

## TERTIARY TREATMENT OF PALM OIL MILL EFFLUENT USING FENTON OXIDATION

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**ABSTRACT:** A study was conducted to determine the feasibility of Fenton oxidation process in treating biologically treated palm oil mill effluent (BT-POME). Two types of Fenton processes were evaluated, namely ambient-Fenton and solar-Fenton. Both were conducted in batch mode at laboratory scale and the efficiency of the processes was assessed based on COD and color removal. The mechanism of removal in the solar-Fenton process was also explored. Both processes were found to be efficient in treating the wastewater. The highest removals of COD and color for ambient-Fenton were 75.2% and 92.4%, respectively. The COD and color removal of 82.4%, and 95.1%, respectively, were achieved by solar-Fenton. The solar-Fenton removal was mainly through oxidation process. Precipitation and coagulation of iron also contributed to the removal of COD and color but at a lesser extent. Enhancement of color removal by the coagulation process is mainly through elimination of the remaining iron rather than removal of the organics. The role of iron and hydrogen peroxide in ambient- and solar-Fenton was statistically evaluated and discussed.

**Keywords:** *Fenton, palm oil mill effluent, solar-Fenton, response surface, coagulation, tertiary treatment*

### 1.0 Introduction

Oil palm plantation is presently covering millions of hectares across Malaysia, Indonesia and Thailand and is becoming the world's number one industrial crop (<http://news.mongabay.com>). In 2006, Malaysian agricultural exports rose to about US\$12.9 billion with palm oil contributing to 51.4% of total agricultural exports (<http://asiacleantech.wordpress.com/2007/08/14/2006-malaysian-palm-oil-market-summary/>). In Indonesia, 11.4 million metric tons of crude palm oil was produced in 2004 which brought in US\$42.4 million to the country treasury. While oil palm plantation is an important economic activity in these countries, to some extent, it also

contributes a significant amount of pollutants to the environment. It is estimated that for every tonne of crude palm oil produced, about 2.5 to 3.5 tonnes of palm oil mill effluent (POME) is generated (Ahmad et al., 2005). Furthermore, the effluent is characterized by high temperature (i.e. 80 to 90°C), highly acidic (i.e. pH 3.8 to 4.5) and contains very high concentration of biodegradable organic matter with COD of 40,000 to 50,000 mg L<sup>-1</sup> and BOD of 20,000 to 30,000 mg L<sup>-1</sup>. Hence, it is vital that such effluent is properly treated before it could be discharged into natural stream in order to protect the environment.

Due to the facts that POME is biodegradable in nature, biological treatment system is found to be the most suitable method to be practiced and hence, become the most popular method used these days. In Malaysia, the typical biological treatment plant comprised the use of anaerobic, facultative and aerobic ponds arranged in series. The choice of pond is greatly influenced by the cost of operation and maintenance of the system, and the availability of land space within the plantation area. The allowable treated effluent to be discharged is governed by the standards stipulated by the Department of Environment of Malaysia as given in Table 1 (Legal Research Board, 2005).

Table 1: Water quality standards for watercourse discharge from palm oil mill<sup>a</sup>

Parameters	Allowable limit (as from January 1984 and thereafter)
Biochemical Oxygen Demand (BOD <sub>3</sub> ) @ 30°C, mg/L	100
Chemical Oxygen Demand (COD), mg/L	(1000) <sup>b</sup>
Total Solids, mg/L	(1500) <sup>b</sup>
Suspended Solids, mg/L	400
Oil and Grease, mg/L	50
pH	5.0-9.0
Temperature, °C	45

<sup>a</sup>Environmental Quality (Prescribed Premises) (Crude Palm-oil) Regulations, 1977 (amended by P.U. (A) 183/82)

<sup>b</sup>Values for the period of 1-7-1981 to 30-6-1982. No new value stipulated since then.

Based on the BOD<sub>3</sub> discharge requirement of 100 mg L<sup>-1</sup> and COD of 1000 mg L<sup>-1</sup> and considering the large volume of the wastewater being discharged, the treated effluent still has the tendency of damaging the environment despite fulfilling the requirement. Moreover, the treated effluent is still seen colored and subjected to complaint from the public residing nearby the receiving river. A tertiary treatment is therefore needed to remove the color and further enhance the quality of the biologically treated POME (BT-POME).

Many treatment techniques are available to be considered to further treat the BT-POME. Among them, advanced oxidation processes (AOPs) may offer better alternative due to their ability to diminish the pollutants in the wastewater as compared to other techniques which merely transfer them from one phase to another. The advantage of the AOPs over traditional chemical oxidation is that they produce and utilize hydroxyl radical (HO<sup>•</sup>) as their oxidizing agent. The radical is one of the most reactive species known to mankind with reaction rate constant of 10<sup>6</sup> - 10<sup>9</sup> M<sup>-1</sup>·s<sup>-1</sup> and is up to 10<sup>9</sup> more powerful than ozone (Farhataziz and Ross, 1977; Andreozzi et al., 1999).

Of the AOPs, Fenton oxidation is the simplest technique to generate HO<sup>•</sup> (Gueneva-Boucheva, 1999; Gernjak et al., 2006). HO<sup>•</sup> is generated through the reaction between ferrous (Fe<sup>2+</sup>) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) at acidic condition via Equation (1).



Numerous studies on the applications of Fenton oxidation process with various pollutants and wastewaters have been reported. The process is dependent on several factors such as pH, reagent dosage, properties of the pollutant and temperature. The use of ultra-violet (UV) light or even visible light ( $\lambda < 580$  nm) to enhance the process has also been widely studied and documented (Legrini et al., 1993; Sun and Pignatello, 1993; Perez et al., 2006).

Sychev and Isak (1995) provide a detailed review on the oxidation mechanism of Fenton oxidation. With respect to the radical-chain mechanism, the following set of consecutive reactions has been widely accepted in the literature.



In the presence of organic substrate (RH), the primary product of the oxidation would be the organic radical, R<sup>•</sup>, which possesses mainly reducing properties of what and may be consumed through the reactions with H<sub>2</sub>O<sub>2</sub>, Fe<sup>3+</sup>, and O<sub>2</sub>.



In addition to oxidation, Fenton oxidation is also capable of removing pollutants via coagulation (Kuo, 1992; Lin and Lo, 1997; Kang and Hwang, 2000). The ferric ions generated in Fenton reaction may form hydroxo complexes with  $\text{OH}^-$  and start to polymerise at pH between 3.5 and 7 which causing coagulation. In practice, precipitation of iron is the required step in Fenton oxidation process. The added iron needs to be removed from the treated wastewater before the wastewater can be discharged to the subsequent process or to the water body. This would provide an additional removal mechanism that partly compensates the inefficiency that may occur during the oxidation stage. However, the precipitation stage generates iron sludge which needs further treatment and disposal.

While the efficiencies of Fenton oxidation in organics removal have been widely studied, the contribution of oxidation and coagulation to the total removal remains unknown for most of the treatment methods (Kang et al., 2002). Englehardt et al. (2006) reported that under the maximum COD removal condition for leachate treatment, removal by oxidation was approximately twice of the coagulation. On the other hands, decolorization of textile wastewater was mainly contributed by oxidation, whereas the COD was removed mainly by coagulation (Kang et al., 2002). It is worthy to note again that one of the main advantages of the AOPs is the ability of the processes to destroy the pollutants rather than transferring them from one phase to another. Hence, although removal in Fenton oxidation can occur through oxidation and coagulation stages, removal of contaminants via the latter should be minimized. A significant removal via coagulation will render the Fenton oxidation less attractive. Besides, with lesser contaminants being trapped in the iron sludge, the potential for iron recovery from the sludge becomes more feasible. Therefore, it is imperative to characterize the degree of the removal mechanisms that are involved in the process.

A study was conducted to determine the viability of Fenton' reagent process as POME tertiary treatment. Since tropical countries are abundance with sunlight throughout the year, the effects of solar light on the process were evaluated. The role of oxidation and coagulation mechanisms in the removal of organics and color was also investigated.

## **2.0 Experimental**

### *2.1 Materials and Analysis*

The BT-POME samples were collected from a nearby palm oil mill. Stock solution of  $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$  was freshly prepared while  $\text{H}_2\text{O}_2$  solution (35% w/w) was used without dilution. A magnetic stirrer was used to mix the solution throughout the experiment. The performance of the Fenton oxidation process was characterized based on COD and color removal. Both were analyzed using Hach DR-4000U Spectrophotometer. Due to the interference of COD analysis by iron, total organic carbon (TOC) was used to

characterize the removal mechanism of the organics. The analysis of TOC was conducted using Shimadzu TOC Analyser V<sub>CSH</sub>.

## 2.2 Experimental Procedures

Two types of Fenton oxidation process were studied namely ambient-Fenton and solar-Fenton. For ambient-Fenton the experiments were conducted inside the laboratory under ambient light. The experiments for solar-Fenton were conducted in an open space directly under the sunlight irradiation during sunny days.

The first part of the experiments was designed using Central Composite Rotatable Design (CCRD) with the aid of Minitab 13.32 statistical software. A set of 11 experimental runs was carried out randomly for each type of Fenton's process. The H<sub>2</sub>O<sub>2</sub> dosage ranged from 800 mg L<sup>-1</sup> to 1500 mg L<sup>-1</sup> while Fe<sup>2+</sup> dosage ranged from 50 mg L<sup>-1</sup> to 400 mg L<sup>-1</sup>. The complete experimental design is outlined in Table 2.

The experiments were conducted batch-wise using a 2-L beaker with BT-POME volume of 1 L. The pH of the wastewater was initially set at 3.0 ± 0.1 using sulphuric acid (50% concentration). Ferrous salt was then added to the solution at the prescribed dosage. Fenton's reaction started with the addition of predetermined dosage of H<sub>2</sub>O<sub>2</sub> and was let run for 1 hour. Then, the wastewater was set to pH 7.0 ± 0.1 using sodium hydroxide (50% concentration). The wastewater was then slow-mixed for 15 minutes and was let stand for another 30 minutes. The supernatant of the treated BT-POME was then sampled and analyzed for COD and color.

Table 2: Experimental runs with their respective dosage of Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub>

Run	Dosage	
	Fe <sup>2+</sup> , mg L <sup>-1</sup>	H <sub>2</sub> O <sub>2</sub> , mg L <sup>-1</sup>
N1	101.3	902.5
N2	348.7	902.5
N3	101.3	1397.5
N4	348.7	1397.5
N5	50.0	1150.0
N6	400.0	1150.0
N7	225.0	800.0
N8	225.0	1500.0
N9	225.0	1150.0
N10	225.0	1150.0
N11	225.0	1150.0

Another set of experiment was conducted using solar-Fenton to characterize the mechanism of removal. In these experimental runs, Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> dosages of 200 mg L<sup>-1</sup> and 1150 mg L<sup>-1</sup>, respectively were used. Experiments were conducted at reaction times of 10, 20, 30, 45 and 60 minutes using separate beakers. After the completion of each

reaction time, samples were withdrawn from the beaker and immediately filtered and analyzed for TOC and color. These samples represented the removal via oxidation. The pH of the remaining solution was raised to  $7.0 \pm 0.1$  followed by slow mixing and settling. The supernatants were sampled, filtered and analyzed for TOC and color. These represent the removal via both oxidation and coagulation. For comparison purposes, coagulation of BT-POME using only ferrous salt was also carried out.

### 3.0 Results and Discussion

#### 3.1 Wastewater Characteristics

The BT-POME obtained for the study has undergone a series of biological treatment at the palm oil mill. Despite the treatment, the wastewater was still colored and high in organic content. The averages COD and TOC were  $1500 \text{ mg L}^{-1}$  and  $430 \text{ mg/L}$ , respectively, with color of 1800 ADMI.

#### 3.2 Removal of COD and Color

The results of the Fenton oxidation treatment on the BT-POME with respect to the COD and color removals are given in Table 3. The removal of COD for ambient-Fenton ranged from 6.5% to 75% while the removal for solar-Fenton ranged from 16.0% to 82.4%. The color removal for ambient-Fenton and solar-Fenton ranged from 56.3% to 92.4% and 60% to 95.1%, respectively. The variation in the results is expected to be due to the various dosages of  $\text{Fe}^{2+}$  and  $\text{H}_2\text{O}_2$  used in the study.

As expected, color appears to have higher removal percentage as compared to COD since the former represents the destruction of color causing compounds only while the latter represents the total mineralization of the contaminants in the wastewater. Solar-Fenton gave better removal of COD than ambient-Fenton but no significant difference between the two Fenton processes was observed with regards to color removal. The highest removal of COD and color for ambient-Fenton were 75.2% and 92.4%, respectively. The COD and color removal of 82.4%, and 95.1% respectively, were achieved by solar-Fenton. These results indicate the viability of Fenton oxidation process in treating the BT-POME. Irradiation of the readily available solar light appears to have positive effect on the performance of the process.

Factorial analysis was carried out to quantify the significance of  $\text{Fe}^{2+}$  and  $\text{H}_2\text{O}_2$  dosage and the synergistic effect that they have on the removal of COD and color by the Fenton oxidation process. The analysis was carried out on the Hadamard matrix runs (run N1 to N4) and center point runs (run N9 to N11). The significance of  $\text{Fe}^{2+}$  dosage and  $\text{H}_2\text{O}_2$  dosage and the combination of both were based on the p-value generated from analysis of variance (ANOVA). However, it should be noted that these results are only valid within the experimental conditions conducted in the study.

The results of the analysis are summarized in Table 4. The dosage of Fe<sup>2+</sup> used in the experiments was found to be significant to the performance of the both Fenton processes. The dosage of H<sub>2</sub>O<sub>2</sub> was significant only for solar-Fenton in removing COD. It was not significant for ambient-Fenton and also solar-Fenton in color removal. The synergistic effect of both Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> dosages was also observed on ambient-Fenton (for COD and color removal) and solar-Fenton for the removal of COD.

Table 3: Removal of COD and color at different reagents dosage for ambient- and solar-Fenton

Run	% COD removal		% Color removal	
	Ambient-Fenton	Solar-Fenton	Ambient-Fenton	Solar-Fenton
N1	6.5	47.9	69.8	74.5
N2	75.2	80.6	92.4	89.7
N3	15.0	25.8	77.7	78.2
N4	66.7	82.4	77.3	92.1
N5	10.3	16.0	69.0	61.5
N6	75.0	75.6	82.5	95.1
N7	52.1	75.6	75.5	78.8
N8	50.9	80.4	74.7	84.2
N9	50.7	75.2	86.3	81.2
N10	48.9	79.6	90.0	83.4
N11	53.3	76.6	89.0	88.7

Table 4: Summary of ANOVA from factorial analysis

Parameter/Response	Factor (s)		
	Fe <sup>2+</sup> dosage	H <sub>2</sub> O <sub>2</sub> dosage	Fe <sup>2+</sup> and H <sub>2</sub> O <sub>2</sub>
	p-value		
COD (ambient- Fenton)	0.001 <sup>a</sup>	1.000	0.062 <sup>b</sup>
COD (solar-Fenton)	0.003 <sup>a</sup>	0.046 <sup>a</sup>	0.034 <sup>a</sup>
Color (ambient- Fenton)	0.028 <sup>a</sup>	0.201	0.027 <sup>a</sup>
Color (solar Fenton)	0.064 <sup>b</sup>	0.512	0.882

<sup>a</sup>Significant at greater than 95% confidence level

<sup>b</sup>Significant at greater than 90% confidence level

The significance of  $\text{Fe}^{2+}$  dosage has been observed by many researchers as reviewed by Aris (2004). The findings of this study are in agreement with those reported earlier. It has generally been observed that the efficiency of the Fenton oxidation process (in term of degradation rate) increases with increasing  $\text{Fe}^{2+}$  dosage in the presence of sufficient  $\text{H}_2\text{O}_2$ . However, as the dosage increases further, the enhancement becomes trivial and at certain stage, reduces the efficiency of the process probably due to the scavenging of the radicals by  $\text{Fe}^{2+}$  as shown in Equation (3).

Similar to iron dosage, increase in  $\text{H}_2\text{O}_2$  dosage has been found to increase the degradation rate of Fenton oxidation process. However, excessive dosage of the peroxide may inhibit the reaction, possibly due to the scavenging effect of  $\text{H}_2\text{O}_2$  producing  $\text{HO}_2^\bullet$  (Equation (4)) which is a less reactive species than  $\text{HO}^\bullet$ , or through recombination of  $\text{HO}^\bullet$  (due to its excessive concentration) reproducing  $\text{H}_2\text{O}_2$  (Equation (5)). In longer term, higher peroxide dosage has been observed to increase the extent of the removal and improve the degree of mineralization.

At many times, optimum dosage of these reagents was observed in Fenton study. These optimum dosage however, strongly depend on the strength and type of the treated wastewater. In this study, the significance of  $\text{H}_2\text{O}_2$  dosage was only observed in COD removal by the solar-Fenton. As mentioned earlier, the presence of UV light from the solar is expected to convert  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$  through Equation (12). With the availability of  $\text{Fe}^{2+}$ , the presence of higher concentration of  $\text{H}_2\text{O}_2$  will therefore enhance the COD removal. Such phenomenon was not observed in ambient-Fenton due to the limitation of  $\text{Fe}^{2+}$ . Although  $\text{Fe}^{3+}$  is also reduced to  $\text{Fe}^{2+}$  through Equation (4), such reaction is much slower as compared to the photo-reduction of  $\text{Fe}^{3+}$  (Sychev and Isak, 1995).



An example of a negative synergistic effect of  $\text{Fe}^{2+}$  and  $\text{H}_2\text{O}_2$  dosage is illustrated in Figure 1. At the iron dosage of  $101.3 \text{ mg L}^{-1}$ , increasing the peroxide dosage from  $902.5 \text{ mg L}^{-1}$  to  $1397.5 \text{ mg L}^{-1}$  increased the color removal from 70% to about 78%. However, at the iron dosage of  $384.7 \text{ mg L}^{-1}$ , increasing the peroxide by the same dosage did not increase the performance but reduced the percentage of removal from more than 90% to about 78%. The reduced performance is apparently caused by the synergistic effect of increasing the dosage of both reagents. Increasing the dosage probably caused the generation of high concentration of  $\text{HO}^\bullet$  which could have scavenged the reactions through Equations (3) to (5) as discussed earlier.

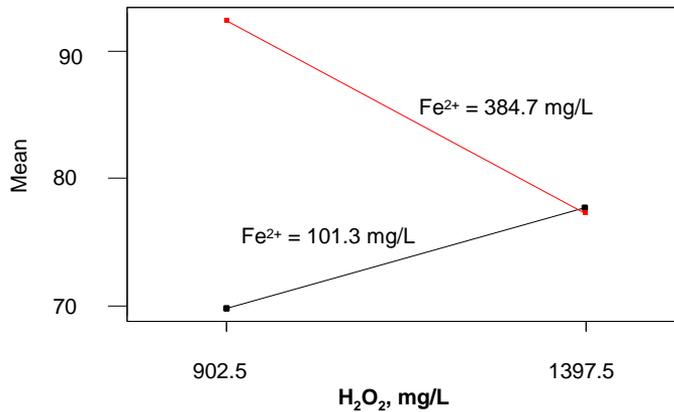


Figure 1: Synergistic effect of H<sub>2</sub>O<sub>2</sub> and Fe<sup>2+</sup> dosage on mean color removal by ambient Fenton

Response surface analysis was conducted to determine the non-linearity of the effect and to develop statistical relationship between the dosage (termed as factor) and the removal of COD and color (termed as responses). The relationship between the factors and the responses can be adequately developed for solar-Fenton and % COD removal (Equation (13)). The relationship holds an R<sup>2</sup> value of 0.975 and lack-of-fit's p-value of greater than 0.1 signifying the adequacy of the relationship.

$$\% \text{ COD removal} = 33.98 + 0.46 (\text{Fe}^{2+}) - 0.044 (\text{H}_2\text{O}_2) - 1.0 \times 10^{-3} (\text{Fe}^{2+})^2 + 1.56 \times 10^{-4} (\text{Fe}^{2+} \times \text{H}_2\text{O}_2) \tag{13}$$

where Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> represent the dosages of ferrous and hydrogen peroxide, respectively. The relationship between Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> dosages on the % COD removal for the solar-Fenton is illustrated in Figure 2.

### 3.3 Removal Mechanisms

In a Fenton oxidation process, the added iron is removed from the solution through precipitation and coagulation at pH 7. At this stage, Fe<sup>2+</sup> and Fe<sup>3+</sup> were considered as coagulant which can cause further reduction of organics and color in the wastewater. The removals of TOC and color after oxidation stage and after coagulation (i.e. after precipitation of Fe<sup>2+</sup> at pH 7) are shown in Figures 3 and 4, respectively. The contribution of oxidation and coagulation towards total removal of TOC is shown in Table 5.

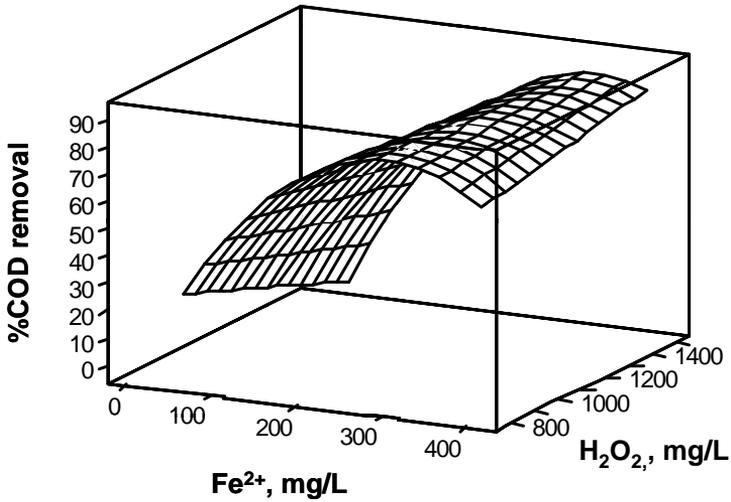


Figure 2: Surface plot generated from response surface analysis between  $\text{Fe}^{2+}$  and  $\text{H}_2\text{O}_2$  and %COD removal for solar-Fenton

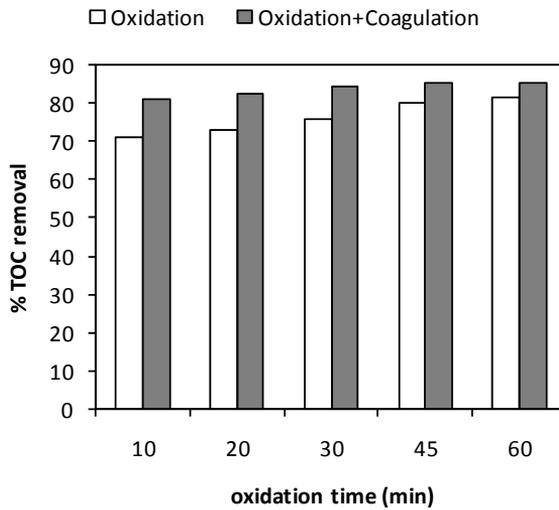


Figure 3: Removal of TOC by oxidation and oxidation+coagulation at different reaction times

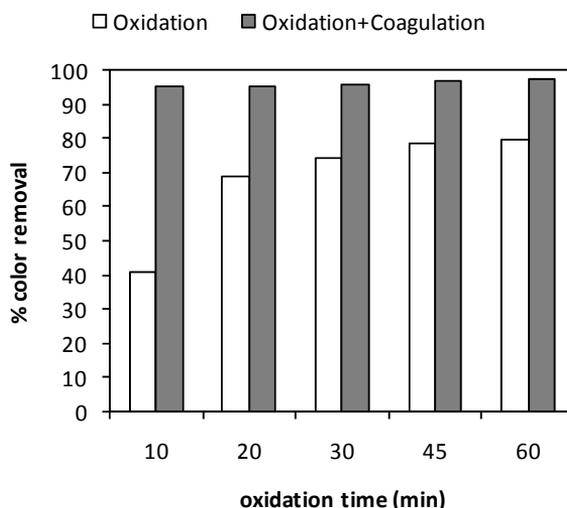


Figure 4: Removal of color by oxidation and oxidation+coagulation at different oxidation times

Table 5: Distribution of oxidation and coagulation in TOC and color removal

Reaction time (min)	TOC Removal		Color Removal	
	% Oxidation <sup>b</sup>	% Coagulation <sup>a</sup>	% Oxidation <sup>b</sup>	% Coagulation <sup>a</sup>
10	87.6	12.4	43.2	56.8
20	88.1	11.9	72.1	27.9
30	90.0	10.0	77.6	22.4
45	94.0	6.0	81.1	18.9
60	95.6	4.4	82.1	17.9

<sup>a</sup>100 x (Total removal – Removal via oxidation)/Total removed

<sup>b</sup>100 - %Removal via coagulation

As observed in Figure 3 and Table 5, most of the TOC removal occurred during the oxidation stage. For 10 minutes of Fenton reaction, 87.6% of the TOC was removed after the oxidation stage and the removal increased to 95.6% as the reaction time was increased to 60 minutes. Coagulation apparently contributes to a maximum of only 12.4% of the TOC removal. As for color, the mechanism of removal depends on the time of reaction. At shorter reaction time, significant color removal was achieved after coagulation was completed. With the increase in oxidation time, the mechanism of removal shifted to oxidation. As shown in Table 5, oxidation contributes to 43% of color removal for 10-

minute reaction time. The contribution significantly increased to 72% for 20-minute reaction time and gradually increased to 82% for 60-minute reaction time.

Precipitation and coagulation of ferric hydroxides and other iron complexes significantly lowered the color of the wastewater especially at shorter oxidation time. However, since more than 70% of TOC has already been removed in the oxidation stage at 10-minute reaction time, the presence of color after oxidation stage appears to be contributed by the iron content in the wastewater. Figure 5 illustrates the removal of TOC and color by coagulation alone with various  $\text{Fe}^{2+}$  dosages. Coagulation with several iron sulphate dosages at pH 7 shows that increasing the iron dosage contributes to a higher TOC removal. As the iron dosage increases, the final color of the solution also increases. With  $\text{Fe}^{2+}$  dosage of  $300 \text{ mg L}^{-1}$ , the color of the treated BT-POME was even higher than the original color of the wastewater despite more than 70% TOC being removed. This supports the hypothesis that the remaining color after the oxidation is mainly caused by the presence of iron complexes rather than the organics. Hence, coagulation in Fenton oxidation process could enhance the color removal mainly through removal of the remaining iron from the wastewater rather than removal of the organics.

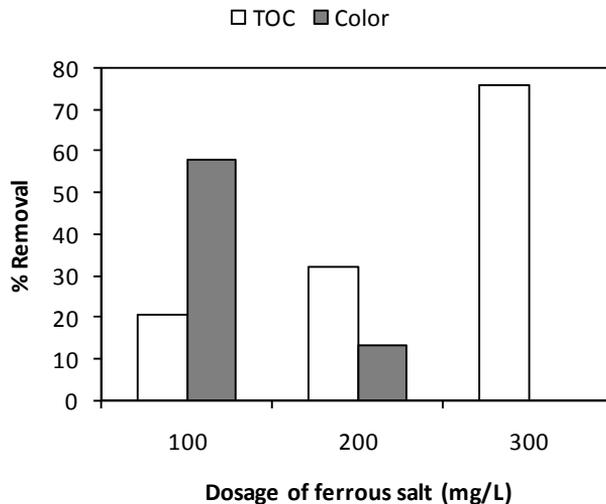


Figure 5: Color and TOC removals by coagulation with various  $\text{Fe}^{2+}$  dosage

#### 4.0 Conclusions

Several conclusions could be derived from the study:

- a. Fenton oxidation is capable of removing COD and color from the BT- POME and thus a viable tertiary treatment option. Solar-Fenton gives a better removal as

compared to ambient Fenton. Color was found to be easily removed as compared to COD.

- b. The removal of COD and color was influenced by the dosage of  $\text{Fe}^{2+}$  and  $\text{H}_2\text{O}_2$ . The relationship between the dosage and the removal was non-linear and synergistic effect was observed.
- c. The main removal mechanism of the solar-Fenton treatment is oxidation. While the oxidation stage of solar-Fenton process provides degradation of the organic compounds in the wastewater (and hence, remove color), the coagulation stage is essential in removing the iron which was applied in the oxidation process. However, the coagulation stage also provides further enhancement in the organics and color removal.

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