Fenton process are given in Table 2. These low and high levels were selected from the ancient work. After generating the polynomial equations, optimization was performed using a desirability function to obtain the levels of X1, X2, and X3, which maximized Y1 while minimizing Y2 and Y3.

Table 2. Variables in Box–Behnken design

Factors	Levels		
	Low	High	
X1: pH	2	4	
X2: [FeSO <sub>4</sub> ] (mM)	0.4	1.2	
X3: [H <sub>2</sub> O <sub>2</sub> ] (mM)	36	40	
Responses	Constraints		
	Low	High	Goal
Y1: Color removal (%)	0	100	Maximize
Y2: MES (mg/L)	30	2000	Minimize
Y3 : DCO (mg de O <sub>2</sub> /L) : Chemical Oxygen Demand	1000	2000	Minimize

## Optimization analysis

For an efficient Fenton process, both MES and DCO are main objectives to be minimized. The desirability function procedure could be utilized to get the maximum color removal. For evaluating several responses in an experimental design, it is important that the optimum points reached individually for each factor coincides in all the cases. A compromise zone was primordial where all the experimental responses fulfill the specifications imposed by the researcher to achieve the same aim. Based on this situation, a desirability function is attributed for each response. The individual desirability varies from zero, undesirable response, to 1 which the response is optimal. Based on these individual functions the overall desirability function is obtained, that will be optimized.

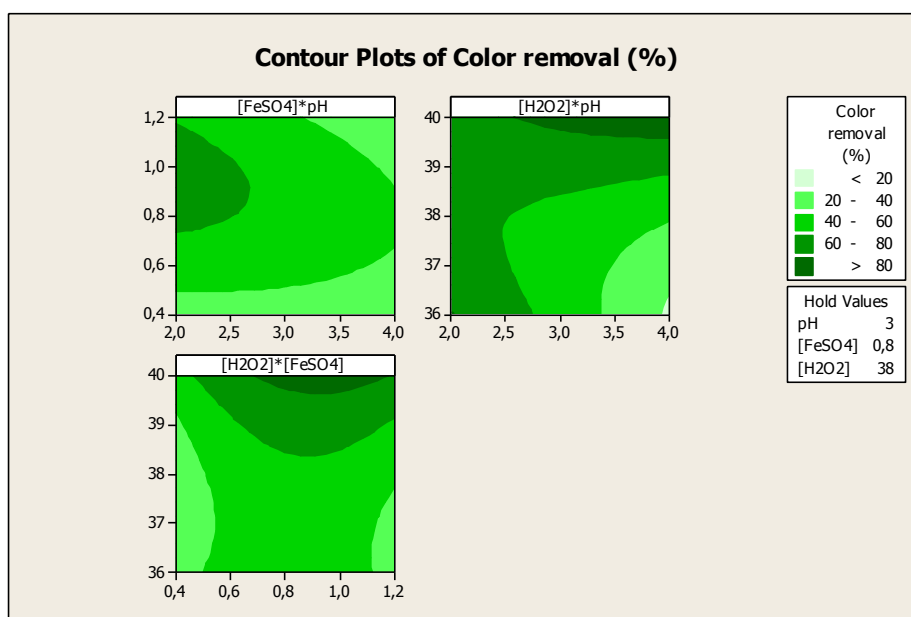
The multi-response optimization included three individuals' desirability: D1 (Color removal), D2 (COD) and D3 (MES).

### *Individual desirability of color removal*

Color removal is the most important property of treated effluent. It determines the efficiency of the Fenton process. The desired values should be reached the value representing the highest importance.

The real effluent has an initial value of pH that can reach 11 or 12. The pH solution is an important parameter for improving removal efficiency in Fenton process. A change of pH values can affect the oxidation dye because it involves a variation of Fe<sup>2+</sup> concentration, hence the production of the radicals OH• will be restricted. The reaction of decomposition of H<sub>2</sub>O<sub>2</sub> was taking to be catalyzed most efficiently by Fe<sup>2+</sup> ions in solutions with pH value between 2 and 4. In order to establish the optimum pH, [H<sub>2</sub>O<sub>2</sub>] and [FeSO<sub>4</sub>] were investigated in a large range of pH values. The use of FeSO<sub>4</sub> iron sulfate as a catalyst accelerates the Fenton reaction by lowering the activation energy of this chemical reaction. The quantity of iron introduced must then be well chosen. In fact, if the catalyst is dosed in a smaller quantity, the

yield will be insufficient. If, on the other hand, this quantity is dosed in excess, we will witness an unpleasant phenomenon which will be detailed later. We observe in [Figure 1](#) that the more the iron concentration increases, the more the discoloration rate increases. We also note that the shape of the curves obtained in the presence of 0.8 mM; 1.2 mM and 2.2 mM is similar.



**Figure 1.** Effect of pH,  $[H_2O_2]$  and  $[FeSO_4]$  on decolorization of industrial effluent (Temperature  $40^\circ C$ )

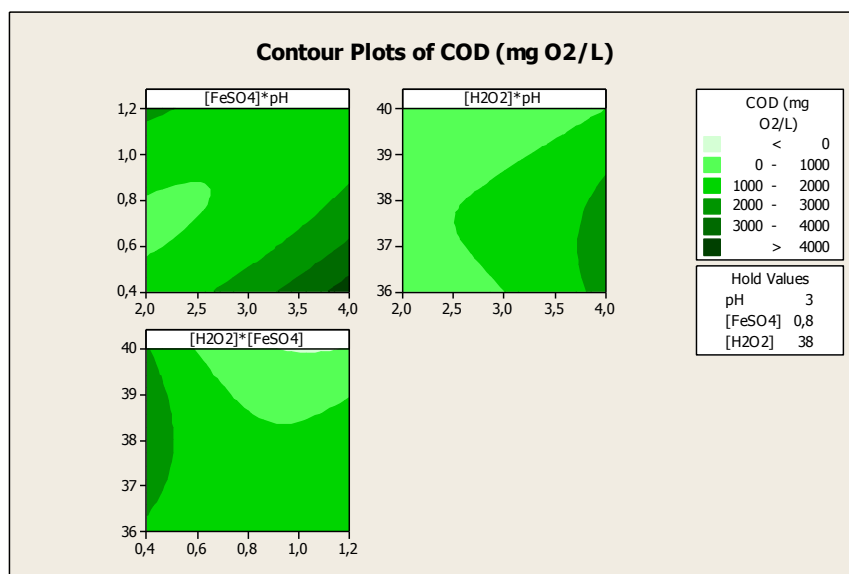
The higher the concentration, the better the discoloration even at the start of the Fenton process. This is not the case for discoloration in the presence of 0.4 mM  $FeSO_4$ . In this case, the best level of discoloration did not exceed 10%. [Figure 1](#) also shows that the discoloration is maximum 70% when the amount of  $FeSO_4$  is 0.4 mM for a period of 40 minutes. In fact, the small quantities of catalyst can be consumed by other reagents, which require a quantity greater than that introduced, hence the ineffectiveness of the Fenton reagent. These low doses of  $FeSO_4$  can also cause the appearance of parasitic reactions between  $H_2O_2$  and  $OH \cdot$  because there is not enough iron to react with  $H_2O_2$  thus the concentration of  $OH \cdot$  which reacts with organic matter and weak.

The effect of the  $H_2O_2$  concentration was studied by varying this concentration from 36 mM to 44 mM. The concentration of  $FeSO_4$  was maintained at 0.8 mM (based on the previous result), the pH of the wastewater is maintained at 3 and at room temperature. Indeed, the effect of temperature on the Fenton reaction has been the subject of several studies which have shown that this parameter (temperature) has no effect on the reaction.

### ***Individual desirability of COD***

Chemical Oxygen Demand is another important factor for the Fenton reaction assessment. It should have an equal importance level when compared with Color removal. As Shown in [Figure 2](#), the optimum pH varies in a scale between 2.5 and 3 for the industrial wastewater. It illustrated also that the COD with Fenton process for the examined effluent proceeded the fastest with pH value equal to 2.5 with an efficiency rate upper than 80%. Many researchers have detected the important use of different ratios of the two reactants  $[H_2O_2]$  and  $[FeSO_4]$ . An excess rate of  $FeSO_4$  or  $H_2O_2$  might be disadvantageous. Hence, these species can react with the radicals  $OH \cdot$ , responsible for the direct oxidation of the organic compounds. In this case it is indicated that for tested effluent increasing the ratio  $[H_2O_2]/[FeSO_4]$  guides to increase the COD, with no effects detected for the highest ratio. On the other hand, the difference

between the COD attained with 30 and 60 ratios demonstrated that enhancement of reaction rate may not give back the large amount of oxidant consumed.

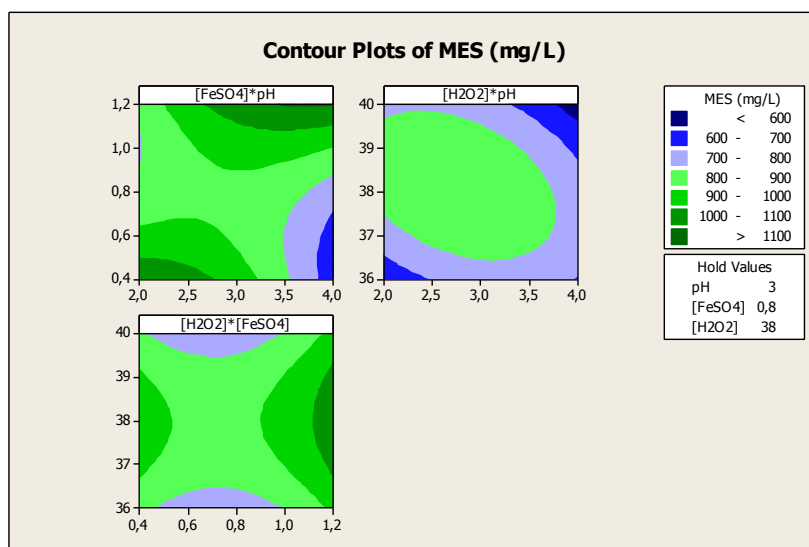


**Figure 2.** Effect of pH, [H<sub>2</sub>O<sub>2</sub>] and [FeSO<sub>4</sub>] on Chemical Oxygen Demand of industrial effluent (Temperature 40°C)

Figure 2 show that with the high ratio of [H<sub>2</sub>O<sub>2</sub>]/[FeSO<sub>4</sub>] the COD decay was low than the rate obtained with lower ratio. This results can be explained that Fenton process, after few seconds, benefits from larger ratio of [H<sub>2</sub>O<sub>2</sub>]/[FeSO<sub>4</sub>]. Whereas for lower ratio, Fenton reaction needs more time to appear oxidation effect.

### *Individual desirability of MES*

Suspended matter MES is a response mentioned for its mandatory to evaluate the Fenton process. Its level of importance is high. We observe in Figure 3 that higher value of pH increases the MES removal. Indeed, very acidic pH (<2) favor the complexation of Fe<sup>2+</sup> by H<sub>2</sub>O<sub>2</sub> and cause a decrease in the concentration of these ions in the reaction medium. This explains a lower level of MES compared to pH = 3.



**Figure 3.** Effect of pH, [H<sub>2</sub>O<sub>2</sub>] and [FeSO<sub>4</sub>] on MES of industrial effluent (Temperature 40°C)

For pH value above 4, ferric ions precipitate in the form of iron hydroxide  $\text{Fe}(\text{OH})_3$ . This precipitate is very stable and the reduction of  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$  becomes very slow and the generation of  $\text{Fe}^{2+}$  as an initiator for the production of  $\text{OH}^\bullet$  radicals, becomes the kinetically limiting step of the process. This justifies the lower discoloration levels at pH = 5.

The ratio  $[\text{H}_2\text{O}_2]/[\text{FeSO}_4]$  is an important factor that determines the efficiency of the mineralization process of organic compounds in the Fenton reaction. It has been the subject of much research. Depending on the nature of the effluent this ratio must be higher or lower. The increase in the rate of MES by the Fenton process remains linked to an increase in the ratio and that of the concentration of the catalyst. When the amount of  $\text{FeSO}_4$  is greater than that of  $\text{H}_2\text{O}_2$ , the treatment tends to have the effect of chemical coagulation. When the two uprights are reversed, the treatment tends to have the effect of chemical oxidation ( $\text{H}_2\text{O}_2 \gg \text{FeSO}_4$ ). Low ratios of  $[\text{H}_2\text{O}_2] / [\text{FeSO}_4]$  and in a sufficiently acid medium ( $0 < \text{pH} < 3$ ), causes the reduction in the rate of MES which may be due to the inhibition of the Fenton system and this has been confirmed by the reduction in the concentration of  $\text{FeSO}_4$ .

The increase in this ratio in a sufficiently acid medium promotes the predominance of propagation and termination reactions and the formation of the hydroperoxyl radical  $\text{HO}_2^\bullet$  able to transform  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$  and propagating the decomposition cycle. However, an excess of reagents can have a limiting factor behavior because  $\text{Fe}^{2+}$  and  $\text{H}_2\text{O}_2$  can become traps for hydroxyl radicals and thus cause a reduction in the degradation of organic matter by inhibiting the Fenton reaction. This finding agreed that obtained elsewhere [27-29].

#### Overall desirability of Fenton process

The methodology for the Fenton process quality is designed relating to the variation of each property from the required value. The main steps followed were property selection, developing of requirement indices, involving weightings and aggregation of the developed indices. The developing equations are based on linear functions which correlate between process quality and selected parameter. Figure 4 presents the results of the optimization for Fenton process. Values of the optimal parameters were as follows: pH= 4, ferrous sulfate concentration  $[\text{FeSO}_4] = 0,97\text{mM}$  and hydrogen peroxide concentration's  $[\text{H}_2\text{O}_2] = 40\text{mM}$ . Weightings were selected based on the literature and the Tunisian standard NT 106 .02. The weightings values for each parameter vary from a country to another.

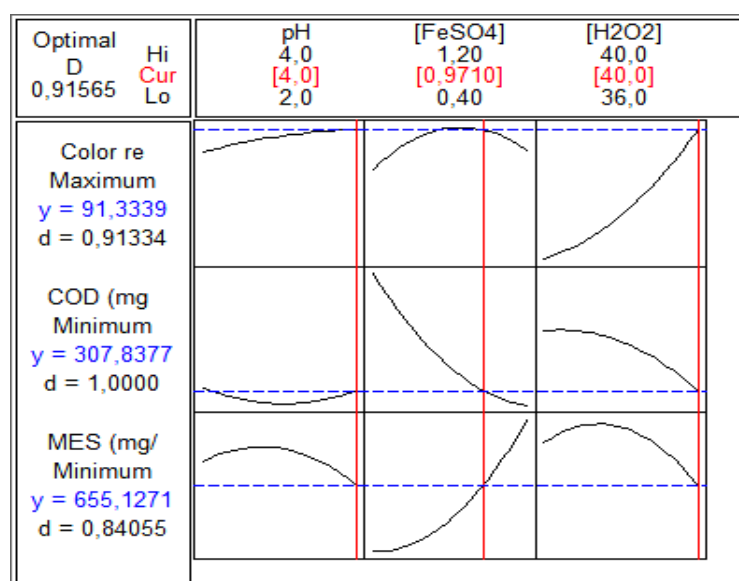


Figure 4. The overall desirability for the optimal Fenton process



From these results, it can be concluded that the three responses yielded acceptable Color removal (d=91%), COD (d=100%) and MES (d=84%). Similarly, it can be deduced from Figure 4 that these three factors have a profound effect on the oval satisfaction of Fenton process. Figure 5 shows the effluent after Fenton treatment. It indicates that there is a significant increase of the color removal. Indeed, the use of reagents in Fenton reaction to destruct the organic compounds creates a dark slurry phase.



**Figure 5.** Application of Fenton process of the industrial effluent ( pH=4,  $[H_2O_2]/[FeSO_4]= 41$ ,  $T=40^\circ C$ )

The formed sludge is mainly reflects a formation of high level of suspended solids. Thus, thanks to the Fenton process, we have managed to considerably clean up this industrial wastewater in terms of discoloration, MES and COD. The discoloration did not exceed 83% and the reduction in MES is around 70%. It should also be noted that the chloride content remains high even after the Fenton process (2911 mg / L). This problem currently concerns all wastewater treatment.

## Conclusion

The Fenton process is a widely studied and a better understanding in many practical fields. This proposed method seems to be available for a large-scale industrial effluent treatment. It has been applied for an industrial textile effluent with the mineralizing of organic matter and removing color. The chemical oxidation was studied through a Fenton process to find out the values of the operating conditions (pH,  $[Fe^{2+}]$  and  $[H_2O_2]$ ) that maximize color removals and DOC also assess the biodegradability of the treated effluent, with the minimum effort in terms of experimentation.

This paper has identified a compromise optimization procedure that considers the correlations between multiple responses using desirability function.

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