

**INDUSTRIAL DETERGENT WASTEWATER TREATMENT  
VIA FENTON REAGENT**

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**INDUSTRIAL DETERGENT WASTEWATER TREATMENT  
VIA FENTON REAGENT**

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**A thesis submitted in fulfillment of the  
requirements for the award of the degree of  
Bachelor of Chemical Engineering**

**Faculty of Chemical & Natural Resources Engineering  
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**APRIL 2010**

“I declare that this thesis entitled “Industrial Detergent Wastewater Treatment via Fenton Reagent” is the result of my own research except as cited in the references.

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*To my beloved parents, my family members, & friends,*

*Thank you for your support, guidance and confidence in me.*

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## ABSTRACT

Production of detergent can generate wastewater containing organic matter which will consume an oxidation demand, surfactants, suspended solids, fat and oil. Besides, sulfate concentration is high in the most detergent plant effluent because of the sulphonation process that has physiological and toxic effects on marine organisms. Therefore, a research must be conducted to find the solution for this problem. The feasibility of Fenton's reagent to treat detergent waste was investigated in this study. The sample of detergent wastewater was taken from FPG Oleochemicals Sdn. Bhd. This experiment studied the effect of temperature towards the feasibility of Fenton's reagent process besides the dosage between hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) and ferrous ion ( $\text{Fe}^{2+}$ ) in the reagent. While, evaluated efficiency of Fenton's reagent in term of chemical oxygen demand (COD), total suspended solid (TSS) and the turbidity reduction within the experimental design. The result found that overall removal was achieved until 96.2% in term of COD, 98.1 % in term of TSS and 99.6 % in term of turbidity using Fenton's reagent process. Besides, also found that this process is optimum at temperature  $35^\circ\text{C}$  able to achieve the Standard A of Parameter Limit of Effluent of Standard A & Standard B were outlined by Department of Environment based on Environment Quality Act 1974.



## ABSTRAK

Penghasilan detergen dikenalpasti menghasilkan air sisa yang mengandungi COD, surfactan, pepejal terampai, lemak dan minyak. Selain itu, kepekatan sulfat juga ditemui tinggi di dalam air sisa yang dikeluarkan oleh pelan detergen di mana dikenalpasti disebabkan oleh proses sulfonasi yang memberi kesan fizikal dan toksid ke atas kehidupan marin. Jadi, satu kajian mesti dijalankan bagi mencari penyelesaian terhadap masalah tersebut. Kajian keberkesanan pengoksidaan sistem Fenton dalam rawatan air sisa detergen telah dijalankan. Sampel air sisa detergen tersebut telah diambil dari FPG Oleochemicals Sdn. Bhd. Eksperimen ini mengkaji kesan suhu terhadap keberkesanan proses reagen Fenton disamping kesan dos antara Hidrogen Peroxida dan ion ferum di dalam reagen Fenton tersebut. Manakala, keberkesanan pengoksidaan Fenton pula ditinjau dari segi penurunan COD, penurunan jumlah pepejal terampai (TSS) dan penurunan kekeruhan sampel selepas eksperimen dijalankan. Keputusan eksperimen mendapati penurunan keseluruhan telah mencapai sehingga 96.2% bagi COD, 98.1% bagi TSS dan 99.6% bagi kekeruhan sampel dengan menggunakan proses reagen Fenton. Selain itu, hasil kajian ini juga menemui kajian keberkesanan ini adalah paling optimum pada suhu 35°C dan mampu mencapai piawai A berdasarkan Had-had Parameter Bagi Effluen Piawai A & Piawai B yang telah digariskan oleh Jabatan Alam Sekitar di bawah akta Kualiti Alam Sekeliling 1974.

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### LIST OF ABBREVIATIONS/ SYMBOLS/ TERMS

% Removal	-	Percentage of removal
AOPs	-	Advanced oxidation process
BDH	-	BDH Chemicals supplier
BOD <sub>5</sub>	-	Biochemical oxygen demand in five days
BOD <sub>0</sub>	-	Initial BOD
BOD <sub>t</sub>	-	BOD at time, t
CO <sub>2</sub>	-	Carbon dioxide
COD	-	Chemical oxygen demand
COD <sub>0</sub>	-	Initial COD
COD <sub>t</sub>	-	COD at t time
Fe <sup>2+</sup>	-	Ion ferum (+2)
Fe <sup>3+</sup>	-	Ion ferum (+3)
FeSO <sub>4</sub> .7H <sub>2</sub> O	-	Ferrous sulphate
h	-	Hour
H <sup>+</sup>	-	Hydogen ion
HCl	-	Hydrochloric Acid
H <sub>2</sub> O <sub>2</sub>	-	Hydrogen peroxide
[H <sub>3</sub> O <sub>2</sub> ] <sup>+</sup>	-	Peroxone ion
HO•	-	Hydroxyl radical
LAS	-	Linear Alkylbenzene Sulfonate
NaOH	-	Sodium hydroxide
NTU	-	Nephelometric <i>Turbidity</i> Units
ppm	-	part per million
t	-	Time

TSS	-	Total suspended solid
TSS <sub>0</sub>	-	Initial total suspended solid
TSS <sub>t</sub>	-	Total suspended solid at time, t
T <sub>0</sub>	-	Initial turbidity
T <sub>t</sub>	-	Turbidity at time, t
UV	-	Ultraviolet



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## CHAPTER 1

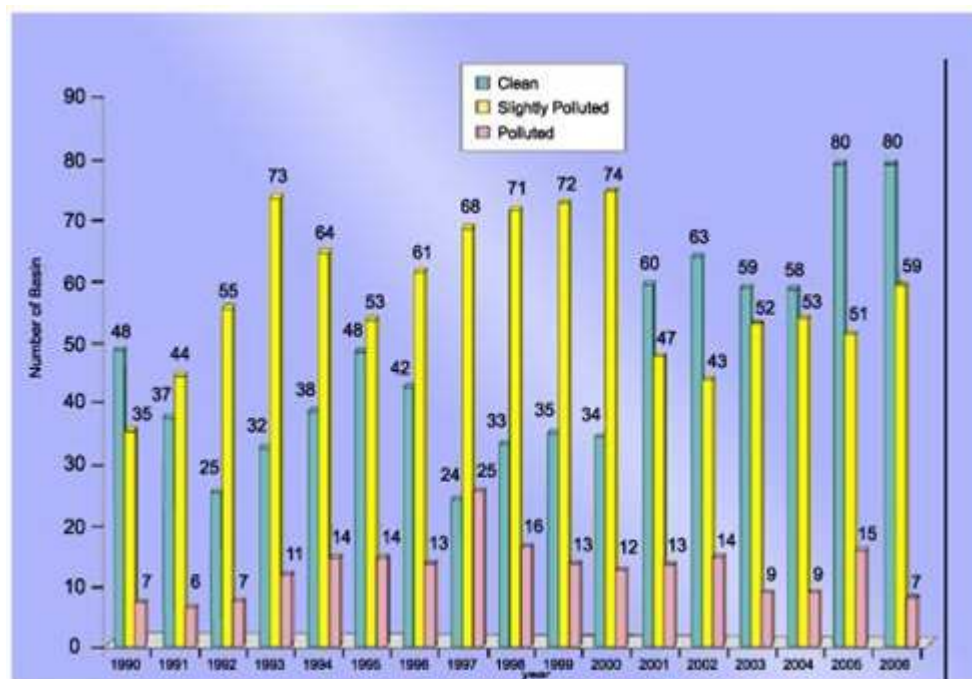
### INTRODUCTION

#### 1.1 Background of study

New developments in the variety of fields to meet the ever-increasing requirements of human beings have also led to the presence of new compounds in the effluent streams of processing plants, which are not readily degraded by the conventional effluent treatment methods (Bauer and Fallmann, 1997; Mantzavinos et al., 1997; Otal et al. 1997; Feigelson et al., 2000). The focus on waste minimization and water conservation in recent years has also resulted in the production of concentrated or toxic residues.

Water pollution is a major problem in the global context. It has been suggested that it is the leading worldwide cause of deaths and diseases. The effect of water pollution are varied which is to humans, animals, plants and ecosystem. They include poisonous of drinking water, poisonous of food animals, unbalanced river and lake ecosystem that can no longer support full biological diversity, deforestation from acid rain and many other effects. So it's not a weird when wastewater discharged from diverse industries plays an important role in environmental pollution; wastewater recycling is now an emerging global issue, and contributes critically to the sustainability of environment (Jie-Chung Lou and Yu-Jen Huang, 2008). Based on research by *Japanese Consulting Institute (JCI)* on water pollution in Malaysia, rivers in Malaysia

generally appear to have high organic pollution loads and high SS concentrations. Low rainfall, which resulted in reduced flow rates in the rivers, was cited as one of the reasons for the increased pollution. In addition, the agriculture-based industries (natural rubber and palm oil production, for instance), manufacturing industry, and livestock industry was identified as the sources of pollution that contributing to BOD loading in water in Malaysia. Figure below shows the water quality in Malaysia:



**Figure 1.1:** River Basins Water Quality Trend, 1990-2006.

Wastewater from the pigment industry has various organic characteristics and is one of the most difficult types of wastewater to treat. Pigment wastewater is almost non-soluble in any solvent and contains high levels of COD, BOD<sub>5</sub>, color and suspended solids (Young-O Kim et al., 2004). Besides that, most wastewater contains heavy metals and organic compounds, which are not only harmful to human health but also dangerous to nature life. Technology for treating industrial wastewater can be divided into three categories: chemical methods, physical methods and biological methods. Chemical methods are include chemical precipitation, chemical oxidation or reduction, formation of an insoluble gas followed by stripping, and other chemical reaction that involve

exchanging or sharing electrons between atoms. Physical treatment methods include sedimentation, floatation, filtering, stripping, ion exchange, adsorption, and other process that accomplish removal of dissolved and undissolved substance without necessarily changing their chemical structure. Meanwhile biological treatment methods are those that involve living organism using organic or in some instances, inorganic, substances for food, completely changing their chemical and physical characteristics (Metcalf & Eddy, 2003).

The most common treatment methods for removing heavy metals and organic compounds are chemical precipitation and chemical oxidation (Jie-Chung Lou et al., 2008). In fact, Fenton's reagent, that is one of the chemical oxidation methods, has been widely used to treat textile of wastewater. It's because of cost effective, easy to treat, reacts well with organic compounds and does not produce toxic compounds during oxidation (Young-O Kim et al., 2004). Therefore, in this research, Fenton's reagent treatment process also will be used as pre-treatment method to remove toxic materials before biological treatment. But this experiment, conduct with batch experiment to study the industrial waste of surfactant that consists of Linear Alkylbenzene Sulfonate (LAS) that have high levels of COD, BOD, TSS and not stabilize in pH and also low biodegradability. (Jie-Chung Lou et al., 2008) So before it can be discharge to the sea, need to minimize their chemical composition in this waste in order to obey the National Water Quality Standard.

## **1.2 Problem Statement**

Production of detergent or surfactant involved several processes each every of proces will contribute to the water pollution and environment. In fact, surfactants have also been widely used in textiles, fibers, food, paints, polymers, plant protection, cosmetics, pharmaceuticals, mining, oil recovery and pulp and paper industries (Hellston, 1986). This fact shows that many kind of industries use surfactant and will

produce surfactants waste. As noted by Swisher (1987), a large number of surfactants, including the anionic types employed in the present study, have relatively low biodegradability.

Based on Xiao-Jun Wang et al., 2008 study, found that a detergent plant generates wastewater containing chemical oxidation demand (COD), surfactant, suspended solids (SS), fat and oil. Besides, sulfate concentration is high in the most detergent plant effluent because of the sulphonation process. For the treatment of the surfactant waste from this plant, they use anaerobic process. But, if this kind of wastewater containing abundant sulfate is treated by an anaerobic process, the sulfate will be converted to sulfides by sulfate reducing bacteria (SRB) (A. Visser et al., 1996 and A. Rinzema et al, 1988). The prevailing SRB inhibit the performance of acidogenic and methanogenic bacteria, so it will affect anaerobic treatment processes (K.K. Samir et al., 2003). Consequently, most of the sulfides can be converted to sulfur under aerobic condition. This will subsequently consume a lot of dissolved oxygen. And sulfur is also harmful to aerobic bacteria in aerobic process. As a result of the high residual surfactants, the wastewater will foam during aerobic biological process. Therefore, it is not feasible to destroy the surfactant by aerobic treatment process directly.

Due primarily to economic reasons, it is impractical to replace those low biodegradable surfactants in all household and industrial application. Besides that, this process easier to treat, reacts well with organic compounds and does not produce toxic compounds during oxidation (Kuo, 1992; Lin and Peng, 1995). So it suitable in application of treatment surfactant wastes. Another method like UV, ozone and others are identified increasing the cost in treatment of wastewater. Pretreatment methods of surfactant wastewater thus need to be developed which allow safe uses of low biodegradable surfactant (Swisher, 1987). Advanced oxidation pretreatment using Fenton reagent was very effective at enhancing the biodegradability of this kind of wastewater. In fact, the Fenton oxidation process has also been employed successfully to treat different industrial wastewaters in many previous investigations (Barbeni et al.,

1987; Sedlak and Andren, 1991; Kuo, 1992; Potter and Roth, 1993; Lin and Peng, 1995; Lin and Chen, 1997; Lin and Lo, 1997).

### **1.3 Research Contribution**

There are some purposes why industrial detergent wastewater entering collecting systems or wastewater treatment plants shall be subject to such pre-treatment. These because of in order to:

- Protect the health of staff working in collecting systems and treatment plants,
- Ensure that collecting systems, waste water treatment plants and associated equipment are not damaged,
- Ensure that the operation of the wastewater treatment plant and the treatment of sludge are not impeded,
- Ensure that discharges from the treatment plants do not adversely affect the environment, or prevent receiving water from complying with other Community Directives,
- Ensure that sludge can be disposed of safely in an environmentally acceptable manner.

### **1.4 Objectives**

The major objective in this study is to investigate the performance of Fenton reagent oxidation in treatment of industrial detergent wastewater.

## 1.5 Scopes of Study

In order to achieve the objectives, the following scopes have been identified:

- Characterizations of industrial detergent wastewater contains before and after treatment in terms of turbidity, COD and TSS removal.
- Effect of  $\text{H}_2\text{O}_2$  dosage
- Effect of  $\text{FeSO}_4$  dosage
- Effect of temperature

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Introduction**

In this chapter will discuss about what the wastewater, detergent waste is and what is LAS that contain in surfactant waste. For the next section, it will discuss more in detail on Fenton's reagent process description and lastly shows the standard water quality index in Malaysia that must follow to discharge wastewater that was treated.

#### **2.2 Wastewater**

Wastewater is any water that has been adversely affected in quality by anthropogenic influence. It comprises liquid waste discharged by domestic residences, commercial properties, industry, and/or agriculture and can encompass a wide range of potential contaminants and concentrations. Wastewater can be divided in three categories:

1. Domestic (sewage) — wastewater is the water that contains mainly human and animal wastes, household wastes, small amounts of groundwater infiltration and small amounts of industrial wastes (Metcalf & Eddy, 2003).

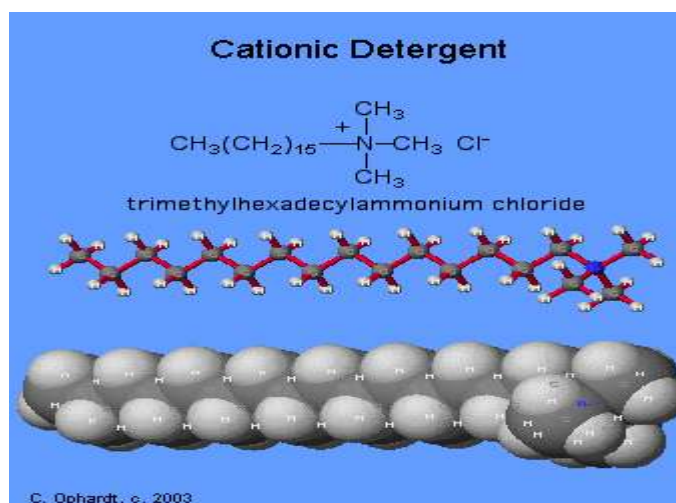


2. Sanitary wastewater — Consists of domestic wastes and significant amounts of industrial wastes. In many cases, the industrial wastes can be treated without special precautions. However, in some cases, the industrial wastes will require special precautions or a pretreatment program to ensure the wastes do not cause compliance problems for the wastewater treatment plant (Metcalf & Eddy, 2003).
3. Industrial wastewater — Consists of industrial wastes only. Often the industry will determine that it is safer and more economical to treat its waste independent of domestic waste (Metcalf & Eddy, 2003).
4. Combined wastewater — Consists of a combination of sanitary wastewater and storm water runoff. All the wastewater and storm water of the community is transported through one system to the treatment plant (Metcalf & Eddy, 2003).
5. Storm water — Contains a separate collection system (no sanitary waste) that carries storm water runoff including street debris, road salt, and grit (Metcalf & Eddy, 2003).

### **2.2.1 Detergent waste**

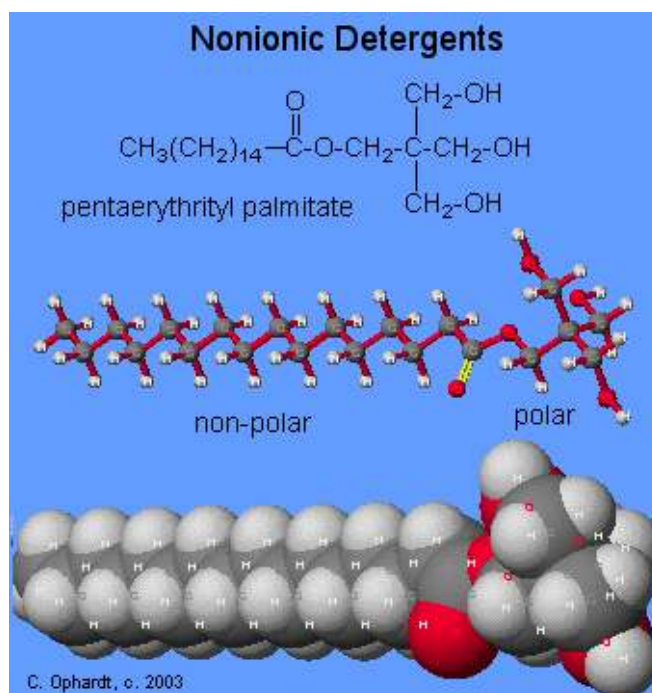
Detergents have similar molecular structures and properties as soap. Although the cleansing action is similar, the detergents do not react as readily with hard water ions of calcium and magnesium. Detergent molecular structures consist of a long hydrocarbon chain and a water soluble ionic group. Most detergents have a negative ionic group and are called anionic detergents (figure 2.1). The majority are alky sulfates. Others are "surfactants" (from surface active agents) which are generally known as alkyl benzene sulfonates (Charles E. Ophardt, 2003).





**Figure 2.2:** Structure of Cationic Detergent

Nonionic detergents are used in dish washing liquids. Since the detergent does not have any ionic groups, it does not react with hard water ions. In addition, nonionic detergents foam less than ionic detergents. The detergent molecules must have some polar parts to provide the necessary water solubility. In the graphic on the left, the polar part of the molecule consists of three alcohol groups and an ester group. The non-polar part is the usual long hydrocarbon chain (Charles E. Ophardt, 2003).



**Figure 2.3:** Structure of Nonionic Detergent

Standard laundry detergent powders contain levels of chemicals such as sodium and boron, which can be damaging to plants and should not be applied to soils. Commercial soil wetting agents will continue to work for a considerable period, but they will eventually be degraded by soil micro-organisms. Some can, however, interfere with the life-cycles of some aquatic organisms, so care should be taken to prevent run-off of these products into streams, and excess product should not be washed down (Baeurle SA and Kroener J, 2004). Besides that, some surfactants are known to be toxic to animals, ecosystems and humans, and can increase the diffusion of other environmental contaminants.

The surfactants present in detergent products remain chemically unchanged during the washing process and they are discharged down the drain with the dirty wash water. In the vast majority of cases, the drain is connected to a sewer and ultimately to a wastewater treatment plant; where the surfactants present in the sewage can be removed by biological and physical-chemical processes. European Law now requires efficient treatment of urban waste water and all but the smallest conurbations must comply before the end of 2005 (Council Directive 91/271/EEC of 21 May 1991).

In the early 1960's occasional instances of foaming were observed both in wastewater treatment plants and in surface waters receiving effluents. This phenomenon was shown to be due to the use in detergent products of a poorly biodegradable surfactant; which, consequently, was insufficiently removed during the treatment process. Industry reacted rapidly by replacing the problematic surfactant with a biodegradable equivalent. Since this time the removal of detergent surfactants in waste-water treatment plants has been the subject of much research; by industry, academia and regulatory authorities (T.C.J. Feijtel et al., 1995).

In fact, surfactant have been widely used in textiles, fibers, food, paints, polymers, plant protection, cosmetics, pharmaceuticals, mining, oil recovery and pulp and paper industries (Hellston, 1986). It uses to induce aggregation, wetting, lathering as

emulsifier by the pharmaceutical industry, cosmetic industry, chemical industry, agriculture industry, food industry, etc. Based on C. Crabb (2000), the worldwide surfactant consumption was of the order of 10.4M tons in year 2000 and predicted large amount of surfactant waste will be discharge to the environment. This situation shows a very important issue to the environment.

### **2.2.2 Linear Alkylbenzene Sulfonate (LAS)**

Linear alkylbenzene sulfonate (LAS) is produced by sulfonation of linear alkylbenzene with sulfur trioxide. LAS is the one of the major constituents of synthetic detergent, and most widely used for domestic and industrial purposes, in both percentage and absolute amount (Inoue et al., 1978; He et al., 1991). But at the same time, it is also the most common pollutant found in almost all environmental compartments (Cullivan and Swisher, 1969; McEvoy and Giger, 1986; Takada and Ishiwatari, 1987; Yediler et al., 1989; Papapon and Eckhoff, 1990; McAvoy et al., 1993). Composition of LAS in the detergent was found that about 5 - 25 %.

Linear alkylbenzene sulfonate (LAS) also is the most important anionic surfactant used in laundry detergent. Although there is high separation efficiency in sewage treatment plants, the increasing use can result in outlet concentration to estuaries and near-shore marine waters in the range of 0.02–1 mg LAS/l (Berna et al., 1991; Stalmans et al., 1991). LAS have been reported to have physiological and toxic effects on marine organisms at this concentration range (H.F. Bjerregaard et al., 2001). Besides, it also are more toxic than the hard alkybenzene sulfonates, (Edison Barbieri et al., 2002).

### 2.3 Fenton Reagent

Fenton reagent process is one of the Advance Oxidation Process (AOPs) that widely used to treat the wastes that not easy to decompose. This Fenton reagent process involves producing of very effective oxidation agent in the removal of organic compound in the wastewater. Besides that, this process is cheaper, easier and suitable in application of treatment detergent waste. Another method like UV, ozone and others are identified increasing the cost in treatment of wastewater.

Fenton reagent was discovered about 100 years ago, but its application as an oxidizing process for destroying toxic organics was not applied until the late 1960s (Huang et al.,1993). Fenton found that using catalyst ions and hydrogen peroxide, most of organic compound easy to oxidize. That process doesn't need high pressure and high temperature. It also doesn't need complex equipment. After varies studies done, found that Fenton mechanism involve hydroxyl radical ( $\text{OH}^\cdot$ ) that produce from decomposition of  $\text{H}_2\text{O}_2$ .

After that many research have done involving this method. In Malaysia, Mohd Rizalman Abd. Aziz, 1996 has used this reagent to study about treatment of industrial coconut milk waste. He said, this method can remove COD parameter about 89%. Besides that, it can remove  $\text{BOD}_5$  more than 95% and suspended solid until 97%.

Fenton reaction wastewater treatment processes are known to be very effective in the removal of many hazardous organic pollutants from water. The main advantage is the complete destruction of contaminants to harmless compounds, e.g.  $\text{CO}_2$ , water and inorganic salts. The Fenton reaction causes the dissociation of the oxidant and the formation of highly reactive hydroxyl radicals that attack and destroy the organic pollutants.

### 2.3.1 Hydrogen Peroxide

Hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) is a strong oxidant and its application in the treatment of various inorganic and organic pollutants is well established. Still  $\text{H}_2\text{O}_2$  alone is not effective for high concentrations of certain refractory contaminants because of low rates of reaction at reasonable  $\text{H}_2\text{O}_2$  concentrations. Improvements can be achieved by using transition metal salts (e.g. iron salts) or ozone and UV-light can activate  $\text{H}_2\text{O}_2$  to form hydroxyl radicals, which are strong oxidants. Oxidation processes utilizing activation of  $\text{H}_2\text{O}_2$  by iron salts, classically referred to as Fenton's reagent is known to be very effective in the destruction of many hazardous organic pollutants in water.

$\text{H}_2\text{O}_2$  is also useful in the treatment of the gaseous sulphur oxides and nitrogen oxides being converted to the corresponding acids. Other related uses include the bleaching of pulp and paper and organic synthesis.  $\text{H}_2\text{O}_2$  has applications in the surface treatment industry involving cleaning, decorating, protecting and etching of metals (L'air Liquide). By dissociation into oxygen and water  $\text{H}_2\text{O}_2$  can also supply oxygen to micro organisms in biological treatment facilities and in the bioremediation of contaminated sites. It can be used as a disinfecting agent in the control of undesirable biofilm growth. Since the oxygen concentration is generally rate limiting during the in situ biodegradation of organic contaminants, several applications using injection of  $\text{H}_2\text{O}_2$  into the subsurface have been successfully attempted to enhance the biodegradation activity (E.J. Calabrese et al., 1989).  $\text{H}_2\text{O}_2$  can be decomposed into water and oxygen by enzymatic and nonenzymatic routes. Oxidation by  $\text{H}_2\text{O}_2$  alone is not effective for high concentrations of certain refractory contaminants, such as highly chlorinated aromatic compounds and inorganic compounds (e.g. cyanides), because of low rates of reaction at reasonable  $\text{H}_2\text{O}_2$  concentrations. Transition salts (e.g. iron salts), ozone and UV-light can activate  $\text{H}_2\text{O}_2$  to form hydroxyl radicals which are strong oxidants:

- Ozone and hydrogen peroxide



- Iron salts and hydrogen peroxide



- UV-light and hydrogen peroxide



The oxidation processes utilizing activation of  $\text{H}_2\text{O}_2$  by iron salts, referred to as Fenton's reagent, are discussed below.

In general, oxidation processes which are based on the generation of radical intermediates are termed Advanced Oxidation Process (AOPs) (Venkatadri et al., 1993). Hydroxyl radicals (oxidation potential: 2.8 V) are stronger oxidants than ozone and  $\text{H}_2\text{O}_2$ . Hydroxyl radicals non-specifically oxidize target compounds at high reaction rates (of the order of  $10^9 \text{M}^{-1} \text{s}^{-1}$ )

### 2.3.2 Theory and Application Principles

Fenton's reagent involve homogenous reaction have a stronger oxidation potential (2.8 V) than ozone (2.07 V). The Fenton reactions at acidic pH lead to the production of ferric ion and of the hydroxyl radical (Eqn. 2.4) (Benatti *et.al.*, 2006).



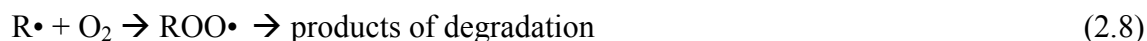
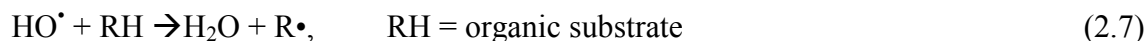
Hydroxyl radicals may be scavenged by reaction with another  $\text{Fe}^{2+}$  or with  $\text{H}_2\text{O}_2$ :







Hydroxyl radicals may react with organics starting a chain reaction:



Ferrous ions and radicals are produced during the reactions as shown below:



The basic mechanism of the Fenton treatment process consists of chemical oxidation and chemical coagulation of organic compounds. Fenton's oxidation process is normally composed of four stages, which are: pH adjustment, oxidation reaction, neutralization and coagulation, and precipitation. Thus, the organic substances are removed at two stages i.e. oxidation and coagulation. In addition, the completion of the oxidation is dependent on the ratio of hydrogen peroxide to organic, while the rate of oxidation is determined by the initial iron concentration and temperature (Benatti et.al., 2006).

In Fenton "like" ( $\text{Fe}^0/\text{H}_2\text{O}_2$ ) processes, ferric salts or iron powder is used as a source of catalytic iron. In acid conditions, iron powder reacts with hydrogen peroxide producing ferrous ions.



From the environmental point of view, the advantage of the implementation of iron powder ( $\text{Fe}^0$ ) instead of iron salts is the avoidance of unnecessary loading of aquatic system with counter anions. Furthermore, the concentration of ferrous and ferric ions in wastewater treated by Fenton “like” process with iron powder is significantly lower in comparison to Fenton type processes that utilize iron salts. The efficiency of Fenton and Fenton”like” processes depend on the generation rate and concentration of the hydroxyl radicals produced through the Fenton reaction (Kusic et.al., 2006).

The advantages of Fenton’s reagent include relatively cheap, high efficiency, simplicity of operating and possible complete destruction contaminants (Benatti et.al.,2006). Recent studies in Fenton reagent involve reaction modifications, including the use of high concentrations of hydrogen peroxide, the substitution of different catalysts such as ferric iron and naturally occurring iron oxides, and the use of phosphate-buffered media and metal-chelating agents. These conditions, although not as stoichiometrically efficient as the standard Fenton’s reactions, are often necessary to treat industrial waste streams and contaminants in soils and groundwater (Benatti et.al., 2006).

### **2.3.3 Factors Affecting Fenton System**

There are several factors that affect Fenton’s system efficiency, which are pH, both iron and hydrogen peroxide dosage, temperature and characteristic of pollutants. The effect of pH on the efficiency of the Fenton reagent system has been widely documented in the literature. The best pH for the system generally ranges from 2.5 to 3.5. Kavitha et al. (2005) observed an optimal pH of 3 appeared to be effective on nitrophenol degradation. They observed more than 92% colour removal at pH 3. Noguiera et al. (2005) also observed that highest percentage removal of colour (70%) can be achieved at pH 2.5 on chlorophenol degradation. The narrow pH requirement by

the Fenton reagent process is apparently attributed to the sensitivity of  $\text{Fe}^{2+}$  or  $\text{Fe}^{3+}$  and  $\text{H}_2\text{O}_2$  to pH. The lower efficiency of the process at pH less than 2.5 is anticipated due to the formation of the complex iron species which react more slowly with  $\text{H}_2\text{O}_2$  and therefore, produces less  $\text{HO}\cdot$ . At high pH, in addition to the formation of Fe(II) complexes with the buffer inhibiting the radical formation, the retardation is expected to be caused by the precipitation of ferric oxyhydroxides ( $\text{Fe}_2\text{O}_2\cdot n\text{H}_2\text{O}$ ). Moreover,  $\text{H}_2\text{O}_2$  is more unstable at high pH as it readily decomposes to  $\text{H}_2\text{O}$  and oxygen.

In longer term, higher initial peroxide dosage has been observed to increase the extent of removal and improve the degree of mineralization (Miguel et al., 2005). The short-term benefit of increasing  $\text{H}_2\text{O}_2$  dosage tends to reduce if a higher dosage is applied. Excessive dosage of the peroxide may inhibit the reaction, possibly due to the scavenging effect of  $\text{H}_2\text{O}_2$  producing  $\text{HO}_2\cdot$  (Eqn. 2.3), which is a less reactive species than  $\text{HO}\cdot$ , or through recombination of  $\text{HO}\cdot$  (due to its excessive concentration) reproducing  $\text{H}_2\text{O}_2$  (Eqn. 2.3.5.1). In longer term, higher initial peroxide dosage has been observed to increase the extent of removal and improve the degree of mineralization (Aris, 2004).



Similar to  $\text{H}_2\text{O}_2$  dosage, increase in iron dosage has been found to increase the degradation rate of the Fenton reagent process. As the dosage increases further, the enhancement becomes trivial and at a certain point, it reduces the efficiency of the process, probably due to the scavenging of the radicals by  $\text{Fe}^{2+}$ .

Different temperature will give different efficiency of Fenton process. According to Hideyuki et al. (2005), Miguel et al. (2005) and Mariana et al. (2003), they reported their most effective temperature at 25°C while Lu et.al. (2003) and Torrades et al. (2004) is at 30°C to 40°C.

The efficiency of the Fenton reagent system is also affected by the characteristic of the pollutants or wastewaters to be treated. The degradation rate and the extent of the removal of the pollutants were observed to reduce with the increase in the pollutant concentration. Structurally, unsaturated compounds were found to degrade faster than the saturated compound. Degradation also tends to be faster for the aromatic compounds as compared to the aliphatic or cyclic organic substances. Aromatic compounds were also observed to use less oxidant than the aliphatic and degrade more effectively at neutral pH. Inhibition of degradation reactions may take place in the presence of scavengers. Some scavengers reduce the rate by complexation with iron while others may react with the HO•, forming less reactive radical species. Different concentrations at which scavengers become significant have been reported (Aris, 2004).

#### **2.3.4 Fenton's reagent Limitation**

Fenton processes have many limitations. One of these limitations is an acidic background. In neutral or alkali background the reaction is either much slower or does not work at all. The other limitation is with substances, which can be degraded by these reactions. Some substances are not oxidized by Fenton reaction, e.g. acetone. The other disadvantage is the possibility of water toxicity increasing within the reaction. That is why it is necessary to know all the oxidation levels. Many times these levels are unpredictable mainly when more substances are present (Š. Tutter et al., 2007).

## 2.4 Limit of Effluent

Effluent guidelines are national standards for wastewater discharges to surface waters and publicly owned treatment works (sometimes called municipal sewage treatment plants). We issue effluent guidelines for categories of existing sources and new sources under Title III of the Clean Water Act. The standards are technology-based (i.e. they are based on the performance of treatment and control technologies); they are not based on risk or impacts upon receiving waters. Limitation of effluent analyses to estimate process parameters whose evaluation cannot be performed through only one batch, unless the sensitivity of effluent characteristic to process parameter is higher than that from sample heterogeneity (Santos *et al.*, 2005). The effluent quality after treatment was compared against specified effluent quality limits to determine the plant's performance in terms of Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Suspended Solids (SS), ammonia-nitrogen, nitrate-nitrogen, and phosphorus. Table 1 shows the Environmental Quality Act 1974, Environmental Quality (Sewage and Industrial Effluents) Regulations, 1979 in Malaysia with selected parameter limits of effluent of standard A and B.

Under this regulation, 'Effluent' is defined as sewage or industrial effluent. However, 'Industrial Effluent' means liquid water or wastewater produced by reason of the production processes taking place at any industrial premises. Sewage is defined as any liquid water or wastewater discharge containing animal or vegetable matter in suspension or solution and may include liquids containing chemicals in solution. Standard A is chosen because the discharge occurs at a watershed. In general, most water quality parameters examined did not violate the water quality of the Third Schedule Environmental Quality Act, 1974. The bulk of industrial effluents that reach the marine environment can be said to come from industries situated in coastal and estuarine areas. These effluents are discharged directly into the sea by way of drains and pipelines or through rivers which serve as the intermediate receiving waters.

**THIRD SCHEDULE**  
**ENVIRONMENTAL QUALITY ACT 1974**  
**ENVIRONMENTAL QUALITY (SEWAGE AND INDUSTRIAL EFFLUENTS)**  
**REGULATIONS 1979**  
**(REGULATIONS 8(1), 8(2), 8(3))**  
**PARAMETER LIMITS OF EFFLUENTS OF STANDARDS A AND B**

Parameter	Unit	Standard	
		A	B
(i) Temperature	°C	40	40
(ii) pH value	-	6.0 - 9.0	5.5 - 9.0
(iii) BOD at 20°C	mg/ l	20	50
(iv) COD	mg/ l	50	100
(v) Suspended Solids	mg/ l	50	100
(vi) Mercury	mg/ l	0.005	0.05
(vii) Cadmium	mg/ l	0.01	0.02
(viii) Chromium, Hexavalent	mg/ l	0.05	0.05
(ix) Arsenic	mg/ l	0.05	0.10
(x) Cyanide	mg/ l	0.05	0.10
(xi) Lead	mg/ l	0.10	0.5
(xii) Chromium Trivalent	mg/ l	0.20	1.0
(xiii) Copper	mg/ l	0.20	1.0
(xiv) Manganese	mg/ l	0.20	1.0
(xv) Nickel	mg/ l	0.20	1.0
(xvi) Tin	mg/ l	0.20	1.0
(xvii) Zinc	mg/ l	2.0	2.0
(xviii) Boron	mg/ l	1.0	4.0
(xix) Iron (Fe)	mg/ l	1.0	5.0
(xx) Phenol	mg/ l	0.001	1.0
(xxi) Free Chlorine	mg/ l	1.0	2.0
(xxii) Sulphide	mg/ l	0.50	0.50
(xxiii) Oil and Grease	mg/ l	Not Detectable	10.0

**Figure 2.4:** Parameter Limit of Effluent Standard A and B (EQA 1974)

## CHAPTER 3

### METHODOLOGY

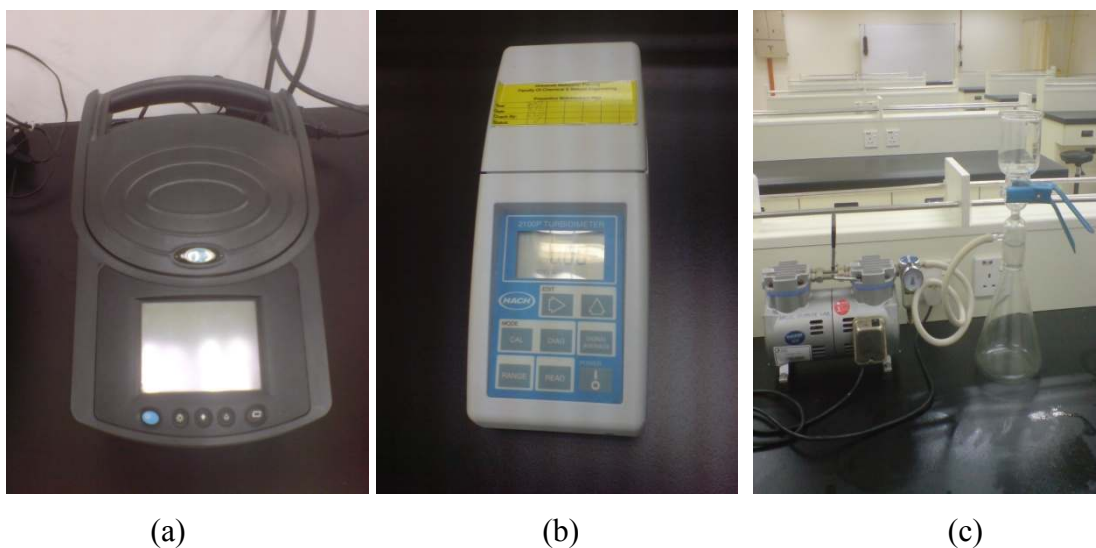
#### 3.1 Introduction

This chapter will discuss about material used for this study, the method used to prepare the sample and treatment chemical and also will explain how to run the experiment and analyze the samples.

#### 3.2 Materials and Equipments

Hydrogen Peroxide (30 % w/w, density 1.11 kg/ L) was obtained from BDH. Stock solutions of  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  were freshly prepared in the range of 1000 mg/L to 1650 mg/L, while  $\text{H}_2\text{O}_2$  solution (30% w/w) was used without any dilution. 0.5M of HCl and NaOH were used for adjust the pH of the sample during and after treatment. Solutions were prepared using distilled water. The range of  $\text{H}_2\text{O}_2$  concentration was from 500 mg/L to 1300 mg/L. A Jar Test apparatus was used to study the effect of dosages while Incubator Shaker used to study effect of temperature. Both were used continuously so that the mixture of sample and Fenton reagent can be uniformly dispersed in the solution. pH meter was used to adjust the pH of sample. HACH DR2400 Spectrophotometer was used to analyze COD value. HACH 2100P Turbidimeter was

used to analyze turbidity value of wastewater, while vacuum pump (Rocker 600) was used to analyze suspended solids value contain in the wastewater. The instruments used were shown in Figure 3.1(a), 3.1(b) and 3.1(c). Industrial detergent wastewater was taken from FPG Oleochemicals Sdn. Bhd. in Pelabuhan Kuantan, Pahang. Fenton oxidation will be used to treat this industrial detergent wastewater in order to determine the efficiency of Fenton oxidation on turbidity, COD and total suspended solids (TSS) removal of industrial detergent wastewater.



**Figure 3.1** Photo of the instruments used (a) HACH DR2400 Spectrophotometer (b) HACH 2100P Turbidimeter (c) Vacuum pump (Rocker 600)

### 3.3 Analytical Method

Initially the samples were measured for pH, analyzed for turbidity, chemical oxygen demand (COD) and total suspended solids (TSS). The performance of the Fenton reagent oxidation was characterized later based on four parameters, namely turbidity, COD and TSS using the methods as described in the Standard Method for the Examination of Water and Wastewater (APHA, 1985). Turbidity was analyzed using Turbidity Meter. UV absorbance was analyzed based on absorbance of wavelength



286.5nm using Shimadzu UV 2450 UV/ Vis Spectrophotometer. COD value was analyzed using HACH DR-2400 Spectrophotometer. TSS value was analyzed using METTLER TOLEDO analytical balance.

### **3.3.1 Turbidity**

Turbidity of the sample was analyzed using turbidity meter. Seventeen round sample cell were use to determine turbidity value of each sample. The first round sample cell was used as blank and another sixteen were used as sample run. 10 mL of deionized water was added into the first round sample cell as blank and 10 mL of sample was added into the each sixteen round sample cell. Then samples were analyzed using that turbidity meter to determine turbidity value of each sample.

### **3.3.2 Chemical Oxygen Demand (COD)**

Chemical Oxygen Demand (COD) was analyzed using HACH Method. Seventeen HACH test tubes were prepared (consists of concentrated sulphuric acid and standard potassium dichromate). The first test tube was used as blank and other sixteen were used as sample run. Sample was diluted into with the dilution factor of 9. Then, 2 mL of deionized water was added into the first test tube as blank and 2 mL of diluted sample was added into the each sixteen test tube. The mixtures of seventeen test tubes were refluxed for 2 hours in HACH COD Digestion Reactor. Then the samples were analyzed using DR2400 Spectrophotometer to determine the COD content in each sample. For details analytical method for COD refer to Appendix A.

### 3.3.3 Total Suspended Solids (TSS)

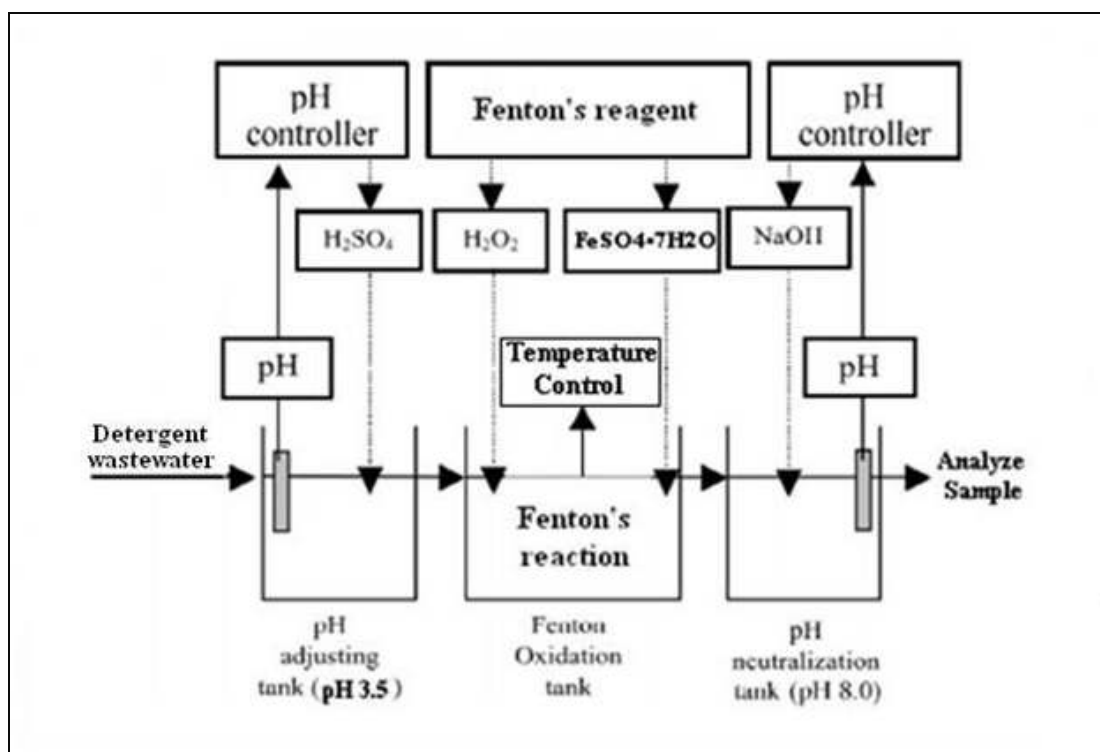
Suspended solid was analyzed using gravimetric method (APHA, 1985). Sixteen glass fiber filter papers were weighted and placed in an oven for 1 hour at 105°C. 10 mL sample was then poured into the pump vacuum and left it for a few minutes. After the sample was pumped entirely then the filter paper was placed back in an oven for 1 hour at 105°C again. The filter paper was weighted for second time. The mass of the suspended solids was measured by subtracting the first measuring mass from second measuring mass. For details analytical method for TSS refer to Appendix A.

## 3.4 Sample Preparation

As mentioned earlier, samples of industrial detergent wastewater used in the experiments were collected from a detergent production plant owned by FPG Oleochemicals Sdn. Bhd. on 17 December 2009. The sample used in the oxidation experiments was firstly characterized in terms of pH, TSS, COD and turbidity.

## 3.5 Experimental Procedure

The experimental work was mainly divided into three parts with a total of 15 experimental runs. They were Fenton reagent study on effect of H<sub>2</sub>O<sub>2</sub> dosages, Fe<sup>2+</sup> dosages and effect of temperature towards turbidity, COD and TSS removals. A set of 15 experimental runs was carried out at different H<sub>2</sub>O<sub>2</sub> and Fe<sup>2+</sup> concentration. The H<sub>2</sub>O<sub>2</sub> concentration ranged from 1000 mg/L to 1650 mg/L and Fe<sup>2+</sup> ranged from 500 mg/L to 1300 mg/L. While temperature ranged from 25°C until 65°C. The Schematic diagram of experiment procedure can see in Figure 3.2.



**Figure 3.2** Schematic diagram of experiment procedure

### 3.5.1 Effect of Dosages

The experiment (Figure 3.3(a)) was conducted batch-wise using Jar Test apparatus with industrial detergent wastewater volume of 1 L.



**Figure 3.3 (a)** Photo of the reactor used in the Fenton reagent study on effect of dosages

The pH was set at pH 3.5 using hydrochloric acid (HCl) and the iron dosage at appropriate dosage was initially added to the solution. The reaction was carried out under ambient laboratory light. Fenton reactions started with the addition of predetermined dosage of  $\text{H}_2\text{O}_2$ . After 3 hour, the solution was set to pH 7.5 using sodium hydroxide (NaOH). The solution was then slow mixed for 15 minutes and was let settled for another 45 minutes. Sample was then taken from the solution and analyzed for turbidity, COD and TSS. The total design outlined in Table 3.1 below.

**Table 3.1:** Experimental design used in the Fenton oxidation study on effect of dosages

Number	Dosage mg/L		Number	Dosage mg/L	
	$\text{Fe}^{2+}$	$\text{H}_2\text{O}_2$		$\text{Fe}^{2+}$	$\text{H}_2\text{O}_2$
<b>1</b>	<b>500</b>	1000	<b>6</b>	500	<b>1650</b>
<b>2</b>	<b>500</b>	1200	<b>7</b>	700	<b>1650</b>
<b>3</b>	<b>500</b>	1400	<b>8</b>	900	<b>1650</b>
<b>4</b>	<b>500</b>	1600	<b>9</b>	1100	<b>1650</b>
<b>5</b>	<b>500</b>	1800	<b>10</b>	1300	<b>1650</b>

### 3.5.2 Effect of Temperature

The experiment (Figure 3.3 (b)) was conducted batch-wise using 2L conical flask with industrial detergent wastewater volume of 1L in the incubator shaker with different temperature.



**Figure 3.3 (b)** Photo of the reactor used in the Fenton reagent study on effect of temperature

Experiment runs by selected the best dosages in removal the turbidity, COD and TSS of the samples based on the effect of dosages experiment. The pH was set at pH 3.5 using hydrochloric acid (HCl) and the iron dosage at appropriate dosage was initially added to the solution. Fenton reactions started with the addition of predetermined dosage of  $H_2O_2$ . After 3 hour, the solution was set to pH 7.5 using sodium hydroxide (NaOH). The solution was then slow mixed for 15 minutes and was let settled for another 45 minutes. The total design outlined in Table 3.2.

**Table 3.2:** Experimental design used in the Fenton oxidation study on effect of temperature

Number	Dosage mg/L		Temperature, °C
	Fe <sup>2+</sup>	H <sub>2</sub> O <sub>2</sub>	
11	1300	1650	25
12	1300	1650	35
13	1300	1650	45
14	1300	1650	55
15	1300	1650	65

### 3.6 Data Analysis

For quantitative analysis, the percentage of removal of industrial detergent wastewater can be calculated by using Equation 3.1-3.4.

#### 3.6.1 Turbidity

$$\% \text{ Removal} = \frac{T_o - T_t}{T_o} \times 100\% \quad 3.1$$

Where,

T<sub>o</sub> = initial turbidity of sample

T<sub>t</sub> = turbidity of sample at time t

### 3.6.2 Chemical Oxygen Demand (COD)

$$\% \text{ Removal} = \frac{COD_o - COD_t}{COD_o} \times 100\% \quad 3.2$$

Where,

$COD_o$  = initial turbidity of sample

$COD_t$  = turbidity of sample at time t

### 3.6.3 Total Suspended Solids (TSS)

$$mg \frac{TSS}{L} = \frac{(A - B) \times 100}{\text{sample volume, mL}} \quad 3.3$$

Where,

A = weight of filter + dried residue,mg

B = weight of filter

$$\% \text{ Removal} = \frac{TSS_o - TSS_t}{TSS_o} \times 100\% \quad 3.4$$

Where,

$TSS_o$  = initial turbidity of sample

$TSS_t$  = turbidity of sample at time t

## **CHAPTER 4**

### **RESULTS AND DISCUSSION**

#### **4.1 Introduction**

This chapter presents the experimental results of the study. As mentioned earlier, the experiments were conducted to explore the behavior of the Fenton reagent in treating industrial detergent wastewater. In particular, the effect of H<sub>2</sub>O<sub>2</sub> dosage, Fe<sup>2+</sup> dosage and temperature on this chemical treatment behavior was investigated. The parameters used to assess the performance of the reagent were COD, TSS and turbidity of the sample.

#### **4.2 Wastewater Characteristics**

The detergent wastewater was taken from FPG Oleochemicals Sdn. Bhd., in Kuantan Port area. The characteristic of the industrial detergent wastewater are shown in Table 4.1 below.



**Table 4.1:** Characteristics of the industrial detergent wastewater

Parameters	unit	Detergent wastewater effluent	Standards	
			A	B
pH	pH	8.1	6.0 – 9.0	5.5 – 9.0
Chemical Oxygen Demand	mg/L	12 920	< 50	< 100
Suspended Solid	mg/L	161	< 50	< 100
Turbidity	NTU	1320	< 50	< 50

### 4.3 General Overview

The results of the study were shown in Table 4.2.1 and 4.2.2. The range of percentage removal in the effect of dosages and temperature in Fenton reagent was 77-96.2% for COD, 85.1-98.1% for suspended solids and 81.1-99.6% for turbidity. The range of final COD was 49-297 mg/L, 3-24 mg/L for suspended solids and 5.1-250 NTU for turbidity. The highest percentage of COD, TSS and turbidity removal was 96.2%, 87.6% and 99.5%, respectively. These were achieved at  $\text{Fe}^{2+}$  and  $\text{H}_2\text{O}_2$  dosage of 1300 mg/L and 1650 mg/L, with temperature 35°C respectively.

**Table 4.2.1:** The percentage removal of COD, Suspended Solids and turbidity for Fenton reagent on effect of dosages.

Number	Dosages, mg/L		% Removal		
	Fe <sup>2+</sup>	H <sub>2</sub> O <sub>2</sub>	COD	TSS	Turbidity
1	<b>500</b>	1000	77.0	96.9	81.1
2	<b>500</b>	1200	77.6	92.5	93.4
3	<b>500</b>	1400	86.8	93.8	96.9
4	<b>500</b>	1600	88.8	95.0	98.4
5	<b>500</b>	1800	91.4	96.3	99.6
6	500	<b>1650</b>	83.4	91.3	99.6
7	700	<b>1650</b>	84.9	94.4	98.3
8	900	<b>1650</b>	83.5	91.9	98.8
9	1100	<b>1650</b>	79.5	89.4	99.2
10	1300	<b>1650</b>	93.0	98.1	99.5

**Table 4.2.2:** The percentage removal of COD, Suspended Solids and turbidity for Fenton reagent on effect of temperature.

Number	Temperature, °C	Dosages, mg/L		% Removal		
		Fe <sup>2+</sup>	H <sub>2</sub> O <sub>2</sub>	COD	TSS	Turbidity
11	25	<b>1300</b>	<b>1650</b>	80.6	85.1	99.6
12	35	<b>1300</b>	<b>1650</b>	96.2	87.6	99.5
13	45	<b>1300</b>	<b>1650</b>	94.7	86.3	92.3
14	55	<b>1300</b>	<b>1650</b>	88.4	85.7	86.2
15	65	<b>1300</b>	<b>1650</b>	84.9	89.4	98.7

#### 4.4 Effect of pH

In this study, pH 3.5 is used because of from C-T Wang et.al, 2008 studied, pH 3.5 give high removal efficiency compare than other pH. This happened because of a low pH is favorable for the production of hydrogen peroxide where the conversion of dissolved oxygen to hydrogen peroxide consumes protons in acidic solution, according to equation ( $O_2 + 2H^+ + 2e^- \rightarrow H_2O_2$ ).

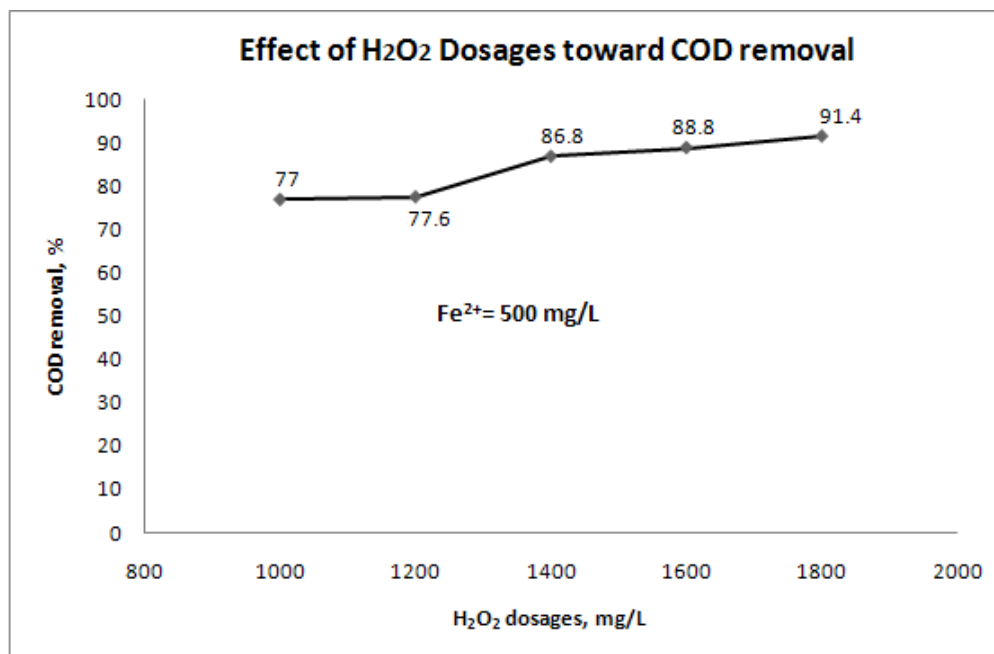
However, a low pH also promotes hydrogen evolution, according to equation ( $H^+ + 2e^- \rightarrow H_2$ ), reducing the number of active sites for generating hydrogen peroxide. So in a mildly acidic solution the removal efficiency increased with solution pH.

Based on Marco S. Lucas and José A. Peres (2009), for pH values lower than 2.0 the reaction of hydrogen peroxide with  $Fe^{2+}$  is seriously affected due to the formation of complex species  $[Fe(H_2O)_6]^{2+}$ , which reacts slower with peroxide when compared to that of  $[Fe(OH)(H_2O)_5]^{2+}$ .

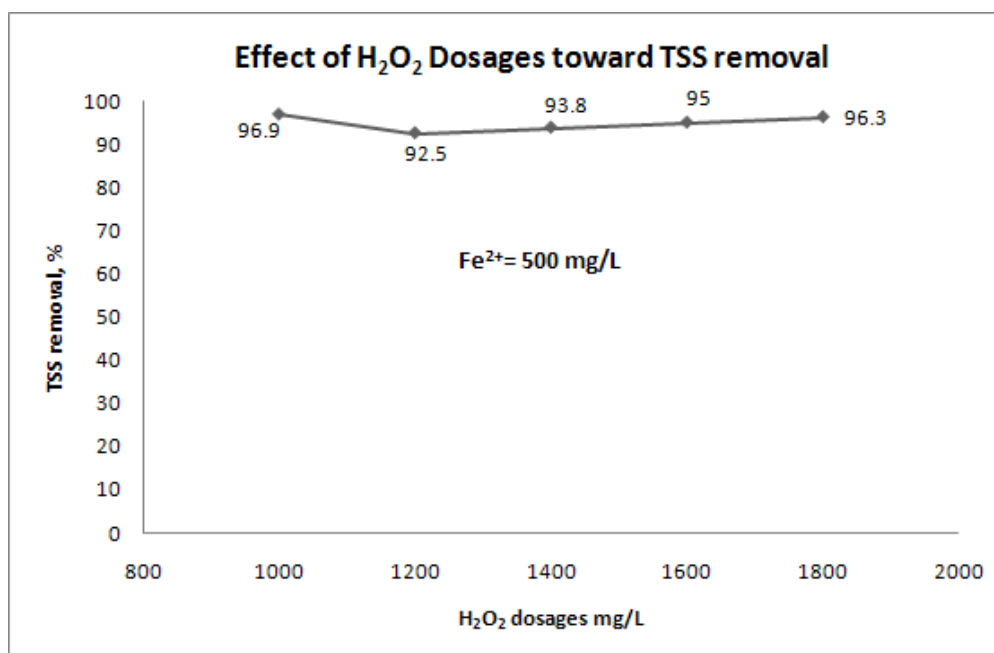
In addition, the peroxide gets solvated in the presence of high concentration of  $H^+$  ion to form stable peroxone ion  $[H_3O_2]^+$ . The peroxone ion it leads to an electrolytic behaviour on the part of hydrogen peroxide improving its stability and substantially reducing the reactivity with ferrous ion.

Therefore, the initial pH value has to be in the acidic range (3–4) to generate the maximum amount of  $HO\bullet$  and oxidize the organic compounds. In particular the optimal value of pH is 3.5, which is in agreement with previous results obtained in other studies using Fenton's reagent.

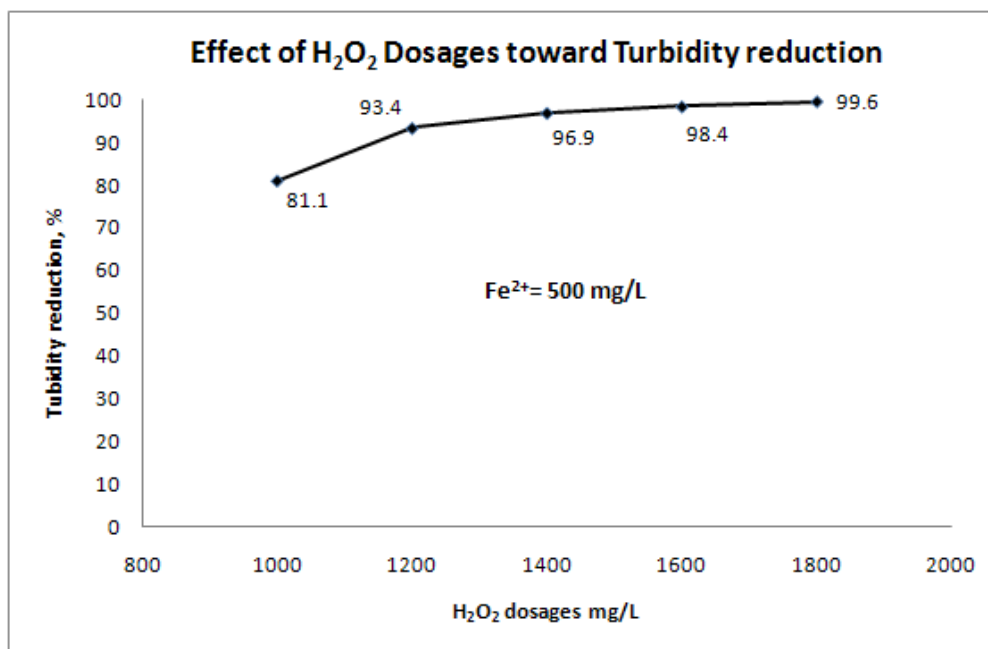
#### 4.5 Effect of H<sub>2</sub>O<sub>2</sub> Dosages



(a)



(b)



(c)

**Figure 4.1:** Chart for the effect of H<sub>2</sub>O<sub>2</sub> dosages toward (a) COD removal  
(b) TSS removal (c) turbidity reduction

The first parameter to consider in this Fenton oxidation is the amount of H<sub>2</sub>O<sub>2</sub> required to obtain the best efficiency in the treatment. In this sense, experiments were performed by fixing the Fe<sup>2+</sup> concentration and change the value of H<sub>2</sub>O<sub>2</sub> concentration as shown in figure 4.1 (a) (b) (c).

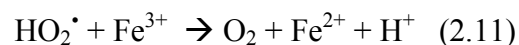
Based on that figure, significant enhancement of efficiency was observed when the H<sub>2</sub>O<sub>2</sub> concentration was increased from 1000 to 1800 mg/L where the removal of COD, TSS and turbidity increased progressively. Percentage removals were achieved until 91.4% in term of COD, 96.3 in term of TSS and 99.6% in term of turbidity.

Based on Y.Yang et. al (2009), increasing the dosage of hydrogen peroxide would result in more production of hydroxyl radicals (have high oxidation potential), which could enhance the oxidation capacity of the system, so that more organic compounds were eliminated.

However, at the end of the graph shown the percentages removal not increase to much compare than when dosages from 1000 mg/L until 1400 mg/L. This happened because of some of hydroxyl radicals would be scavenged by excess  $\text{H}_2\text{O}_2$  through the equation (2.6) which is

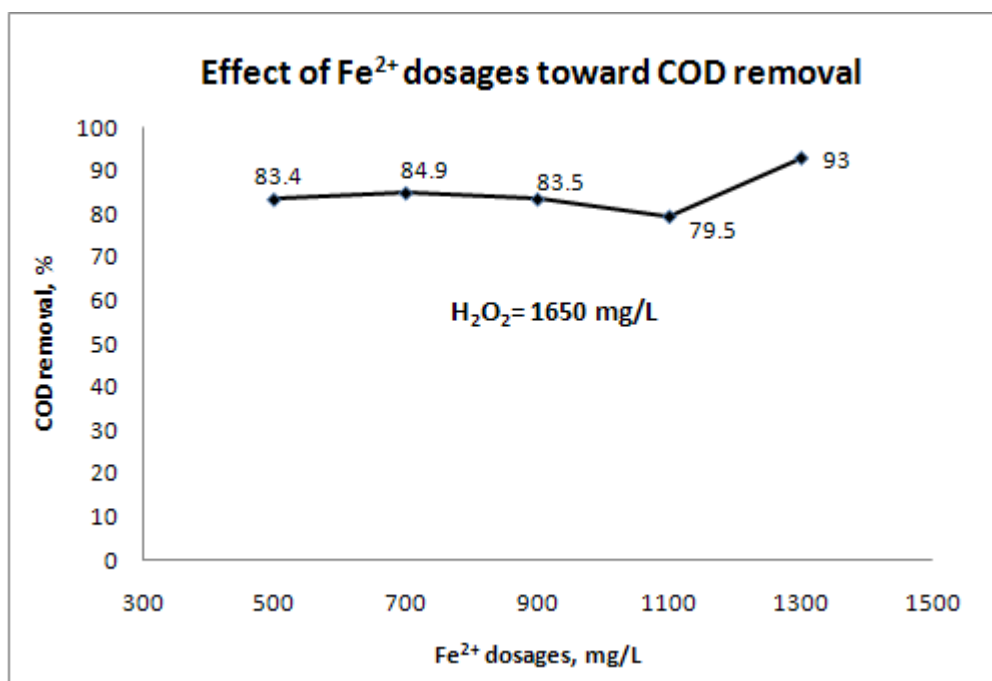


In addition, equation (2.11) would become independent of hydrogen peroxide when excess  $\text{H}_2\text{O}_2$  existed in the system (L. Lunar et. al, 2000).

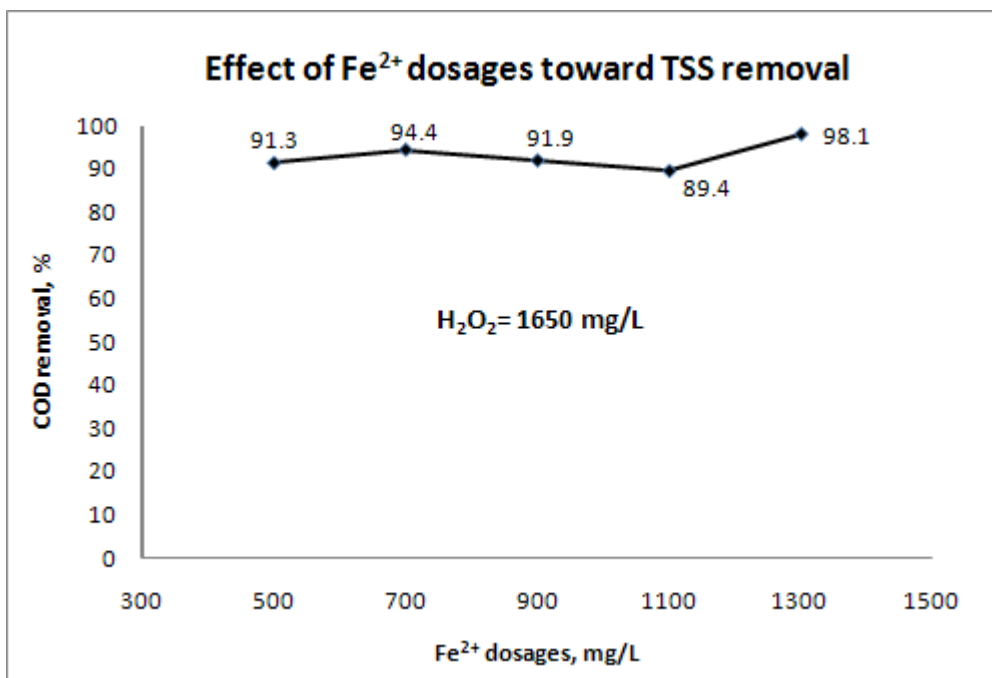


As a consequence, the variation of removal was slight when excess  $\text{H}_2\text{O}_2$  was added into the system. But the fact is the importance of  $\text{H}_2\text{O}_2$  is very much related to the concentration of  $\bullet\text{OH}$  generated. High activity of  $\bullet\text{OH}$ , removal will high.

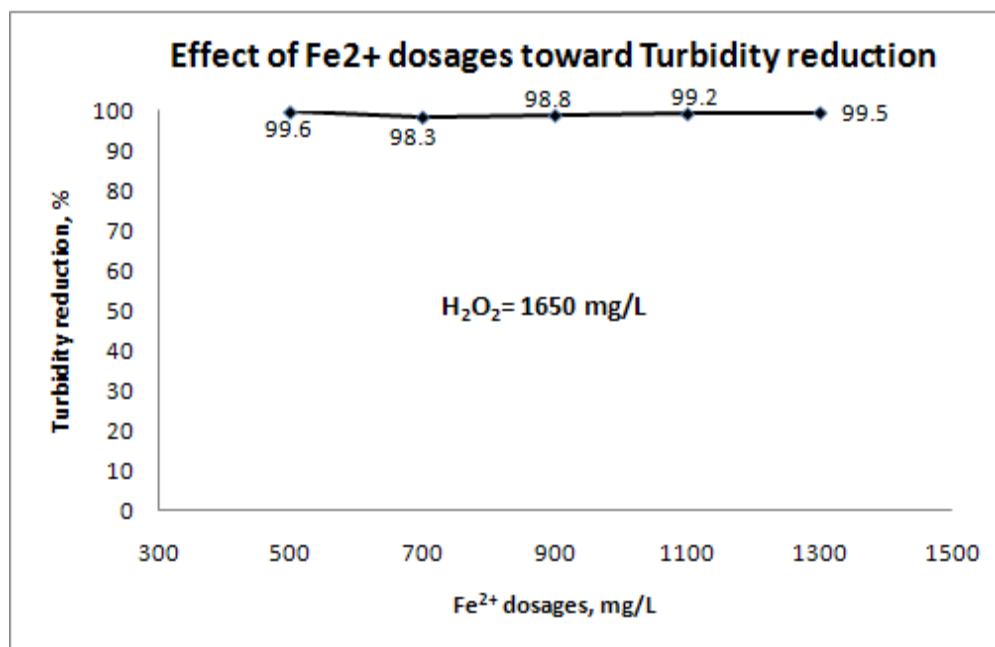
#### 4.6 Effect of $\text{Fe}^{2+}$ Dosages



(a)



(b)



(c)

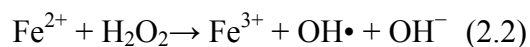
**Figure 4.2:** Chart for the effect of Fe<sup>2+</sup> dosages toward (a) COD removal (b) TSS removal (c) turbidity reduction

Another important effect to consider in this study is the amount of Fe<sup>2+</sup> required to obtain the best percentage removal efficiency in the treatment. Different from the previous experiment, this experiment were performed by fixing the H<sub>2</sub>O<sub>2</sub> concentration and change the amount of Fe<sup>2+</sup> concentration as shown in figure 4.2 (a) (b) (c).

Based on the figure 4.2 all the parameters are mutually proportional as Fe<sup>2+</sup> ion increase, COD, TSS and turbidity removal also increase. For this experiment, percentage removal of all the parameters is higher than the previous. As can see, percentage removal of COD was achieved until 93%, 98.1% for TSS and 99.5% in term of turbidity.

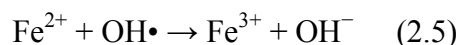
Increasing the amount of Fe<sup>2+</sup> concentration was effected the reaction of the reagent of Fenton in this treatment. So the results give more significant. Based on Z.P. Xing et al., 2009, oxidation rate increases with Fe<sup>2+</sup> concentration as the result of higher production of hydroxyl radicals according to equation (2.2):





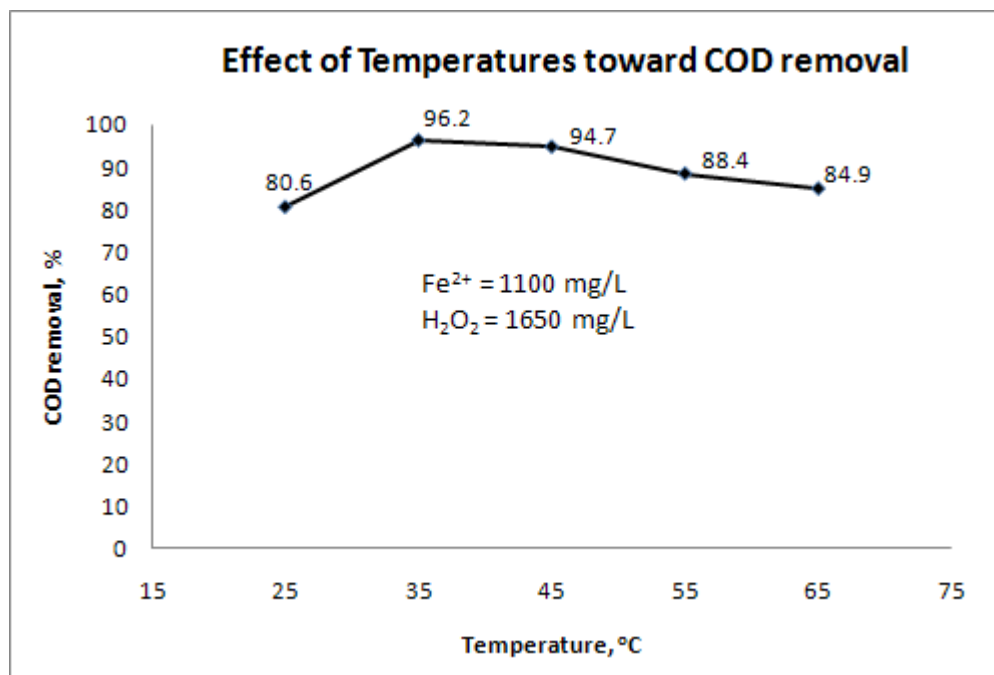
In fact, hydrogen peroxide is not a very strong oxidant. The oxidizing power of hydrogen peroxide was not enough to destroy large molecules. So it needs another chemical react with hydrogen peroxide to produce hydroxyl free radicals ions which is very strong oxidation agent. From the previous study using electrolysis by Xing et. al, 2009, amounts of ferrous ions added were related to the amounts of hydroxyl free radicals produced. This statement supported also according to equation (2.2). So can make assumption that, the removal efficiency of all the parameters should increase with the amount of added ferrous ions.

However, the COD and TSS removal efficiency did not increase significantly as the dosage of ferrous ions increased from 900 mg/L to 1300 mg/L and different with the graph of turbidity removal. This can be described by when the concentrations of the ferrous ions and hydroxyl free radicals are high; the ferrous ions can react with the hydroxyl free radicals according to Eq. (2.5).

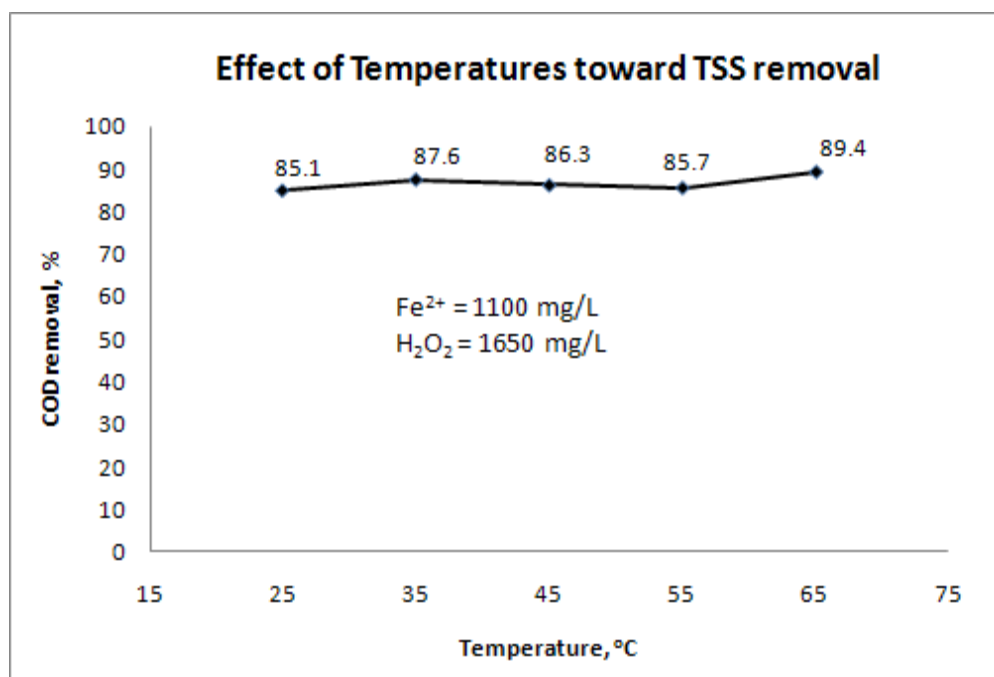


Hence, the excess ferrous ions consumed the hydroxyl free radicals with a high oxidative potential. In spite of the improvement in reaction kinetics achieved with higher  $\text{Fe}^{2+}$  concentration, the production of  $\text{Fe}^{3+}$  that will probably precipitate as  $\text{Fe}(\text{OH})_3$ . So increasing the precipitate down to the bottom, turbidity of water will decrease.

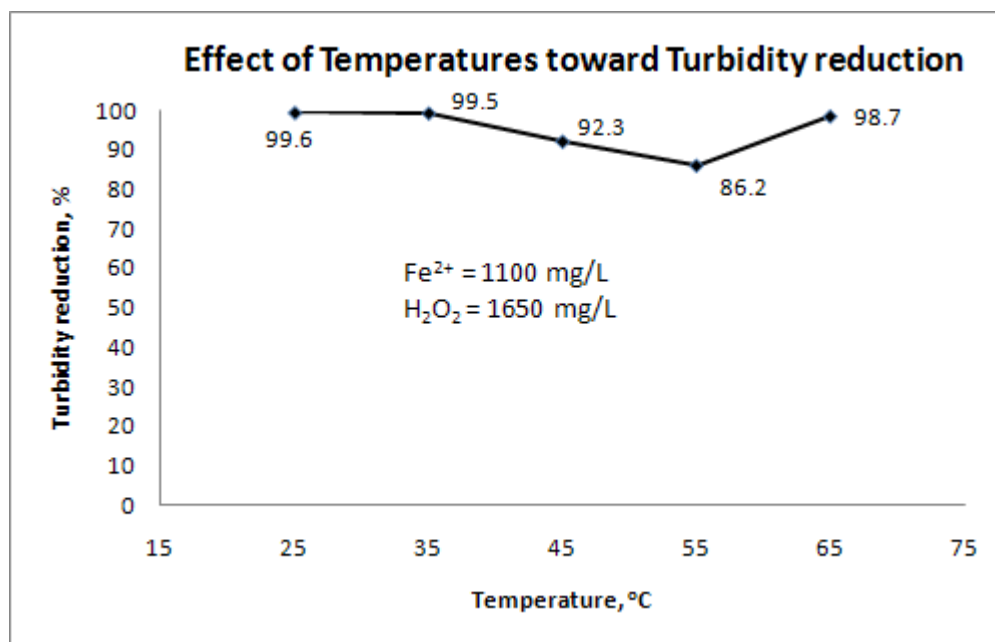
#### 4.7 Effect of Temperature



(a)



(b)



(c)

**Figure 4.3:** Chart for the effect of temperature toward (a) COD removal  
 (b) TSS removal (c) turbidity reduction

Figure 4.3 displays the effect of temperature on the removal of COD, TSS and turbidity of the sample. The temperatures used herein were 25°C, 35°C, 45°C, 55°C and 65°C. The result shows that the temperature negatively affected the COD, TSS and turbidity removal efficiency. The removal efficiencies decreased from 96.2% to 84.9% for COD, 87.6 % to 85.7% for TSS and 99.5% to 86.2% for turbidity as the temperature increased from 35°C to 65°C.

But by refer from the graph, at the temperature 25 °C, percentages of COD, TSS and turbidity removal are smaller than percentages removal for the temperature as increase from 35 °C to 65 °C . In this case, based on M.S. Lucas, J.A. Peres, 2009 for the lower temperature study, these things happened because of all the parameters reduction indicates that the extent of reaction was not complete within 3 h of reaction.

Since the COD, TSS and turbidity was removed by an indirect process, the removal rate was essentially limited by the rate of production of hydrogen peroxide. The negative effect of temperature on the production of hydrogen peroxide can be explained by the lower concentration of dissolved oxygen and the self-decomposition of hydrogen peroxide.

The concentration of hydrogen peroxide decreased as the temperature was increased because of the decrease in the concentration of dissolved oxygen. Hence, increasing the temperature lowered the COD, TSS and turbidity removal efficiency.

Additionally, the rate of self decomposition of the hydrogen peroxide to water and oxygen increased with the temperature. In this respectively, a lower temperature favored the production and accumulation of hydrogen peroxide, thereby increasing the rate of removal of COD.

From the result obtained for the turbidity and TSS reduction, there are errors at temperature 65°C. Based on theory, the reduction for both parameters must be decrease as temperature increase. This happened because of the equipment was breakdown almost 10 minutes during the experiment. So probably it was disturbed the reaction of the Fenton reagent in the sample during experiment progress.

#### 4.8 Comparison with Environmental Quality (Sewage and Industrial Effluents)

**Table 4.3.1:** Parameter Limits of Effluents of Standards A and B

Parameters	unit	Standards	
		A	B
pH	pH	6.0 – 9.0	5.5 – 9.0
Chemical Oxygen Demand (COD)	mg/L	< 50	< 100
Suspended Solid	mg/L	< 50	< 100
Turbidity	NTU	< 50	< 50

For full table of Parameter Limits of Effluents of Standards A and B, refer to the Appendix C.

**Table 4.3.2:** COD, TSS and turbidity value of the sample after treatment

Number	Effluent After Treatment, mg/L		
	COD	TSS	Turbidity
1	297	5	250
2	289	12	79.4
3	171	10	40.7
4	145	8	21.6
5	111	6	50
6	215	14	52
7	195	9	18.1
8	213	13	15.5
9	265	17	10.1
10	91	3	7.1
11	251	24	5.1
12	49	20	6.3
13	68	22	103
14	150	23	182
15	195	17	17.5

From the result obtained in the table 4.3.2, three of the COD and all the TSS value are obeying the standard B of the effluent. While at the optimum condition of this treatment which is at  $\text{Fe}^{2+}$  and  $\text{H}_2\text{O}_2$  dosage of 1300 mg/L and 1650 mg/L, at temperature  $35^\circ\text{C}$ , the effluent were achieved the standard A (as can see at the 12<sup>th</sup> sample in the table 4.3.2).

Here means that this effluent was fully treated and can release to the sea or river either along the stream have water intake or not. Therefore, proved that, Fenton reagent was a feasible treatment for industrial detergent wastewater.

## CHAPTER 5

### CONCLUSION & RECOMMENDATIONS

#### 5.1 Conclusion

From the results obtained, we can see the trend of the results in removing all the parameters are in between 77% until 99.6 %. The best dosage of Fenton reagent are when  $\text{H}_2\text{O}_2 = 1650 \text{ mg/L}$  and  $\text{Fe}^{2+} = 1300 \text{ mg/L}$  and also found, this treatment is optimum at temperature  $35^\circ\text{C}$ . These results are following the previous study by Marco S. Lucas et. al, 2009 where state that treatment efficiency was best at temperature  $35^\circ\text{C}$ .

In overall, feasibility or effectiveness of this treatment was controlled by the activity of hydroxyl free radicals ions (very strong oxidation agent) produce. Increasing the number of free radicals ions produce, increasing the ability of Fenton reagent treatment to destroy large molecules of pollutants in the wastewater. Found that, this reactivity of hydroxyl ions also depend on pH and temperature of the sample.

After compared with the Standard Limit of Effluent (EQA 1974), the COD and TSS value also was achieved the standard A of the effluent at the optimum condition. Means it is safe to be discharge to the river/downstream although have water supply intake in that area. The effluent will not harm the environment and human body.

As a conclusion, Fenton reagent was a feasible treatment for wastewater containing a large amount of surfactants and sulfate, allowing a significant decrease of COD, Suspended solids and turbidity of the wastewater. Besides approved that, this Fenton reagent was found to be very efficient for treatment industrial detergent wastewater.

## **5.2 Recommendations**

Based on the findings of this study, the followings are recommended for future researcher.

- I. This treatment can be use for treat another type of wastewater.
- II. Other parameters can be test in study the feasibility of this Fenton reagent.
- III. In order to achieve standard A and Standard B were outlined by the Department of Environment Malaysia (DOE), another treatment must be done (filtration, aerobic biological processes or other) after using this treatment.
- IV. Flocculation can be done after treatment to increase the effectiveness of removal suspended and insoluble matter then will reduce the settling time of the sludge.
- V. Further studies should be carried out on the sedimentation to determine the exact percentage of reduction is taken by hydroxyl oxidation or iron precipitation.
- VI. Alternative combination of UV + Fenton reagent, Solar + Fenton reagent and Ozone + Fenton reagent can be used to get more effective result.



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# **APPENDIX A**

## **Analytical Methodology**

## **A.1 CHEMICAL OXYGEN DEMAND (COD)**

### **APPARATUS/INSTRUMENTS**

COD Digestion Reactor  
Spectrophotometer, HACH DR/2400 @ DR/2800  
COD Digestion Reagent Vial LR @ HR  
Volumetric pipette, 2 mL  
Paper towel

### **PROCEDURE**

1. Homogenize 100 mL of sample for 30 seconds in a blender.  
  
\*For samples containing large amounts of solids, increase the homogenization time.
2. For the 200-15,000 mg/L range or to improve accuracy and reproducibility of the other ranges, pour the homogenized sample into a 250-mL beaker and gently stir with a magnetic stir plate.  
  
\*If the sample does not contain suspended solids, omit step 1 and step 2.
3. Turn on the COD Reactor. Preheat to 150°C. Place the safety shield in front of the reactor.
4. Remove the caps from two COD Digestion Reagent Vials.  
\*Be sure to use vials for the appropriate range.
5. Hold one vial at a 45-degree angle. Use a clean volumetric pipet to add 2.00 mL of sample to the vial. This is the prepared sample.
6. Hold a second vial at a 45-degree angle. Use a clean volumetric pipet to add 2.00 mL deionized water to the vial. This is the blank.
7. Cap the vials tightly. Rinse them with de-ionized water and wipe with a clean paper towel.
8. Hold the vials by the cap over a sink. Invert gently several times to mix. Place the vials in the preheated COD Reactor.  
  
\*The sample vials will become very hot during mixing.

9. Heat the vials for two hours.
10. Turn the reactor off. Wait about 20 minutes for the vials to cool to 120°C or less.
11. Invert each vial several times while still warm. Place the vials into a rack and cool to room temperature.
12. Proceed with Colorimetric Determination Method 8000.
13. Touch **Hach Programs**. Select program **430 COD LR** (Low Range) or **435 COD HR** (High Range/High Range Plus). Touch **Start**.
14. Clean the outside of the vials with a damp towel followed by a dry one to remove fingerprints or other marks.
15. Install the 16-mm adapter. Place the blank into the adapter.
16. Touch **Zero**. The display will show: **0 mg/L COD**.
17. When the timer beeps, place the sample vial into the adapter. Touch **Read**. Results will appear in mg/L COD.

## **A.2 TOTAL SUSPENDED SOLIDS (TSS)**

### **APPARATUS/INSTRUMENTS**

Glass fiber filter disk, 70 mm @ 90 mm – pre dry in the oven

Measuring cylinder, 100 mL

Pipette, 10 mL

Analytical balance

Oven – preheated to 103°C to 105°C

Desiccators

Buchner flask and funnel

Vacuum pump

Aluminum weighing dishes/Crucible dish

### **PROCEDURE**

1. Dry the filter disk in the oven at 103°C to 105°C for 1 hour, cool in a desiccator and weigh.
2. Assemble filtering apparatus and filter and begin suction. Wet filter with a small volume of distilled water to seat it.
3. Pipette 50 mL of water sample (mixed to ensure homogeneity) onto centre of filter disk in a buchner flask, using gentle suction (under vacuum).
4. Wash filter with three successive 10 mL volumes of distilled water, allowing complete drainage between washings, and continue suction for about 3 min after filtration is complete.
5. Carefully remove filter from filtration apparatus and transfer to aluminum weighing dish/crucible dish as a support.
6. Dry at least 1 hour at 103°C to 105°C in an oven, cool in a desiccator to balance temperature, and weigh.
7. Repeat the cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained.
8. Duplicate the test for each sample.

## **APPENDIX B**

### **Stock Solution Calculation**



**(A) Stock Solution of Hydrogen Peroxide**

Volume of H<sub>2</sub>O<sub>2</sub> needed is shown as below (Table 1).

$$\begin{aligned} H_2O_2 &= 30\% \text{ w/w} \\ &= \frac{30 \text{ g } H_2O_2}{100 \text{ g solution } H_2O_2} \end{aligned}$$

Thus, the calculation is shown as follow:

Concentration of H<sub>2</sub>O<sub>2</sub>

$$= \frac{30 \text{ g } H_2O_2}{100 \text{ g solution } H_2O_2} \times \frac{1.11 \times 10^3 \text{ g}}{1 \text{ L solution}}$$

= 333 mg/L

Volume of H<sub>2</sub>O<sub>2</sub>

$$= 1000 \text{ mg} \times \frac{\text{mL}}{333 \text{ mg}}$$

= 3.0 mL

**(B) Stock Solution of Ion Ferum**

Mass needed is shown as below (Table 2).

Concentration of Fe<sup>2+</sup> = 500 mg/L

$$\text{Concentration of Fe}^{2+} \text{ (in mM)} = \frac{500 \text{ mg/L}}{56 \text{ mg/mmol}}$$

= 8.9286 mmol

$$\text{Mass of FeSO}_4 \cdot 7\text{H}_2\text{O needed in 1L} = 8.9286 \text{ mmol} \times \frac{278.10 \text{ mg}}{\text{mL}} \times \frac{1 \text{ mL}}{\text{mmol}}$$

= 2.4822 g

**Table B.1:** Volume of hydrogen peroxide (30%) in 15 runs experiments

<b>No</b>	<b>H<sub>2</sub>O<sub>2</sub> Conc. (mg/L)</b>	<b>Volume (mL)</b>	<b>No</b>	<b>H<sub>2</sub>O<sub>2</sub> Conc. (mg/L)</b>	<b>Volume (mL)</b>
<b>1</b>	1000	3.0	<b>4</b>	1600	4.8
<b>2</b>	1200	3.6	<b>5</b>	1650	5
<b>3</b>	1400	4.2	<b>6</b>	1800	5.4

**Table B.2:** Mass of iron (II) in 15 runs experiments

<b>No</b>	<b>Fe<sup>2+</sup> Conc. (mg/L)</b>	<b>Mass (g)</b>	<b>No</b>	<b>Fe<sup>2+</sup> Conc. (mg/L)</b>	<b>Mass (g)</b>
1	500	2.4822	4	1100	5.4609
2	700	3.4751	5	1300	6.4538
3	900	4.4680			

## **APPENDIX C**

### **Parameter Limits of Effluents of Standards A and B**

**THIRD SCHEDULE**  
**ENVIRONMENTAL QUALITY ACT 1974**  
**ENVIRONMENTAL QUALITY (SEWAGE AND INDUSTRIAL EFFLUENTS)**  
**REGULATIONS 1979**  
**(REGULATIONS 8(1), 8(2), 8(3))**

**PARAMETER LIMITS OF EFFLUENTS OF STANDARDS A AND B**

Parameter	Unit	Standard	
		A	B
(i) Temperature	°C	40	40
(ii) pH value	-	6.0 - 9.0	5.5 - 9.0
(iii) BOD at 20°C	mg/ l	20	50
(iv) COD	mg/ l	50	100
(v) Suspended Solids	mg/ l	50	100
(vi) Mercury	mg/ l	0.005	0.05
(vii) Cadmium	mg/ l	0.01	0.02
(viii) Chromium, Hexavalent	mg/ l	0.05	0.05
(ix) Arsenic	mg/ l	0.05	0.10
(x) Cyanide	mg/ l	0.05	0.10
(xi) Lead	mg/ l	0.10	0.5
(xii) Chromium Trivalent	mg/ l	0.20	1.0
(xiii) Copper	mg/ l	0.20	1.0
(xiv) Manganese	mg/ l	0.20	1.0
(xv) Nickel	mg/ l	0.20	1.0
(xvi) Tin	mg/ l	0.20	1.0
(xvii) Zinc	mg/ l	2.0	2.0
(xviii) Boron	mg/ l	1.0	4.0
(xix) Iron (Fe)	mg/ l	1.0	5.0
(xx) Phenol	mg/ l	0.001	1.0
(xxi) Free Chlorine	mg/ l	1.0	2.0
(xxii) Sulphide	mg/ l	0.50	0.50
(xxiii) Oil and Grease	mg/ l	Not Detectable	10.0

## **APPENDIX D**

### **Results of Experiment**

**Table D.1:** The percentage removal of COD, Suspended Solids and Turbidity for effect of H<sub>2</sub>O<sub>2</sub> dosages.

Number	Dosages, mg/L		COD		TSS		Turbidity	
	Fe <sup>2+</sup>	H <sub>2</sub> O <sub>2</sub>	Value	% Removal	Value	% Removal	Value	% Removal
1	500	1000	297	77.0	5	96.9	250	81.1
2	500	1200	289	77.6	12	92.5	79.4	93.4
3	500	1400	171	86.8	10	93.8	40.7	96.9
4	500	1600	145	88.8	8	95.0	21.6	98.4
5	500	1800	111	91.4	6	96.3	50	99.6

**Table D.2:** The percentage removal of COD, Suspended Solids and Turbidity for effect of Fe<sup>2+</sup> dosages.

Number	Dosages, mg/L		COD		TSS		Turbidity	
	Fe <sup>2+</sup>	H <sub>2</sub> O <sub>2</sub>	Value	% Removal	Value	% Removal	Value	% Removal
1	500	1650	215	83.4	14	91.3	52	99.6
2	700	1650	195	84.9	9	94.4	18.1	98.3
3	900	1650	213	83.5	13	91.9	15.5	98.8
4	1100	1650	265	79.5	17	89.4	10.1	99.2
5	1300	1650	91	93.0	3	98.1	7.1	99.5

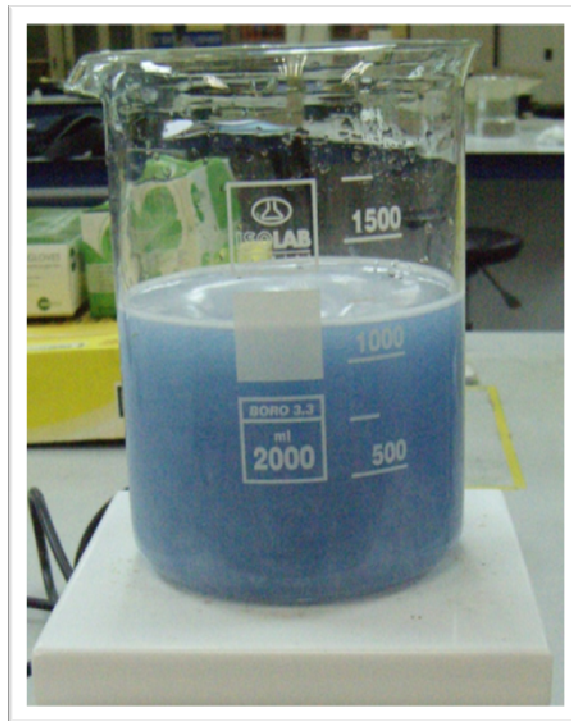
**Table D.3:** The percentage removal of COD, Suspended Solids and Turbidity for effect of Temperature.

Number	Temperature, °C	Dosages, mg/L		COD		ISS		Turbidity	
		Fe <sup>2+</sup>	H <sub>2</sub> O <sub>2</sub>	Value	% Removal	Value	% Removal	Value	% Removal
1	25	1300	1650	251	80.6	24	85.1	5.1	99.6
2	35	1300	1650	49	96.2	20	87.6	6.3	99.5
3	45	1300	1650	68	94.7	22	86.3	103	92.3
4	55	1300	1650	150	88.4	23	85.7	182	86.2
5	65	1300	1650	195	84.9	17	89.4	17.5	98.7

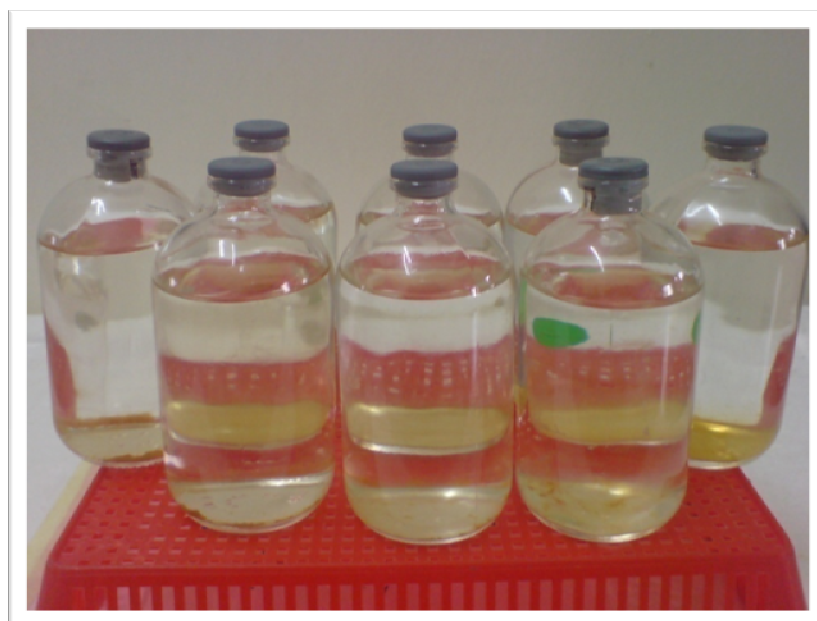
## **APPENDIX E**

### **Pictures of Sample**





**Figure E.1:** Industrial detergent wastewater before treatment



**Figure E.2:** Industrial detergent wastewater after treatment