



## Treatment of Landfill Leachate by Advanced Oxidation Processes

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### Abstract

Organic and inorganic pollutants found in municipal landfill leachate lead to severe problems for the environment when directly discharged to water bodies without treatment. Due to the existence of recalcitrant organics in leachate, advanced oxidation processes (AOP) are mostly applied to biologically treated leachate as a polishing step.

In this study, the effectiveness of Fenton process on leachate treatment was examined. The Fenton process was applied to both young (untreated) and biologically treated leachate provided from Şile Kömürçüoda Landfill in Istanbul. It was treated by using up flow anaerobic sludge blanket (UASB) reactor and membrane bio-reactor (MBR) processes respectively prior to AOP application. In addition, the combination of Ultraviolet (UV)/Fenton and Ultrasound (US)/Fenton processes were applied to biologically treated leachate to improve the efficiency of Fenton process.

The results suggested that, Fenton process is an efficient oxidation method for the treatment of organic matter and color in leachate. It was able to remove 70 % chemical oxygen demand (COD) from the raw leachate, while for biologically treated leachate the COD removal was over 50 %. On the other hand, color removal was observed to be 90 % and 98% in old and young leachate respectively. It was also seen that the additional treatment techniques used in the study did not improve the treatment efficiency of Fenton process.

**Keywords:** Leachate, AOPs, Fenton, UV, US.

## Katı Atık Düzenli Depolama Sahası Sızıntı Suyunun İleri Oksidasyon Yöntemleri ile Arıtımı

### Özet

Katı atık depolama sahası sızıntı suyunda bulunan organik ve inorganik kirlleticiler arıtılmadan direk olarak su kaynaklarına deşarj edildiğinde ciddi çevresel sorunlara yol açmaktadır. Biyolojik arıtmadan geçmiş sızıntı suyunda bulunan inatçı organikler için ileri oksidasyon yöntemleri çoğunlukla cilalama evresi olarak uygulanır.

Bu çalışmada, Fenton prosesinin katı atık depolama sahası sızıntı suyu üzerindeki etkinliği incelenmiştir. Fenton prosesi İstanbul'daki Şile Kömürçüoda katı atık depolama sahasından temin edilen genç (arıtılmamış) ve biyolojik olarak arıtılmış sızıntı suyuna uygulanmıştır. Sızıntı suyu, ileri oksidasyon uygulaması öncesinde sırasıyla anaerobik yukarı akışlı çamur yatağı ve membran biyo-reaktör prosesleri ile arıtılmıştır. Buna ek olarak biyolojik olarak arıtılmış sızıntı suyuna verimi artırabilmek amacı ile ultraviyole/Fenton ve ultrasound/Fenton kombinasyonları da uygulanmıştır.

Çalışmalar sonucunda Fenton prosesinin sızıntı suyunda renk ve organik madde arıtımında etkin bir yöntem olduğu ortaya konmuştur. Ham sızıntı suyunda %70 KOİ giderimi elde edilirken biyolojik olarak arıtılmış sızıntı suyunda bu değer %50'nin üzerindedir. Diğer taraftan renk giderimi ham ve biyolojik olarak arıtılmış sızıntı suyunda sırasıyla %90 ve %98 olarak gözlemlenmiştir. Fenton'a ek olarak uygulanan ultraviyole ışın ve ultrasound yöntemlerinin arıtma veriminde artış yaratmadığı belirlenmiştir.

**AnahtarKelimeler:** Sızıntı suyu, İleri oksidasyon, Fenton, UV, US

## **Introduction**

Landfilling is a common way of disposing solid waste. Leachate produced in landfill requires control and treatment. There is a wide range of organic and inorganic pollutants in municipal landfill leachate, which cause problems for living organisms and environment. Due to the high pollutant loads, leachate treatment has become an important issue and several treatment configurations have been investigated for many years [1,2]. Biological, chemical or physical treatment and advanced technologies such as membrane applications, advanced oxidation techniques are required for the treatment of leachate. AOPs are sufficient treatment methods to convert refractory organics to simpler forms in leachate, so that chemical oxygen demand (COD) and color can be taken under control. In general, the effectiveness of an AOP is proportional with the ability to generate hydroxyl radicals [3].

Fenton is an efficient advanced oxidation process for oxidation of refractory organics in landfill leachate. Fenton reagent, a mixture of Fe salts and H<sub>2</sub>O<sub>2</sub> as shown in equation 1, is an attractive oxidative system owing to the simple production of OH radicals [4].



Especially in biologically pre-treated leachate there are lots of non-biodegradable organic matters. Free hydroxyl radicals formed in Fenton reaction are able to oxidize them and therefore COD of leachate can be treated. Although Fenton is efficient for oxidation, some organic compounds are recalcitrant to Fenton treatment.

Umar et al. (1999), reported that appropriate molar ratio of Fenton reagents and initial pH are the two most important factors to achieve maximum COD removal performance [5]. In Fenton process, researches show that initial pH is effective between 2 and 4.5 [6].

In order to increase OH radicals and treatment efficiency in Fenton process, additional oxidation methods can be used such as UV, US, O<sub>3</sub> and microwave. A combination of hydrogen peroxide and UV radiation with Fe (II), produces more hydroxyl radicals in comparison to the conventional Fenton Method, thus promoting the degradation of organic pollutants [7].

Ultraviolet irradiation promotes degradation of organic compounds in Fenton and photo-Fenton reactions. Several studies have investigated the effects of iron salt dosage, H<sub>2</sub>O<sub>2</sub> dosage, the nature of the iron salt the presence or absence of UV and UV intensity in Fenton-type processes [8].

The higher production of OH radicals due to the combination of oxidant compounds and metallic catalysts in presence of UV radiation and the potential applicability of sunlight as UV light resource are some attractive advantages of this system [9]. US is another process that may enhance the treatment efficiency of Fenton. Ultrasonic waves, when applied to water, causes rapid formation, growth, and violent collapse of cavitation bubbles, resulting in sonochemical effect taking place either due to the pyrolytic decomposition inside the bubbles, or by the reduction and oxidation due to the generation of H and OH radicals [10] Moreover the ultrasonic irradiation is an effective method for the decomposition of ammonia-nitrogen in landfill leachate [11].

The leachate used in this study was pre-treated with up-flow anaerobic sludge blanket reactor and membrane bio-reactor, respectively. Although these techniques provided high treatment efficiency, effluent COD values were still high with respect to discharge regulations. The aim of this study was to determine the effect of Fenton and combination of Fenton with different oxidation methods on biologically pre-treated and raw landfill leachate. In this study, Fenton

process was applied at different concentrations of H<sub>2</sub>O<sub>2</sub> and different H<sub>2</sub>O<sub>2</sub>/Fe ratios to biologically pre-treated leachate. After the determination of optimum concentration and ratio, combination of Ultraviolet (UV)/Fenton, Ultrasound (US)/Fenton were applied to biologically pre-treated leachate to improve the efficiency of Fenton process.

## Material and Method

### Characteristics of Landfill Leachate

Leachate samples in this study were supplied from Şile Kömürçüoda Landfill site which is landed on 233 ha area at Şile and has been in operation till 1995. It is one of the two landfill sites in Istanbul and municipal wastes come to the Landfill from the transfer stations. Landfill is composed of 6 different cells and leachate generated from active cell is collected in lagoon so leachate sample was brought to the laboratory from lagoon periodically at different time intervals, fluctuations were seen in some quality parameters as shown in Table 1.

**Table 1.** Raw leachate characterization

Parameter	Minimum	Maximum	Average
pH	7.5	8.1	7.8
Total Alkalinity (mg/L CaCO <sub>3</sub> )	8,100	11,800	10,581
COD (mg/L)	10,695	37,760	24,040
BOD <sub>5</sub> (mg/L)	4,640	27,465	15,021
BOD <sub>5</sub> /COD	0.43	0.73	0.62
Color (Pt–Co)	15,600	45,000	26,690
Ammonia (mg N/L)	1,830	2,560	2,281

### Experimental Set-up

The biological pre-treatment system of raw leachate was consisted of Up-flow anaerobic sludge blanket (UASB) reactor and Membrane Bio-reactor (MBR) system. The influent of UASB was supplied from landfill leachate and the effluent of UASB was passed through an MBR in order to remove remaining organic content. The MBR effluent was stored at +4°C temperature until used for the study.

According to water pollution control regulations, the COD of 2 hours composited sample should be less than 700 mg/L. As can be seen in Table 2, biologically treated leachate has a higher COD and requires further treatment. Since its BOD<sub>5</sub>/COD ratio is lower than 0.1, which indicates poor biodegradability and hard to remove organic matter by biological means. Fenton, photo-Fenton (Fenton/UV) and Fenton/Ultrasound processes were applied to effluent of MBR in order to determine optimum chemical dosages and conditions.

**Table 2.** Effluent Leachate Characterization of MBR

Parameter	Value
pH	8.4
Total Alkalinity (mg/L CaCO <sub>3</sub> )	2600
COD (mg/L)	1400
BOD <sub>5</sub> (mg/L)	100
BOD/COD	0.07
Color (Pt-Co)	2600
Ammonia (mg N/L)	1400

#### Fenton Process

Fenton experiments were performed at  $20 \pm 3$  °C (room temperature) to eliminate the effect of temperature on the performance of Fenton process. In order to define the optimum dosage of reagents, different  $[\text{H}_2\text{O}_2]/[\text{Fe}^{+2}]$  ratios were applied for several  $\text{H}_2\text{O}_2$  concentrations. Sample volume was 50 ml and pH was adjusted to a desired value ( $\text{pH} = 2.5 \pm 0.05$ ) with concentrated sulfuric acid. After the addition and homogenization of ferrous sulfate, the sample was analyzed for  $\text{Fe}^{+2}$  in order to control  $\text{Fe}^{+2}$  concentrations. With the addition of hydrogen peroxide, the reaction time was started instantaneously. The mixtures were stirred at 200 rpm for 60 min. and at the end of reaction time, it was neutralized to  $\text{pH} 7.5 \pm 0.05$  by adding 10M NaOH solution. The neutralized solution was mixed at 20 rpm for 30 min in order to provide coagulation. After slow mixing; the sample was centrifuged (Hettich 320R) at 9000 rpm for 3 min. Color and COD analyses were performed with the supernatant.

#### Fenton + US

The sample including ferrous sulfate at  $\text{pH} = 2.5 \pm 0.05$  was transferred to a falcon tube. Hydrogen peroxide was instantaneously added and in order to obtain homogeneous mixture, falcon tube was mixed gently and placed into the ultrasound reactor (BANDELIN TK100) with a frequency of 43 kHz and max power of 2x160 W/per. In this study, ultrasound was used instead of rapid mixing and sample was exposed to ultrasonic wave for one hour. After one hour expose to ultrasonic wave, pH was adjusted to  $7.5 \pm 0.05$  by 10 M NaOH. The sample was subjected to stirring at 20 rpm for 30 min and was transferred to falcon tube. After slow mixing; it centrifuged at 9000 rpm during 3 min. The supernatant was analyzed for color and COD.

#### Fenton + UV

Ultraviolet system was composed of an ultraviolet reactor, a sample container, a magnetic stirrer and a recirculation pump. 40-watt low-pressure UV lamp with a wavelength of 254 nm was used to generate UV light. The UV dosage given by the lamp was  $30000 \mu\text{Ws}/\text{cm}^2$ . Lamp and reactor dimensions were 19x450 mm and 80x500 mm respectively. The optimum conditions defined in Fenton process were applied ( $[\text{H}_2\text{O}_2]=900 \text{ mg/L}$ ,  $[\text{H}_2\text{O}_2]/[\text{Fe}]=1$ ). After hydrogen peroxide addition, the sample was stirred to obtain a homogeneous mixture and

ultraviolet lamp was turned on. UV radiation applied to leachate during different time intervals. pH was adjusted to  $7.5 \pm 0.05$  by 10 M NaOH and after neutralization, it was stirred at 20 rpm for 20 min. After slow mixing; it was centrifuged at 9000 rpm for 3 min. Color and COD analyses were performed with the supernatant.

The UV dosage applied to the water is lower than it is given from the lamp depending on the transmittance of sample. Therefore, applied dosage was calculated according to equation 2 [11].

$$D_{UV} = 300 \times \frac{0.01617 \times T (\%) + 0.2071}{Q} \quad (2)$$

Where T (%) is transmittance and Q is the flowrate of leachate into the UV reactor.

### Analytical Methods

The analytical methods used for the characterization of raw leachate and operational parameters of advance oxidation studies were carried out according to Standard Methods [13]. COD was determined by closed reflux, colorimetric method at 620 nm. In this method, potassium dichromate, used as oxidant, was reduced by inorganic specie,  $H_2O_2$ , had reductive ability. The  $H_2O_2$  that was used as chemical coagulant in the Fenton experiment led to interference. The correction of COD data was satisfied by equation 3, 4 [14].

$$COD \text{ (mg/L)} = COD_i - f \times [H_2O_2] \quad (3)$$

$$f = 0.4706 - 4.06 \times 10^{-5} \times [H_2O_2] \quad (4)$$

The concentration of hydrogen peroxide was analyzed using  $H_2O_2$  test strips (Quantofix®). Ferrous iron was determined by 1, 10 - phenanthroline colorimetric method at a wavelength of 455 nm.

### Results and Discussion

Treatment of leachate is very hard due to its components. Advanced oxidation is an effective way of organic treatment due to its high oxidation power. Raw leachate used in this study was a young (fresh) landfill leachate. COD of leachate was approximately 15000 mg/L and  $BOD_5$  was around 10000 mg/L. After biological treatment COD and  $BOD_5$  were decreased to approximately 1500 mg/L and 100 mg/L respectively. Water Pollution Control Regulations in Turkey prohibits the discharge of municipal landfill leachate to receiving body with a COD higher than 700 mg/L and 500 mg/L for 2 and 24 hours composited samples respectively. Due to the fact that, the organic content was removed by Fenton based advanced oxidation techniques.

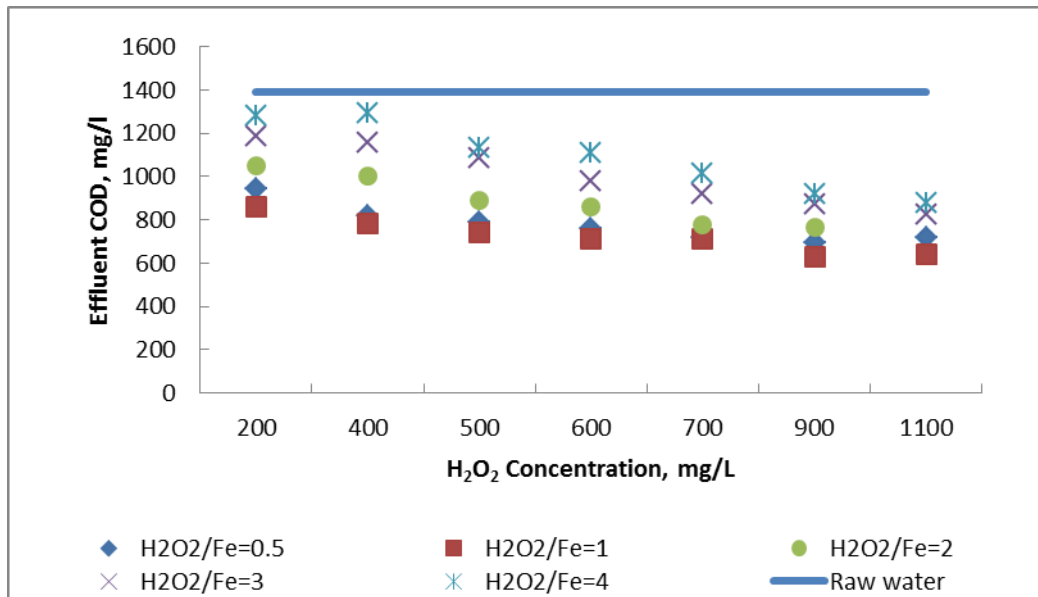
## **Fenton**

Fenton process, which was the first step of this study, was applied to both raw and biologically treated landfill leachate. pH is one of the major factors that limits the performance of Fenton and Fenton related processes. According to Umar et al. (1999) acidic pH conditions is favorable for Fenton process due to the oxidation reactions [5]. Under alkaline conditions, H<sub>2</sub>O<sub>2</sub> does not produce any hydroxyl radicals [15]. Optimum pH condition was reported as 2.5 [16, 17], although in some studies maximum removal efficiency is obtained at pH a bit higher than 2.5 [18].

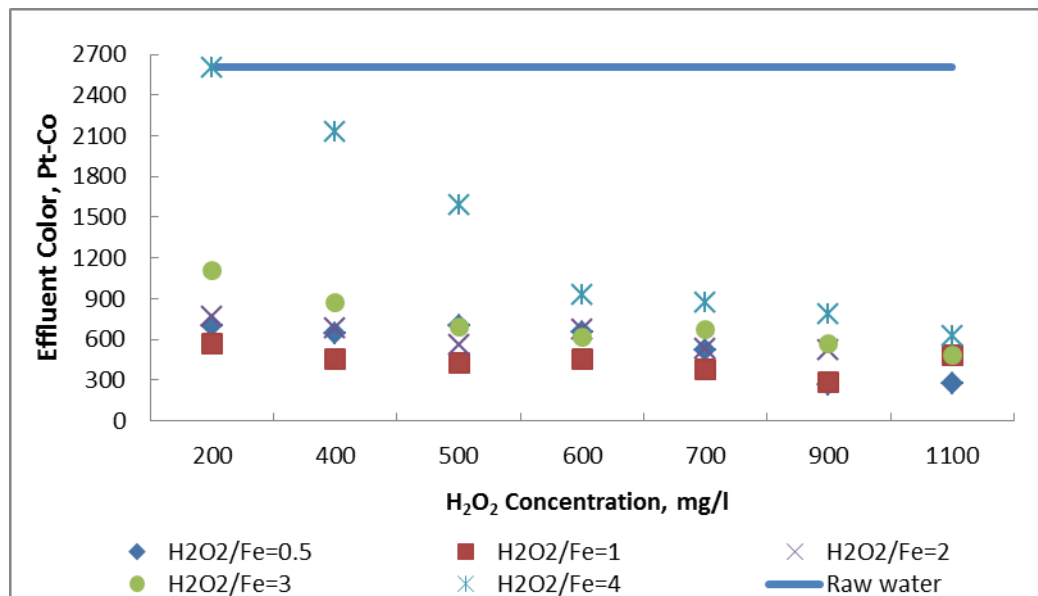
In the Fenton and Fenton related processes, the [H<sub>2</sub>O<sub>2</sub>] / [Fe<sup>2+</sup>] ratio is very important in terms of COD and color removal efficiency of the process. When the dosage of H<sub>2</sub>O<sub>2</sub> is decreased, enough hydroxyl radicals are not generated, so degradation is increased with increasing H<sub>2</sub>O<sub>2</sub> dosages [19]. An optimum [H<sub>2</sub>O<sub>2</sub>] / [Fe<sup>2+</sup>] ratio is necessarily important to avoid scavenging effects and final sludge production. [H<sub>2</sub>O<sub>2</sub>] / [Fe<sup>2+</sup>] ratio should be kept as low as possible to avoid recombination of hydroxyl radicals and final sludge volume [20].

Figure 1 demonstrates the treatment performance of Fenton process conducted for different [H<sub>2</sub>O<sub>2</sub>] / [Fe<sup>2+</sup>] ratios. H<sub>2</sub>O<sub>2</sub> concentrations were changed between 200 mg/L and 1100 mg/L. COD concentration decreased from 1400 mg/L to 630 mg/L with a treatment efficiency of 55% for 900 mg/L H<sub>2</sub>O<sub>2</sub> which provided the maximum efficiency for [H<sub>2</sub>O<sub>2</sub>] / [Fe<sup>2+</sup>] = 1. Zhang et al. (2005), reported that the COD removal increased linearly with the increase of [H<sub>2</sub>O<sub>2</sub>] / [Fe<sup>2+</sup>] ratio by 1.5 [16]. However, Cortez et. al. (2010) determined optimum [H<sub>2</sub>O<sub>2</sub>]/[Fe] ratio as 3 for Fenton oxidation process [21]. Maximum 90% decolorization was obtained for the same dosage and sample color decreased from 2600 Pt-Co to 260 Pt-Co.

As shown in the Figure 2, Fenton process could achieve 30–55% of COD and 70-90% color removal respectively. Zhang et al. (2005), achieved 63.6% efficiency for COD removal in leachate [16] also Roddy and Choi (1999) showed that 85% COD removal efficiency can be achieved under optimum conditions [22]. Finally, Xu et al. (2004), reported that advanced oxidation is an effective alternative while dealing with the refractory organic compounds in old or biologically treated landfill leachate [23]. COD removal efficiency changes due to the leachate characterization and also pre-treatment types. In this study, an efficient biological treatment was applied prior to oxidation studies, so in Fenton process remaining COD was resistant to OH radicals. Although the COD removal efficiency was not as high as the previous studies, effluent leachate satisfied the discharge limits.



(a)



(b)

Figure 1. Fenton application to biologically pre-treated leachate for the determination of optimum dosage for a) COD removal, b) Color removal

Fenton is an efficient advanced oxidation process for oxidation of refractory organics in landfill leachate. Especially in biologically pre-treated leachate there are lots of non-biodegradable portion of the organic matters. Free hydroxyl radicals are combined with them and precipitate so COD of leachate will decrease.

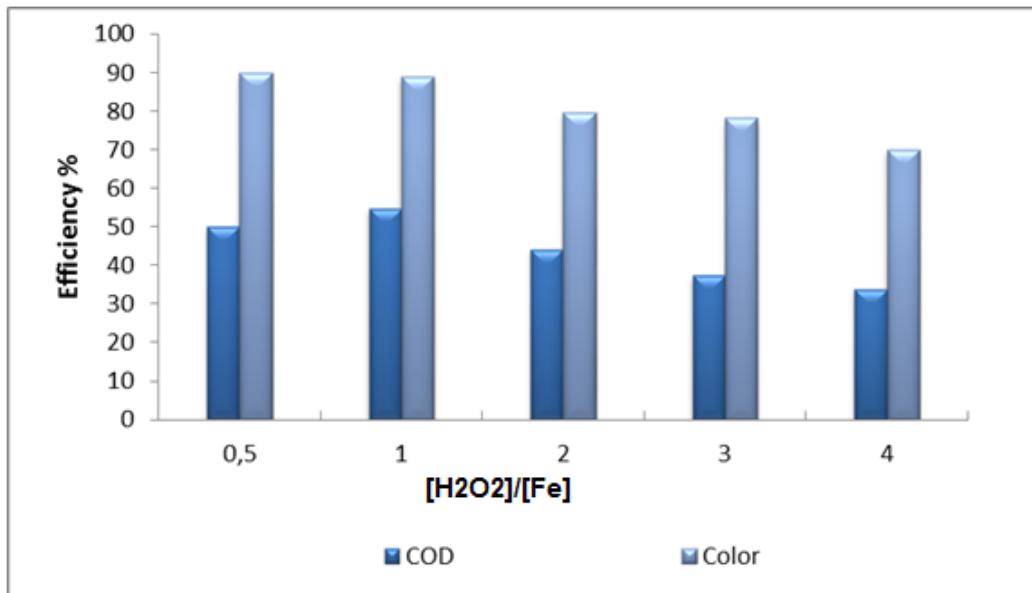


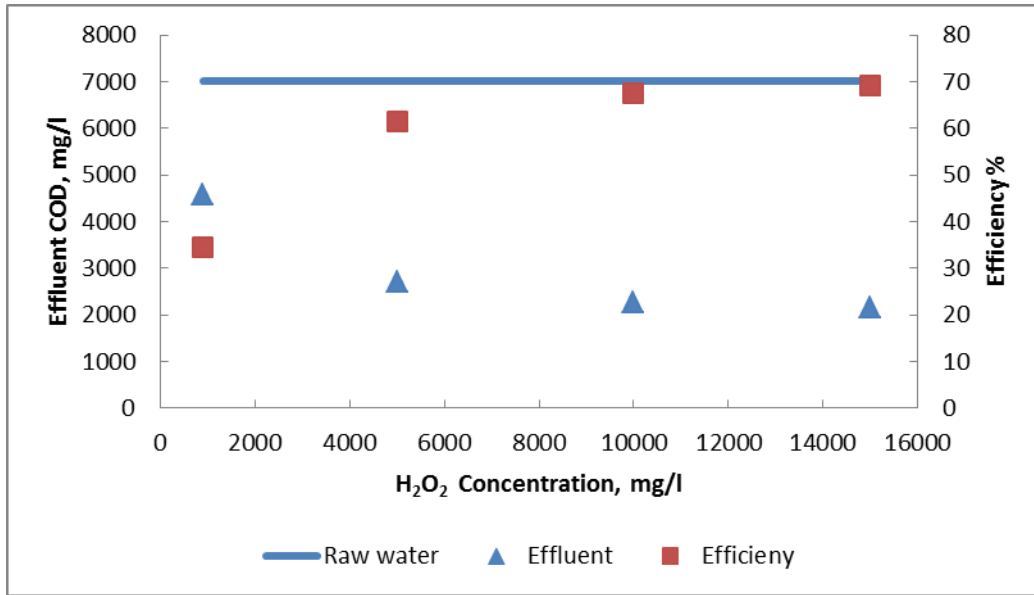
Figure 2. Comparison of COD and color removal efficiencies for Fenton

In this study Fenton process was also studied with raw (untreated) leachate. H<sub>2</sub>O<sub>2</sub> concentrations were changed between 900 mg/L and 15000 mg/L and [H<sub>2</sub>O<sub>2</sub>]/[Fe]=1 (Figure 3). COD concentration was decreased from 7000 mg COD/L to 2150 mg COD/L and the maximum efficiency was observed at the dosage of 15000 mg H<sub>2</sub>O<sub>2</sub>/L as 70%. Color removal efficiency was found to be 98 % so that Fenton process is also effective for raw leachate. However, in raw leachate it requires high amount of chemicals which increase the cost of treatment and sludge production. Moreover, the effluent COD values could not be decreased below stipulated limits in this case. It was clearly seen that application of Fenton to the effluent of biological treatment is more efficient and feasible than raw leachate applications.

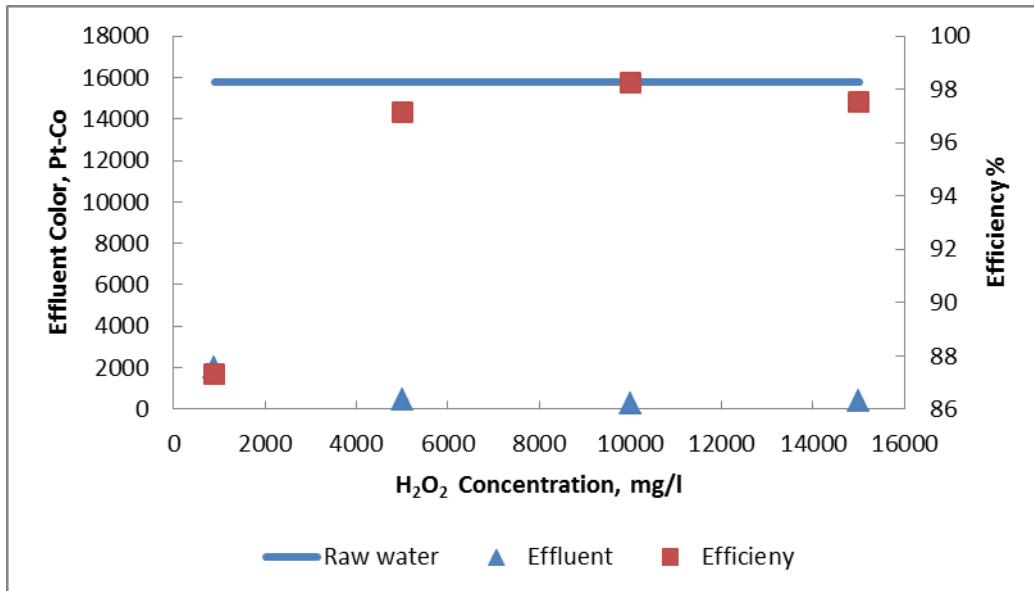
### Fenton/Ultrasound

Fenton-ultrasound combination is a new approach in advanced oxidation and there are limited studies in the literature. Figure 4a and 4b demonstrates the treatment performance of Fenton/US process conducted with the optimum [H<sub>2</sub>O<sub>2</sub>] / [Fe<sup>2+</sup>] ratio which was found in Fenton applications. H<sub>2</sub>O<sub>2</sub> concentrations were changed between 200 mg/L and 1100 mg/L in order to see the effects of H<sub>2</sub>O<sub>2</sub> concentration on the performance of treatment. COD concentration decreased from 1400 mg/L to 735 mg/L with a treatment efficiency of 46% for 900 mg/L H<sub>2</sub>O<sub>2</sub> which provided the maximum efficiency for [H<sub>2</sub>O<sub>2</sub>] / [Fe<sup>2+</sup>] = 1. Color was decreased from 2600 Pt-Co to 370 Pt-Co. For this ratio the maximum efficiency was observed to be 86% at the same dosage of H<sub>2</sub>O<sub>2</sub>.





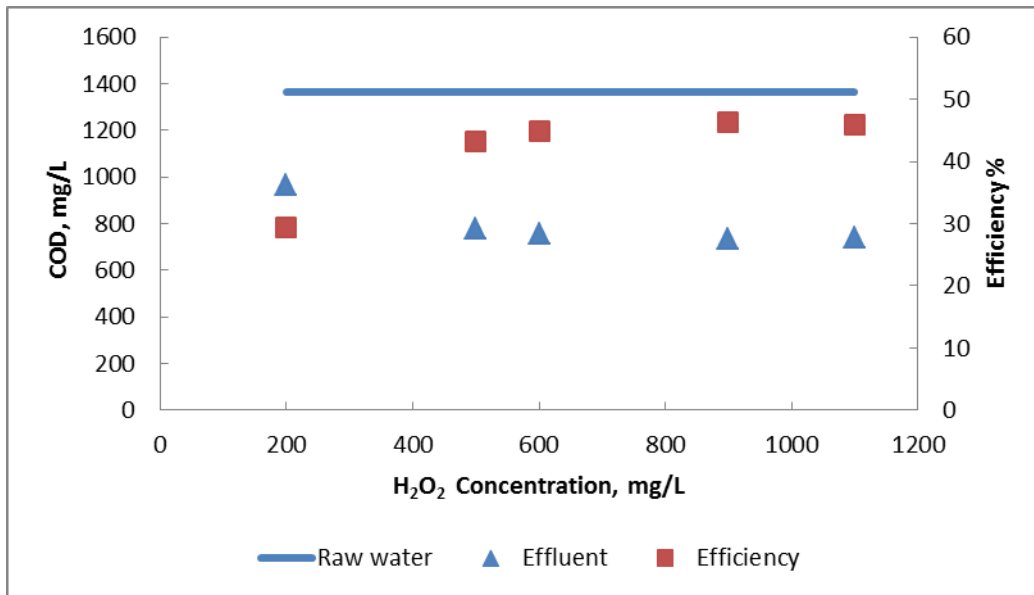
(a)



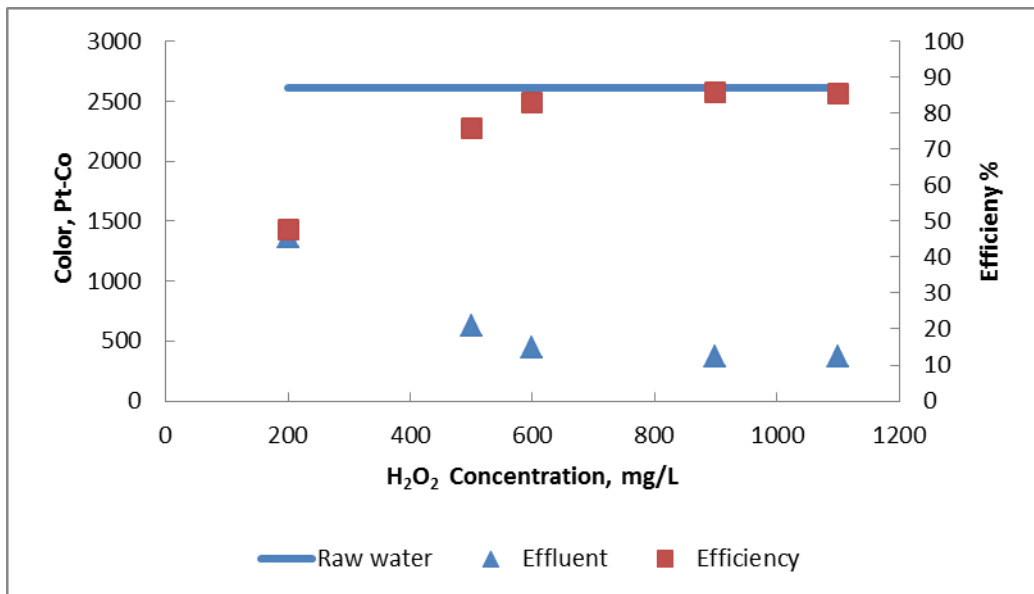
(b)

Figure 3. Fenton application to raw leachate for the determination of optimum dosage for a) COD removal, b) Color removal ( $[H_2O_2]/[Fe]=1$ )

In the previous Fenton/US studies, US applications mostly increased the efficiency of Fenton process in terms of COD removal. Méndez et al. (2009) reported that the presence of ultrasonic irradiation slightly improved the iron catalytic activity for sonophoto-Fenton process [24]. In Fenton/US process, the results showed that organics remained after biological treatment are refractory to OH radicals, thus, ultrasound did not improve the efficiency of Fenton process in pre-treated leachate.



(a)

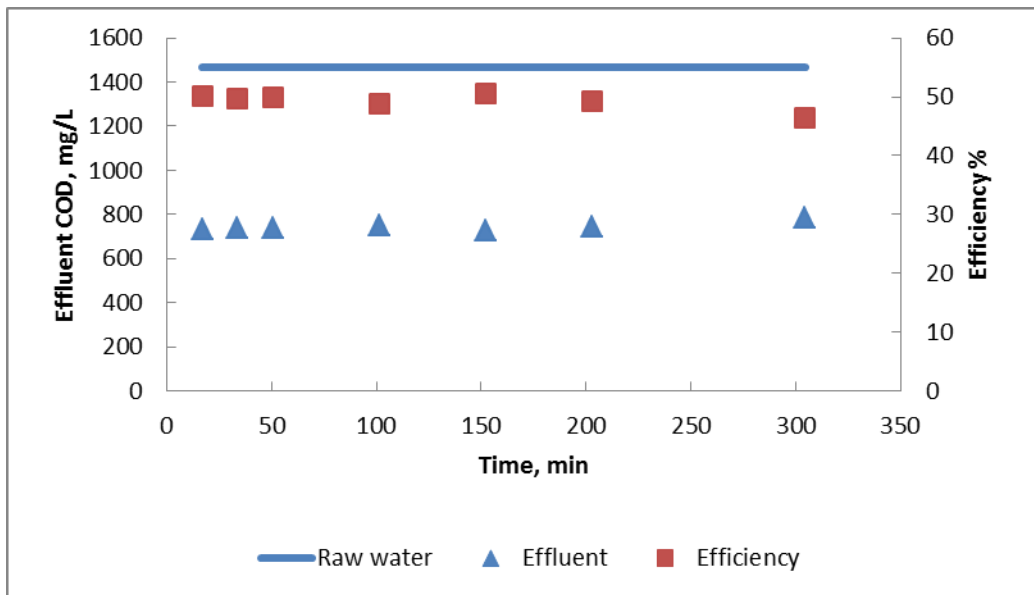


(b)

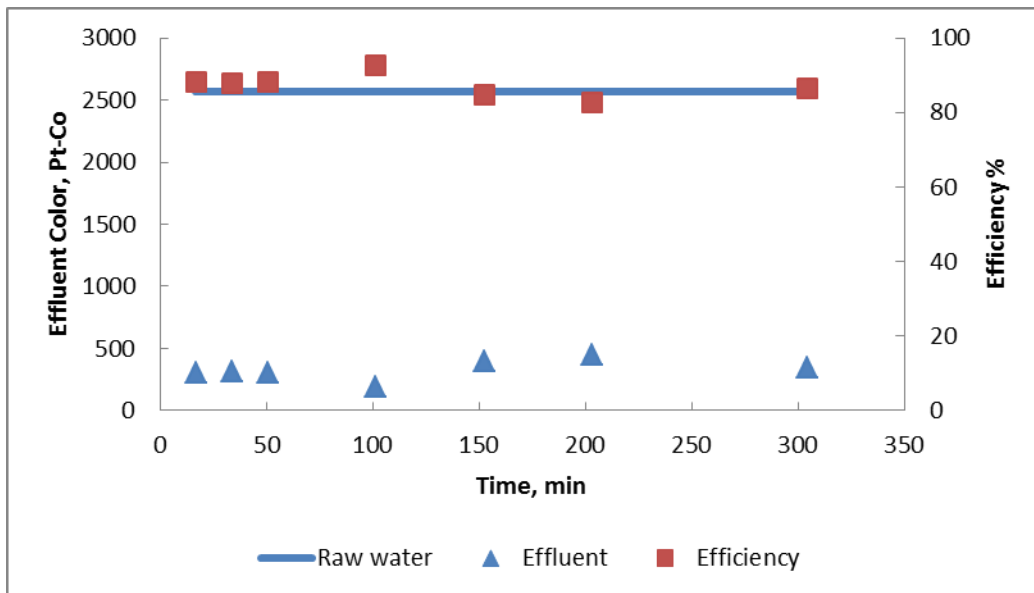
Figure 4. Fenton+US application to biologically pre-treated leachate for the determination of optimum dosage for a) COD removal, b) Color removal ( $[H_2O_2]/[Fe]=1$ )

### Fenton/ Ultraviolet (Photo-Fenton)

In Fenton/UV process, UV irradiation was applied besides Fenton process in order to increase the formation of OH radicals for the advanced oxidation of pre-treated landfill leachate. It was applied during 300 min, however UV application did not improve the treatment performance. Both COD and color remained almost constant during whole process. The highest COD removal efficiency of photo-Fenton application was observed to be 50.5% as shown in Figure 5a.



(a)



(b)

Figure 5. Fenton+UV application to biologically pre-treated leachate for the determination of optimum dosage for a) COD removal, b) Color removal ( $[H_2O_2]/[Fe]=1$ )

Similar organic removal performances observed both Fenton and photo-Fenton processes. It was seen that, UV irradiation did not improve the efficiency of Fenton. It was thought that the reason of having no efficiency with UV was; during Fenton process turbidity occurred and UV light could not affect as it should be [20]. The other reason could be that the leachate was pre-treated and remaining organics were very hard to degrade with the given UV dosage. According to Shu et al. (2006), decolorization and COD removal is decreasing with the strength of leachate [25]. COD removal efficiencies are 59.2%, 61.5% and 88.1% with given

leachate strengths of 100%, 50% and 20%, respectively. Primo et al. (2008) reported that Fenton/UV process is the most efficient process when compared to other advanced oxidation methods [26]. However; Hermosilla et al.(2009), reported no efficient increase in both COD and TOC removal of when UV applied to raw landfill leachate [27].

The experiments showed that the methods combined with Fenton did not provide excess efficiency. According to the results that observed; it is obvious that Fenton is the most effective and applicable oxidation method for treatment of landfill leachate.

## **Conclusion**

For the further treatment of organics remained after biological treatments Fenton, Fenton/UV and Fenton/US processes were used. Among all treatment options, Fenton process is the most suitable technique for complete mineralization of organic matters in leachate. For the optimum conditions, by the application of Fenton process 55% COD and 89% color removal was provided. However, Fenton/UV combination provided 50% COD removal in 330 min UV application and Fenton/US combination could only remove 46% COD. Elevated COD removal percentages (70%) were obtained in raw leachate applications due to high readily biodegradable organics found in raw leachate. Nevertheless, the effluent COD could not be decreased below 2000 mg/L. Moreover, in raw leachate applications, Fenton required very high chemical dosages because of its high alkalinity and scavenger compounds.

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