

**TEXTILE WASTEWATER TREATMENT BY
ADVANCED OXIDATION PROCESS: FENTON PROCESS**

ADVANCED OXIDATION PROCESS: FENTON PROCESS

by

Khairul Amin B Umar @ Adli

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Civil Engineering)

JANUARY 2009

Universiti Teknologi PETRONAS

Bandar Seri Iskandar

31750 Tronoh

Perak Darul Ridzuan

**TEXTILE WASTEWATER TREATMENT BY
ADVANCED OXIDATION PROCESS: FENTON PROCESS**

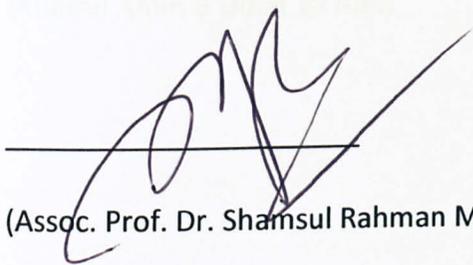
By

Khairul Amin B Umar @ Adli

(8682)

A project dissertation submitted to the
Civil Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfillment of the requirement for the
Bachelor of Engineering (Hons.)
(Civil Engineering)

Approved by,



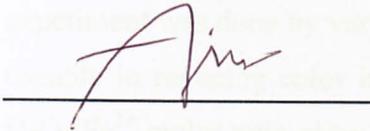
(Assoc. Prof. Dr. Shamsul Rahman Mohamed Kutty)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK

January 2009

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contain herein not been undertaken or done by unspecified sources or persons.



(Khairul Amin B Umar @ Adli)

ABSTRACT

Textile wastewaters are emerging contaminants in the aquatic environment because of their adverse effects on aquatic life and human. The organic substances that may be resistant to biological degradation make it hard to treat. With the existence of large mixture of dyes and chemicals addition, it is not only considered as liquid waste but as chemical composition as well. Textile wastewater has high COD and high color hence is difficult to treat biologically. The research examined the effect of operating conditions ($\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio, reaction time and pH) on the Fenton process of a textile wastewater. The experiment was started by taking the initial value of COD and color as a comparison with the final measurement of COD and color after the Fenton process. First batch of the experiment were done by adjusting the pH to 3 by adding HCl into the sample. The experiment continued by addition of H_2O_2 and FeSO_4 and at the same time, the jar that put under the floc jar tester was started to be stir. 50 ml of samples were taken at intervals 15, 30, 60, 90 and 120 minutes for COD and color measurement. The experiments are varied by using different volume of H_2O_2 and FeSO_4 . The optimum $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio was identified and used for the second batch of experiment. The second batch of experiment was done by varying the pH to 2, 4, 5, and 6. The graphs show that Fenton process is capable in reducing color in textile wastewater but not a great method in treating COD. The $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio plays a major role in this process and variation of pH also influencing the changed of color and COD. Reaction of time is not affecting much in this treatment as the result started to change immediately after the first 15 minutes and not showing a gradual changing for the next minutes. As the conclusion, the Advanced Oxidation Process of Fenton method is an efficient way of treating color but not the COD. The usage of Fenton method can be commercialized and can be applied in the textile wastewater treatment plant with addition of some advance research in dealing with COD. Lastly, the objectives of the study which to investigate the effect of reaction time, Fenton's reagent dosage and pH on the efficiency of COD and color removal and second, to discover the effectiveness of Fenton method in treating textile wastewater were achieved.

ACKNOWLEDGEMENT

Most thankful and grateful to God who gave me the courage and strength to complete this Final Year Project. I will not forget the contribution of my FYP’s supervisor, Assoc. Prof. Dr. Shamsul Rahman Mohamed Kutty. The support that he gave, the guidance and advices to me in making this research a success are priceless. To all technician and staff from Civil Engineering Department, thank you for allowing the use of resources and equipment for this project. Not forget the help and commitment gave from my beloved friends, the time they spend accompanied me, the kindness of them letting me borrow staff and finance to complete this project. For sure, I will not forget their help. And last but not forgotten, to my beloved family, especially my mother for her moral support. May God reward generously all those who have been to guide, teach, support and involve in the journey of completing my project.

1.1 Objectives.....4

1.2 Scope of Project.....4

1.3 Feasibility of Studies.....4

CHAPTER 2: LITERATURE REVIEW.....5

2.1 Previous Research.....5

2.1.1 Non-ferrous Leadful Leachate Treatment through electro-Fenton Oxidation.....5

2.1.2 Adsorption of Textile Wastewater Using MIRA Coagulant.....5

2.1.3 Ferrate and photo-Fenton Oxidation of Textile Effluents.....5

2.2 Impact of Textile Wastewater.....7

2.3 Color.....7

2.4 Chemical Oxygen Demand (COD).....8

TABLE OF CONTENT

ABSTRACT.....	iv
ACKNOWLEDGEMENT.....	v
TABLE OF CONTENT.....	vi
LIST OF FIGURES.....	viii
LIST OF TABLES.....	ix
CHAPTER 1: INTRODUCTION.....	1
1.1 Background Studies.....	2
1.2 Problem Statement.....	3
1.3 Objectives.....	4
1.4 Scope of Studies.....	4
1.5 Feasibility of Studies.....	4
CHAPTER 2: LITERATURE REVIEW.....	5
2.1 Previous Studies.....	5
2.1.1 <i>Semi-aerobic Landfill Leachate Treatment through electro-Fenton Oxidation</i>	5
2.1.2 <i>Adsorption of Textile Wastewater Using MIRHA Columns</i>	5
2.1.3 <i>Fenton and photo-Fenton Oxidation of Textile Effleunts</i>	5
2.2 Impact of Textile Wastewater.....	7
2.3 Color.....	7
2.4 Chemical Oxygen Demand (COD).....	8

LIST OF FIGURES

- Figure 3.2.1 : Color measurement (sample cell)
- Figure 3.2.2 : Color measurement (Spectrometer)
- Figure 3.2.4 : Filtration of sample by vacuum pump
- Figure 3.2.5 : pH meter (METTLER-TOLEDO, MP230)
- Figure 3.2.6(a): pH setting
- Figure 3.2.6(a): Sample been stirred under Floc Jar Tester
- Figure 4.1.1 : Graph of combination of Final SCOD versus Time at pH3
- Figure 4.1.2 : Graph of combination of Final Color versus Time at pH3
- Figure 4.1.3 : Graph of combination of Final SCOD versus $H_2O_2/FeSO_4$ Volume at pH3
- Figure 4.1.4 : Graph of combination of Final Color versus $H_2O_2/FeSO_4$ Volume at pH3
- Figure 4.2.1 : Graph of combination of Final SCOD versus Time at pH2, 3, 4, 5, and 6
- Figure 4.2.2 : Graph of combination of Final Color versus Time at pH2, 3, 4, 5, and 6
- Table 4.3.1 : Result of Final Color at pH2
- Table 4.3.2 : Result of Final SCOD at pH2
- Table 4.3.3 : Result of Final Color at pH3
- Table 4.3.4 : Result of Final SCOD at pH3
- Table 4.3.5 : Result of Final Color at pH4
- Table 4.3.6 : Result of Final SCOD at pH4
- Table 4.3.7 : Result of Final Color at pH5
- Table 4.3.8 : Result of Final SCOD at pH5
- Table 4.3.9 : Result of Final Color at pH6

LIST OF TABLES

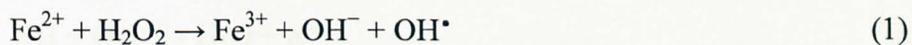
- Table 2.2.1 : Characteristic of Textile Wastewater from Batik Industry at Chemor, Perak
- Table 4.1.1 : Varied Volume of H_2O_2 and FeSO_4 used in the first batch of experiments
- Table 4.1.2 : Result of Final SCOD at pH3 with molar ratio $\text{H}_2\text{O}_2/\text{Fe}^{2+} = 4.098:234.6$
- Table 4.1.3 : Result of Final Color at pH3 with molar ratio $\text{H}_2\text{O}_2/\text{Fe}^{2+} = 4.098:234.6$
- Table 4.1.4 : Result of Final SCOD at pH3 with molar ratio $\text{H}_2\text{O}_2/\text{Fe}^{2+} = 5.464:312.8$
- Table 4.1.5 : Result of Final Color at pH3 with molar ratio $\text{H}_2\text{O}_2/\text{Fe}^{2+} = 5.464:312.8$
- Table 4.1.6 : Result of Final SCOD at pH3 with molar ratio $\text{H}_2\text{O}_2/\text{Fe}^{2+} = 6.83:391$
- Table 4.1.7 : Result of Final Color at pH3 with molar ratio $\text{H}_2\text{O}_2/\text{Fe}^{2+} = 6.83:391$
- Table 4.1.8 : Result of Final SCOD at pH3 with molar ratio $\text{H}_2\text{O}_2/\text{Fe}^{2+} = 8.916:469.2$
- Table 4.1.9 : Result of Final Color at pH3 with molar ratio $\text{H}_2\text{O}_2/\text{Fe}^{2+} = 8.916:469.2$
- Table 4.2.1 : Variation of pH
- Table 4.3.2 : Result of Final SCOD at pH2
- Table 4.3.3 : Result of Final Color at pH2
- Table 4.3.4 : Result of Final SCOD at pH4
- Table 4.3.5 : Result of Final Color at pH4
- Table 4.3.6 : Result of Final SCOD at pH5
- Table 4.3.7 : Result of Final Color at pH5
- Table 4.3.8 : Result of Final SCOD at pH6
- Table 4.3.9 : Result of Final Color at pH6

CHAPTER 1: INTRODUCTION

Textile wastewater can be defined as a high-strength wastewater that is hard to treat because of its organic substances that may be resistant to biological degradation. Advanced oxidation processes (AOPs) have received increased attention in the treatment of industrial wastewaters containing non-biodegradable substances. Basically, AOP's are based on the generation of highly reactive radical species, specifically the hydroxyl radical (OH) to react with organic compounds.

The Fenton process has attracted high interest in view of its high capacity to generate hydroxyl radicals through decomposition of H₂O₂ by Fe²⁺ in acidic conditions (H Lee., 2008). Fenton treatment is a powerful treatment choice for wastewater.

For the period of Fenton reaction, hydrogen peroxide is catalyzed by ferrous ions to generate hydroxyl radicals. In acidic medium, the oxidizing power of H₂O₂ can be strongly enhanced using Fenton method, where a small quantity of Fe²⁺ is added as catalyst to the contaminated solution to generate OH and Fe³⁺ from Fenton's reaction. Fenton process can proceed by the following chain reactions:



Hydroxyl radicals are also produced from water oxidation:



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



Also the produced ferric ion from Equation (1) can be reduced to ferrous ion:



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



The produced hydroxyl radicals would degrade organic matter in the textile wastewater:



Therefore, more ferrous ion dosage is needed to keep the moderate hydroxyl radicals reduction.

1.1 Background Studies

Every community produces both liquid and solid wastes and air emissions. The liquid waste, wastewater, is essentially the water supply of the community after it has been used in a



variety of application. From the standpoint of sources of generation, wastewater may be defined as a combination of the liquid or water-carried wastes removed from residences, institutions, and commercial and industrial establishments, together with such groundwater, surface water, and stormwater as may be present (Metcalf, 2004).

When untreated wastewater accumulates and is allowed to go septic, the decomposition of the organic matter it contains will lead to nuisance conditions including the production of malodorous gases. Wastewater also contains nutrients, which can stimulate the growth of aquatic plants, and may contain toxic compounds or compounds that potentially may be mutagenic or carcinogenic.

Wastewater treatment plants can be divided into three major types such as physical process, biological plant and physical/chemical plant. A physical process usually treats suspended, rather than dissolved pollutants. It may be a passive process, such as simply allowing suspended pollutants to settle out or float to the top naturally depending on whether they are more or less dense than water.

One of the main sources of wastewater is from textile industry. Since textile waste characteristic was similar to toxic waste due to content of dye such as lead, treatment of textile waste is obligatory. Recent, the method used to treat such wastewater is high in cost. Other alternative is to use low cost and easily obtained material. Advanced Oxidation Process was seen to be a potential material in this treatment. The aim of this study is to investigate the effectiveness of Fenton process in textile wastewater treatment.

1.2 Problem Statement

1) Textile wastewater contains a large mixture of dyes and chemicals additions that make the environmental challenge for textile industry not only as liquid waste but also in its chemical composition (Venceslau *et al.*, 1994).



2) The textile industry produces large volumes of bleaching effluents that contain appreciable quantities of organic compounds which are not easily amenable to chemical or biological treatment (Lin S.H et al., 1994).

3) Furthermore, treatment cost of textile wastewaters has been scaling rapidly in recent years. Hence a search for more cost effective treatment methods has to be done.

1.3 Objectives

1. Discover the feasibility of textile wastewater treatment by using Advanced Oxidation Process (AOP) on Fenton method.
2. Investigate the effect of reaction time, Fenton's reagent dosage and $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio on the efficiency of COD and color removal.

1.4 Scope of Studies

In this research, textile wastewater has been used as the specific heavy instead of using the raw wastewater in determining Fenton oxidation process. Investigations were mainly about effect of reaction time, Fenton's reagent dosage and $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio on the efficiency of COD and color removal. The use of Fenton's reagent as an oxidant for textile wastewater treatment is attractive due to the facts that: (1) iron is a highly abundant and non-toxic element, and (2) hydrogen peroxide is an easy to handle and environmentally benevolent oxidant.

1.5 Feasibility of Studies

The feasibility of the project is reasonable. A lot of time is spent during running the experiments of Fenton process.

CHAPTER 2: LITERATURE REVIEW

2.1 Previous Studies

2.1.1 Semi-aerobic Landfill Leachate Treatment through electro-Fenton Oxidation

This study investigates the combination of the classical Fenton reaction with electrochemical oxidation, the electro-Fenton process, for the treatment of semi aerobic landfill leachate collected from Pulau Burung Landfill Site (PBLs), Penang, Malaysia. The investigation was carried out using 500 ml batch reactors with aluminum electrodes to establish the optimal treatment conditions. The effects of applied current, pH, reaction time, electrodes separation distance, $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio, and H_2O_2 and Fe^{2+} concentrations, which are significant process parameters were investigated. According to the results obtained, electro-Fenton process was very efficient for the treatment of landfill leachate. Optimum oxidation efficiency was achieved when neither H_2O_2 nor Fe^{2+} were overdosed, so that the maximum amount of OH^\bullet radicals was available for the oxidation of organic compounds. The highest COD and color removals achieved were 92% and 93% respectively; at initial pH = 3, $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio = 1, applied current = 2 A, treatment duration = 30 min, and electrodes separation distance = 3 cm. (Mohajeri S. et al., 2007).

2.1.2 Adsorption of Textile Wastewater Using Microwave Incinerated Rice Husk Ash (MIRHA) Columns

Microwave Incinerated Rice Husk Ash (MIRHA), produced from the rice husk is one of the low-cost materials that were used as a sorbent of many type of wastewater. It was proven by lots of researchers around the world about the potential of the rice husk ash with different method of preparation. The objective of this project is to determine the effectiveness of MIRHA by using column method in treating textile wastewater. The effects of important factors, such as the flow rate and burning temperatures of MIRHA will be studied. The rice husk ash was burned in different controlled temperature to get them in two states which are amorphous state and silica



state. In addition, to ensure the MIRHA is in adsorption zone, the MIRHA need to be immersed in weak acid for 24hours. The bed heights of the columns are fixed so that the different effectiveness of state of MIRHA can be observed. The result obtained shows that MIRHA was unable to act as medium of absorption for textile wastewater. Instead of using textile wastewater, the effluent should be change by using other wastewater. Conclusion of this research is textile wastewater cannot be treated by using MIRHA column method (Amin K. 2008)

2.1.3 Fenton and photo-Fenton oxidation of textile effluents

The simultaneous use of Fenton reagent and irradiation for the treatment of textile wastewaters generated during a hydrogen peroxide bleaching process is investigated. The experimental conditions tested during this study provide the simultaneous occurrence of Fenton, Fenton-like and photo-Fenton reactions. The batch experimental results are assessed in terms of total organic carbon reduction. Identification of some of the chemical constituents of the effluent was performed by means of GC-MS. Other pollution related features of the initial effluent-like COD and color were also measured. The main parameters that govern the complex reactive system, i.e., light intensity, temperature, pH, Fe(II) and H₂O₂ initial concentrations have been studied. Concentrations of Fe(II) between 0 and 400 ppm, and H₂O₂ between 0 and 10,000 ppm were used. Temperatures above 25°C and up to 70°C show a beneficial effect on organic load reduction. A set of experiments was conducted under different light sources with the aim to ensure the efficiency of using solar light irradiation. The combination of Fenton, Fenton-like and photon-Fenton reactions has been proved to be highly effective for the treatment of such a type of wastewaters, and several advantages for the technique application arise from the study (Perez M. et al., 2001).



2.2 Impact of Textile Wastewater

Textile industry can be classified into three categories, cotton, woolen, and synthetic fibers depending upon the raw materials used (Babu et al., 2000). Textile industries consume large volumes of water and chemicals for wet processing of textiles. The chemical reagents used are very diverse in chemical composition, ranging from inorganic compounds to polymers and organic products. In general, the waste water from textile industry is characterized by high value of BOD, COD, pH, and color. Table 2.2.1 below shows characteristics of sample textile wastewater from Batik Industries at Chemor, Perak.

<i>Parameter</i>	<i>Value</i>
pH	11 - 13
COD	700 – 1200
SCOD	600 – 750
Color (Apparent)	7300 – 7750
Color (True)	7050 - 7500

Table 2.2.1

The major sources of waste from textile operations can be contributed to the following activities, raw wool scouring, yarn and fabric manufacture, wool finishing, woven fabric finishing, and knitted fabric finishing.

2.3 Color

The term “condition” was used along with composition and concentration to describe wastewater. Condition refers to the age of the wastewater, which is determined qualitatively by its color and odor. For textile wastewater, mainly, color comes from dye. (Metcalf, 2004). A very small amount of dye is visible in water, decreasing the transparency of the water. In the environment this leads to inhibition of sunlight penetration and therefore of photosynthesis. The measurement of color of raw textile wastewater before and after the test is being measured to compare the effectiveness of the method used.



2.4 Chemical Oxygen Demand (COD)

Bacteria require oxygen to breathe and oxidize organic substances; the total amount of which is called oxygen demand. The level of COD is a critically important factor in evaluating the extent of organic pollution in textile waste water. Substantial sewer surcharges are often imposed when the local permitted limits are exceeded. Depending on the types of fibers, dyes, and additives, various textile operations will routinely generate water having high levels of COD (Timothy et al., 1998).

In environmental chemistry, the chemical oxygen demand (COD) test is commonly used to indirectly measure the amount of organic compounds in water, making COD a useful measure of water quality. It is expressed in milligrams per liter (mg/L), which indicates the mass of oxygen consumed per liter of solution. Older references may express the units as parts per million (ppm) (Sawyer et al., 2003). Therefore, COD measures the potential overall oxygen requirements of the waste water sample.

2.5 Advanced Oxidation Process

Advanced oxidation processes have been employed for removal of refractory organic matter from wastewater (Mohajeri S. et al., 2007). The goal of any AOPs design is to generate and use hydroxyl free radical (HO) as strong oxidant to destroy compound that can not be oxidized by conventional oxidant. Advanced oxidation processes are characterized by production of OH radicals and selectivity of attack which is a useful attribute for an oxidant. The versatility of AOP is also enhanced by the fact that they offer different possible ways for OH radicals. One of methods in AOP is Fenton reaction that occurs while using hydrogen peroxide (H_2O_2) and iron ions as a catalyst of the process. The reaction leads to catalysis break-down of hydrogen peroxide in presence of ferrous ions Fe^{2+} , what results in free radicals generation OH (Adel Al-Kdasi et al., 2005).

CHAPTER 3: METHODOLOGY

3.1 Sampling

Textile wastewater samples were collected from Chemor Batik Industry in Perak, Malaysia. The textile wastewater characteristics are presented in Table 1. Collected samples were immediately moved to a cold room and stored at 4 °C.

3.2 Experimental Set

In this Fenton experiments, textile wastewater characteristics were measured by several test. This characteristics test results as shown in Table 1, are comparison of raw textile wastewater and textile wastewater after Fenton process.

3.2.1 Color

Textile wastewater was first diluted to ratio 1:50 in a 500ml conical flask by mixing 10ml of the textile wastewater with 490ml of distilled water. A sample cell was filled with 10ml of distilled water for blank sample. A second sample cell was filled with 10mL of wastewater sample (textile wastewater). The blank sample cell was wiped and it was inserted into the cell holder of Spectrometer (DR 2800) with the fill line facing right. The ZERO button was pressed and the display will show: 0 unit PtCo. Three readings were taken down and the average result is taken. The experiment was repeated by using effluent sample. The results then were multiplied with 50 to get the true value.



Figure 3.2.1



Figure 3.2.2

3.2.2 Biodegradable Oxygen Demand (BOD)

Samples were prepared and poured into the BOD bottles according to the volume needed. Blank samples were also prepared. After all samples were prepared, the initial DO for each sample was measured by the DO probe that was equipped with a stirring mechanism. The BOD bottles were then placed in the refrigerator at 20°C temperature and left for 5 days. After 5 days incubation, the final DO is measured by using the DO probe. The experiment was repeated by using effluent sample.

3.2.3 Chemical Oxygen Demand (COD)

A 2ml of wastewater sample (textile wastewater) was measured and poured into a test tube containing potassium dichromate. The test tube was then shaken thoroughly using 37600 Mixer. Heat was produced, indicating an exothermic process due to the reaction. All the test tubes together with a blank as an indicator were then put into the rotator (DRB 200) and left for 2 hours. Three readings are taken down by using Spectrometer (DR 2800) and the average was calculated.

3.2.4 Soluble Chemical Oxygen Demand (SCOD)

Wastewater samples were filtered through a filter paper by using vacuum pump. Only the soluble part is present in the filtrate and its COD was measured.



Figure 3.2.4

3.2.5 pH Measurement

By using METTLER – TOLEDO, MP230 the pH of textile wastewater had been measured. The pH meter was calibrated with standard buffers pH (4, 7 and 10) at 30°C (room temperature) before measurements.



Figure 3.2.5

3.2.6 Advanced Oxidation Process – Fenton Process

Experiment of Fenton oxidation of textile wastewater was carried out at laboratory scale. 500mL beaker was used as a batch reactor and was put under Floc Jar Tester. These experiments have been done in two batches. First batch was to identify the effect based on the ratio of H_2O_2 : Fe by fixing the pH value to pH3. Second batch was according to the best ratio of H_2O_2 : Fe in the first batch of experiment by varying the value of pH by 2, 4, 5 and 6.

For the first batch of experiments, 500ml of sample of textile wastewater was placed in a 500ml beaker and set the sample pH to 3. This step was done by adding HCl of high concentration about 15ml in one pour to reach the pH value under pH7 and continued by adding the HCl drop by drop until it the pH meter shows pH3(± 0.1) while it been stirred above the magnetic stirrer machine. The reason why the process of changing the pH to pH3 cannot be done by adding HCl drop by drop from the start was because if author done it that way, when the pH is reach pH7-8, it will turn the sample into jelly form, and the experiment cannot be continue. The beaker then was placed under the paddle of floc jar tester with the machine was set ran for 120 minutes with the speed 200rpm. Hydrogen Peroxide, H_2O_2 with concentration 35% and Iron Sulphate, $FeSO_4 \cdot 7H_2O$ had been

measured based on data in Table 4.1. The reaction time starts to be record when both reagents, H_2O_2 and $FeSO_4 \cdot 7H_2O$ have been mixed into the textile wastewater. By using pipette, samples of the mixture were taken at 15, 30, 60, 90 and 120 minutes with 50ml for each interval. Samples taken were put in a 100ml beaker and 20 drops of Sodium Hydroxide were added to stop the reaction of H_2O_2 in the samples. The samples then left for 3days to let ferum settled at the bottom of the beaker. Samples were then filtered by using vacuum pump with filter paper (Whatman: Purabid $45\mu m$) and COD and color of the sample were then to be measured and the experiment was repeated by using different ratio of H_2O_2 : Fe based on value in Table 3.1.

From the results got in experiment 1, the optimum ratio of H_2O_2 : Fe was been used to determining the effect of pH in second batch of experiment. Table 2 shows the different pH values used in experiment 2. Based on the optimum ratio of H_2O_2 : Fe which give the most significant change in COD value and color, experiment 3 have been done by varying the pH to 2, 4, 5, and 6. Results of the experiments were recorded and were discussed.

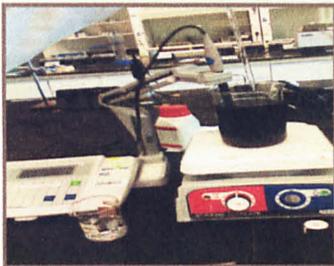


Figure 3.2.6(a)



Figure 3.2.6(b)

3.3 Hazard Analysis

The project conducted must comply with Universiti Teknologi PETRONAS Health, Safety and Environment (HSE) rules and regulation. The HSE rules and regulation are meant to prevent accident or injuries, to avoid any harm to students and people surrounding, to prevent properties



CHAPTER 4: RESULT AND DISCUSSION

4.1 Effect of H_2O_2/Fe^{2+} Molar Ratio

In first batch of experiments, the pH was set to 3 by adding HCl of high concentration. Below is the volume used in every experiment:

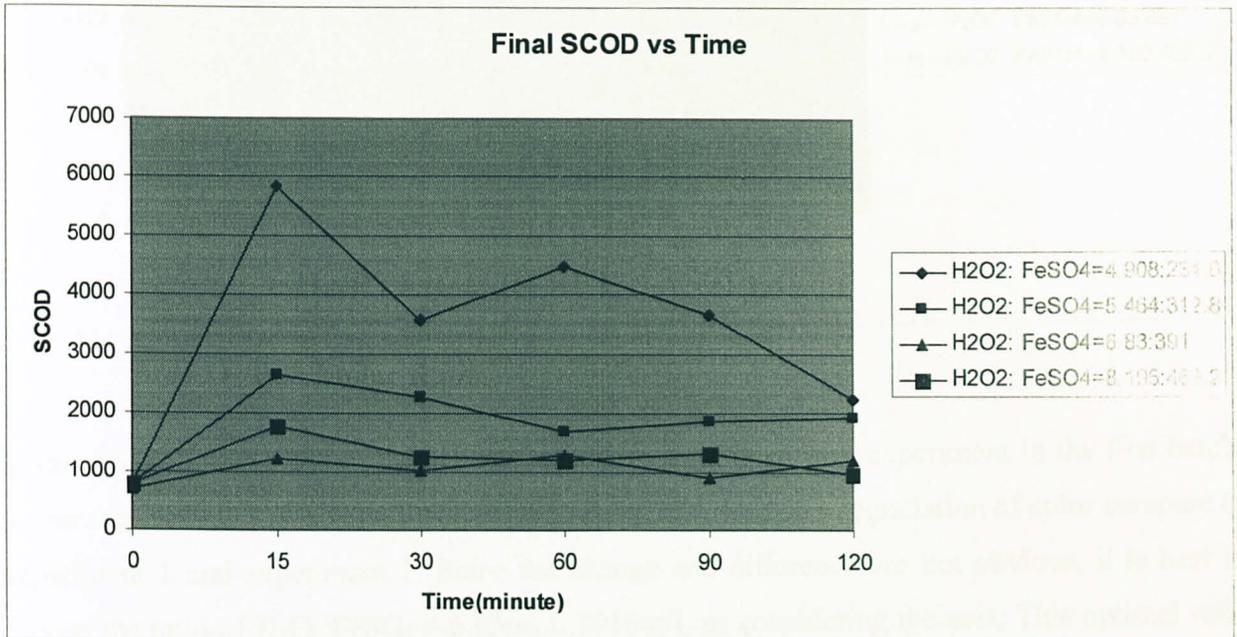


Figure 4.1.1

Fenton process involved two major chemicals that determining the operation cost as well as the efficiency which are iron and hydrogen peroxide (to determine the optimal H_2O_2/Fe^{2+} molar ratio) (Elmolla E., 2008), experiments were conducted at constant pH which is 3. The concentration of H_2O_2 also has been remained constant (35%). Figure 4.9 is a combination of the overall previous experiments. From here, it can be said that the best result is obtained from experiment 3, ratio of $H_2O_2/FeSO_4 = 6.83\text{ml/L}:391\text{mg/L}$ since it shows the lowest average of COD value compared to the other experiments. Even the ratio of $H_2O_2/FeSO_4$ has been increased in experiment 4, the result have no big different instead experiment 3 shows better and lower value of COD.

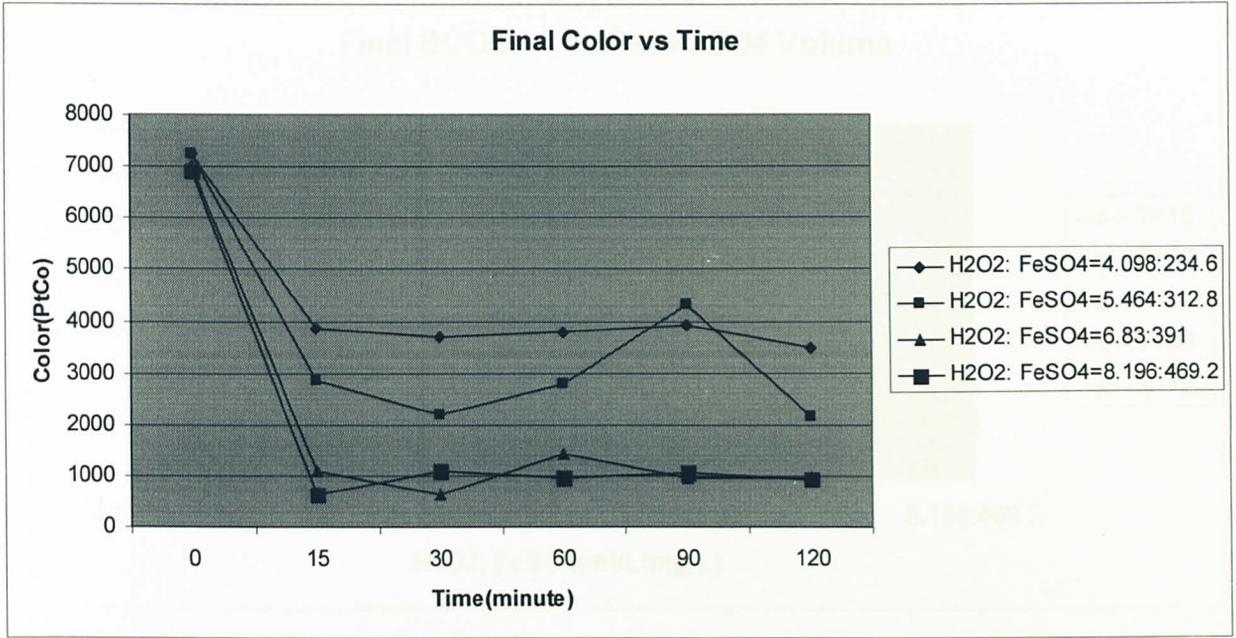


Figure 4.1.2

In this Figure 4.10, it shows the final color versus time of overall experiment in the first batch. As we can see, the experiment 3 and experiment 4 shows a good degradation of color compare to experiment 1 and experiment 2. Since the change and difference are not obvious, it is best to choose the ratio of $H_2O_2/FeSO_4 = 6.83\text{ml/L}:391\text{mg/L}$ as considering the cost. This optimal ratio of $H_2O_2/FeSO_4$ has been used as fixed ratio in the second batch of experiment but with different pH to determine the optimum pH in Fenton process to treat textile wastewater.

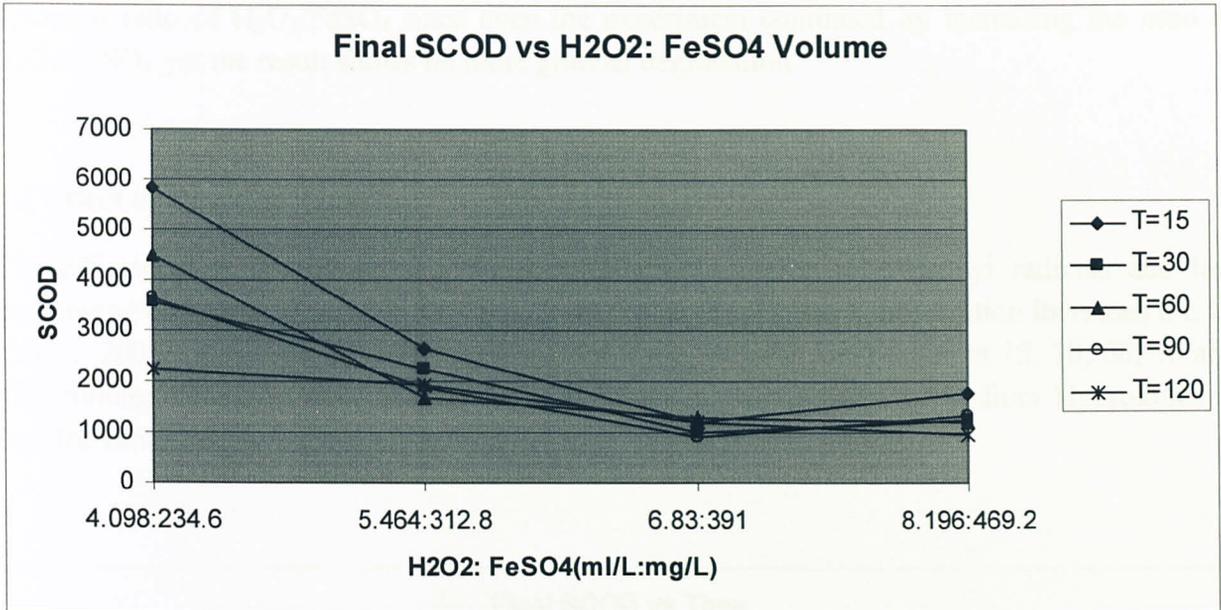


Figure 4.1.3

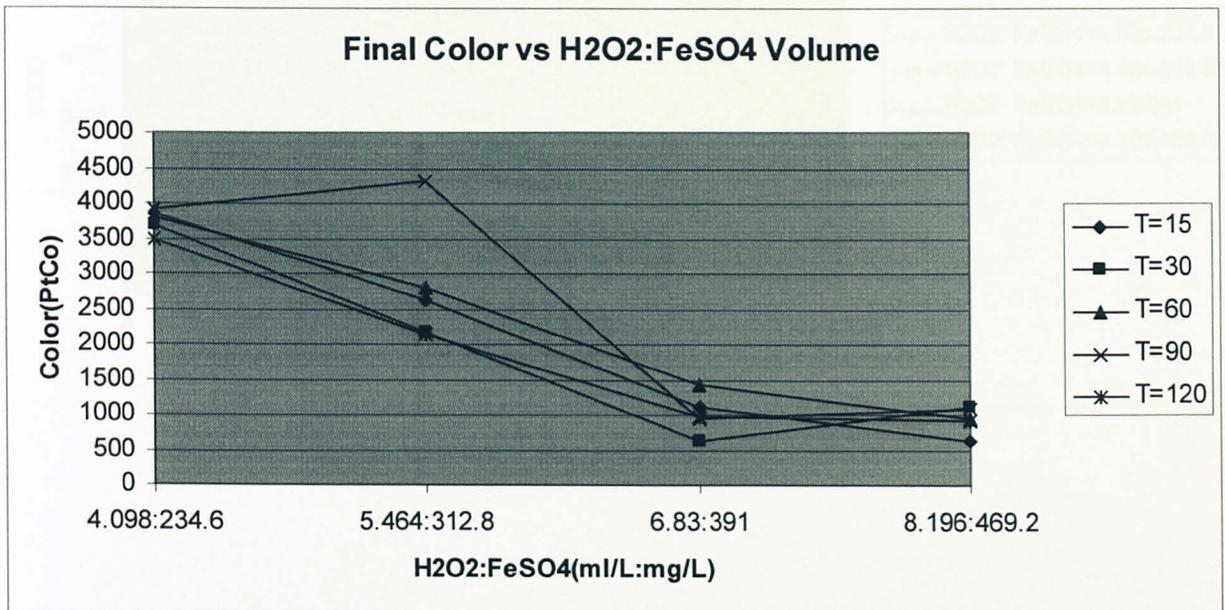


Figure 4.1.4

Figure 4.11 and Figure 4.12 show the final color and COD after Fenton process versus ratio of $H_2O_2/FeSO_4$. The figure above shows the reaction at experiment 3 can be determine as an

optimum ratio of $H_2O_2/FeSO_4$ since even the experiment continued by increasing the ratio of $H_2O_2/FeSO_4$, yet the result shows no more gradual degradation.

4.2 Effect of Reaction Time

The efficiency of Fenton process depends on the formation of hydroxyl radicals and less scavenging of hydroxyl radicals occurs as initial organic substrate concentration increases (A. G. Trovo., 2008). To observe the effect of reaction time, samples were taken at 15, 30, 60, 90 and 120 minutes interval. Every sample taken had been adding 20drops of Sodium Hydroxides to stop the ferum reaction for every interval.

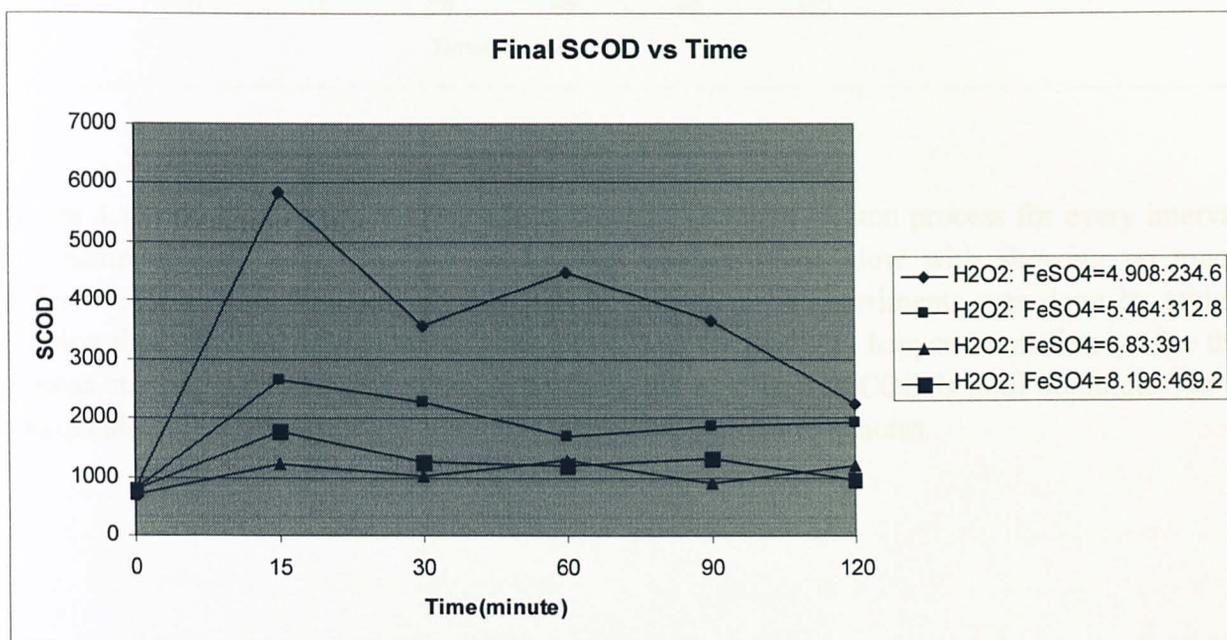


Figure 4.2.1

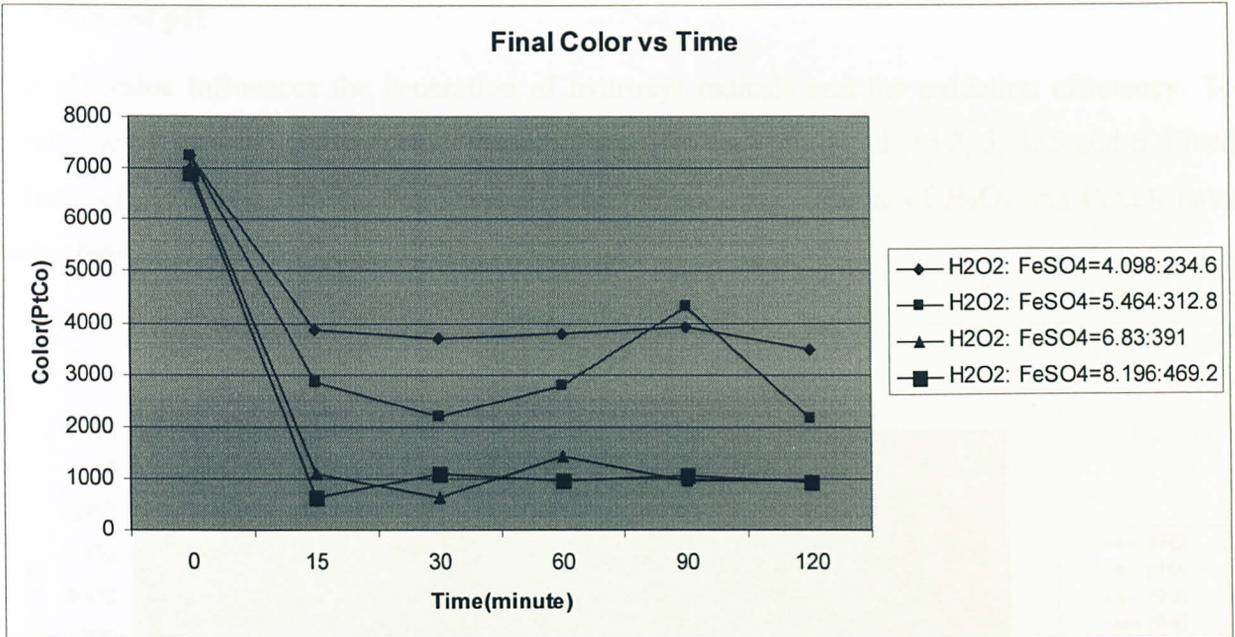


Figure 4.2.2

Figure 4.13 and 4.14 shows the residual color and COD after Fenton process for every interval that samples were taken. In figure 4.13, the reaction going slow with showing no much difference in change after minute 60. The second batch of experiments was done by taking reaction time just until 60minutes. Figure 4.14 tell us the reactions happen immediately after the process started, just like rapid mixing. But since the best time of COD is until 60minutes, so it was decided to take the reaction time until 60minutes and not 15minutes.

4.3 Effect of pH

The pH value influences the generation of hydroxyl radicals and the oxidation efficiency. To obtain the optimal pH, experiments were conducted by varying the pH to 2, 3, 4, 5 and 6. Fixed volume of H_2O_2 and $FeSO_4$ was being used as the optimum volume of H_2O_2 and $FeSO_4$ have been obtained in the first batch of the experiment.

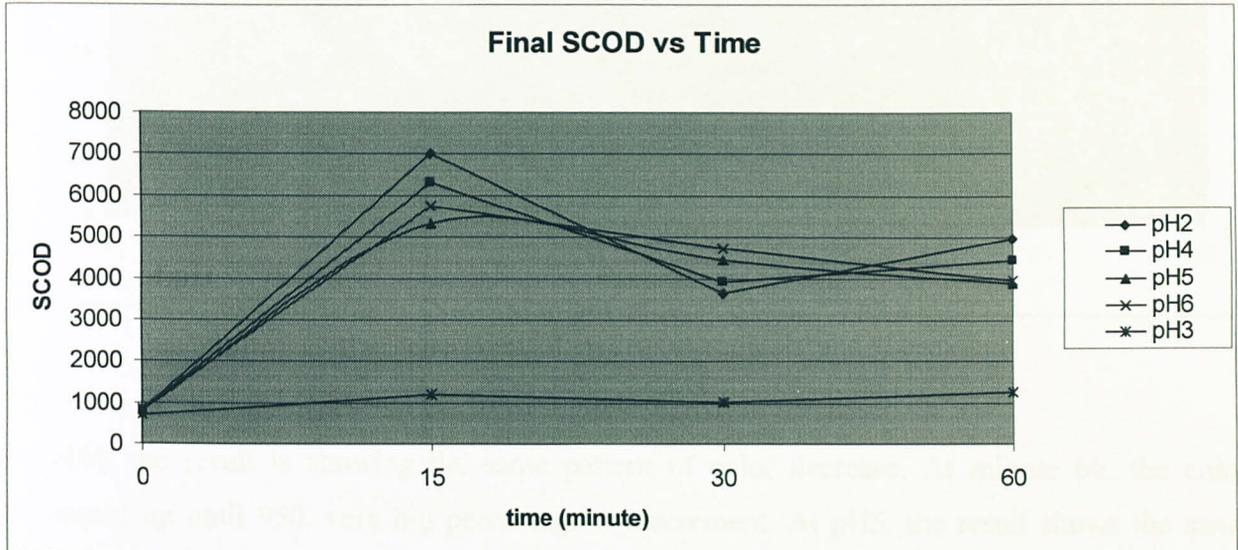


Figure 4.3.1

In this figure 4.3.1, it shows the different results obtained from varying the pH to 2, 3, 4, 5 and 6. Fenton process at pH2, compared to Fenton process at pH3, the SCOD results are larger. The initial SCOD(795) increased to 7015 which is much more higher than pH3. We can see clearly that experiment done at pH3 give the best result of all. The COD is not increased much compare to other. These results show that pH significantly influences COD degradation. At low pH, hydrogen peroxide can stay stable, probably because it solvates a proton to form oxonium ion (H_3O^+). An oxonium ion makes hydrogen peroxide electrophilic to enhance its stability and presumably to reduce substantially the reactivity with ferrous ion (Kwon, B.G., 1999).

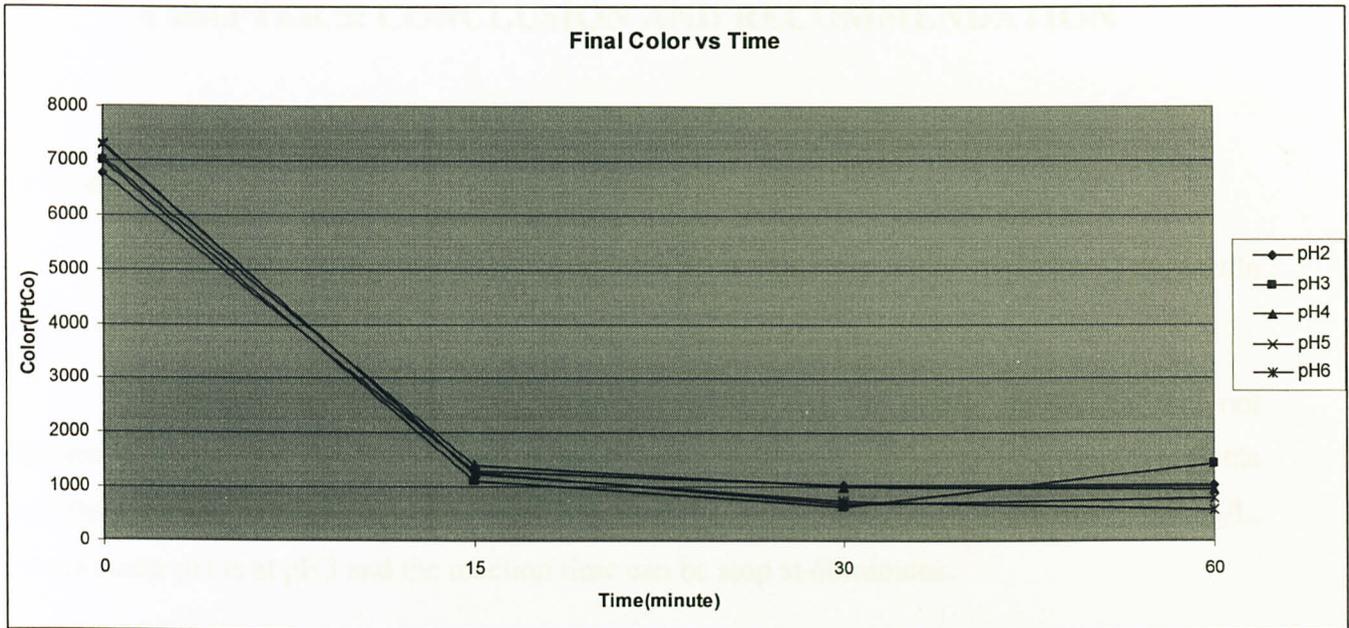


Figure 4.3.2

At pH4, the result is showing the same pattern of color decrease. At minute 60, the color decreased up until 950, very big percentage of decrement. At pH5, the result shows the same pattern as pattern in experiment done at pH4 in color decrement. But at minute 30, it reached the value of color which is 650. As been discussed above, pH significantly influences color decrement also. In figure 4.3.2, pH6 shows the lowest decrement at minute 60 which is 575.

CHAPTER 5: CONCLUSION AND RECOMMENDATION

5.1 Conclusion

From the analyses that have been conducted for textile wastewater, the color of the textile wastewater is decrease a lot but not the COD.

The Fenton method has a capability in treating color of textile wastewater but not efficient in degradation of COD. The $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio, pH used and time reaction affects the effectiveness of the Fenton process. The best $\text{H}_2\text{O}_2/\text{Fe}^{2+}$ molar ratio is 6.83ml/L:391mg/L, the optimum pH is at pH3 and the reaction time can be stop at 60minutes.

5.2 Recommendation

There is no restriction in improvement for this research. The recommendation for better advancement will benefit the wastewater industry as well as the researchers. Thus, the recommendations are as follows:

- Do advance research of why COD does not degrade in Fenton process.
- Experiment can be done by fixing either H_2O_2 or Fe^{2+} to find the optimum dosage and volume.
- Study the reasons why textile wastewater turns into jelly form at pH7-8 after additional of acid.

REFERENCE

- Al-Kdasi A., Idris A., Saed K. and Chuah T. G. (2005) *Treatment of Textile Wastewater by Advanced Oxidation Process – A Review*, 1
- A. G. Trovo., S. A. S. Melo, R. F. P. Nogueira,(2008) *Photodegradation of the Pharmacueticals Amoxicillin, Bezafibrate and Paracetamol by the photo-Fenton Process- Application to sewage treatment plant effluent*, Chemistry 198, p215-220.
- Amin K. (2008) *Adsorption of Textile Wastewater Using Microwave Incinerated Rice Husk Ash (MIRHA) Columns*, 1.
- Babu, B.V., Rana, H.T., Krishna, V.R., and Sharma, M. (2000). *COD Reduction of Reactive Dyeing Effluent from Cotton Textile Industry*. India: Birla Institute of Technology & Science.
- Elmolla E. (2008) *Optimization of Fenton Process for Treatment of Simulated Amoxicillin, Ampicillin and Cloxacillin Wastewater*, 5.
- H. Lee, M. Shoda, (2008) *Removal of COD and color from livestock wastewater by the Fenton method*, J. Hazard. Mater.1314-1319
- Lin SH, Peng CF. (1994) *Treatment of Textile Wastewater by Electrochemical Method*. Water Res 277–282.
- Kwon, B.G., Lee, D.S., Kang, N., Yoon, J., (1999) *Characteristics of p-chlorophenol Oxidation by Fenton’s reagent*. 2110-2118.
- Metcalf, Eddy, (2004). *Wastewater Engineering Treatment and Reuse*. Mc Graw Hill, p 1-2.



Mohajeri S., Hamidi A. A., Hasnain I., Rahman S. and Hung Y. T. (2007) *Semi-aerobic landfill leachate treatment through electro-Fenton oxidation*, 1.

Perez M., Torrades F., Domenech X., and Peral J. (2001) *Fenton and Photo-Fenton oxidation of textile effluent*, 1.

R. Munter. (2001) *Advanced oxidation processes-current status and prospects*, *Proc. Estonian Acad. Sci. Chem.* 59–80.

Venceslau M.C., Tom S. and Simon J.J. (1994), *Characterization of textile wastewaters- a review*, *Environmental Technology*, 15, 917-929

Table 4.1.1

Time (min)	Efficiency (%)
0	0
15	77.5
30	80.0
60	85.0
90	86.0
120	87.0

Table 4.1.2

APPENDIX

Experiment	H ₂ O ₂ Dosage (ml)	FeSO ₄ Dosage (mg)	H ₂ O ₂ Volume (ml/L)	FeSO ₄ Volume (mg/L)
Experiment 1	2.049	117.3	4.098	234.6
Experiment 2	2.732	156.4	5.464	312.8
Experiment 3	3.415	195.5	6.83	391
Experiment 4	4.098	234.6	8.196	469.2

Table 4.1.1

Time(minute)	SCOD
0	775
15	5830
30	3560
60	4475
90	3650
120	2235

Table 4.1.2

Time(minute)	Color(PtCo)
0	1445
15	770
30	740
60	760
90	785
120	695

Table 4.1.3

Time(minute)	COD
0	770
15	2640
30	2260
60	1680
90	1870
120	1920

Table 4.1.4

Time(minute)	Color(PtCo)
0	7225
15	2850
30	2175
60	2800
90	4300
120	2150

Table 4.1.5

Time(minute)	COD
0	710
15	1206.7
30	1006.7
60	1280
90	913.3
120	1190

Table 4.1.6

Time(minute)	Color
0	7000
15	1100
30	625
60	1425
90	950
120	975

Table 4.1.7

Time(minute)	COD
0	773.3
15	1763.3
30	1236.7
60	1183.3
90	1306.7
120	946.7

Table 4.1.8

Time(minute)	Color
0	6900
15	625
30	1100
60	950
90	1075
120	925

Table 4.1.9

Experiment	pH	H ₂ O ₂ /FeSO ₄ ratio (ml/L:mg/L)
Experiment	2	3.415:195.5
Experiment	4	3.415:195.5
Experiment	5	3.415:195.5
Experiment	6	3.415:195.5

Table 4.3.1

Time(minute)	COD
0	795
15	7015
30	3635
60	4960

Table 4.3.2

Time(minute)	Color(PtCo)
0	6750
15	1300
30	1025
60	1025

Table 4.3.3

Time(minute)	COD
0	800
15	6300
30	3900
60	4425

Table 4.3.4

Time(minute)	Color(PtCo)
0	7050
15	1400
30	1000
60	950

Table 4.3.5

Time(minute)	COD
0	805
15	5335
30	4425
60	3870

Table 4.3.6

Time(minute)	Color(PtCo)
0	7275
15	1275
30	675
60	800

Table 4.3.7

Time(minute)	COD
0	850
15	5735
30	4720
60	3945

Table 4.3.8

Time(minute)	Color(PtCo)
0	7325
15	1250
30	750
60	575

Table 4.3.9