

**"Development of an Empirical Rate Law using Initial Rate for the
Degradation of Diisopropanolamine using Photo-Fenton"**

by

Amirah Raihana binti Haris Fadzilah

DISSERTATION
Submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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Universiti Teknologi PETRONAS
in partial fulfilment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(CHEMICAL ENGINEERING)

Approved by,

(Mrs. Putri Nazrol Faizura Bt Megat Khamaruddin)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
September 2012

CERTIFICATION OF ORIGINALITY

This is to certify 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 contained herein have not been undertaken or done by unspecified sources or persons.

AMIRAH RAIHANA BINTI HARIS FADZILAH

ABSTRACT

Alkanolamines such as diisopropanolamine are used to treat natural gas so as to remove the acid gases, carbon dioxide etc in it. The wastewater of this process will contain high amount of the amines in which causes high COD reading in it. Therefore, methods of degrading the recalcitrant pollutant were introduced, in which photo-Fenton is added into the wastewater and undergo oxidation process producing •OH radical which later will react with the amines and degrade them.

Focusing on the effect of concentration of both reagents and amines, empirical rate law is developed to obtain the optimum condition parameter for the degradation of diisopropanolamine in natural gas processing plant wastewater using photo-Fenton.

$$r = k \frac{[H_2O_2]^{0.141}[DIPA]^{1.561}}{[Fe^{2+}]^{-0.260}}$$

The proportionality constant, k, or the rate constant is 0.779. It is safe to say that this reaction is (141/1000)th order in H₂O₂ and (1561/1000)th order in DIPA. The overall order of this reaction is (1442/1000) order which is the sum of all the orders of the reactants. This indicates a fractional order reaction, in which indicates the complexity of the reaction mechanism

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

1.1.1 Wastewater

Wastewater is liquid waste generated by various human activities and contains impurities or pollutants and is harmful if disposed into the environment. For safe disposal of wastewater to the receiving bodies, it is routed to treatment system to remove the pollutants to the dischargeable limits. Treatment of wastewater produced by industry is the most challenging tasks due to the wide variety of pollutants. Selection of treatment technologies depends on the target pollutants as well as degree of treatment to be achieved.

In natural gas processing plant, raw natural gas extracted from reservoir contains impurities such as acid gases. As the removal of these acid gases are important, amine gas treating (also known as gas sweetening or acid gas removal) is introduced using alkanolamines as the absorbent for the acidic gases. Discovered by R. R. Bottoms in 1930, triethanolamine (TEA) is the first absorbent to be used for the purposes. Since then, various other members of alkanolamines were introduced and available such as monoethanolamine (MEA), diethanolamine (DEA), diisopropanolamine (DIPA) etc. After the process is completed, lean amine solution is either being recycled or carried over in water to the wastewater treatment system. These traces of amine in the wastewater will eventually increase the chemical oxygen demand (COD) to unacceptable level and violating the regulation.

1.1.2 Diisopropanolamine (DIPA)

Diisopropanolamine (DIPA) is a secondary amine of aliphatic amine group. It's a colourless liquid or white to yellow crystalline solid with an odour of ammonia and highly soluble in water. DIPA has been used widely in many industries; cosmetic, personal care and hygiene, pharmaceutical, natural gas industries and several others. In natural gas industries, DIPA is used to remove carbon dioxide (CO₂) and hydrogen sulphide (H₂S) from natural gas and refinery process stream in amine gas treating. Therefore, due to its usefulness in the industries, wastewater contaminated of DIPA is constantly generated. As DIPA is an organic compound, a recalcitrant pollutant, it suggested a high amount of chemical oxygen demand (COD) in the wastewater. Even with only one gram of DIPA in a litre of wastewater will contribute to nearly 2,000mg/L of COD (Ramli, 2010). In fact, it is highly soluble in water thus the removal of DIPA from the wastewater generated can be very tricky. Therefore, before entering the wastewater treatment system, DIPA needs to undergo pre-treatment by degrading it into biodegradable product. Lots of researches has been conducted by the researchers including advanced oxidation process (AOP) in which it has been identified to be able to degrade wide range of recalcitrant organic compounds. For degradation of DIPA using AOP method, studies have proved that Fenton's reagent are able to degrade the DIPA into a biodegradable substance by producing high concentration of •OH radical to be reacted actively with organic compound to produce CO₂ and water thus decreasing the COD of the wastewater.

1.1.3 Photo-Fenton in Degradation of DIPA

Infamous for its ability to oxidize contaminants in wastewater, Fenton's reagent is a mixture consists of hydrogen peroxide and ferrous catalyst. Oxidation of Fenton's reagent as per stated involves generating •OH radicals to degrade organic compound. Developed by Henry John Horstman Fenton in 1890s, many studies have been conducted to vary the applicable of Fenton's reagent oxidation for wastewater treatment one of which is Fe-UV Radiation-Hydrogen Peroxide. This is similar to the Fenton's oxidation except for the addition feature, UV radiation. This variation has been proved to be able to improve the degradation of recalcitrant pollutant in the wastewater in which the presence of UV light increased the production rate of •OH radical for the degradation of the pollutant.

1.2 PROBLEM STATEMENT

The application of alkanolamines in wastewater pre-treatment for natural gas processing plant has been widely used by the industry. Wastewater from the plant usually contains alkanolamines such as DIPA, TEA etc has an extremely high COD which exceeds the limit set by local authorities. Thus AOP is introduced in which Fenton's reagent is used to degrade the recalcitrant pollutant in the wastewater. Many researches and studies have been conducted related to the applicability of Fenton's reagent oxidation in degradation of recalcitrant pollutant in wastewater. However, little are focusing on the empirical rate law and the kinetic of the process for an optimum result of the degradation. For that reason, this study intends to determine the kinetic and reaction's empirical rate law for an optimum degradation using initial rates approaches by focusing on the effects of concentration of both reagents and subject towards the degradation process

1.3 OBJECTIVES OF STUDY

This project aims to determine the reaction's kinetic and empirical rate law for the degradation of DIPA using photo-Fenton. The objectives are:

- a) To conduct experiment for the degradation of diisopropanolamine.
- b) To investigate the effect of concentration of ferrous, hydrogen peroxide and initial concentration of DIPA towards the degradation process.
- c) To develop the empirical rate law using initial rate based on the effect of concentration for the degradation of DIPA in synthetic wastewater and industrial wastewater samples.
- d) To prove the developed empirical rate law and reaction kinetic as the optimum condition for the degradation of the DIPA.

1.4 SCOPE OF STUDY

This study focuses on the development of empirical rate law for the degradation of diisopropanolamine (DIPA) using photo-Fenton. Focusing on the concentration of Fenton and the initial concentration of DIPA, data will be analysed and simulated using initial approaches such as least-square methods to provide the optimum kinetic for the reaction, provided other parameters such as temperature, pressure etc are controlled.

CHAPTER 2

LITERATURE REVIEW

2.1 DIISOPROPANOLAMINE (DIPA)

Diisopropanolamine (DIPA) is a secondary alkanolamine, organic derivative of ammonia categorized based on the number of substituent groups that are attached to the nitrogen atom. DIPA is produced through reaction between propylene oxide (C_3H_6O) and ammonia (NH_3) as in Figure 2.1 below:

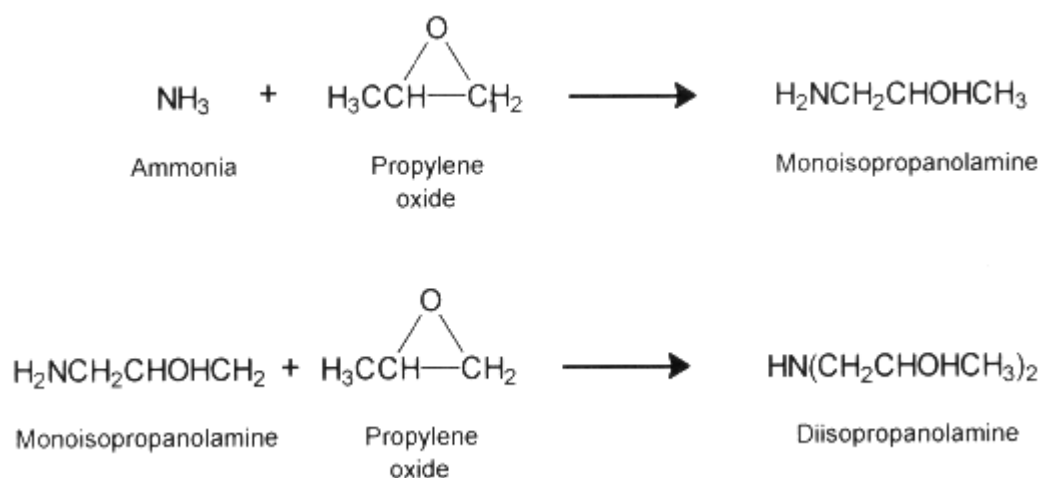


Figure 1: Synthesis of DIPA

Widely used in many industries such as cosmetic, metalworking fluid, pharmaceutical industry etc, wastewater contaminated of DIPA is constantly generated and contains extremely high chemical oxygen demand (COD) which exceeds limits set by local authorities.

2.2 ADVANCED OXIDATION PROCESS (AOP)

2.2.1 Theory of AOP

In wastewater treatment, advanced oxidation processes are used to oxidize complex organic constituents in the wastewater which are non-biodegradable. In some cases, partial oxidation is enough to make the pollutant less toxic and amenable to biological treatment. The oxidation of specific compound can be characterized by the extent of degradation of it as follows (Rice, 1996):

- a) Primary degradation – structural change in the parent compound
- b) Acceptable degradation (defusing) – structural change in the parent compound to an extent that the toxicity is reduced.
- c) Ultimate degradation (mineralization – complete conversion of organic compound to inorganic CO₂)
- d) Unacceptable degradation (fusing) - structural change in the parent compound that results in increasing toxicity.

AOP involves the generation of •OH hydroxyl radical, a highly reactive oxidant which react with dissolved constituents, initiating a series of oxidation reactions until to an extent of complete mineralization, despite the specific classes or group of the compound. Able to operate at standard temperature and pressure, AOP is different than other treatment processes such as ion exchange or stripping because wastewater compounds are degraded rather than concentrated or transferred into different phase.

2.2.2 Technology in producing hydroxyl radicals (•OH)

Due to its usefulness in the industry, variety of technologies has been introduced in order to produce •OH hydroxyl radicals in aqueous phase. Example such as Ozone + UV, Ozone + H₂O₂, Ozone + UV₂₅₄ + H₂O₂, H₂O₂ + UV, Fenton's Reagent Photocatalysis (UV + TiO₂) and several others.

Focusing on the production of hydroxyl radicals using Fenton's reagent, a mixture of ferrous and H₂O₂ this methods has been widely used to degrade recalcitrant pollutant in wastewater. As H₂O₂ alone as a strong oxidant is not effective in high concentration of recalcitrant contaminant such as amine, ferrous ion is introduced as catalyst to increase the rate of radical •OH production as in equation below:



For photo-Fenton reaction in producing $\bullet\text{OH}$, the basis will be the same as for Fenton's reagent, with additional in the reaction which increases the production rate of $\bullet\text{OH}$ radical. The reactions are as below:



Where L is organic ligand. For this project, the $\bullet\text{OH}$ produces from all these reactions will degrade the diisopropanolamine and forming product such as formic acid, oxalic acid, acetic acid and glycine. However, in doing this, effect of other parameters such as pH, temperature, concentration of reagent, UV power dosage etc needs to be taken into considerations as well.

2.3 RATE LAW

One of the first steps in studying the kinetics of a chemical reaction is to determine the rate law for the reaction. The rate law also known as rate equation is the expression of chemical reaction linking the reaction rate with concentrations of reactants and constant parameters. Determining the rate equation for a particular system require mass balance and reaction rate. A simple demonstration of rate equation is as below :



the rate is given by:

$$r = k[\text{A}]^\alpha[\text{B}]^\beta \quad (2.5)$$

where [A] = concentration of species A
 [B] = concentration of species B
 α, β = reaction orders
 k = rate constant of the reaction

One method for determining the rate law is by measuring how concentration of a reactant or product varies with time experimentally, and then make characteristic kinetic plots. The other methods would be using methods of initial rates

2.3.1 Methods of Initial Rates

The method of Initial Rates involves measuring the rate of reaction at very short times before any significant changes in concentration occur. This method is introduced due to the presence of effects such as significant reverse reaction which could render ineffectiveness. Using this method, series of experiment is conducted manipulating the initial concentration of the reagent, C_{A0} , C_{B0} or product, and the initial rate of reaction, $-r_{A0}$ is determined. For example from equation (2.5), the expression of the initial rates is as follow:

$$-r_{A0} = kC_{A0}^{\alpha}C_{B0}^{\beta}C_{C0}^{\gamma} \quad (2.6)$$

Where α and β is the order of reaction. This equation will then be used for a plot of graph to obtain the value of α and β .

2.3.2 Least-square methods

Using method of initial rates, least-square method is used as a data analysis useful in determining the best value of the rate law parameters from a series of measurements when three or more parameters are involved. From equation (2.6) with rate of expression expressed in the form of reaction of concentration over time:

$$\left(-\frac{dC_A}{dt}\right)_0 = -r_{A0} = kC_{A0}^{\alpha}C_{B0}^{\beta}C_{C0}^{\gamma} \quad (2.7)$$

$$\ln \left(-\frac{dC_A}{dt}\right)_0 = \ln k + \alpha \ln C_{A0} + \beta \ln C_{B0} + \gamma \ln C_{C0} \quad (2.8)$$

Letting $Y = \ln -(dC_A/dt)_0$, $X_1 = \ln C_{A0}$, $X_2 = \ln C_{B0}$, $a_0 = \ln k$, $a_1 = \alpha$ and $a_2 = \beta$, $a_3 = \gamma$ then

$$Y = a_0 + a_1X_1 + a_2X_2 + a_3X_3 \quad (2.9)$$

CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

This chapter explains on the materials, experimental and analytical simulation used during the projects.

3.2 MATERIALS, EQUIPMENTS AND SOFTWARE

Several materials are used throughout the project. The key materials would be diisopropanolamine (DIPA) which is contained in the wastewater sample taken from a sour gas plant in Malaysia. Table 1 listed the chemical used in this project and Table 2 listed on the equipment and software used for the experimental and analytical procedure.

Table 1: List of Chemical Used for the Experiment

CHEMICALS	MW g.mol⁻¹	T_m °C	T_b °C	ρ (T=25 °C) kg/m³
DIPA	133.19	44	249	0.989
Hydrogen Peroxide (H ₂ O ₂)	34.00	-	-	1.11
Iron (II) Sulphate 7- Hydrate (FeSO ₄ .7H ₂ O)	278.20	-	-	1.04
Sulphuric Acid (H ₂ SO ₄)	98.04	-15	330	1.84
Sodium Hydroxide (NaOH)	40.00	65	-	1.04

Table 2: List of Equipment and Software used

Parameters/Characteristic	Equipment	Software
Ph	SENSion 4	-
Degradation Rate	HPLC	-
Kinetic of Reaction and Rate Law Simulation	-	Microsoft Excel Polymath

3.3 EXPERIMENTAL PROCEDURE

3.3.1 Experimental Set-up

The work flow of the experimental procedure is as shown in Figure 3. Figure 2 show the diagram of the experimental set-up for the degradation of DIPA using photo-Fenton.

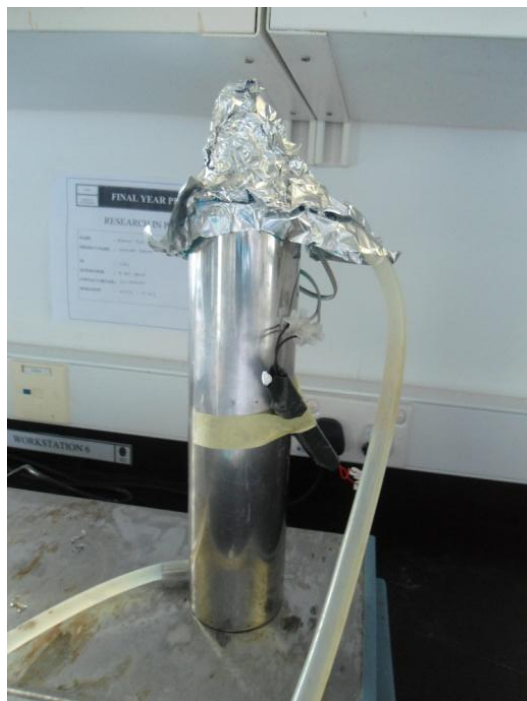


Figure 2: Diagram of Experimental Set-up

As stated, this experiment is conducted focusing on the effect of concentration of the key materials; Iron, Hydrogen Peroxide and Diisopropanolamine on the degradation rate of the DIPA. Thus, it is conducted with several variations as following assuming that other parameters such as catalyst, temperature, pressure, pH etc are constant:

- a) Effect of different concentration of FeSO_4
- b) Effect of different concentration of H_2O_2
- c) Effect of different initial concentration of DIPA

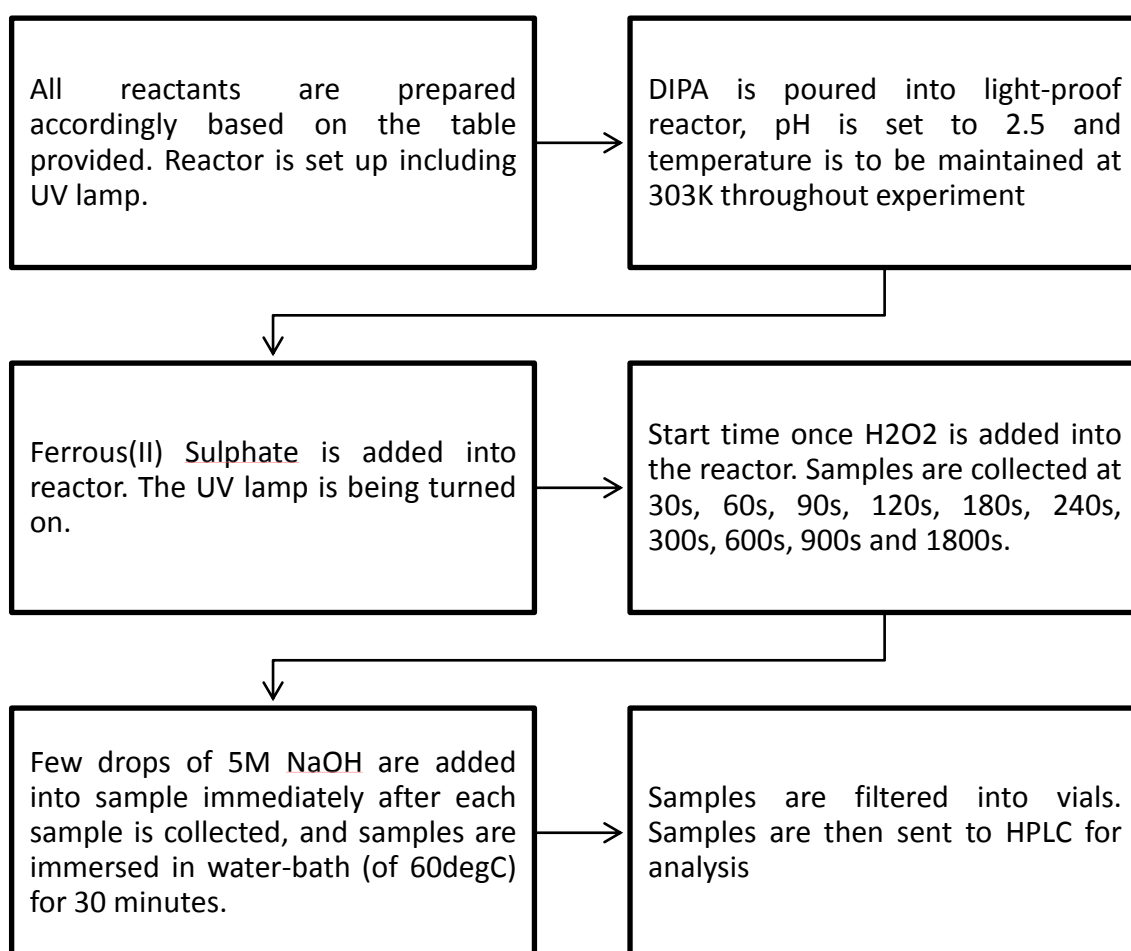


Figure 3: Experimental Procedure

3.3.2 Experimental Procedure

- a) Amine solution (DIPA) is prepared in a beaker.
- b) The pH and temperature is tested using pH meter. Based on literature review, the pH is to be maintained at pH 2.5 throughout the experiment. If the pH is too high, H_2SO_4 will be added while lower pH requires NaOH.
- c) The sample is continuously stirred during the experiment using magnetic stirrer. The addition of the Ferrous Sulphate and Hydrogen Sulphate will be varied base on Table 3.
- d) Sample is collected from the beaker at corresponding time, 30, 60, 90, 120, 180, 240, 300, 600, 900 and 1800 seconds. For each sampling time, the sample is replicated trice for more accurate result.
- e) The sample is sent to the HPLC for analysis of the concentration of the DIPA in the sample. From the result, the percentage of the different in DIPA concentration from initial to final will be assumed as the percentage as the degradation.
- f) Data obtained from the HPLC analysis is analysed using computer software as stated in Table 2.



Figure 4: HPLC Instrument

3.3.3 Calculation

3.3.3.1 Pre-experiment calculation

Before doing the experiment, calculation of the exact amount of Hydrogen Peroxide (in ml), DIPA (in g/L) and Fe²⁺ (in g) need to be done to ensure that it meets the required concentration decided. For the calculation to be made, volume of Hydrogen Peroxide is used as the base volume.

Calculation for volume of Hydrogen Peroxide (with concentration of x M) and DIPA is as below (assuming that total used for the reaction is 250mL):

$$\text{Volume of H}_2\text{O}_2, y \text{ (mL)} = x \frac{\text{mol}}{\text{L}} \times 34.01 \frac{\text{g}}{\text{mol}} \times 1/1.135 \frac{\text{mL}}{\text{g}} \times \frac{100\%}{30\%} \times 0.25\text{L} \quad (3.1)$$

$$\begin{aligned} \text{Volume of DIPA (mL) required} &= 250\text{mL} - y \text{ mL} \\ &= z \text{ mL} \end{aligned} \quad (3.2)$$

As DIPA is solid at room temperature, it is important to first calculate the amount of DIPA required. The total weight required to meet the concentration requirement of a M is calculated as below:

$$\begin{aligned} &\text{Weight of DIPA (g)} \\ &= a \frac{\text{mol}}{\text{L}} \times 133.2 \frac{\text{g}}{\text{mol}} \times 1 \text{ L (to prepare 1L of a M DIPA)} \end{aligned} \quad (3.3)$$

The DIPA calculated is then diluted in 1L of distilled water and z mL of the DIPA is taken from this solution.

For Fe²⁺, the amount of Ferrous (II) Sulphate requires to meet the concentration of p M is calculated as below:

$$\begin{aligned} &\text{Weight of Ferrous (II) Sulphate (g)} \\ &= p \frac{\text{mol}}{\text{L}} \times 55.845 \frac{\text{g}}{\text{mol}} \text{Fe}^{2+} \times 0.25 \text{ L} \times \frac{278.15 \text{ g (Ferrous (II) Sulphate)}}{55.545 \text{ g (Fe}^{2+})} \end{aligned} \quad (3.4)$$

Sample calculation is shown in appendix.

3.3.3.2 Analytical calculation

From the experiment, each and every data obtained is expressed in the form of table created in the software Microsoft Office Excel. Set of data obtained is from the three experiments conducted to:

- a) Effect of different concentration of Ferrous on degradation rate
- b) Effect of different concentration of H₂O₂ on degradation rate
- c) Effect of different initial concentration of DIPA on degradation rate

All the data will be collected and tabulated as below:

Table 3: Table for Data Collected Throughout Experiment

H2O2		DIPA		Fe2+		Temp (K)	pH
M	ml	M	ml	M	g		
0.000	0.000	0.023	250.000	0.007	0.452	303	2.5
0.055	1.373	0.023	248.627	0.007	0.452	303	2.5
0.110	2.747	0.023	247.253	0.007	0.452	303	2.5
0.215	5.369	0.023	244.631	0.007	0.452	303	2.5
0.320	7.991	0.023	242.009	0.007	0.452	303	2.5
0.430	10.737	0.023	239.263	0.007	0.452	303	2.5
0.800	19.977	0.023	230.023	0.007	0.452	303	2.5
1.600	39.953	0.023	210.047	0.007	0.452	303	2.5
0.215	5.369	0.023	244.631	0.000	0.000	303	2.5
0.215	5.369	0.023	244.631	0.002	0.139	303	2.5
0.215	5.369	0.023	244.631	0.004	0.278	303	2.5
0.215	5.369	0.023	244.631	0.007	0.452	303	2.5
0.215	5.369	0.023	244.631	0.008	0.556	303	2.5
0.215	5.369	0.023	244.631	0.010	0.695	303	2.5
0.215	5.369	0.023	244.631	0.020	1.390	303	2.5
0.215	5.369	0.008	244.631	0.007	0.452	303	2.5
0.215	5.369	0.015	244.631	0.007	0.452	303	2.5
0.215	5.369	0.023	244.631	0.007	0.452	303	2.5
0.215	5.369	0.030	244.631	0.007	0.452	303	2.5
0.215	5.369	0.038	244.631	0.007	0.452	303	2.5
0.215	5.369	0.045	244.631	0.007	0.452	303	2.5

Once all the data are collected, plots of concentration profile and degradation Percentage against Concentration of Reagents is created.

The data next will be analyzed in the Microsoft Excel using Data Analysis ToolPak and Solver Add-in. Calculation can be solved by Solve Adds-in whereas data can be analyze by the ToolPak. This Toolpak will generate a complete analysis of the data obtained and providing users with final coefficient value to be used for the regression equation.

Example of analyzed data by Analysis Toolpak is as in the figure below:

SUMMARY OUTPUT								
<i>Regression Statistics</i>								
Multiple R	0.907032167							
R Square	0.822707353							
Adjusted R Square	0.734061029							
Standard Error	0.123932431							
Observations	10							
<i>ANOVA</i>								
	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>gnificance F</i>			
Regression	3	0.427638	0.142546	9.280784	0.011349			
Residual	6	0.092155	0.015359					
Total	9	0.519793						
	<i>Coefficients</i>	<i>andard Err</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>	<i>ower 99.0%</i>	<i>pper 99.0%</i>
Intercept	3.568353157	2.094378	1.703777	0.139311	-1.5564	8.693111	-4.1964	11.33311
X Variable 1	0.58415558	0.154508	3.780739	0.009172	0.206087	0.962224	0.011327	1.156984
X Variable 2	-0.13628979	0.507686	-0.26845	0.797342	-1.37855	1.105973	-2.0185	1.745919
X Variable 3	-0.09305815	0.053725	-1.73212	0.133962	-0.22452	0.038402	-0.29224	0.106124

Figure 5: Random Analyzed Data Created by using Analysis ToolPak (Regression)

From the above random analysis, the regression equation based on equation (2.9) is:

$$Y = 0.568 + 0.584X_1 + (-0.136)X_2 + (-0.09)X_3 \quad (3.5)$$

From this, further calculation can be made to come out with the kinetic and empirical law of the reaction.

CHAPTER 4

RESULTS & DISCUSSION

4.1 EXPERIMENTAL

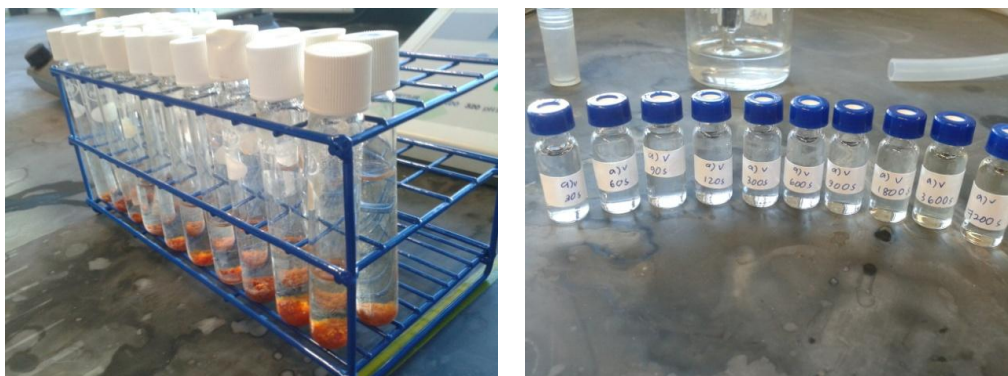


Figure 6: Pictures of Experimental Works and Final Sample Before HPLC Analysis

Once the experimental work is done, all the samples were sent to HPLC for analysis. However, the peak at the specific retention time obtained will be compared with the prepared calibration curve. This is done automatically by the HPLC. In before, to obtain the calibration curve, samples of standard for DIPA ranging from 1g/L to 4g/L were prepared and analysed first to obtain the peak and retention time.

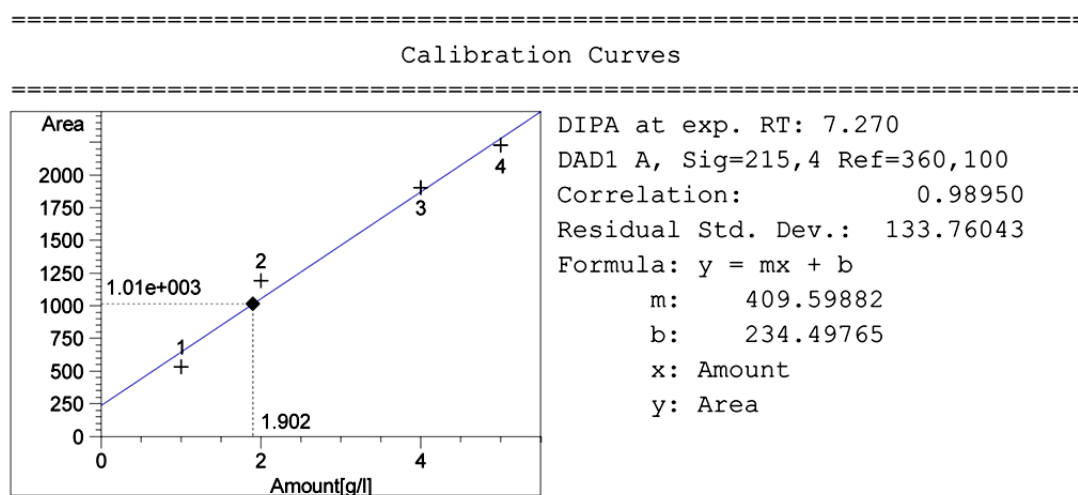


Figure 7: Calibration Curves of DIPA

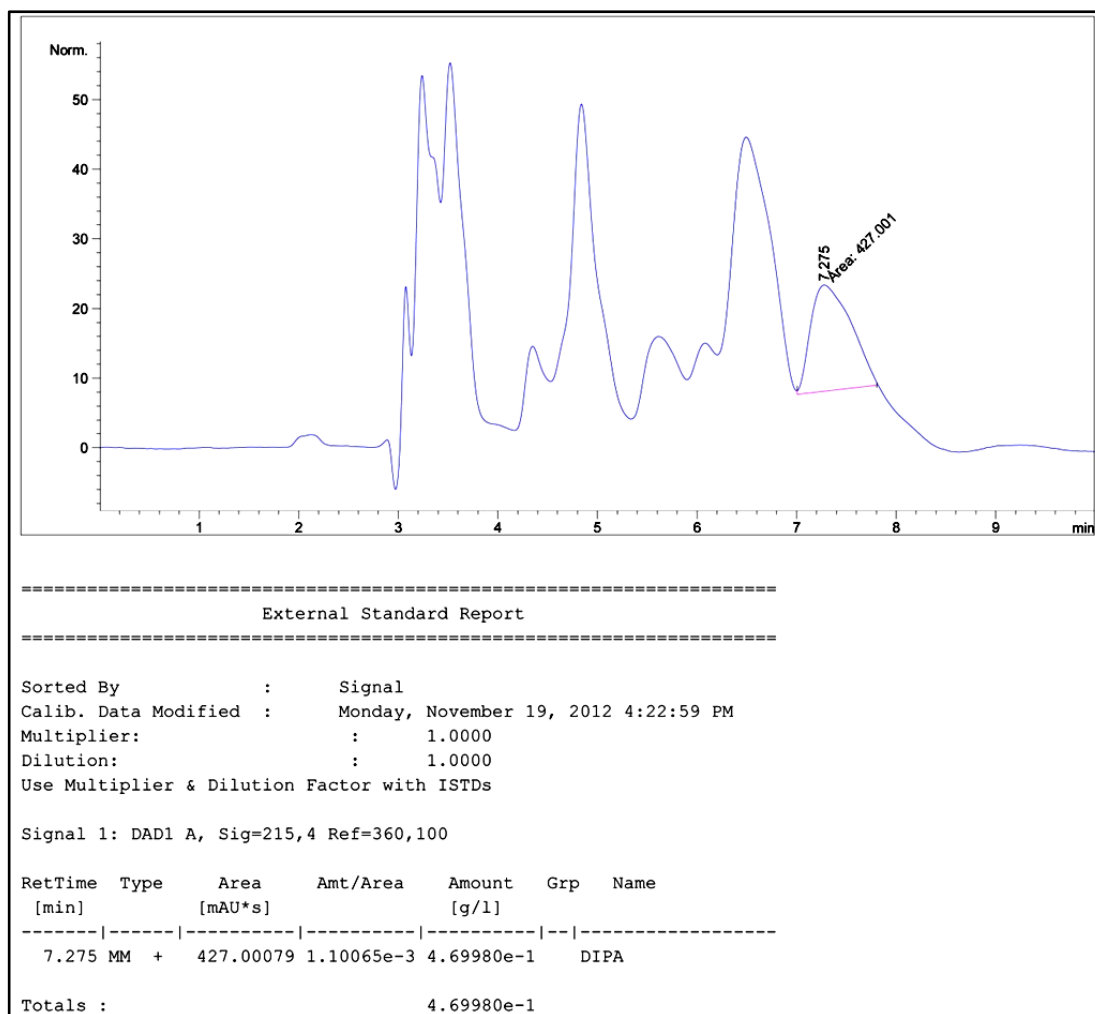


Figure 8: Example of Report Obtained from HPLC

The report as in Figure 8 above is obtained from the HPLC, as the analysis result of experiment for Hydrogen Peroxide's varied concentration of 0.430 M , with concentration of DIPA and Fe²⁺ is fixed at 0.023M and 0.007 M respectively.

4.2 RESULTS OF EXPERIMENT

From the report, we would be able to know the amount of degraded concentration of DIPA by subtracting value obtained to the initial concentration of DIPA as below:

$$\text{Degraded DIPA (g/L)} = \text{Initial Dipa} \left(\frac{\text{g}}{\text{L}} \right) - \text{Final DIPA} \left(\frac{\text{g}}{\text{L}} \right) \quad (4.1)$$

Full results are as summarised in Table 4 and Table 5.

Table 4: Overall Results of Experiment (Final DIP A(g/L) Concentration at Time(s))

[H2O2]	[DIP A]	[Fe2+]	Temp (K)	pH	[DIP A] at Time (s) (g/L)									
M	M	M			30	60	90	120	180	240	300	600	900	1800
0.000	0.023	0.007	303	2.5	1.832	1.830	1.802	1.781	1.774	1.665	1.646	1.640	1.517	1.509
0.055	0.023	0.007	303	2.5	1.596	1.054	0.858	0.799	0.772	0.770	0.751	0.544	0.243	0.000
0.110	0.023	0.007	303	2.5	1.479	1.354	1.293	1.238	1.166	1.113	1.167	1.055	1.038	0.593
0.215	0.023	0.007	303	2.5	1.403	1.267	1.075	1.022	0.954	0.832	0.825	0.503	0.464	0.023
0.320	0.023	0.007	303	2.5	0.814	0.784	0.765	0.649	0.604	0.521	0.458	0.075	0.000	0.000
0.430	0.023	0.007	303	2.5	0.983	0.970	0.758	0.746	0.716	0.646	0.592	0.469	0.349	0.000
0.800	0.023	0.007	303	2.5	0.748	0.718	0.462	0.364	0.394	0.316	0.208	0.072	0.000	0.000
1.600	0.023	0.007	303	2.5	0.834	0.667	0.500	0.401	0.225	0.120	0.000	0.000	0.000	0.000
0.215	0.023	0.000	303	2.5	0.809	0.606	0.479	0.422	0.397	0.390	0.056	0.000	0.000	0.000
0.215	0.023	0.002	303	2.5	1.251	1.152	1.024	0.972	0.819	0.537	0.290	0.000	0.000	0.000
0.215	0.023	0.004	303	2.5	0.913	0.865	0.832	0.796	0.759	0.755	0.568	0.411	0.283	0.149
0.215	0.023	0.007	303	2.5	0.608	0.606	0.598	0.592	0.542	0.528	0.461	0.259	0.213	0.000
0.215	0.023	0.008	303	2.5	0.771	0.722	0.609	0.564	0.453	0.436	0.422	0.414	0.237	0.000
0.215	0.023	0.010	303	2.5	0.689	0.680	0.645	0.611	0.535	0.501	0.452	0.296	0.137	0.000
0.215	0.023	0.020	303	2.5	0.393	0.391	0.337	0.335	0.333	0.282	0.241	0.126	0.000	0.000
0.215	0.008	0.007	303	2.5	0.037	0.037	0.037	0.035	0.033	0.029	0.026	0.016	0.007	0.001
0.215	0.015	0.007	303	2.5	0.089	0.052	0.046	0.042	0.038	0.024	0.019	0.005	0.003	0.001
0.215	0.023	0.007	303	2.5	0.509	0.504	0.448	0.434	0.406	0.369	0.345	0.144	0.020	0.000
0.215	0.030	0.007	303	2.5	1.164	1.103	1.060	0.942	0.940	0.939	0.809	0.673	0.496	0.137
0.215	0.038	0.007	303	2.5	1.594	1.571	1.565	1.505	1.467	1.402	1.345	1.108	0.943	0.409
0.215	0.045	0.007	303	2.5	1.891	1.880	1.873	1.785	1.783	1.766	1.616	1.504	1.418	0.918

Table 5: Total Degraded DIPA (g/L) at Time (s)

[H2O2]	[DIPA]	[Fe2+]	Degraded [DIPA] at Time (s) (g/L)										-r _{DIPA} at t=0s	
M	M	M	30	60	90	120	180	240	300	600	900	1800	-r _{DIPA}	LN (-r)
0.000	0.023	0.007	1.165	1.167	1.195	1.216	1.223	1.332	1.351	1.357	1.480	1.488	0.0004	-7.724
0.055	0.023	0.007	1.401	1.943	2.139	2.198	2.225	2.227	2.246	2.453	2.754	2.997	0.0013	-6.658
0.110	0.023	0.007	1.518	1.643	1.704	1.759	1.831	1.884	1.830	1.942	1.959	2.404	0.0020	-6.221
0.215	0.023	0.007	1.594	1.730	1.922	1.975	2.043	2.165	2.172	2.494	2.533	2.974	0.0030	-5.807
0.320	0.023	0.007	2.183	2.213	2.232	2.348	2.393	2.476	2.539	2.922	2.997	2.997	0.0015	-6.488
0.430	0.023	0.007	2.014	2.027	2.239	2.251	2.281	2.351	2.405	2.533	2.648	2.997	0.0020	-6.228
0.800	0.023	0.007	2.249	2.279	2.535	2.633	2.603	2.681	2.789	2.925	2.997	2.997	0.0027	-5.909
1.600	0.023	0.007	2.163	2.330	2.497	2.596	2.772	2.877	2.997	2.997	2.997	2.997	0.0040	-5.517
0.215	0.023	0.000	2.188	2.391	2.518	2.575	2.600	2.607	2.941	2.997	2.997	2.997	0.0021	-6.154
0.215	0.023	0.002	1.746	1.845	1.973	2.025	2.178	2.460	2.707	2.997	2.997	2.997	0.0023	-6.086
0.215	0.023	0.004	2.084	2.132	2.165	2.201	2.238	2.242	2.429	2.586	2.714	2.848	0.0011	-6.850
0.215	0.023	0.007	2.389	2.391	2.399	2.405	2.455	2.469	2.536	2.738	2.784	2.997	0.0005	-7.531
0.215	0.023	0.008	2.226	2.275	2.388	2.433	2.544	2.561	2.575	2.583	2.760	2.997	0.0013	-6.619
0.215	0.023	0.010	2.308	2.317	2.352	2.386	2.462	2.496	2.545	2.701	2.860	2.997	0.0009	-6.978
0.215	0.023	0.020	2.604	2.606	2.660	2.662	2.664	2.715	2.756	2.871	2.997	2.997	0.0005	-7.537
0.215	0.008	0.007	0.962	0.962	0.962	0.964	0.966	0.970	0.973	0.983	0.992	0.998	0.0001	-9.903
0.215	0.015	0.007	1.922	1.959	1.966	1.969	1.973	1.987	1.992	2.011	2.011	2.011	0.0001	-8.874
0.215	0.023	0.007	2.488	2.493	2.549	2.563	2.591	2.628	2.652	2.853	2.977	2.997	0.0003	-8.063
0.215	0.030	0.007	2.832	2.893	2.937	3.054	3.056	3.057	3.187	3.323	3.500	3.859	0.0006	-7.497
0.215	0.038	0.007	3.401	3.424	3.430	3.490	3.528	3.593	3.650	3.887	4.052	4.586	0.0007	-7.299
0.215	0.045	0.007	4.103	4.114	4.121	4.209	4.211	4.228	4.378	4.490	4.576	5.076	0.0005	-7.522

4.3 DATA ANALYSIS

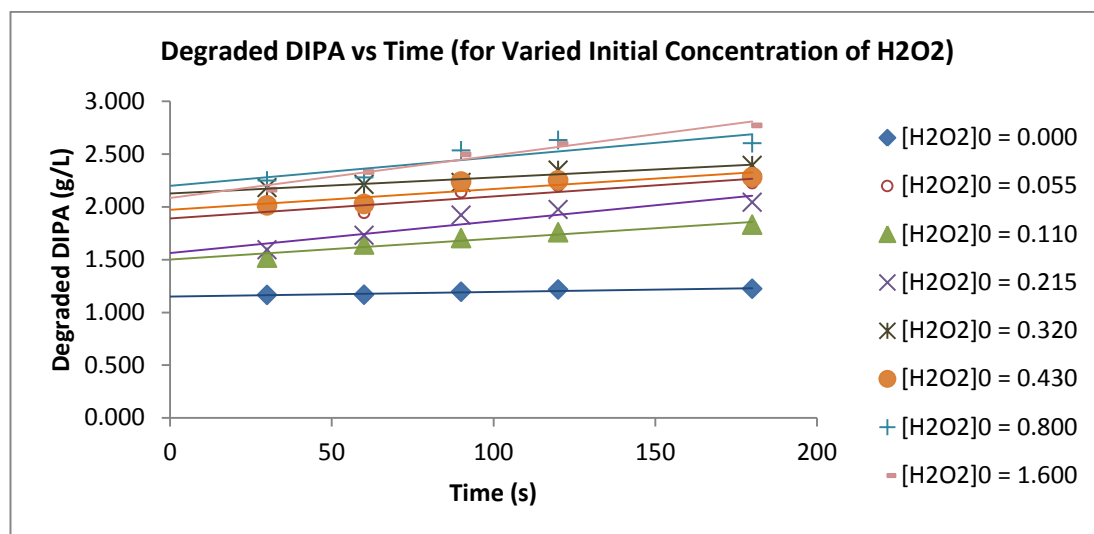


Figure 9: Degraded Time vs Time (for Varies [H2O2]0)

From the result obtained, linear graphs of degraded DIPA over time are plotted for each set of concentration variation. The linear plot is extrapolated up to when the time is 0s and linear slope of the graph is obtained as the rate of reaction.

Figure 9 shows the degradation of DIPA over time with different initial concentration of Hydrogen Peroxide. For these experiments, the initial concentration of DIPA and Fe^{2+} are kept at constant 0.023M and 0.007M respectively, including temperature at 303K and pH at 2.5. It can be seen from Figure 9 that the degradation of DIPA increase as we increase the concentration of hydrogen peroxide however, it is increasing at different slope. This shows that different concentration of Hydrogen Peroxide does affect the rate of degradation.

Figure 10 shows the degradation of DIPA over time with different initial concentration of Ferrous (II) Sulphate. Different than the first batch of experiments, for these experiments, the initial concentration of DIPA and H_2O_2 are kept at constant 0.023M and 0.215M respectively, including temperature at 303K and pH at 2.5. Figure 10 shows that the degradation of DIPA increases with time however, the trend is varying randomly at different Ferrous (II) Sulphate concentration. This might be due to the effect of reversible reaction. This shows that different concentration of Fe^{2+} does affect the rate of degradation too.

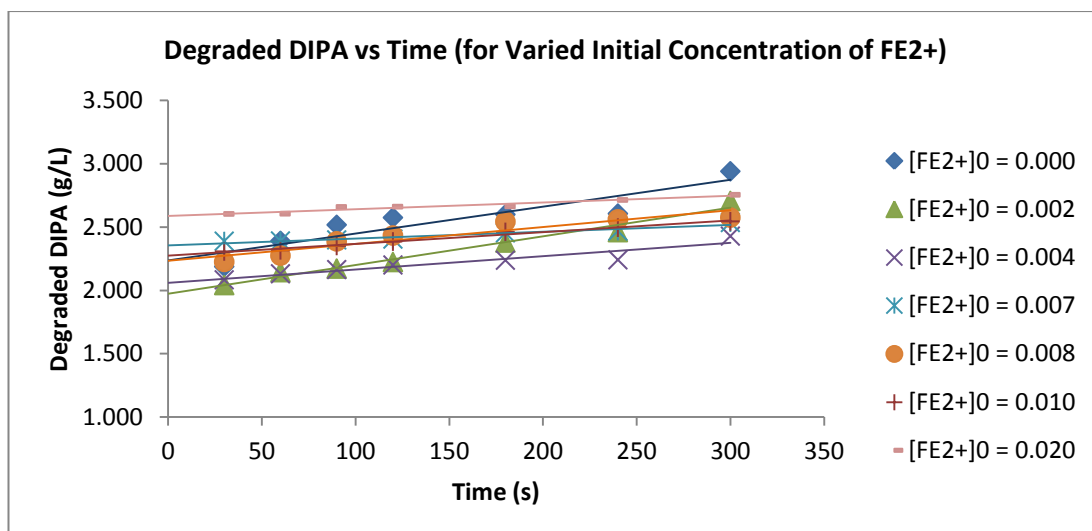


Figure 10: Degraded Time vs Time (for Varies [Fe²⁺]₀)

The degradation of DIPA over time with different initial concentration of DIPA is as shown in Figure 11. For these experiments, the initial concentration of H₂O₂ and Fe²⁺ are kept at constant at 0.213M and 0.007M respectively, including temperature at 303K and pH at 2.5. Figure 11 shows an obvious observation that the degradation of DIPA increase as we increase the initial concentration of the DIPA. This might be due to the increase in amount of DIPA to be degraded by the photo-fenton. It also shows that different concentration of DIPA does affect the rate of degradation.

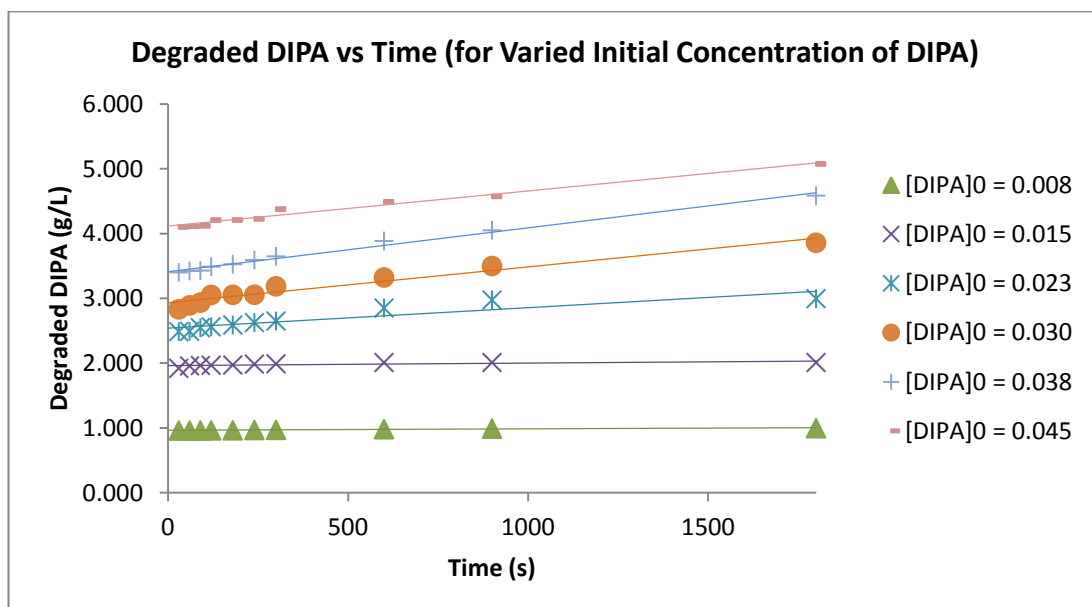


Figure 11: Degraded Time vs Time (for Varies [DIPA]₀)

From Figure 9, 10 and 11, the linear slope of each plot are obtained and recorded in the Table 5 as the initial rate of experiments. Next, a graph of collected Initial Rates versus different concentration of H₂O₂, DIPA and Fe²⁺ is plotted as in Figure 12 to examine the changes in the degradation of DIPA.

From Figure 12, we can see that varying concentration of H₂O₂ shows significant changes in the rate of reaction. The rate of reaction continue to increase as the concentration of Hydrogen Peroxide increase, however it will drop significantly. Figure 12 also shows that different concentration of Fe²⁺ will cause the reaction to react inversely. Increasing amount of Fe²⁺ favours reversible reaction throughout the process thus decreasing the rate of degradation. Besides, increasing initial concentration of DIPA will increase the rate of reaction until an optimum rate is achieved. Further increment will decrease the rate of reaction.

Next, LN of the initial rate is then used to be analysed using Toolpak Analysis Solver. The data are as in below:

Table 6: Data for Toolpak Analysis

LN [H ₂ O ₂]	LN [DIPA]	LN [Fe ²⁺]	-r _{DIPA}	LN (-r)
-9.210	-3.772	-4.962	0.0004	-7.724
-2.900	-3.772	-4.962	0.0013	-6.658
-2.207	-3.772	-4.962	0.0020	-6.221
-1.537	-3.772	-4.962	0.0030	-5.807
-1.139	-3.772	-4.962	0.0015	-6.488
-0.844	-3.772	-4.962	0.0020	-6.228
-0.223	-3.772	-4.962	0.0027	-5.909
0.470	-3.772	-4.962	0.0040	-5.517
-1.537	-3.772	-9.210	0.0021	-6.154
-1.537	-3.772	-6.215	0.0023	-6.086
-1.537	-3.772	-5.521	0.0011	-6.850
-1.537	-3.772	-4.962	0.0005	-7.531
-1.537	-3.772	-4.828	0.0013	-6.619
-1.537	-3.772	-4.605	0.0009	-6.978
-1.537	-3.772	-3.912	0.0005	-7.537
-1.537	-4.828	-4.962	0.0001	-9.903
-1.537	-4.200	-4.962	0.0001	-8.874
-1.537	-3.772	-4.962	0.0003	-8.063
-1.537	-3.507	-4.962	0.0006	-7.497
-1.537	-3.270	-4.962	0.0007	-7.299
-1.537	-3.101	-4.962	0.0005	-7.522

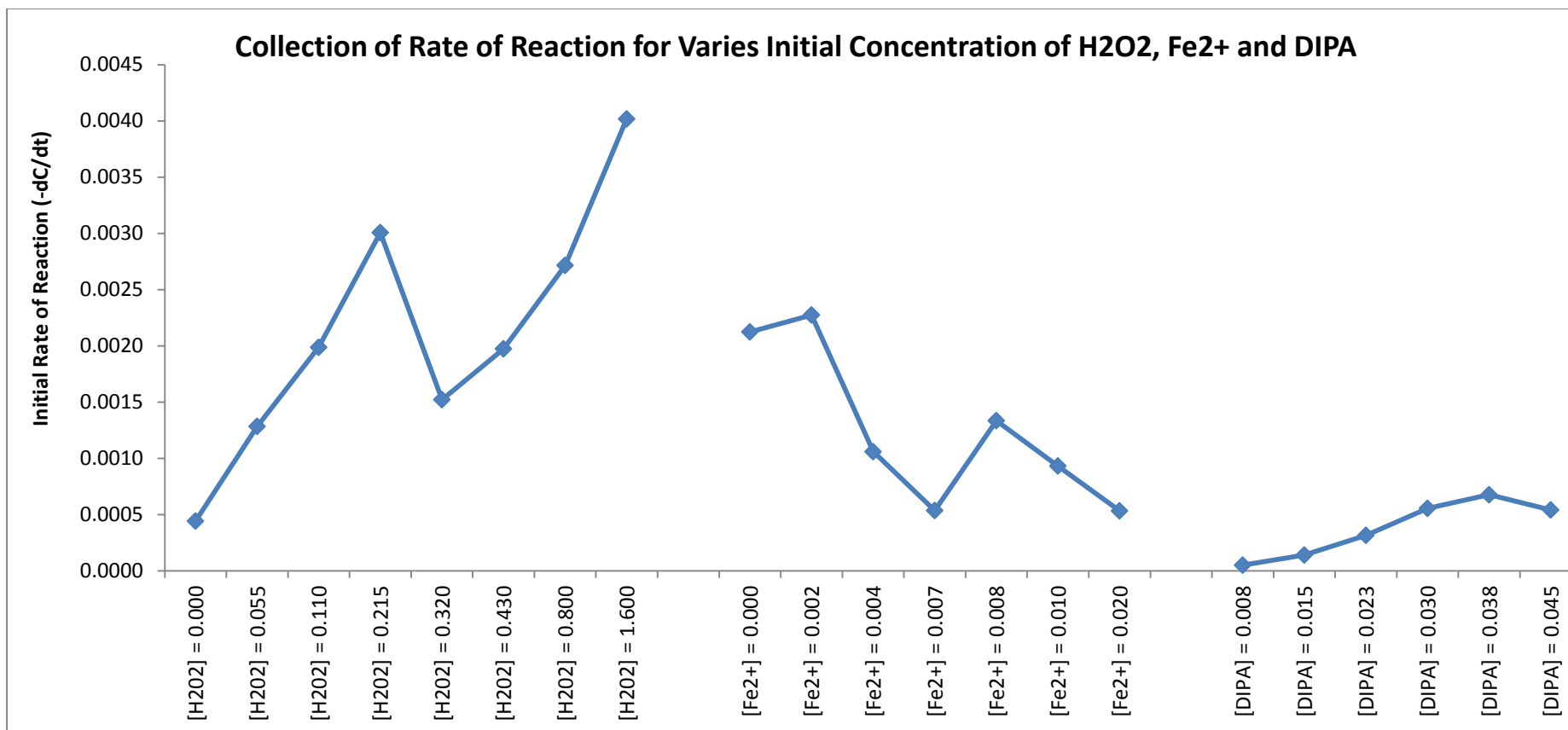


Figure 12: Collection of Initial Rates

SUMMARY OUTPUT								
<i>Regression Statistics</i>								
Multiple R	0.584187241							
R Square	0.341274733							
Adjusted R Square	0.225029097							
Standard Error	0.941593857							
Observations	21							
ANOVA								
	<i>df</i>	<i>SS</i>	<i>MS</i>	<i>F</i>	<i>Significance F</i>			
Regression	3	7.808650186	2.602883395	2.935806852	0.063118272			
Residual	17	15.07218286	0.886598992					
Total	20	22.88083305						
	<i>Coefficients</i>	<i>Standard Error</i>	<i>t Stat</i>	<i>P-value</i>	<i>Lower 95%</i>	<i>Upper 95%</i>	<i>Lower 95.0%</i>	<i>Upper 95.0%</i>
Intercept	-2.224303753	2.717368912	-0.818550526	0.424369067	-7.957450962	3.508843456	-7.957450962	3.508843456
[H2O2] M	0.140723429	0.115744127	1.215814856	0.240669728	-0.10347533	0.384922188	-0.10347533	0.384922188
[DIPA] M	1.560911432	0.654292437	2.385647983	0.028954205	0.180475069	2.941347794	0.180475069	2.941347794
[Fe2+] M	-0.259877352	0.209571554	-1.240041156	0.231804371	-0.702034678	0.182279973	-0.702034678	0.182279973

Figure 13: Data Analysis Using Toolpak Solver

Therefore, based on Figure 12, from the analysis of Toolpak, the initial rate generated will be in the form of Equation (2.9) as below:

$$Y = -2.224 + 0.141X_1 + 1.561X_2 + (-0.260)X_3 \quad (4.2)$$

In which, based on equation (2.8)

$$Y = \ln(-dC_{DIPA}/dt) \quad (4.3)$$

$$\ln k = -2.224 \text{ therefore,}$$

$$k = 0.779 \quad (4.4)$$

$$X_1 = \ln[H_2O_2] \text{ and}$$

$$\alpha = 0.141 \quad (4.5)$$

$$X_2 = \ln[DIPA] \text{ and}$$

$$\beta = 1.561 \quad (4.6)$$

$$X_3 = \ln[Fe^{2+}] \text{ and}$$

$$\theta = -0.26 \quad (4.7)$$

Substituting (4.4), (4.5), (4.6) and (4.7) into Equation (2.7), we get the **empirical rate law** as in below:

$$\left(-\frac{dC_{DIPA}}{dt}\right)_0 = -r_{DIPA0} = 0.779 C_{H_2O_2}^{0.141} C_{DIPA}^{1.561} C_{Fe^{2+}}^{-0.26} \quad (4.7)$$

$$r = k \frac{[H_2O_2]^{0.141} [DIPA]^{1.561}}{[Fe^{2+}]^{-0.26}} \quad (4.8)$$

Therefore, the order of the reaction will be $0.141 + 1.561 + (-0.26) = 1.442$, indicating a complex *fractional order reaction*.

The empirical rate law obtained shows that the degradation of DIPA's rate of reaction is directly proportional to the initial concentration of Hydrogen Peroxide to the power of 0.141 and initial concentration of DIPA to the power of 1.561. The rate is however inversely affected to the initial concentration of Fe^{2+} to the power of 0.260. The proportionality constant, k, or the rate constant is 0.779. It is safe to say that this reaction is (141/1000)th order in H_2O_2 and (1561/1000)th order in DIPA. From this, we can see that the rate of reaction is much depending on initial concentration of DIPA followed by Hydrogen Peroxide. Though concentration of Fe^{2+} also affects the rate of concentration, it affects inversely due to the reversible reaction that might occur. Increasing Fe^{2+} will up to one point, decrease the rate of reaction.

The overall order of this reaction is (1442/1000) order which is the sum of all the orders of the reactants. This indicates a fractional order reaction, in which indicates the complexity of the reaction mechanism

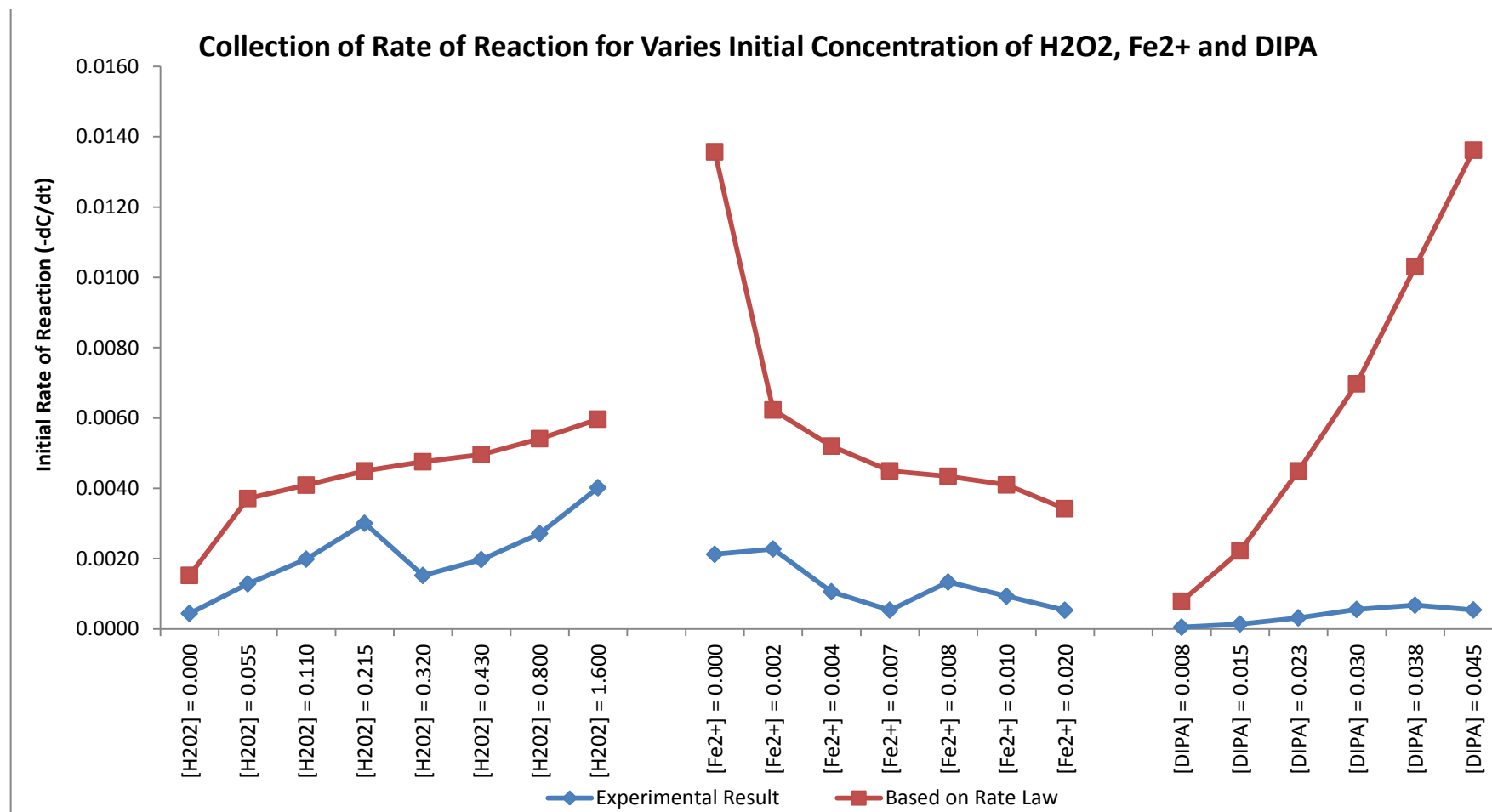


Figure 14: Comparison of Experimental Rate of Reaction with Theoretical Rate

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

As a conclusion, by studying the effect of different initial concentration of ferrous, hydrogen peroxide and DIPA, we were able to determine the empirical rate law for the degradation of DIPA using photo-Fenton. In order to do so, it is important to first conducting the experiment for the degradation of DIPA using photo-Fenton by varying the initial concentration of Fe^{2+} , Hydrogen Peroxide and DIPA itself.

Based on the results obtained, the initial concentration of diisopropanolamine (DIPA) gives the most direct effect to the rate of reaction, followed by initial concentration of Hydrogen Peroxide. However, negative order shown by Fe^{2+} indicates that the initial concentration of Fe^{2+} gives inverse effect towards the rate of reaction. Overall empirical rate law shows that the reaction is a complex, fractional order reaction.

Therefore, the objectives are achieved.

5.2 RECOMMENDATIONS

- a) The experiment is to be repeated at least three times for each sample for a better result. The value used would be the average from all three samples.
- b) No catalyst would be used for the experiment as the time taken to analyse the sample can be shorten neglecting the catalyst effect.

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APPENDICES

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APPENDIX 1: PROPERTIES OF DIPA

Property	Unit	Value
CAS registry number	-	110-97-4
Molecular formula	-	C ₆ H ₁₅ NO ₂
Molecular weight	g mol ⁻¹	133.19
Melting point	° C	44
Boiling point	° C	249
Specific gravity 20° C (DIPA) /4° C (Water)	-	1.004
40° C (DIPA) /4° C (Water)	-	0.992
Flashpoint	° C	126
Density at 25° C	g cm ⁻³	0.989
Vapour density (air=1)	g L ⁻¹	4.6
Vapour pressure 42° C	mm Hg	0.02
50° C	mm Hg	0.035
100° C	hPA	3
n-Octanol-water partition coefficient (Kow)	log	0.79
Organic carbon partition coefficient (Koc)	log	21.77
Henry's law constant	atm m ³ . mol ⁻¹	1.72 x 10 ⁻⁷
Solubility in water 25° C	g L ⁻¹	870
Water soil partition coefficient (Kd) montmorillonite	L/kg	16-42
kaolinite	L/kg	3.5
humus-rich soil	L/kg	2.0
low carbon content surface soils	L/kg	0.73-4.0
till	L/kg	3.2
sandstone, shale/sandstone	L/kg	0.54-1.1
mean value for nine soils/sediments	L/kg	2.2
pKa	-log K	8.88
Viscosity 30° C	centipoise	870
54° C	centipoise	86

APPENDIX 2: SAMPLE CALCULATION

For total reactor volume of 250mL

Considering concentration of **H₂O₂ = 0.215 M, DIPA = 0.023M and Fe²⁺ = 0.007M**

$$\begin{aligned}\text{Volume of H}_2\text{O}_2 \text{ (mL)} &= 0.215 \frac{\text{mol}}{\text{L}} \times 34.01 \frac{\text{g}}{\text{mol}} \times 1/1.135 \frac{\text{mL}}{\text{g}} \times \frac{100\%}{30\%} \times 0.25\text{L} \\ &= 5.369\text{mL}\end{aligned}$$

$$\begin{aligned}\text{Volume of DIPA (mL) required} &= 250\text{mL} - 5.369 \text{ mL} \\ &= 244.631 \text{ mL}\end{aligned}$$

As DIPA is solid at room temperature, it is important to first calculate the amount of DIPA required. The total weight required to meet the concentration requirement of 0.023 M is calculated as below:

$$\begin{aligned}\text{Weight of DIPA (g)} \\ &= 0.023 \frac{\text{mol}}{\text{L}} \times 133.2 \frac{\text{g}}{\text{mol}} \times 1 \text{ L (to prepare 1L of a M DIPA)} \\ &= 2.997\text{g}\end{aligned}$$

2.997g of DIPA is then diluted in 1L of distilled water and 244.631 mL of the DIPA is taken from this solution.

For Fe²⁺, the amount of Ferrous (II) Sulphate requires to meet the concentration of 0.007 M is calculated as below:

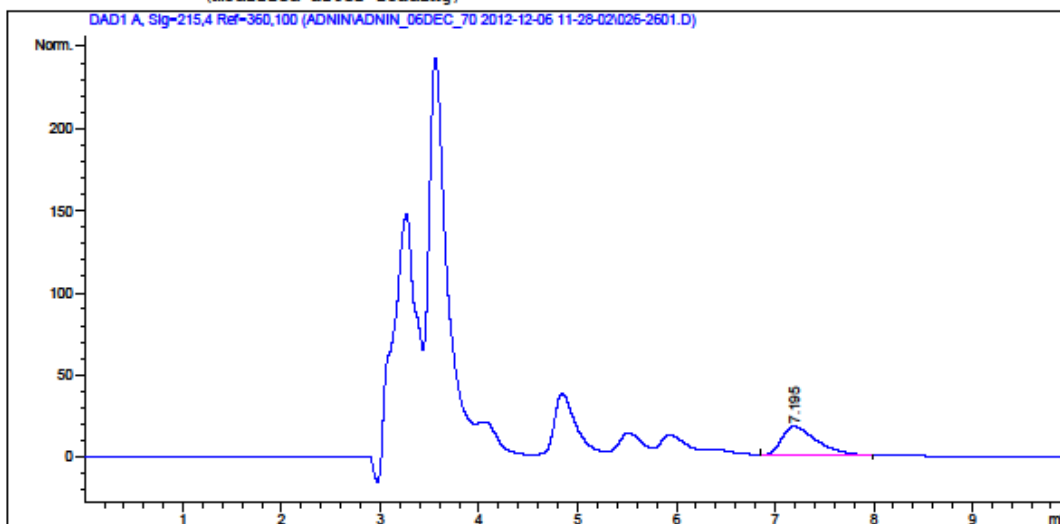
$$\begin{aligned}\text{Weight of Ferrous (II) Sulphate (g)} \\ &= 0.007 \frac{\text{mol}}{\text{L}} \times 55.845 \frac{\text{g}}{\text{mol}} \text{Fe}^{2+} \times 0.25 \text{ L} \times \frac{278.15 \text{ g (Ferrous (II) Sulphate)}}{55.845 \text{ g (Fe}^{2+}\text{)}} \\ &= 0.452\text{g}\end{aligned}$$

APPENDIX 3: EXAMPLE OF ANALYSIS RESULT FROM HPLC

Data File C:\CHEM32\1\DATA\ADNIN\ADNIN_06DEC_70 2012-12-06 11-28-02\026-2601.D
 Sample Name: RAI_B5-6

```

=====
Acq. Operator   : NIAMNAH                      Seq. Line : 26
Acq. Instrument : Instrument 1                  Location  : Vial 26
Injection Date  : 12/6/2012 4:23:38 PM        Inj       : 1
                                           Inj Volume: 20 µl
Acq. Method     : C:\CHEM32\1\DATA\ADNIN\ADNIN_06DEC_70 2012-12-06 11-28-02\MALAYA_DEA.M
Last changed    : 11/26/2012 12:22:19 PM by ADNIN
Analysis Method : C:\CHEM32\1\METHODS\ADNIN_DIPASTD.M
Last changed    : 12/6/2012 4:55:52 PM by NIAMNAH
                  (modified after loading)
=====
  
```



External Standard Report

```

=====
Sorted By       : Signal
Calib. Data Modified : 12/6/2012 4:55:54 PM
Multiplier:     : 1.0000
Dilution:       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
  
```

Signal 1: DAD1 A, Sig=215,4 Ref=360,100

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [g/l]	Grp	Name
7.195	BB +	413.08163	1.05547e-2	4.35997e-1		DIPA

Totals : 4.35997e-1

APPENDIX 4: TOOLPAK ANALYSIS SOLVER IN MICROSOFT OFFICE EXCEL

The screenshot displays the Microsoft Excel interface with the following components:

- Ribbon:** Home, Insert, Page Layout, Formulas, Data, Review, View, Acrobat.
- Data Tools Group:** Contains 'Data Analysis Solver' and 'Analysis' buttons, both highlighted with black boxes. An arrow points from the 'Data Analysis Solver' button in the ribbon to the dialog box.
- Data Analysis Solver Dialog Box:** A floating window titled 'Data Analysis Solver' with 'Analysis' at the bottom.
- Spreadsheet Content:**
 - Row 20: SUMMARY OUTPUT
 - Row 22: *Regression Statistics*
 - Row 23: Multiple R: 0.907032167
 - Row 24: R Square: 0.822707353
 - Row 25: Adjusted R Square: 0.734061029
 - Row 26: Standard Error: 0.123932431
 - Row 27: Observations: 10
 - Row 29: ANOVA
 - Row 30: ANOVA table with columns: *df*, *SS*, *MS*, *F*, *Significance F*
 - Row 31: Regression: 3, 0.427638, 0.142546, 9.280784, 0.011349
 - Row 32: Residual: 6, 0.092155, 0.015359
 - Row 33: Total: 9, 0.519793
 - Row 35: *Coefficients*, *Standard Error*, *t Stat*, *P-value*, *Lower 95%*, *Upper 95%*, *Lower 99.0%*, *Upper 99.0%*
 - Row 36: Intercept: 3.568353157, 2.094378, 1.703777, 0.139311, -1.5564, 8.693111, -4.1964, 11.33311
 - Row 37: X Variable 1: 0.58415558, 0.154508, 3.780739, 0.009172, 0.206087, 0.962224, 0.011327, 1.156984
 - Row 38: X Variable 2: -0.13628979, 0.507686, -0.26845, 0.797342, -1.37855, 1.105973, -2.0185, 1.745919
 - Row 39: X Variable 3: -0.09305815, 0.053725, -1.73212, 0.133962, -0.22452, 0.038402, -0.29224, 0.106124