

Landfill Leachate Treatment through Electro-Fenton Oxidation

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ABSTRACT: Advanced Oxidation Processes (AOPs) have been employed to degrade biorefractory organic matters. This study investigates the combination of classical Fenton reaction with electrochemical oxidation, the electro-Fenton process, for the treatment of semi aerobic landfill leachate, collected from Pulau Burung Landfill Site (PBLs), Penang, Malaysia. The investigation has been carried out in batch reactors with aluminum electrodes to establish the optimal treatment conditions. The effects of applied current, pH, reaction time, electrodes separation distance, $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio, and H_2O_2 and Fe^{2+} concentrations, significant process parameters by themselves, have also been investigated. According to the obtained results, electro-Fenton process is very efficient for the treatment of landfill leachate. Optimum oxidation efficiency has been achieved when neither H_2O_2 nor Fe^{2+} are overdosed, so that the maximum amount of $\text{OH}\cdot$ radicals is available for the oxidation of organic compounds. The highest COD and color removals have been 92% and 93%, respectively; obtained at initial pH=3, $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio=1, applied current= 2A, treatment duration= 30 min, and electrodes separation distance= 3 cm. The current efficiency declines from 94% to 38% when the current rises from 0.5A to 2A.

Keywords: Landfill, wastewater, treatment, AOP.

INTRODUCTION

Landfill leachate is recognized as a high-strength wastewater that is very difficult to deal with, because, along with heavy metals, it contains organic substances, resistant to biological degradation. Advanced Oxidation Processes (AOPs) have received increased interest in the treatment of municipal landfill leachate, containing non-biodegradable organics.

AOPs are essentially based on the generation of highly reactive radical species,

specifically the hydroxyl radical ($\text{OH}\cdot$) to react with organic compounds (Brillas et al., 2009). Oxidation via H_2O_2 alone has been found ineffective for high concentrations of certain refractory contaminants, due to low rates of reaction at reasonable H_2O_2 concentrations (Mohajeri et al., 2011). Transition metal salts (e.g. iron salts), ozone, and UV-light can activate H_2O_2 to form hydroxyl radicals (Neyens and Baeyens, 2003).

In recent years there has been an increased focus on the use of electrochemical methods, which can produce oxidizing

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agents by different ways (Montanaro and Petrucci, 2009; Panizza et al., 2010). Anglada et al. (2009) and Panizza et al. (2010) employed electrochemical method to decompose refractory organic and inorganic components in the leachate. Both studies found efficient removal of chemical oxygen demand (COD) in leachate.

The Fenton process has attracted great interest in terms of its high capacity to generate hydroxyl radicals through decomposition of H₂O₂ by Fe²⁺ in acidic conditions (De Morais and Zamora, 2005, Brillas et al., 2009; Li et al., 2010). The efficiency of this process can be enhanced by coupling with electricity (electro-Fenton), the basis of this paper. Electro-Fenton process includes the combined advantages of electrochemical and Fenton treatment methods, each a powerful treatment choice. Under electrically-assisted Fenton reaction, more OH• radicals are produced and the oxidation of organics to CO₂ can be enhanced in the same period (Liu et al., 2007a).

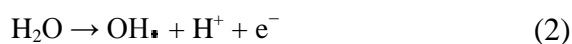
During Fenton reaction, hydrogen peroxide is catalyzed with ferrous ions to produce hydroxyl radicals. In acidic medium, the oxidizing power of H₂O₂ can be strongly enhanced, using electro-Fenton method, where a small quantity of Fe²⁺ is added as catalyst to the contaminated solution to generate OH• and Fe³⁺ from Fenton's reaction.

The use of Fenton's reagent as an oxidant for wastewater treatment is attractive due to the facts that (1) iron is a highly abundant and non-toxic element, and (2) hydrogen peroxide is an environmentally-benign oxidant, easy to handle (Zazo et al., 2005).

Electro-Fenton can proceed by the following chain reactions (Guinea et al., 2008; Lee and Shoda, 2008; Liu et al., 2007b; Mohajeri et al., 2010a, Zhou et al., 2017; Ding et al., 2018):



Hydroxyl radicals are also generated at the surface of a high-oxygen overvoltage anode from water oxidation:



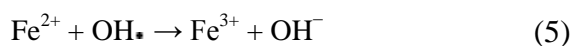
Equation (1) is propagated from ferrous ion regeneration mainly through reduction of the produced ferric species with hydrogen peroxide:



Also the produced ferric ion from Equation (1) can be reduced to ferrous ion via electrochemical regeneration of Fe²⁺ ions on the cathode surface:



In addition, ferrous ions can also be rapidly destroyed by hydroxyl radicals:



The produced hydroxyl radicals would degrade organic matter in wastewaters:



The main objective of this investigation is to explore the feasibility of using electro-Fenton oxidation for the treatment of a mature landfill leachate. The effect of important process parameters, namely, reaction time, pH, electrodes separation distance, applied current, dosages of Fenton's reagent (H₂O₂ and Fe²⁺), and H₂O₂/Fe²⁺ molar ratio on the efficiency of COD and color removal from landfill leachate was determined. Furthermore, current efficiency and energy consumption were also calculated.

MATERIAL AND METHODS

Stabilized landfill leachate samples were collected from the Pulau Burung Landfill Site (PBLs) in Nibong Tebal, Penang, Malaysia. Table 1 shows the leachate characteristics. Collected samples were immediately moved to a cool room and stored at 4°C.

Table 1. Characteristics of leachate, collected from PBLs

Parameters	Range	Average	StD.*
pH	8.3-8.8	8.5	0.19
COD (mg/L)	2380-2480	2450	40.62
BOD ₅	210-270	230	14.33
BOD ₅ /COD	0.09-0.11	0.10	0.01
Color (PtCo.)	3020-3150	3100	48.48
Turbidity (FAU)	220-255	240	13.69
TSS (mg/L)	120-135	130	6.12
Temperature (°C)	27-29	28	0.84

*StD. = Standard Deviation

The characteristics of landfill leachate can vary from time to time, due to environmental conditions (i.e., waste composition, soil properties, age of the waste, seasonal weather variations, etc.) as well as landfill chemical/biological activities. The PBLs leachate characteristics indicated that most of the contaminant levels were relatively high, capable of negatively affecting the environment. As can be seen in Table 1, the high COD (2380–2480 mg/L) and low BOD concentrations (210–270 mg/L) resulted in a very low BOD₅/COD average ratio of about 0.10, which implies that this leachate is highly stabilized and not amenable to biological treatment. It also showed that the color intensity was very high at 3020-3150 Pt-Co.

Experiments on electro-Fenton oxidation of landfill leachate were conducted at laboratory scale in batch system. A direct current (DC) power supply (DAZHENG, PS-305D, 30V, 3A) was employed to provide the desired current. A pair of anodic and cathodic aluminum electrodes, 3 cm by 5 cm in size (active electrode area dipped in leachate) was used. For each run, 500 ml of leachate was placed in an electrolytic cell and desired amounts of iron (Fe²⁺) and hydrogen peroxide (H₂O₂) were added before the electrical current was turned on. All tests were conducted at room temperature (28±2°C) and atmospheric pressure. Initial leachate pH was adjusted to the desired values with concentrated sulfuric acid or sodium hydroxide before adding Fenton reagents. The leachate was stirred with a

magnetic stirrer throughout the reaction duration. Thereafter, the samples were allowed to stand for 30 min (to allow solids sedimentation) and the supernatant was then taken for water quality measurements.

Reagent grade sulfuric acid and sodium hydroxide (Merck, Darmstadt, Germany) were used for pH adjustments. For each run, pre-decided amounts of ferrous sulfate heptahydrate (FeSO₄.7H₂O) and hydrogen peroxide 31% (Merck, Darmstadt, Germany) were used to trigger the electro-Fenton reactions. A HACH DR2010 spectrophotometer was employed to determine COD and color (at 465 nm wavelength) and a METTLER-TOLEDO to measure the pH. Prior to these measurements, the pH meter was calibrated with standard buffers (pH = 4.0, 7.0, and 10.0) at room temperature (30°C). Sampling, sample custody, and chemical analysis took place in accordance with the Standard Methods for Examination of Water and Wastewater (APHA, 2017), followed by Quality Assurance/Quality Control (QA/QC), by means of the same procedure. All samples were in triplicate or more. Laboratory and instrument blanks were tested. Relative standard deviation (RSD) was 2.33 (N=7.) A Certified Reference Material (CRM) was used (Sigma-Aldrich, St. Louis, Mo.). The CRM was produced and certified in accordance with ISO Guide 34:2009 and ISO/IEC 17025:2005. Figure 1 illustrates the flowcharts of the experiments.

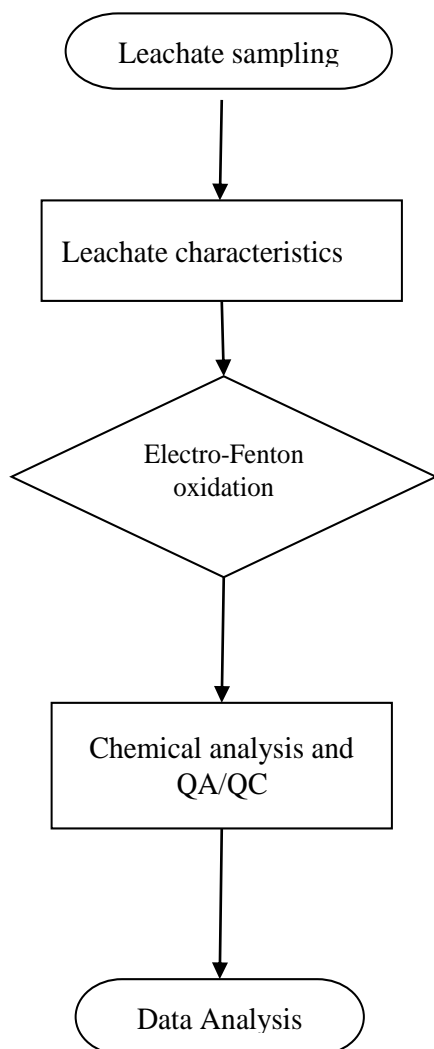


Fig. 1. Flowchart of the experiments

RESULTS AND DISCUSSION

The impact of Reaction Time (RT) on electro-Fenton process got tested in order to determine the duration, required for contaminant destruction. In this study, the efficacy of electro-Fenton process was evaluated in terms of COD and color removal, with the RT varying between 10 and 120 min. The initial pH, H_2O_2 dosage, Fe^{2+} dosage, and applied current were kept constant at 3, 0.01 mol/L, 0.01 mol/L ($\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio = 1), and 1 A, respectively. The results (Fig. 2)

demonstrated that organic materials were rapidly degraded by electro-Fenton process. The removal of COD and color reached 76% and 84%, respectively, in 30 min and after that the removal efficiencies did not change considerably. Thus, the optimum reaction time was adopted as 30 min.

The pH is an important parameter in Fenton process, as the solution's pH controls the production of the hydroxyl radical and the concentration of ferrous ions. Acidic conditions have been reported to be preferable for $\text{OH}\cdot$ generation (Altin, 2008; Swaminathan et al., 2003). Here, several tests were carried out at different pH values within the range of 2-6 with Fig. 3 showing the effect of pH on the degradation and decolorization of leachate.

The highest electro-Fenton activity was obtained at pH = 3, where the COD and color removal percentages were nearly 76% and 84% in 30 min. When pH increased, the iron ions (especially Fe^{3+}) precipitated in the form of $\text{Fe}(\text{OH})_3$. Therefore, the amount of catalyst of Fenton's reaction decreased. Besides, hydrogen peroxide was unstable at high pH rates and might decompose to water and oxygen, losing its oxidation ability (Eren and Acar, 2006; Mohajeri et al., 2010b).

It was also observed that at pH below 3, the degradation efficiency decreased, perhaps due to scavenging of hydroxyl radicals by H^+ ions. At lower pH rates, the formation of $[\text{Fe}(\text{H}_2\text{O})]^{2+}$ occurred, which reacted more slowly with hydrogen peroxide and, consequently, produced less amount of reactive hydroxyl radicals, thereby reducing the degradation efficiency (Bautista et al., 2007; Iglesias et al., 2015). All subsequent experiments were performed at an initial pH value of 3.

Fig. 4 shows the effect of separation distance between the electrodes on COD and color removal efficiencies.

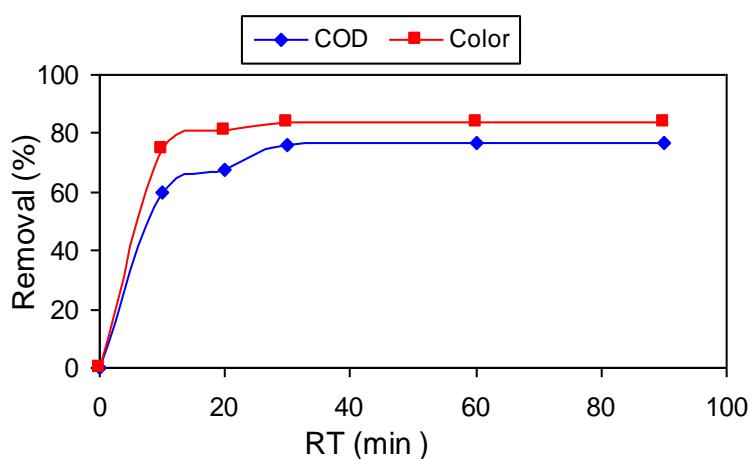


Fig. 2. Impact of reaction time (RT) on COD and color removal efficiencies. ($\text{H}_2\text{O}_2 = 0.01 \text{ mol/L}$, $\text{Fe}^{2+} = 0.01 \text{ mol/L}$, $\text{pH} = 3$, Applied current = 1 A)

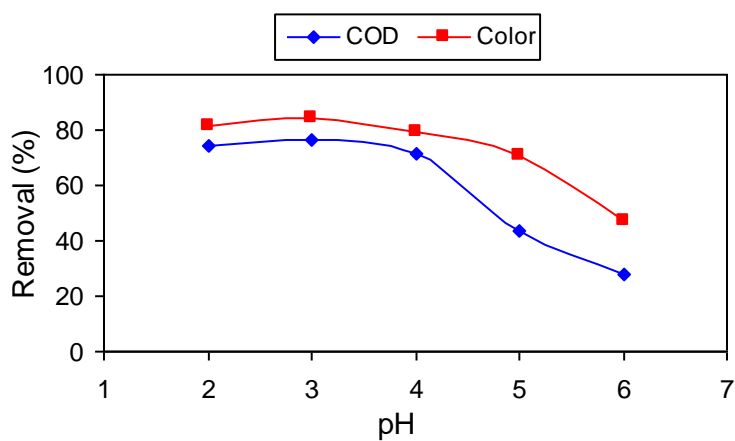


Fig. 3. Effect of pH on COD and color removal efficiencies ($\text{H}_2\text{O}_2 = 0.01 \text{ mol/L}$, $\text{Fe}^{2+} = 0.01 \text{ mol/L}$, applied current = 1 A, RT = 30 min)

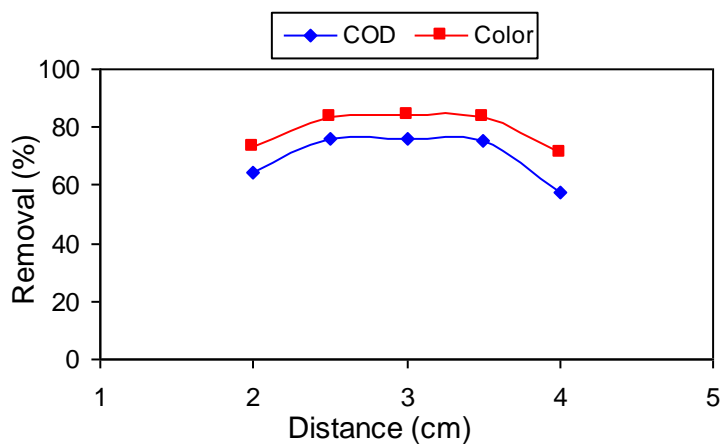


Fig. 4. Effect of distance between the electrodes on COD and color removal efficiencies ($\text{H}_2\text{O}_2 = 0.01 \text{ mol/L}$, $\text{Fe}^{2+} = 0.01 \text{ mol/L}$, applied current = 1 A, RT = 30 min, $\text{pH} = 3$)

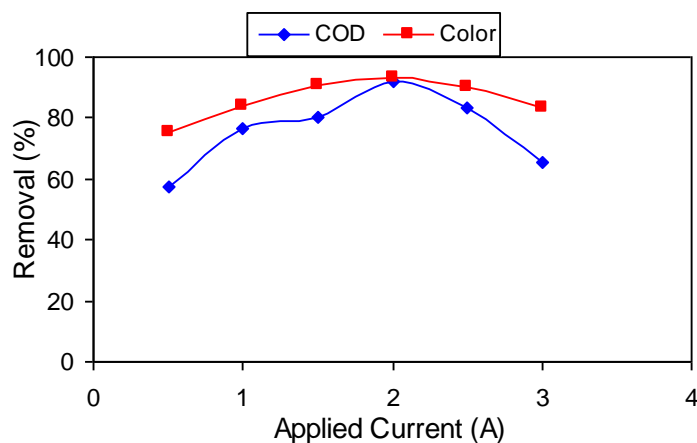


Fig. 5. Effect of applied current on COD and color removal efficiencies ($\text{H}_2\text{O}_2 = 0.01 \text{ mol/L}$, $\text{Fe}^{2+} = 0.01 \text{ mol/L}$, $\text{pH} = 3$, $\text{RT} = 30 \text{ min}$)

As can be seen, COD and color removal efficiencies were fairly constant at 76% and 84% respectively, when the electrodes separation distance was between 2.5 cm and 3.5 cm. Lower efficiencies of COD and color removal were observed outside this range; the drop being higher at increased pH. A separation distance of 3 cm was thus, adopted as the optimum.

In electro-Fenton process, ferric ions can be reduced to ferrous ions at the cathode in accordance with Equation (4). This would encourage Fenton chain reactions. When the electrodes are placed too far apart, mass transfer of ferric ion to the cathode surface can be limited and result in decreased ferrous ion regeneration. Therefore, hydroxyl radicals could not be produced efficiently from ferrous ion according to Equation (1). Also, increasing the distance between the electrodes, gives rise to energy expenses of the method significantly. On the other hand, when the electrodes are placed too close, ferrous ions can be easily oxidized to ferric ions at the anode, according to the following reaction, and inhibit the efficacy of Fenton chain reactions:

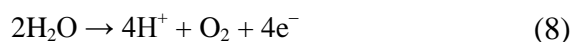


A series of experiments was carried out to test the influence of applied current on

the efficiency of the electro-Fenton process in leachate treatment.

As can be seen in Fig. 5, COD and color removal efficiencies increased when the current was raised from 0.5 to 2.0 A, representing an improvement in mineralization. This trend can be attributed to greater production of $\text{OH}\cdot$ at the surface of the anode and also higher electro-regeneration of ferrous ions from ferric ions (Equation 4) at high applied current, which increased the efficiency of Fenton chain reactions. The continuous electrical conversion of Fe^{3+} into Fe^{2+} is a great advantage, compared to chemical Fenton systems.

On the other hand, further increase of electrical current caused lower COD and color removal. The COD and color removal efficiencies were 65.7% and 83.5% at 3.0 A, respectively, compared with 92% and 93% at 2 A. At higher current applied, the competitive electrode reactions such as the discharge of oxygen at the anode via Equation (8) and the evolution hydrogen at the cathode via Equation (9) would occur (Körbahti and Tanyolaç, 2008):



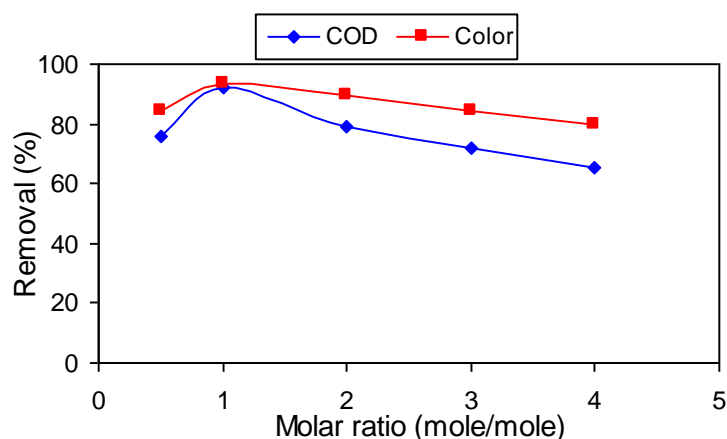


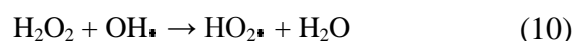
Fig. 6. Effect of molar ratio on the COD and color removal efficiencies ($\text{Fe}^{2+} = 0.01 \text{ mol/L}$, $\text{pH} = 3$, applied current = 2 A, RT = 30 min)

These can inhibit the main reactions such as Equations (2) and (4). When a low current was applied ($I = 0.5 \text{ A}$) an inhibition effect of the degradation rate was observed; presumably because of the low concentration of oxidants produced. Thus, current density plays an important role in degradation and decolorization of leachate.

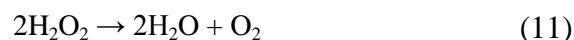
In Fenton process, iron and hydrogen peroxide are the two major chemicals that determine operational costs as well as treatment efficiency. In order to maximize the effectiveness of the process, it is highly important to determine the optimal operational $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio. In order to find the optimum $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio (mol/mol), COD and color removals were plotted against different $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ ratios tested for leachate treatment (Fig. 6).

The $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio varied from 0.5 to 4 at a constant Fe^{2+} dose of 0.01 mol/L, with the maximum removals of COD and color being 92% and 93%, respectively at an $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio of 1.

An increase in $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio beyond 1 decreased the removal efficiency. This may be due to the fact that Fenton's reaction mechanisms would change and some side reactions would occur. It seems that excessive hydrogen peroxide has a scavenging effect on hydroxyl radicals (Equation 10) (Deng, 2007):



This reaction leads to the generation of hydroperoxyl radical, a species with much weaker oxidizing power, compared to hydroxyl radical (Lee and Shoda, 2008; Ting, 2009). Also an excess amount of hydrogen peroxide can cause the auto decomposition of H_2O_2 to oxygen and water, and the recombination of $\text{OH}\cdot$ radicals, thereby decreasing the concentration of hydroxyl radicals and reducing pollutant elimination efficiency (Equations 11 and 12):

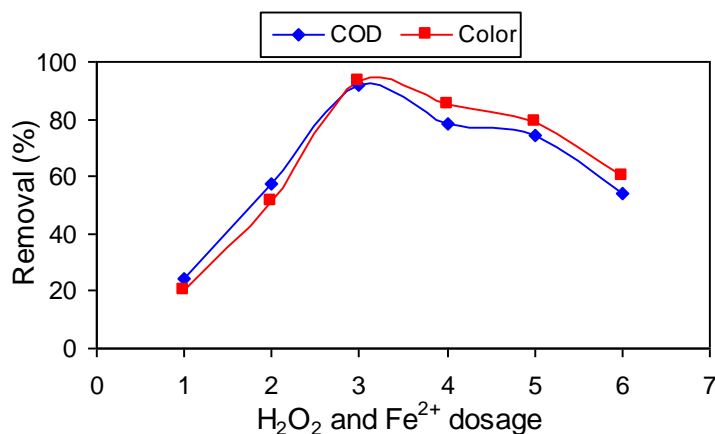


This phenomenon was also reported by Lodha and Chaudhari (2007), Bautista et al. (2007), and Ay et al. (2008). On the other hand, when the molar ratio was low (below 1 in this case), COD and color removals were decreased because of scavenging effect of excess Fe^{2+} (Equation 5). There was a competition between Fe^{2+} and organic compounds for hydroxyl radicals, resulting in reduced COD and color removal efficiencies (Zhang et al., 2005). In addition, the Fe^{3+} formed can react with H_2O_2 (Equation 3) to generate Fe^{2+} and hydroperoxyl radicals ($\text{HO}_2\cdot$) in solution. (Meric et al., 2004)

The discrepancies in optimal ratios of H₂O₂ to Fe²⁺ reported in literature are attributed to variations in landfill leachate characteristics such as landfill age, nature of waste, and depth of fills.

The amount of Fenton's reagent, necessary for an effective treatment, must be determined. Maintaining a constant H₂O₂/Fe²⁺ ratio, the steady state

concentration of hydroxyl radicals depends on the absolute amounts of H₂O₂ and Fe²⁺. Therefore, in order to assess the relationship between the treatment efficiencies and such amounts, a set of experiments was carried out by fixing the H₂O₂/Fe²⁺ molar ratio at 1 and treating the leachate with varying amounts of both reagents.



Case	H ₂ O ₂ (mol/L)	Fe ²⁺ (mol/L)
1	0.0025	0.0025
2	0.005	0.005
3	0.01	0.01
4	0.02	0.02
5	0.03	0.03
6	0.04	0.04

Fig. 7. Effect of varying dosages of H₂O₂ and Fe²⁺, at constant molar ratio (H₂O₂/Fe²⁺ = 1), on COD and color removal (pH = 3, RT = 30 min, applied current I = 2 A).

As can be seen in Fig.7, the COD and color removal efficiencies increased with increasing Fenton reagent concentration up to the optimum dosage (H₂O₂ = Fe²⁺ = 0.01 mol/L). Thereafter, COD and color removals started to decrease. This is because the ultimate products of oxidation reactions were mainly composed of short chain organic acids that were difficult to be oxidized further (Lopez et al., 2004). The reduction in COD and color removal efficiencies beyond a critical concentration of Fenton reagents can be explained considering that the hydroxyl radical may be scavenged by the reaction with excess Fe²⁺ and H₂O₂ (Equations 5 and 10).

The results demonstrated that optimum oxidation efficiency was achieved when neither H₂O₂ nor Fe²⁺ was overdosed, so that the maximum amount of OH• radicals belonged to the oxidation of organic compounds. The highest COD and color

removals were 92% and 93% at 340 mg/L H₂O₂ and 560 mg/L Fe²⁺ respectively. Any further addition of hydrogen peroxide and ferrous ions only caused a progressive reduction in the degree of oxidation of the organic matter.

Current efficiency was used to indicate the overall oxidation efficiency, including the action of hydroxyl radicals and other oxidants (such as chlorine), produced in the electro-Fenton process. The average current efficiency (ACE) percentage was calculated based on COD values, using the following expression: (Mohajeri et al., 2010c; Montanaro and Petrucci, 2009).

$$ACE = \frac{(COD_0 - COD_t)FV_s}{8It} \times 100 \quad (13)$$

where COD₀ (g O₂/L) donates the initial chemical oxygen demand, COD_t (g O₂/L) is the chemical oxygen demands at a given time *t* (s), *F* stands for the Faraday constant

(96487 C/mole), V_s represents the volume of the solution (L), I stands for the current applied (A), and 8 is the oxygen equivalent mass (g/eq).

The highest ACE of 94% was observed for applied current of 0.5 A in 30 min experimentation. In general, an increase in current density would lead to the decrease in current efficiency (Chou et al., 1999). At an optimum operation condition of this study, i.e., applied current of 2 A, the current efficiency reduced to 38%.

The energy consumption for the removal of 1 kg of COD was calculated and expressed in kWh. The required electrical energy consumption (EE_c) per volume of leachate was calculated as kWh/m³ according to (Brillas et al., 2009; El-Ashtoukhya et al., 2009), using Equation (14):

$$EE_c = \frac{IVt}{V_s} \quad (14)$$

where I is current (A); V , the voltage (V); t , the treatment time (h); and V_s , the solution volume (L).

In this study the operational parameters for the experiment including electrolysis current, cell voltage, and the reaction time in optimum condition turned out to be 2 A, 8V, and 30 min, respectively. The volume of the leachate treated was 0.5 L, therefore energy consumption were determined to be 16 kWh/m³ for PBLs leachate.

As indicated in this study, electro-Fenton process is highly efficient in the treatment of mature leachate with low biodegradability. Moraes and Bertazzoli (2005) reported achieving 73% and 86% removal of COD and color, respectively, using oxide-coated titanium anode at a current density of 116.0 mA/cm² and 180 min reaction time. Kochany and Lipczynska-Kochany (2009) stated that 66% of COD was removed at pH = 3.5 and 650 mg/L of H₂O₂. In another landfill leachate treatment study (Barnes et al., 2007), COD was lowered by 76%, using

Fenton oxidation at optimized conditions of H₂O₂ dosage of 3500 mg/L, Fe²⁺ dosage of 437.5 mg/L, pH = 4, a mass ratio of 8 for H₂O₂: Fe²⁺ dosage, and reaction time of 30 minutes. Ilhan et al. (2008) also applied electrochemical oxidation and observed over 59% COD and 14% ammonia removals within 30 min reaction time for a current density of 631 A/m², using aluminum electrode.

CONCLUSIONS

Electro-Fenton is a fast and easy technique to eliminate refractory organics from mature landfill leachate. This study showed an efficient mineralization of above 92% after only 30 min of treatment. The optimum COD and color removals of 92% and 93%, respectively, were obtained at initial pH of 3, H₂O₂/Fe²⁺ molar ratio of 1, initial H₂O₂ and Fe²⁺ concentrations of 340 and 560 mg/L respectively, constant DC current of 2 A, treatment duration of 30 min, and electrodes separation distance of 3 cm.

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