



## DETERMINATION OF COD AND COLOR REDUCTION OF STABILIZED LANDFILL LEACHATE BY FENTON PROCESS



Anita Maslahati  
Roudi<sup>1\*</sup>  
Shreeshivadasan  
Chelliapan<sup>2</sup>  
Samaneh Sadat  
Mohseni Armaki<sup>3</sup>

<sup>1</sup>Department of Environmental Engineering, Faculty of Civil Engineering, Universiti Teknologi Malaysia, Johor Bahru, Malaysia

<sup>2</sup>Engineering Department, UTM Razak School of Engineering & Advanced, Universiti Teknologi Malaysia, Malaysia

<sup>3</sup>Environmental pollution, Islamic Azad University, Damavand Branch, Tehran Province, Iran.



(+Corresponding author)

### ABSTRACT

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The Fenton process on landfill leachate was done in a batch reactor. The application of Fenton treatment technology for landfill leachate treatment greatly depends on the optimum Fenton operating conditions. The present study investigated the chemical oxygen demand (COD) and color removal efficiency of a non-biodegradable leachate by Fenton process. Determining optimum Fenton conditions requires multiple experiments using variable reaction parameters (pH, temperature, and H<sub>2</sub>O<sub>2</sub> and Fe<sup>2+</sup> doses). The treatment showed that the leachate organics can be broke down by the use of Fenton's reagent. The result showed that with increasing H<sub>2</sub>O<sub>2</sub> dosage, H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> ratio and decreasing the pH and contact time, the COD and color removal increased. The laboratory result determined that the best functional situation to catch the goals \_ (i.e., BOD<sub>5</sub>/COD =0.1) resulted; pH=3, Contact time=30 minutes, H<sub>2</sub>O<sub>2</sub> concentration= 40000 mgL<sup>-1</sup> and the favorable H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> ratio = 10.

**Contribution/ Originality:** The main contribution of this research is a better understanding of the treatment of landfill leachate using Fenton process for COD and Color removal to meet effluent discharge standards.

## 1. INTRODUCTION

The landfill leachate made from solid waste in cities and landfill sites are known as wastewaters which are dangerous and extremely polluted. There exist a large amount of organic materials as both kinds of biodegradable and bio refractory carbon, ammonia-nitrogen, heavy metals, and chlorinated organic and inorganic salts in leachates [1]. Environmental problems can be resulted from discharging the landfill leachate in the way of percolating through soils and sub-soils with an inappropriate and bad dispose of the landfill leachate such problems causes' serious ground and surface water pollution [2, 3].

AOP are generally explain as the processes of oxidation producing hydroxyl radicals in an acceptable amount to have an effect on water and wastewater treatment [4]. The hydroxyl radical is defined as one of the most reactive free radicals and one of the strongest oxidants (HO<sup>o</sup>+H<sup>+</sup>+e<sup>-</sup>→H<sub>2</sub>O; E<sup>o</sup>= 2.33 mV). Except the Fenton process, a large number of systems can be classified as AOP, and most of such systems use a combination [5]. Fenton process (FP) removes a wide range of OM and has been studied by several researchers for treatment of

various types of industrial and residential wastewaters including landfill leachate [6-9]. Hydrogen peroxide, in the Fenton process, is added to waste water and ferrous salt is presented to generate species which are strongly oxidative when the organic compounds are existed [10]. Hydroxyl radicals ( $^{\circ}\text{OH}$ ) that traditionally are considered as the main oxidizing species in the Fenton processes [11, 12] though high valence iron species and alkoxy radicals ( $\text{RO}^{\circ}$ ) have also been proposed [13, 14]. The mechanism of the classical Fenton free radical lacking the organic compounds mostly includes the series of reactions below [6]. Fenton's reagent ( $\text{Fe}^{2+} + \text{H}_2\text{O}_2 + \text{H}^+$ ) includes main advantages which are presented as: 1) non-poisonous and cheap iron and hydrogen peroxide; 2) no limitation in mass transfer for its homogeneity in catalytic nature; 3) no energy from involved as a catalyst; 4) the technological simplicity of the process. Many areas of study has used the Fenton's process, [15] for instance the recalcitrant of wastewater and/or landfill leachates treatment [16-24]. Landfill leachate treatment using the FP has shown 30–95% total COD removal for initial COD ( $\text{COD}_0$ ) in the range of 93–34,920  $\text{mg L}^{-1}$  and  $\text{BOD}_5/\text{COD}$  has increased from 0.01–0.44 to 0.1–0.70 [9, 25].

Therefore, the objective of this research was to evaluate landfill leachate treatment using the Fenton process for COD and Color removal to meet effluent discharge standards.

## 2. MATERIALS AND METHODS

### 2.1. Materials

The collected samples were gathered from Jeram Landfill, which is located in an oil palm plantation at Kuala Selangor, Malaysia. When the samples were collected based on the standard methods. The leachate was kept in polyethylene bottles and then in a refrigerator in order to protect from any light. Zha, et al. [26] when the samples were brought out of the refrigerator and were placed at about 22 °C to be conditioned for about 2 hours [27]. Before any experiments filtration was done to remove large particles and debris in order to eliminate the special effects in reactions of oxidation. The major features of leachate which was used in the experiments are demonstrated in Table 1. Standard Methods were the base for any analyses [26, 28]. In order to adjust the pH, Sulphuric acid and Sodium were used. In order to determine the color, COD, suspended solids, and turbidity, Samples were withdrawn by the use of plastic syringe from about 2 cm below the liquid level where the point located. The analyses were assumed to be duplicated. A pH meter (SCHOTT Instruments-Lab 850) was used to measure the pH, while turbidity was measured using 2020 Turbid meter (LaMotte) [27]. Based on the standard methods, COD were determined by both Hach vials and a closed reflux, colorimetric method at 600 nm with Hach spectrophotometer (Hach DR/2800) [28, 29].

Color measurements were reported as true color (filtered using 0.45  $\mu\text{m}$  filter paper) assayed at 455 nm using DR 2800 HACH spectrophotometer following Standard Method [28] Method No. 2120C reported in Platinum-cobalt (Pt Co), the unit of color being produced by 1 mg platinum/l by the use of central sample, The effect of color removal filtration was corrected. Removal efficiency of color was obtained using the following formula [27].

$$\text{Removal \%} = \left( \frac{C_i - C_f}{C_i} \right) * 100 \quad (1)$$

where  $C_i$  and  $C_f$  are the initial and final color concentration of leachate, respectively [27].

### 2.2. Fenton's Treatment Procedure

Bench-scale batch experiments were carried out at room temperature to determine optimum operating conditions of the reaction [30]. Shaking the Leachate samples helped to re-suspension of settled solids and the desired aliquots of leachate were transferred to jar-test beaker [25]. 500mL cylindrical glass vessel was used for this purpose. The predetermined ferrous sulfate dose was added to 300 ml leachate sample. The mixture was acidified using sulfuric acid to enhance the oxidation [31, 32]. This was followed by the addition of hydrogen peroxide under vigorous stirring to start Fenton reaction. The desired amounts of reaction times were selected

based on each step for starting oxidation. The pH of the leachate samples were raised up to eight by NaOH for two reasons; precipitation of dissolved  $\text{Fe}^{3+}$  and degradation of residual  $\text{H}_2\text{O}_2$ . By increasing the pH, coagulation was provided. When the Fenton process finished, sludge was allowed to sediment for 30 minutes for coagulation-flocculation step. Samples which were filtered were used in all analyses of treated leachate [30].

### 2.3. Analytical Method

TOC analyses, total organic carbon, were accomplished by an Aurora 1030 W TOC analyzer from Shimadzu, Japan. The iodometric method was used to determine  $\text{H}_2\text{O}_2$  concentration. Metals were analyzed by PerkinElmer ICP-Mass Spectrometer, NexION 350 Q.  $\text{BOD}_5$ , COD, TS, TSS,  $\text{PO}_4\text{-P}$ ,  $\text{NH}_4\text{-N}$ , alkalinity and chlorides were measured according to standard methods [28]. COD were determined by both Hach vials and a closed reflux, colorimetric method at 600 nm with Hach spectrophotometer (Hach DR/2800) according to the Standard Methods [29].

### 2.4. Experimental Procedures

In the present study, Fenton oxidation and coagulation process were modified by use of batch-scale tests which were carried out in a reactor to reduce COD and Color from landfill leachate treatment [33]. A magnetic device was used to mix the leachate. The treatment was performed through some experiments, and the functional characters were optimized below: the reaction time, the temperature, the initial pH, and the reagent concentrations [26]. First, in order to determine the value of suitable PH for Fenton oxidation and coagulation process,  $\text{H}_2\text{SO}_4$  was added an appropriate value of PH is 3.0 [33]. an automatic pH controller was used to control the acidic condition on the reactor by the use of 0.02 M sulfuric acid and 0.02 M sodium hydroxide [29]. Primary tests of batch were carried out in order to discover the best dosage of Fenton reagents. Maximum COD and color removals of Shah Alam landfill leachate were recognized when  $\text{H}_2\text{O}_2/\text{Fe}^{2+}$  was added at the molar ratio of 10. The form of Ferrous Sulfate solution was used as the iron to be added. Respectively the amounts of 40000 and 4000 mg/L were used for  $\text{H}_2\text{O}_2$  and  $\text{Fe}^{2+}$  of to be added. In order for happening the oxidation process, the samples were mixed by the use of a glass stick. The reports showed that Fenton oxidation rapidly reacted at the first steps that chemicals has added, followed by a decrease of oxidation rate with time [34]. A modified Fenton oxidation was continued by predetermined time. After the Fenton oxidation process took place over Leachate, it was sent into the sedimentation reactor of which pH was changed to 7.0 by NaOH. Sedimentation was supplied within 30 minutes to fix the sludge which were produced. Realizing the effectiveness of the Fenton oxidation-coagulation process, the supernatant were measured using COD and Color. Through the sludge resulted from Fenton oxidation which contain iron salt, partly existing as ferric hydroxo complexes [35] therefore, the sludge may play an further role in the process of coagulating [33].

## 3. RESULTS AND DISCUSSIONS

### 3.1. Leachate Characteristics

The chemical compound of the Leachate which was examined in the study is reported in table 1. The leachate was classified as "old" and non-biodegradable through considering the value of pH, the low value of  $\text{BOD}_5/\text{COD}$  ratio [ $(2096/10516) = 0.1$ ] and the  $\text{NH}_4\text{-N}$  contents of (781 mg  $\text{L}^{-1}$ ). Baig, et al. [36] based on Fenton process indicates clearly that higher hydrogen peroxide to substrate ratios leads to a wider degradation of substrate, when faster rates are produced by higher concentrations of iron ions [24]. it also shows that the intensity of the color was higher than 500 Pt-Co with the dark color [27] stated that there would be high organic substances (measured as COD) which is related to suspended solids and turbidity when there is a high amount of color in the landfill leachate. Due to its low  $\text{BOD}_5/\text{COD}$  ratio and high concentration of  $\text{NH}_3\text{-N}$  (781 mg  $\text{L}^{-1}$ ), Jeram raw leachate is recognized high in stabilized leachate and low in biodegradability [37].

Table-1. Comparison of different parameters of Jeram landfill leachate with Malaysia standards

Parameters	Units	Value	Standard B
PH	-	7.5	5.5-9
Temperature	°C	25.5	40
COD	mg/L	10516	#200
BOD <sub>5</sub>	mg/L	2096	50
Total Suspended Solid	mg/L	810	100
Color	Pt.Co	> 500	200
Ammonical Nitrogen	mg/L	781	20
Zinc as Zn	mg/L	2.48	2.0
Iron as Fe	mg/L	28.2	5.0
Oil and Grease	mg/L	24.5	10

Source: Jeram Landfill, Palm Oil plantation in Kuta Selangor, Malaysia.

### 3.2. Effect of Fenton Oxidation Reaction Time on COD and Colour Removal

In order to find an experimental condition for further research, the reaction time effect on Fenton process was examined. In the present study, the efficacy of Fenton process was evaluated in terms of COD and Color. Although it is assumed in COD test that all organic substances can be oxidized. The oxidation process is done by a strong oxidizing agent and under acidic conditions COD test may include some limitations. Organic nitrogen will be changed to ammonia. In addition, some reduced substances such as sulfides, sulfites, ferrous iron, and hydrogen peroxide will be oxidized. They are all known as COD [28, 29]. A function of oxidation reaction time is shown in figure 1 that demonstrates an increase of COD and color removal efficiencies. Based on the results, Fenton reagents degraded the organic substances rapidly. In the first 30 minutes most of the organic removal has been occurred, and then residual COD and color changes become inconsiderable. The COD and color removals stabilized at

In addition inorganic carbon concentration of untreated leachate changed into carbonic acid from bicarbonate ions when pH was adjusted to acidic. And color removals stabilized at 73.25% and 90.0%, respectively. More foam was observed on the top layer of leachate as the oxidation proceeded. It was an evidence of formation of carbon dioxide. COD reduced from 10516 mg/L<sup>-1</sup> to 2570 mg/L<sup>-1</sup> during 30 minutes reaction.

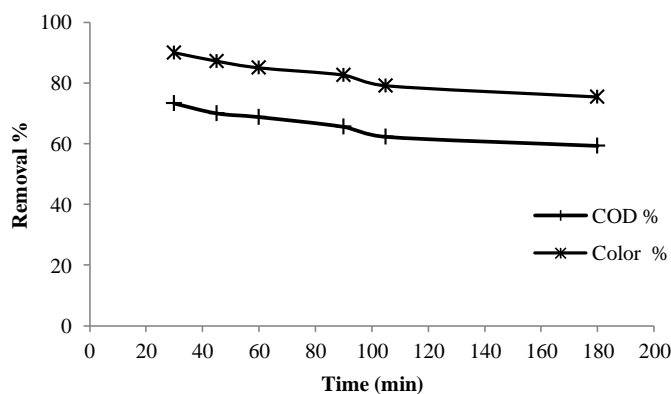


Figure-1. Effect of Fenton oxidation reaction time on COD and colour removal. (H<sub>2</sub>O<sub>2</sub>: 40000 mg L<sup>-1</sup>, pH=3, Fe<sup>2+</sup>: 24000 mg L<sup>-1</sup>)

It turned to inorganic carbon as carbon dioxide. In addition, inorganic carbon concentration of untreated leachate changed into carbonic acid from bicarbonate ions when pH was adjusted to acidic. Carbon dioxide results from dissolving the carbonic acid in liquid or escaping to air. The main organic substances were rapidly changed to other by products that are more highly oxidized by-Fenton reagent. Though, the organic carbon was not completely converted to inorganic carbon. this shows that the first reactions of oxidation were not only partial oxidations (primary degradation and/or acceptable degradation) also ultimate conversion to inorganic carbon with a large amount of hydroxyl radicals, but further reactions with residual hydroxyl radicals prevailed partial

oxidations rather than ultimate conversion [29]. The best reaction time (30 minutes) for the Fenton process with batch reactor was found by Zhang et al. with the same result.

### 3.3. Optimum pH

To test the degradation of COD and color reduction, pH has been examined closely [3]. pH of solution, as a significant factor in Fenton process, directs the hydroxyl radical production and Ferrous ions when concentrating [37].

The activity of oxidant (iron specification), and substrate (hydrogen peroxide decomposition) are both affected by pH. According to the explanation of Sedlak and Andren [38] producing higher hydroxyl radical happens in a pH within 2 to 4 through a reaction which involves the organometallic complex of where an increase happened in the reaction rate and when hydrogen peroxide is regenerated. As inorganic carbons of the wastewater can scavenge the hydroxyl radicals, they should be removed. Such Carbons (inorganic ones) can be eliminated easily when the pH is controlled in an acidic condition. Fig 2 demonstrates how pH can affect the removal effectiveness of the COD and color. Best removal efficiency was obtained at pH 3.0. By increasing the pH, the soluble species have been extremely decreased. Considering the added total iron concentration, most irons precipitated as Haag and Yao [39] found, and oxidation became less efficient. The reactions were tested in three different ranges (3,6, and 9) of pH value.

Figure 2 demonstrates that most removals of COD and color were taken win pH value of 3, increasing the pH value made them decrease. As in Figure 2, the maximum COD removal was 57.09% and 69.6% for maximum color removal. In order to produce the maximum amount of hydroxyl radicals to oxidize organic complexes; the pH value must exist in the acidic range. As at a very low pH (<2.0) the reaction happens slowly due to the formation of complex iron species and oxonium ions  $[H_3O_2]^+$ , so the pH value should not be too slow. At high pH (pH > 4), in contrast; there would be a slow generation of hydroxyl radicals, because the ferric hydroxo compounds are forming.

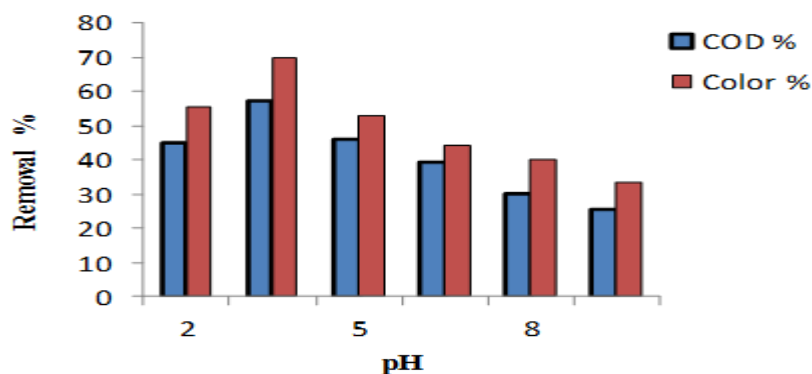


Figure-2. COD and Color removal efficiencies at different reaction pH values (Contact time=30 min,  $H_2O_2$  concentration= 40000 mg  $L^{-1}$ ,  $Fe^{2+}$ =24000 mg  $L^{-1}$ )

### 3.4. Effect of Temperature

The effect of temperature has been ignored in all studies, because this factor enhanced the COD removal slightly. There is no need to pay attention to the temperature in optimization of Fenton's reaction for leachate to be treated. [29, 40] investigated 10–30 °C and 13–37 °C, respectively, and discovered that an increase in temperature will increase the final COD removal. An extreme increase in temperature leads to an ineffective composition of  $H_2O_2$  that offsets COD removal increase. As a consequence, the increase of COD removal is marginal in a high temperature. In compare with other factors, increasing the organic removal due to the increase in temperature is relatively small [6, 29].

### 3.5. Effect of H<sub>2</sub>O<sub>2</sub> Concentration

Investigating optimum H<sub>2</sub>O<sub>2</sub> concentration, different initial concentrations were tested. The H<sub>2</sub>O<sub>2</sub> concentrations from 8000 mg L<sup>-1</sup>, 15000 mg L<sup>-1</sup>, 20000 mg L<sup>-1</sup>, 24000 mg L<sup>-1</sup>, 30000 mg L<sup>-1</sup> and 40000 mg L<sup>-1</sup> were used. Figure 3 shows the final COD and Color removal effectiveness is affected by the initial hydrogen peroxide concentration. It was determined that COD removal effectiveness increased from 33.3% to 70.67% when H<sub>2</sub>O<sub>2</sub> concentration has been raised to 40000 mg/L<sup>-1</sup>. The optimal concentration of H<sub>2</sub>O<sub>2</sub> was obtained as 40000 mg/L<sup>-1</sup> for the Fenton processes.

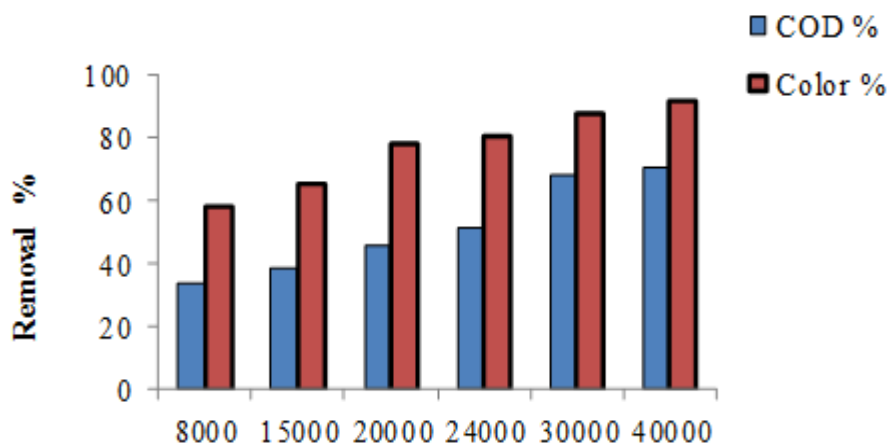


Figure-3. Effect of H<sub>2</sub>O<sub>2</sub> concentration on COD and Color removal efficiency (Contact Time= 30 min, pH=3, H<sub>2</sub>O<sub>2</sub>: Fe<sup>2+</sup> ratio=10)

### 3.3. Effect of H<sub>2</sub>O<sub>2</sub> to Fe (II) Molar Ratio

In Fenton process none of both H<sub>2</sub>O<sub>2</sub> and Fe<sup>2+</sup> has to be observed, so the maximum amount of OH radicals is available for the organic complex oxidation. Thus, two main chemicals such as hydrogen peroxide and iron determine the operation costs and efficacy. To investigate the process efficacy it is so significant that the optional operational H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> molar ratio to be determined. The role of H<sub>2</sub>O<sub>2</sub> and iron in removal of organic complexes by Fenton helps us to find out the dosages of optimal reagent as they are utilized in producing the hydroxyl radicals which are essential in organic materials oxidization.

In order to find out the optimum H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> molar ratio, three disparate H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> molar ratios i e., 2, 4, 5, 6, 8 and 10 were tested. The maximum removal effectiveness of COD and color were 80.25% and 95.55% respectively at a Fe<sup>2+</sup> dosage of 4000 mg L<sup>-1</sup> and H<sub>2</sub>O<sub>2</sub>/Fe<sup>2+</sup> molar ratio of 10.

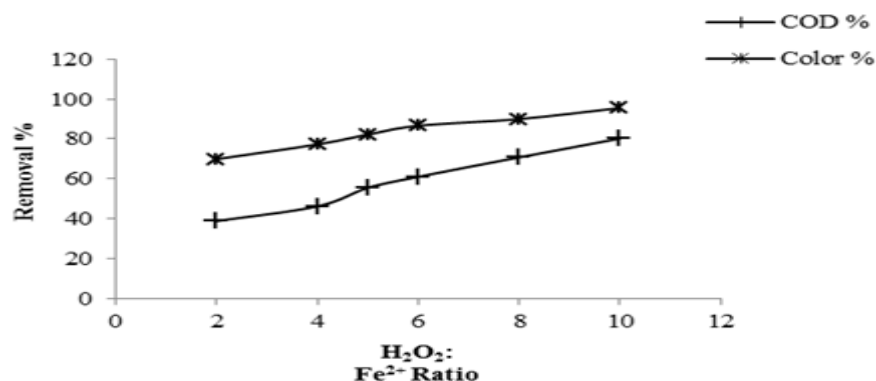


Figure-4. Effect of molar ratio on COD and color removal (pH =3, Time=30 min, Fe<sup>2+</sup>=24000 mg L<sup>-1</sup>, H<sub>2</sub>O<sub>2</sub>= 40000 mg L<sup>-1</sup>)

#### 4. CONCLUSION

Fenton was effectively used to reduce concentrations of organic contaminants and color of landfill leachate wastewater. A batch reactor was used to estimate the effects of the main parameters on Fenton process. It was established that Fenton reagent causes the oxidation of organic substances to be successfully removed. Such oxidation happens so fast (within 30 min) by Fenton's reagent with batch experiments. The organic substances oxidation in the leachate demonstrated that such oxidation depended on pH and was the most effective factor in the pH range of 3. An acceptable  $H_2O_2/Fe(II)$  molar ratio was 10 and dosage increase at an acceptable  $H_2O_2/Fe(II)$  molar ratio leads to an increase in organic removal.

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**Contributors/Acknowledgement:** All authors contributed equally to the conception and design of the study.

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