

## Chemical Treatment of Industrial Wastewater Using Fenton Reaction

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### Abstract

The treatment is an economic tool to meet the diversity of industrial activities in developing country like Egypt. The present study introduces serious trials to partially remove organic matter in industrial wastewater and their using advanced oxidation process (AOP). Sampling program was carried out according to standard international methods for treating industrial wastewater in terms of wastewater quality parameters such as Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD) to meet the optimum conditions using advanced oxidation process (AOP).

The study aims to apply advanced oxidation process, through Fenton reaction, in secondary treatment of both mixed and industrial wastewater. The research focused on studying the optimum dose of hydrogen peroxide, initial reaction pH value and the retention time to treat influent fresh industrial and mixed wastewater samples. The study samples were fresh industrial and mixed wastewater samples collected from the influent of three different industries. The chosen samples were, namely, Dairy Products Industry, Olive Oil Industry

and Mixed Wastewater located in industrial area in El-Obour City, Qalioubeya, Egypt. The study experiments were carried out at the laboratories of National Research Center (NRC) according to the. The results showed that the overall optimum dose of H<sub>2</sub>O<sub>2</sub> to treat dairy products is 25 ml /l at contact time of 10 minutes and initial reaction pH value of (4). Whereas, the overall optimum dose of H<sub>2</sub>O<sub>2</sub> to treat olive oil industry is 10 ml /l at contact time of 20 minutes and initial reaction pH value of (4). Also, the

overall optimum dose of  $H_2O_2$  to treat mixed industrial- domestic wastewater is 10 ml/l at contact time of 10 minutes and initial reaction pH value of (4).

**Keywords:** Industrial Wastewater, Organic Matter, Fenton reaction, Advanced Oxidation Process.

## 1. Introduction

The removal of organic pollutants involves one or more basic techniques such as chemical oxidation, adsorption and biodegradation. The choice of method depends on process costs and other factors such as type of pollutant, concentration and effluent volume. Advanced oxidation processes (AOPs) are suitable for the degradation of hazardous aqueous pollutants [Zapata et al., 2010]. The AOP processes are capable technology as an alternative treatment to conventional wastewater treatment methods, improvement current biological treatment methods and characterized by their capacity for total/partial oxidation of organic content and/or improvement of the biodegradability [Moreira et al., 2012].

The traditional treatment could be less efficient and slower than chemical treatment in case of mixed wastewater, so it was found that chemical treatment would give higher efficiency if we used it beside traditional treatment. Also, for improving the treatment of sewage, advanced chemical oxidation technologies (AOTs) were applied [ I.G Rahed, 2005] as a successful technique which can degrade the non-biodegradable substance. Oxidation with ozone or hydrogen peroxide has been found to be an important alternative to chlorination because the oxidation doesn't result in toxic chlorinated organic compounds [Chen R, 1997], Fenton reaction is considered as one of the most successful process in advanced chemical oxidation.

The oxidation of organic and natural substrates by iron (II) and hydrogen peroxide is called the "Fenton chemistry", as it was first described by H. J. H. Fenton who first observed the oxidation process of tartaric acid by  $H_2O_2$  in the occurrence of ferrous iron ions [Jian Chen, 1997]. Fenton's reagent ( $H_2O_2/Fe(II)$ ) technique is widely, effectively and easily applied to treat different industrial wastewaters [Sawyer et al., 2003].

Following this time comprehensive inspections showed that the Fenton reagent is effective in treating various industrial sewage components including aromatic amines [Casero I., Sicilia D, 1993], an extensive variety of dyes [Kuo W.G, 1992], [Barbusiński K, 2005] pesticides [Huston P. L, 1999], [Ikehata K. and Gamal El-Din M, 2006] surfactants [Lin S.H., 1999], explosives [Casero I., Sicilia D, 1993]. as well as many other substances. Consequently, the Fenton reagent has been applied to treat many different wastes such as those associated with the textile industry, chemical substance manufacturing, refinery and energy terminals, engine and steel cleaning etc. [ Bigda R, 1996].

The oxidation method is based on a mixture of special property of ferrous ion ( $\text{Fe}^{+2}$ ) and hydrogen peroxide to generate the hydroxyl radical ( $\text{OH}^{\cdot}$ ) at acidic pH in optimum conditions and kinetically the mechanism of the Fenton reaction enhancing the oxidation of the substrate [Liu et al., 2012]. Both optimal dosage of hydrogen peroxide and ferrous ion must be done to complete oxidation reactions and prevent inhibition of the reaction if either of them is not optimal dosage [Muruganandham et al., 2014]. Generally, Fenton's oxidation process is pH adjustment, oxidation response, neutralization and coagulation for precipitation. Therefore, the organic substances are removed in two stages of the oxidation [Kange and Hwang, 2000]. The scavenging effect of Phosphate and bicarbonate anions on the degradation of organic and natural pollutants through the Fenton process may be relatively reduced by the requirement of the application of this Technique at relatively low pH [Nogueira et al., 2005]. Therefore, Chemical treatment by Fenton oxidization process had been studied in this research. The research focused on studying the optimum dose of hydrogen peroxide, pH value and the retention time to treat influent fresh industrial wastewater samples. The used fresh industrial wastewater samples were collected from the influent of Industrial Area of El-Obour City that is located in El-Obour City-Qaliobeya governorate, Egypt.

## **2. Materials and Methods**

All environmental measurements that include chemical and microbial parameters measured and performed according to the instructions of international examinations for collection and preservation samples [APHA, 2012]. The wastewater quality was compared with water quality standards. The work involved advanced oxidation process (AOPs) including Fenton  $\text{H}_2\text{O}_2/\text{Fe}$  reagents technique to evaluate their oxidative abilities. These techniques were applied to industrial wastewater and mixed wastewater that characterized by high load oxidizable demand and high toxicity waste.

## **3. Experimental Work**

### **3.1 Wastewater Sampling**

The used Fresh industrial and mixed wastewater samples were taken from the influent of Three different industries. The three sampling were taken from Dairy Products Industry (A), Olive Oil Industry (B), and Mixed Wastewater (C) which located in industrial area in El-Obour City Qalioubeya, Egypt. Four testes are selected to represent water quality, characterization of industrial wastewater and concentrations of environmental measurements that are listed in Table 1.

<b>Table 1. The Characterization of Wastewater</b>				
<b>Environmental Measurements</b>				
		<b>A</b>	<b>B</b>	<b>C</b>
<b>Physical Analyses</b>	<b>pH</b>	6.45	5.82	7.82
	<b>TSS</b>	183	141	112
<b>Chemical Analyses</b>	<b>BOD</b>	1900	2100	880
	<b>COD</b>	7040	3300	918

### 3.2. Experimental Set-up

For Fenton reaction the material used in this study include Ferrous Sulfate Heptahydrate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ ) which is used as a catalyst and Hydrogen Peroxide solution ( $\text{H}_2\text{O}_2$ ) was provided from El Gomhouria Company for chemicals (El-kasr El-Aini Street, Cairo). In addition, conical glass, one liter, flasks were used. A burette and a stopwatch were, also, used.

### 3.3. Procedure

The investigation was based on the different doses of  $\text{H}_2\text{O}_2$ , pH value and the reaction time. To perform this experimental study, three conical flasks were used. First fresh wastewater was mixed well together, and then samples were taken from it to measure the main chemical, and physical parameters before the treatment by Fenton reaction, which was mentioned in Table 1 previously. Then the three flasks were filled with fresh wastewater samples one liter each. Each sample was later subjected to a different  $\text{H}_2\text{O}_2$  dose. But first, a 0.8 g/L of  $\text{FeSO}_4$  (ferrous sulfate) was added to the flasks. After that, three different concentrations of  $\text{H}_2\text{O}_2$  were used for treatment. The used doses were, 10, 25, and 40 ml/l, respectively, to find out the optimum dose of  $\text{H}_2\text{O}_2$  for each sample and treated it for two pH value of 4 and 10 to find out the optimum pH value for each sample and treated it for three different reaction times of 10, 20, and 30 mins. This arrangement produced twenty-four different samples. Each sample was tested for Chemical Oxygen Demand (COD) and Biological Oxygen Demand (BOD).

### 3.4. Analytical Method

Laboratory Experimental for water sample analyses were carried out at the Central Laboratory for Environmental Quality Monitoring, National Water Research Center (CLEQM-NWRC) at El-Qanter El-Khyria, Qalubiya, Egypt. Many organic measurements are selected to represent organic pollution which are Gross fraction of organic matter include Chemical Oxygen Demand (COD –

colorimetric method) using (HACH DR-3900 spectrophotometer U.S.A.), Biological Oxygen Demand (BOD) using BOD fast respirometry system model TS 606/2 with a measuring range 0-4000 mg/L at 20 °C incubation in a thermostatic incubator model WTW, dryer and sensitive electrical balance according to the standard method.

## 4. Results and Discussion

Advanced Oxidation Processes (AOPs) are very efficient compared to conventional treatment methods for degradation and mineralization of recalcitrant organic pollutants present in wastewater [Wu et al., 2011]. It generates a powerful oxidizing agent, hydroxyl radical, which can react with most of the pollutants present in wastewater.

### 4.1. Technical Evaluation

In order to investigate the optimum dose of  $H_2O_2$ , optimum value of pH and the optimum retention time for COD and BOD removal, a series of experiments were carried out at different Hydrogen Peroxide initial concentrations. The percentages of COD and BOD removal have been determined throughout pH value of 4 and 10 and have been determined throughout the reaction period of 10, 20, and 30 minutes. COD removal efficiency at different doses, different pH value and different retention times were calculated, analyzed, and presented. In order to obtain the optimal pH value and optimal time, the investigation was carried out at various  $H_2O_2$  doses, The treatment was proceeding by a preliminary settling for 2 hours. The preliminary settling was employed to decrease the inorganic content and enhance the efficiency of treatment. **Figure (1)** showed the removal efficiency percentage for all samples treated by Fenton reaction, versus Hydrogen peroxide ( $H_2O_2$ ) dose then compared to raw untreated sample at zero dose of  $H_2O_2$ . The optimum Hydrogen peroxide ( $H_2O_2$ ) dose ranged between 10 and 25 ml/l for all samples.

**For Dairy Products Industry** in general, the maximum COD and BOD removal efficiency (%) for treated wastewater was corresponded an optimal Hydrogen peroxide ( $H_2O_2$ ) dose of 25 ml/l. However, the COD and BOD removal efficiency (%) that corresponded the maximum Hydrogen peroxide ( $H_2O_2$ ) dose of 40 ml/l, was, slightly less than that of the optimal dose of 25 ml/l as shown in Figure (1). This might be attributed to increase of TSS and convert the sample to sludge sample.

**For Olive Oil Industry** in general, the COD and BOD removal efficiency (%) decreased by increasing the dose of ( $H_2O_2$ ). The minimum COD and BOD for treated wastewater were corresponded an optimal Hydrogen peroxide ( $H_2O_2$ ) dose of 10 ml/l than others doses. This might be attributable to the fact that pH of the sample decreased by increasing  $H_2O_2$  doses at that point. The acidity medium led more dissolving of

Olive Oil of wastewater that increased dissolved Olive Oil and organic matter (COD` mg/l) and decreased of organisms count that decreased BOD mg/l.

**For Mixed Wastewater** in general, the COD and BOD removal efficiency (%) decreased by increasing the dose of ( $H_2O_2$ ). The minimum COD and BOD for treated wastewater were corresponded an optimal Hydrogen peroxide ( $H_2O_2$ ) dose of 10 ml/l than others doses. This might be attributable to the fact that pH of the sample decreased by increasing  $H_2O_2$  doses. The acidity medium led more dissolving of fats or oil and grease of wastewater that increased organic matter mg/l.

These results might encourage the wide application of Fenton process in treatment of industrial wastewater with acceptable cost as shown in Figure (1)

**Figure (2)** showed the removal efficiency percentage for all samples treated by Fenton reaction, versus effect of the value of pH (4&10). The optimum pH value of 4 for all samples in general, the increase in COD and BOD removal efficiency (%) was much more dramatic at a pH value of 4 (acidic medium) This might be indicated that the efficiency of the Fenton reaction increases in the acidic medium. These results might encourage the wide application of Fenton process in treatment of industrial wastewater with acceptable cost as shown in Figure (2).

**Figure (3)** showed the removal efficiency percentage for all samples treated by Fenton reaction, versus Changing Contact Time then compared to raw untreated sample. The optimum time ranged between 10 and 20 min. for all samples.

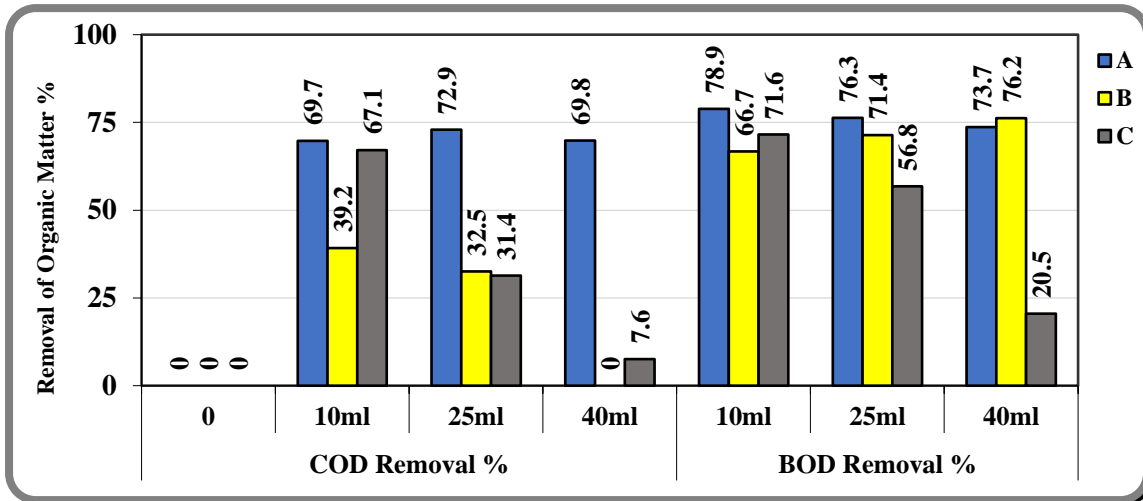
**For Dairy Products Industry** in general, the COD increase by increasing the time and the data of BOD mg/l decreased at 10 min. (100 mg/l). The minimum COD for treated wastewater was corresponded an optimal time of 10 min. However, the COD that was corresponded the maximum time of 30 min. was higher than that of the optimal time of 10min., This might be attributable to the fact that pH at that point was less 1 and the medium of solution became acid. The acid medium for led more dissolving of dairy products of wastewater and increased the organic matter (COD) and decreased of organisms count that decreased (BOD), in general, the increase in COD and BOD removal efficiency (%) was much more dramatic at 10 min.

**For Olive Oil Industry** in general, the COD and BOD decreased by increasing the Time. The minimum COD and BOD for treated wastewater were corresponded an optimal Time of 20 min. than others times. This might be attributable to the fact that pH of the sample decreased by increasing time at that point. The acidity medium led more dissolving of Olive Oil of wastewater that increased dissolved Olive Oil and organic matter (COD mg/l) and decreased of organisms count that decreased BOD mg/l

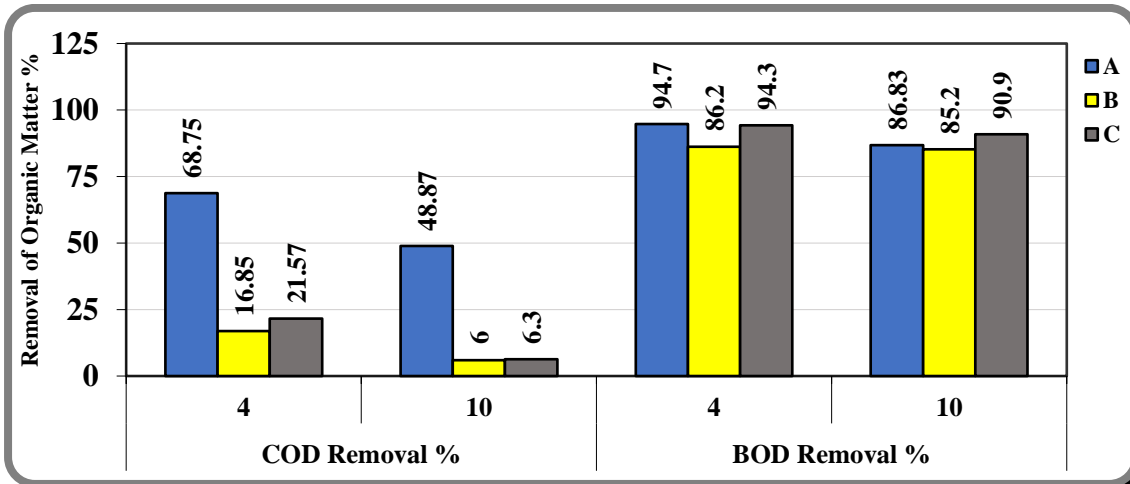
**For Mixed Wastewater** in general, the COD increase by increasing the time and the optimum time was 10 min. That removed BOD concentrations to (80mg/l) and removed COD concentrations to (860mg/l) for treated wastewater. This might be attributable to the fact that pH of the sample decreased by increasing time. The acidity

medium led more dissolving of fats or oil and grease of wastewater that increased organic matter mg/l.

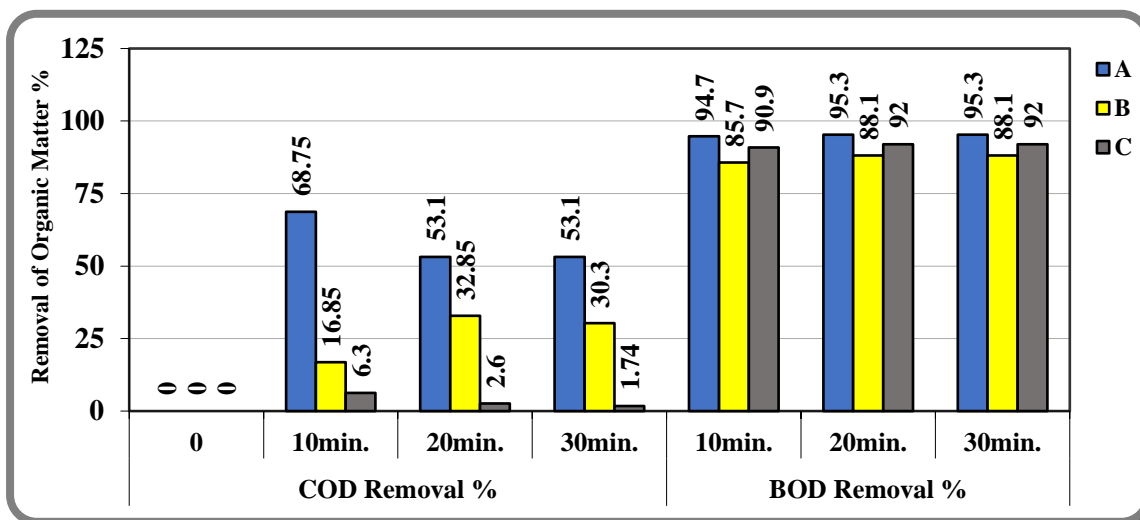
These results might encourage the wide application of Fenton process in treatment of industrial wastewater with acceptable cost as shown in Figure (3).



**Figure 1: Removal Efficiency (%) for COD and BOD at Fenton's Reagent dose (10, 25 and 40ml of H<sub>2</sub>O<sub>2</sub>+ 0.8g/l FeSO<sub>4</sub>) & Fixed pH and Contact Time (10 Min.) for all Samples.**



**Figure 2: Removal Efficiency (%) for COD and BOD at different pH (4 & 10) & Fixed Fenton Reagent and Contact Time (10 Min.) for all Samples.**



**Figure 3: Removal Efficiency (%) for COD and BOD at Changing Contact Time (10, 20, 30 Min.) at Fixed Fenton Reagent and pH value (4) on Organic Matter (COD & BOD) for all Samples.**

## 5. Conclusion

The research studied the effect of  $H_2O_2$  dose variation with pH value and the retention time on enhancing the BOD and COD. The main target was to investigate the suitability of using Fenton reaction in treatment of industrial wastewater and mixed wastewater. These tests were executed to achieve, both, efficiency and economical benefit. Based on the experimental work program executed in this research, and limited to the tested materials and testing procedures, the following conclusions can be achieved:

Fenton reaction can be used in the treatment of industrial and mixed wastewater with great variation in organic matter content.

### For Dairy Products Industry Wastewater

- The removal efficiency (%) for COD pollutant under  $H_2O_2$  doses (10ml/l, 25 ml/l and 40 ml/l) at fixed pH of sample and contact time (10 min.) was 69.7 ,72.9 and 69.8 respectively.
- The removal efficiency (%) for BOD pollutant under  $H_2O_2$  doses (10ml/l, 25 ml/l and 40 ml/l) at fixed pH of sample and contact time (10 min.) was 78.9 ,76.3 and 73.7 respectively.

Hence the optimum dose For Dairy Products Industry Wastewater is 25 ml/l.

- The removal efficiency (%) for COD pollutant under changing the value of pH (4 & 10) at fixed Fenton Reagent (25 ml of  $H_2O_2$ + 0.8 g/l  $FeSO_4$ ) and contact time (10 min.) was 68.75 and 48.87 respectively.



- The removal efficiency (%) for BOD pollutant under changing the value of pH (4 & 10) at fixed Fenton Reagent (25 ml of  $\text{H}_2\text{O}_2$ + 0.8 g/l  $\text{FeSO}_4$ ) and contact time (10 min.) was 94.7 and 86.83 respectively.  
Hence the optimum pH value For Dairy Products Industry Wastewater is 4.
- The removal efficiency (%) for COD pollutant under changing contact time (10, 20 and 30 min.) at Fixed Fenton Reagent (25 ml of  $\text{H}_2\text{O}_2$ + 0.8 g/l  $\text{FeSO}_4$ ) and pH value (4) was 68.75 ,53.1 and 53.1 respectively.
- The removal efficiency (%) for BOD pollutant under changing contact time (10, 20 and 30 min.) at Fixed Fenton Reagent (25 ml of  $\text{H}_2\text{O}_2$ + 0.8 g/l  $\text{FeSO}_4$ ) and pH value (4) was 94.7 ,95.3 and 95.3 respectively.  
Hence the optimum time For Dairy Products Industry Wastewater is 10 min.
- The overall optimum dose for  $\text{H}_2\text{O}_2$  is 25 ml /l with contact time = 10 minutes and pH value (4).

#### **For Olive Oil Industry Wastewater**

- The removal efficiency (%) for COD pollutant under  $\text{H}_2\text{O}_2$  doses (10ml/l, 25 ml/l and 40 ml/l) at fixed pH of sample and contact time (10 min.) was 39.2 ,32.5 and 0.0 respectively.
- The removal efficiency (%) for BOD pollutant under  $\text{H}_2\text{O}_2$  doses (10ml/l, 25 ml/l and 40 ml/l) at fixed pH of sample and contact time (10 min.) was 66.7 ,71.4 and 76.2 respectively.  
Hence the optimum dose For Olive Oil Industry Wastewater is 10 ml/l.
- The removal efficiency (%) for COD pollutant under changing the value of pH (4 & 10) at fixed Fenton Reagent (10 ml of  $\text{H}_2\text{O}_2$ + 0.8 g/l  $\text{FeSO}_4$ ) and contact time (10 min.) was 16.85 and 6 respectively.
- The removal efficiency (%) for BOD pollutant under changing the value of pH (4 & 10) at fixed Fenton Reagent (10 ml of  $\text{H}_2\text{O}_2$ + 0.8 g/l  $\text{FeSO}_4$ ) and contact time (10 min.) was 86.2 and 85.2 respectively.  
Hence the optimum pH value For Olive Oil Industry Wastewater is 4.
- The removal efficiency (%) for COD pollutant under changing contact time (10, 20 and 30 min.) at Fixed Fenton Reagent (10 ml of  $\text{H}_2\text{O}_2$ + 0.8 g/l  $\text{FeSO}_4$ ) and pH value (4) was 16.85 ,32.85 and 30.3 respectively.
- The removal efficiency (%) for BOD pollutant under changing contact time (10, 20 and 30 min.) at Fixed Fenton Reagent (10 ml of  $\text{H}_2\text{O}_2$ + 0.8 g/l  $\text{FeSO}_4$ ) and pH value (4) was 85.7 ,88.1 and 88.1 respectively.  
Hence the optimum time For Olive Oil Industry Wastewater is 20 min.
- The overall optimum dose for  $\text{H}_2\text{O}_2$  is 10 ml /l with contact time = 20 minutes and pH value (4).

### **For Mixed Industrial-Domestic Wastewater**

- The removal efficiency (%) for COD pollutant under  $H_2O_2$  doses (10ml/l, 25 ml/l and 40 ml/l) at fixed pH of sample and contact time (10 min.) was 67.1 ,31.4 and 7.63 respectively.
- The removal efficiency (%) for BOD pollutant under  $H_2O_2$  doses (10ml/l, 25 ml/l and 40 ml/l) at fixed pH of sample and contact time (10 min.) was 71.6 ,56.8 and 20.5 respectively.

Hence the optimum dose For Mixed Industrial-Domestic Wastewater is 10 ml/l.

- The removal efficiency (%) for COD pollutant under changing the value of pH (4 & 10) at fixed Fenton Reagent (10 ml of  $H_2O_2$ + 0.8 g/l  $FeSO_4$ ) and contact time (10 min.) was 21.57 and 6.3 respectively.
- The removal efficiency (%) for BOD pollutant under changing the value of pH (4 & 10) at fixed Fenton Reagent (10 ml of  $H_2O_2$ + 0.8 g/l  $FeSO_4$ ) and contact time (10 min.) was 94.3 and 90.9 respectively.

Hence the optimum pH value For Mixed Industrial-Domestic Wastewater is 4.

- The removal efficiency (%) for COD pollutant under changing contact time (10, 20 and 30 min.) at Fixed Fenton Reagent (10 ml of  $H_2O_2$ + 0.8 g/l  $FeSO_4$ ) and pH value (4) was 6.3 ,2.6 and 1.74 respectively.
- The removal efficiency (%) for BOD pollutant under changing contact time (10, 20 and 30 min.) at Fixed Fenton Reagent (10 ml of  $H_2O_2$ + 0.8 g/l  $FeSO_4$ ) and pH value (4) was 90.9 ,92 and 92 respectively.

Hence the optimum time For Mixed Industrial-Domestic Wastewater is 10 min.

- The overall optimum dose for  $H_2O_2$  is 10 ml /l with contact time = 10 minutes and pH value (4).

### **5.2. Recommendations for Further Researches**

The work of this research was mainly based on studying the possibility of using Fenton's reaction in different industrial wastewater in Egypt. The tests were carried out and evaluated and the results led to the following recommendations:

The wastewater of oxidation ponds in El-Obour City is highly polluted with different variables that must be treated to increase their efficiency and utilization in water reuse. Then, the environmental studies must be focused on:

- Renew and develop factories in-situ treatment plant that has pretreatment unit before discharge its effluents into oxidation ponds.
- Environmental monitoring along new cities to check their water quality, avoid pollution sources and their wastes must not be used for irrigation.
- Environmental monitoring for groundwater quality to avoid pollution sources.
- Separation for agricultural and domestic drainage water from industrial wastewater to construct suitable domestic sewage facilities to avoid their

effluents into agricultural drains.

- Fenton reaction efficiency in removing TSS pollutant are incompetent. It is therefore advisable to use sedimentation tanks before treatment process to insure best results

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