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APPENDICES

APPENDIX A
Analytical Method for H_2O_2

Analytical Method of Hydrogen Peroxide
(Spectrophotometer Determination of Hydrogen Peroxide using Potassium Titanium (IV) Oxalate, Sellers, 1980)

1. General Discussion

A specific spectrophotometric test based on the formation of a complex, often written as TiO_2^{2+}, between hydrogen peroxide and the titanium (IV) ion. Organic peroxides and many other oxidizing agents convert I^- into I_3^-. One of the most sensitive and widely used is based on the oxidation of I^- to I_3^{2-}, although the analysis is usually performed in the presence of molybdate, a specific catalyst of the reaction. These too measure total peroxides, rather than hydrogen peroxide alone.

The titanium (IV) - peroxide complex is yellow to orange in color and absorbs with a λ_{max} of about 400 nm. The intensity of this absorption was found to be dependent on the concentrations of titanium (IV) oxalate and sulfuric acid. At higher concentrations the intensity of the absorbance of the titanium (IV) - peroxide complex was much reduced. The λ_{max} for the absorption increased with decreasing titanium (IV) oxalate concentration or increasing sulfuric acid concentration.

2. Apparatus

2.1 Colorimetric Equipment

Spectrophotometer (Shimadsu UV-1201): The absorbance used for hydrogen peroxide (H_2O_2) analysis was 400 nm.

2.2 Reagent

Titanium reagent: Mix 27.2 ml of concentrated H_2SO_4 with about 300 ml of DI water. Dissolve in this mixture 35.4 g potassium titanium(IV) oxalate, $K_2TiO(C_2O_4)_2.2H_2O$, and made up to 1 L.

3. Procedure

3.1 Sample Preparation for Calibration Curves: The standard H_2O_2 solutions were prepared in the range 0.1 to 5 mM as H_2O_2. Pipette 5 ml of titanium reagent and 1 ml 0.1, 0.5, 1, 2 and 5 mM as H_2O_2 into 25 ml volumetric flask and made up to the mark, mixed thoroughly.

3.2 Hydrogen Peroxide Analysis: To determine H_2O_2, 5 ml of titanium from stock solution was prepared in 25 ml volumetric flask. Then, 1 ml of sample was pipetted and diluted to 25 ml with DI water. After that, the samples were immediately measured by spectrophotometer at 400 nm.

4. Preparing for Calibration Curve

The standard H_2O_2 solution were prepared in the range of 0.1 to 5 mM

1. 5 ml of $K_2TiO(C_2O_4)_2$ from stock solution was prepared in five 25 ml volumetric flasks.

2. 1 ml of 0.1,0.5, 1, 2 and 5 mM as H_2O_2 standard solution were pipetted, respectively.

3. The samples were diluted to 25 ml with DI water, mixed thoroughly.

4. The samples were immediately measured by spectrophotometer at 400 nm.

5. For blank, was followed step 1-4 and was added 2 drops of 1:4 H_2SO_4

6.Calculation

$Y = 25.391x$

Where $Y = H_2O_2$, mM

X = absorbance at 400 nm

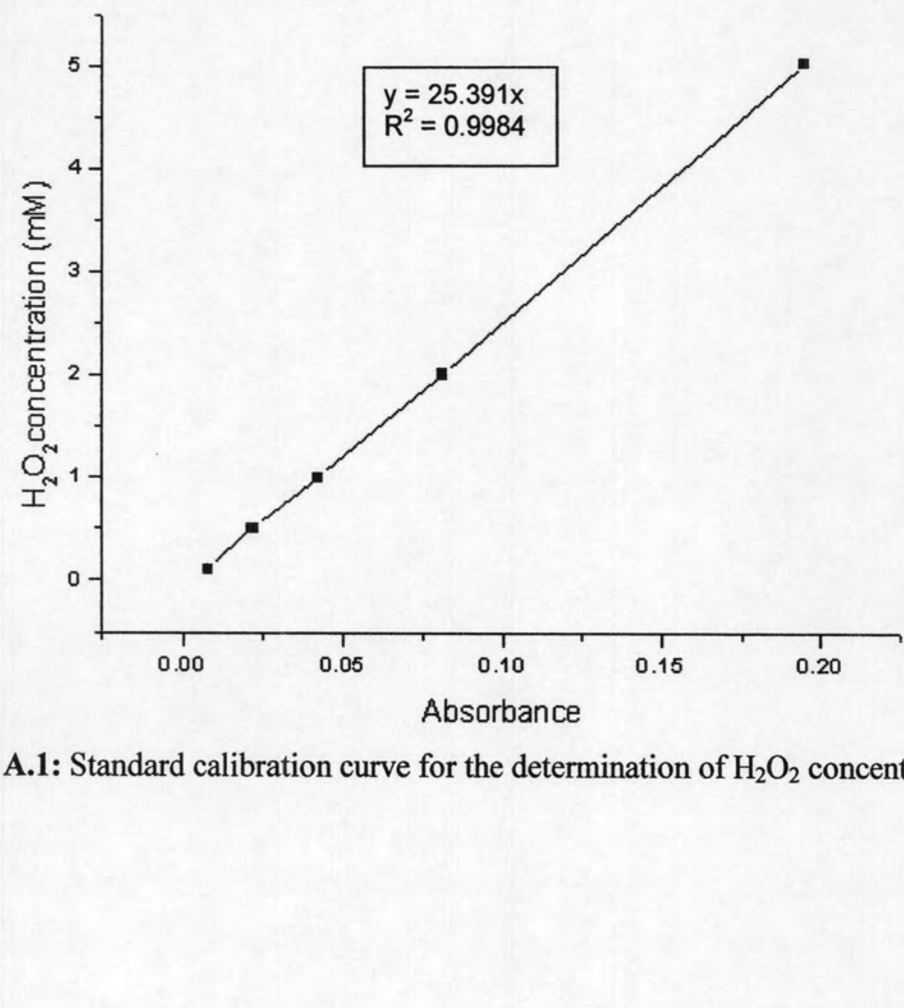

Figure A.1: Standard calibration curve for the determination of H_2O_2 concentration

APPENDIX B
Experimental Figures

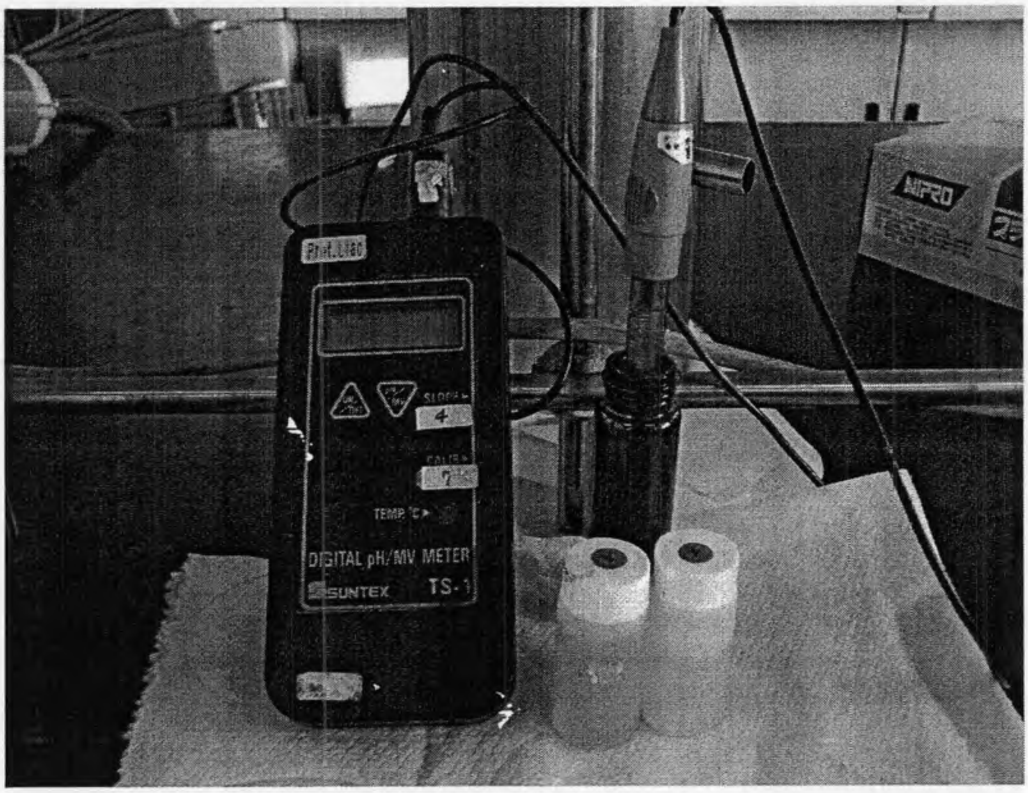

Figure B.1: pH meter

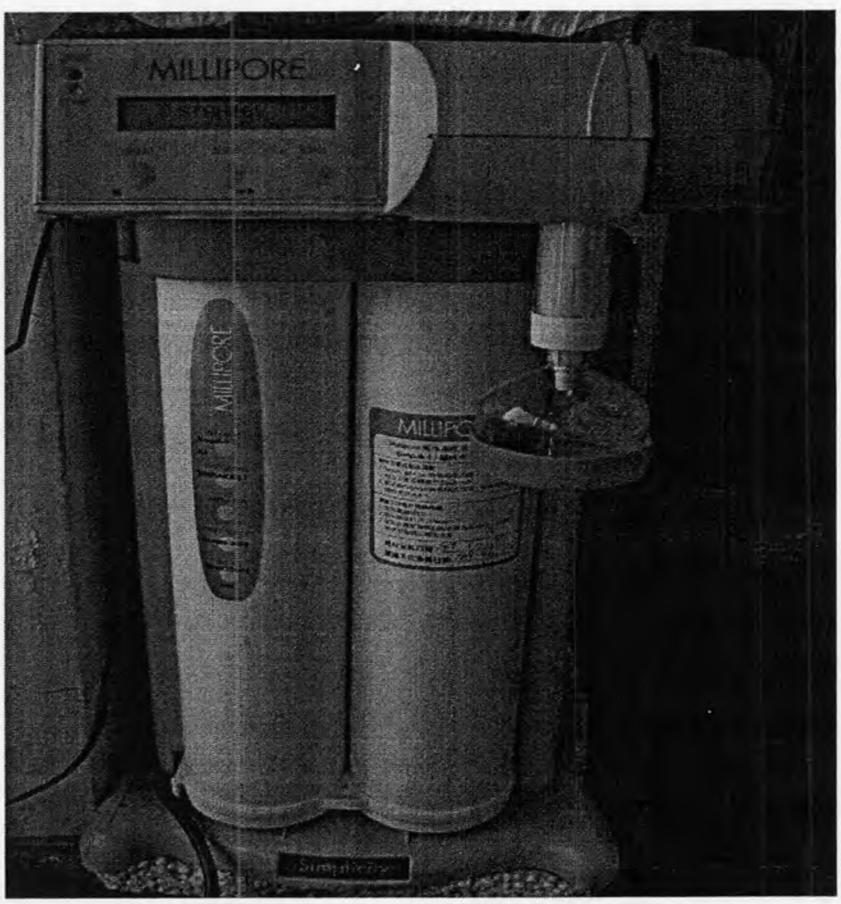

Figure B.2: DI water

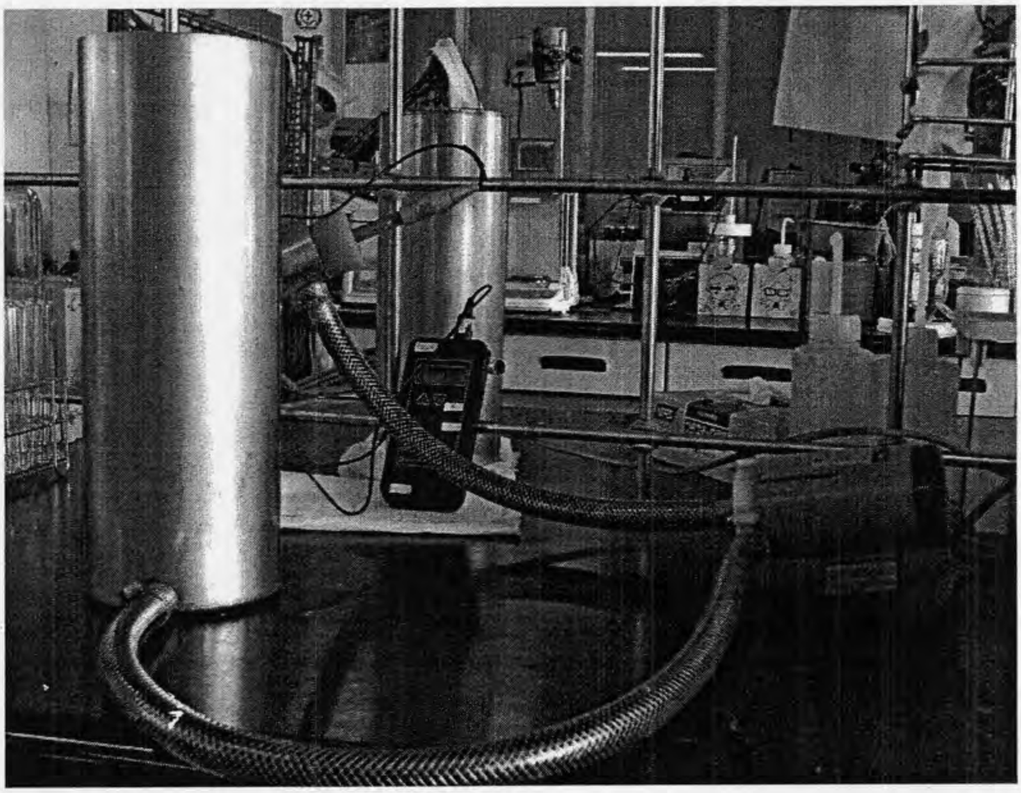

Figure B.3: Fenton reactor setup

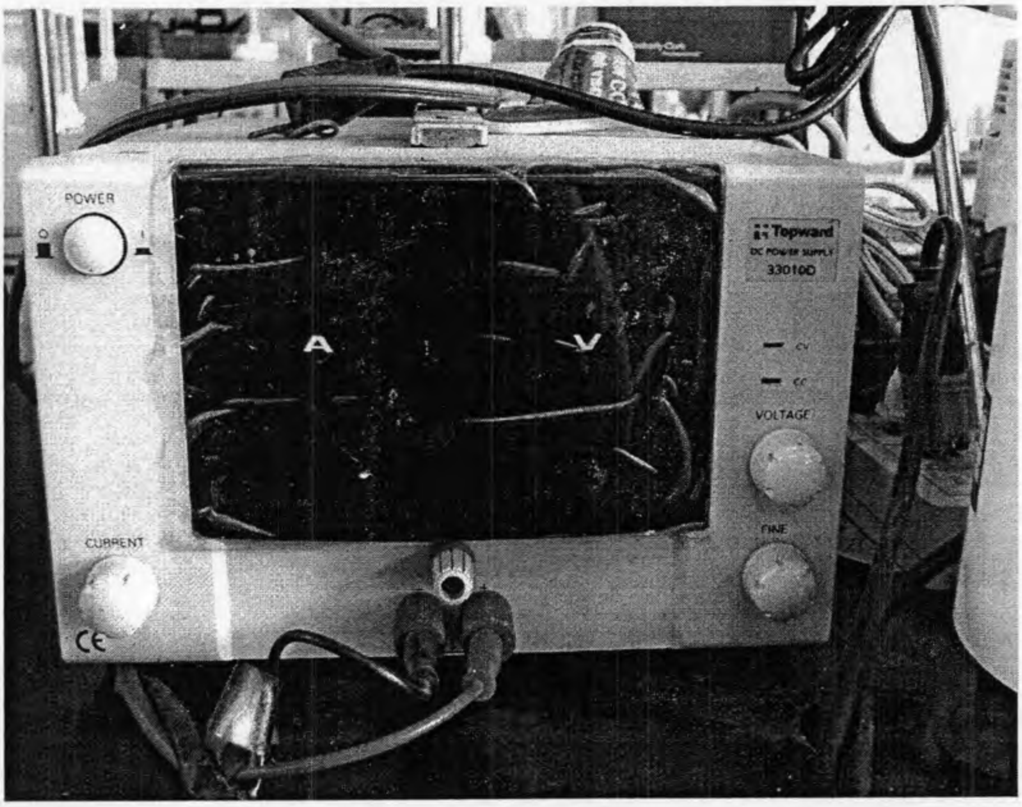

Figure B.4: DC power supply

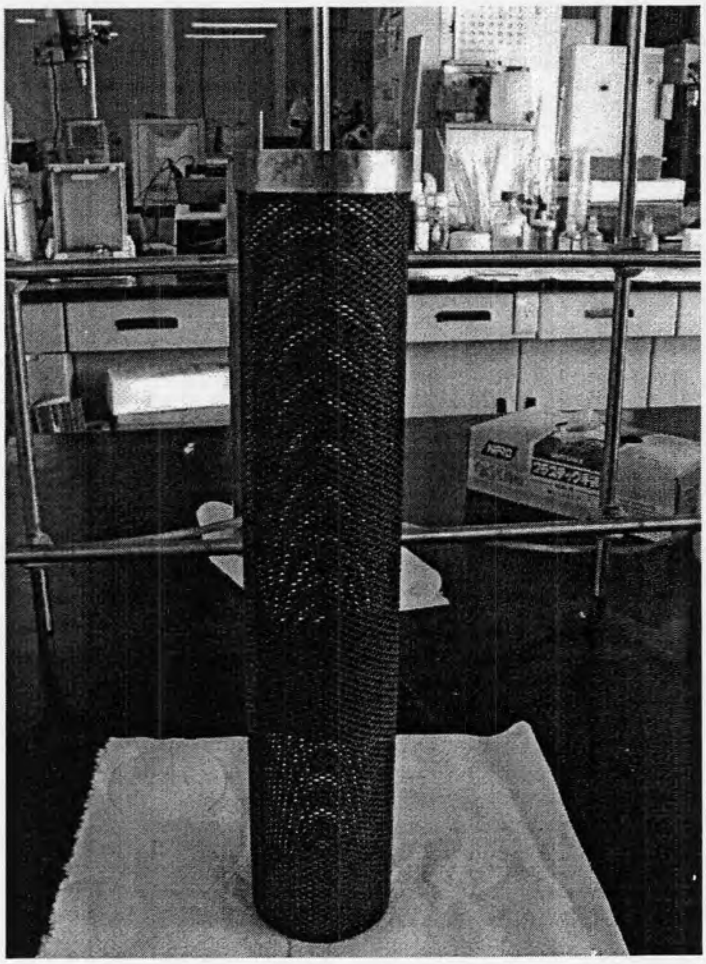

Figure B.5: Electrode (Anodic)

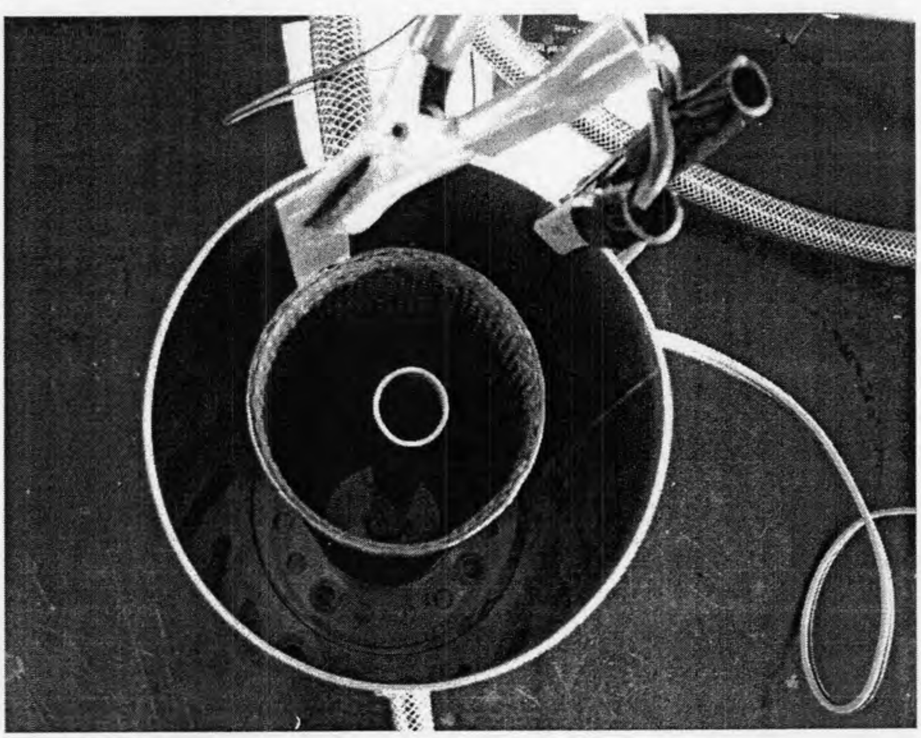

Figure B.6: Top view of electro-Fenton setup

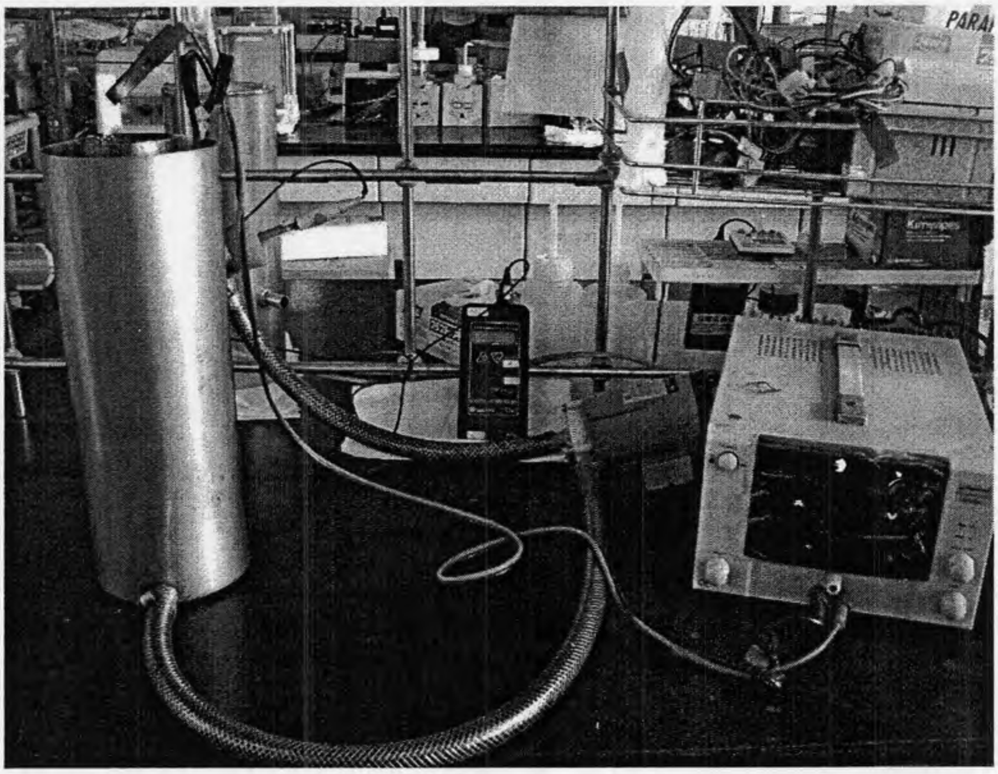

Figure B.7: Electro-Fenton setup

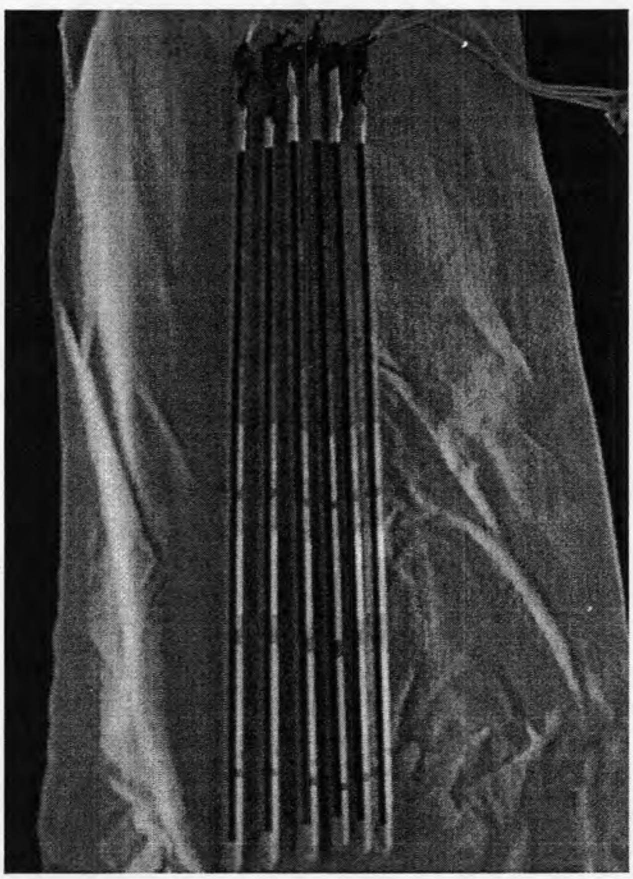

Figure B.8: UV lamps

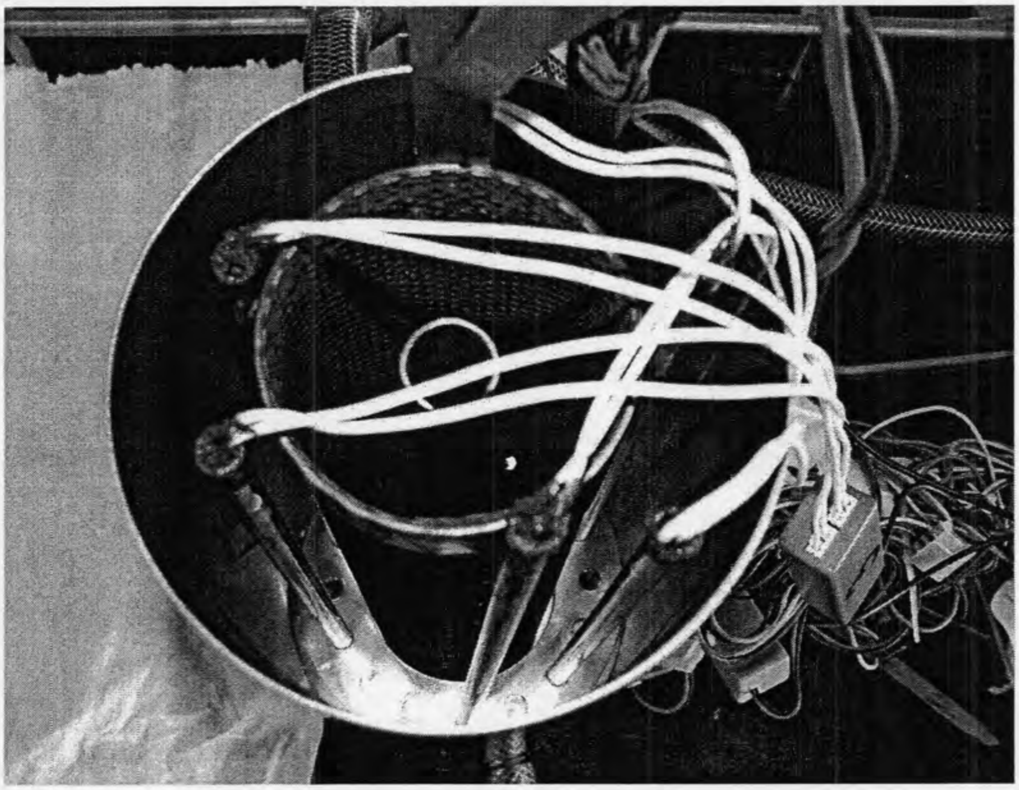

Figure B.9: Top view of photoelectro-Fenton setup

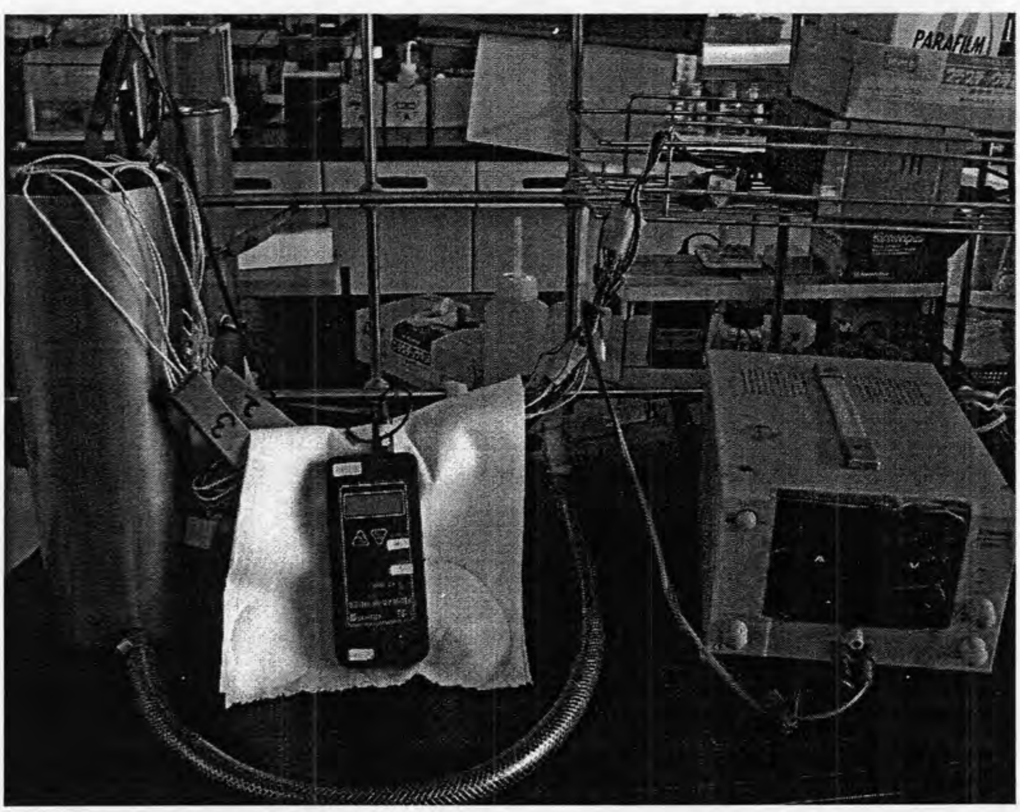

Figure B.10: Photoelectro-Fenton setup

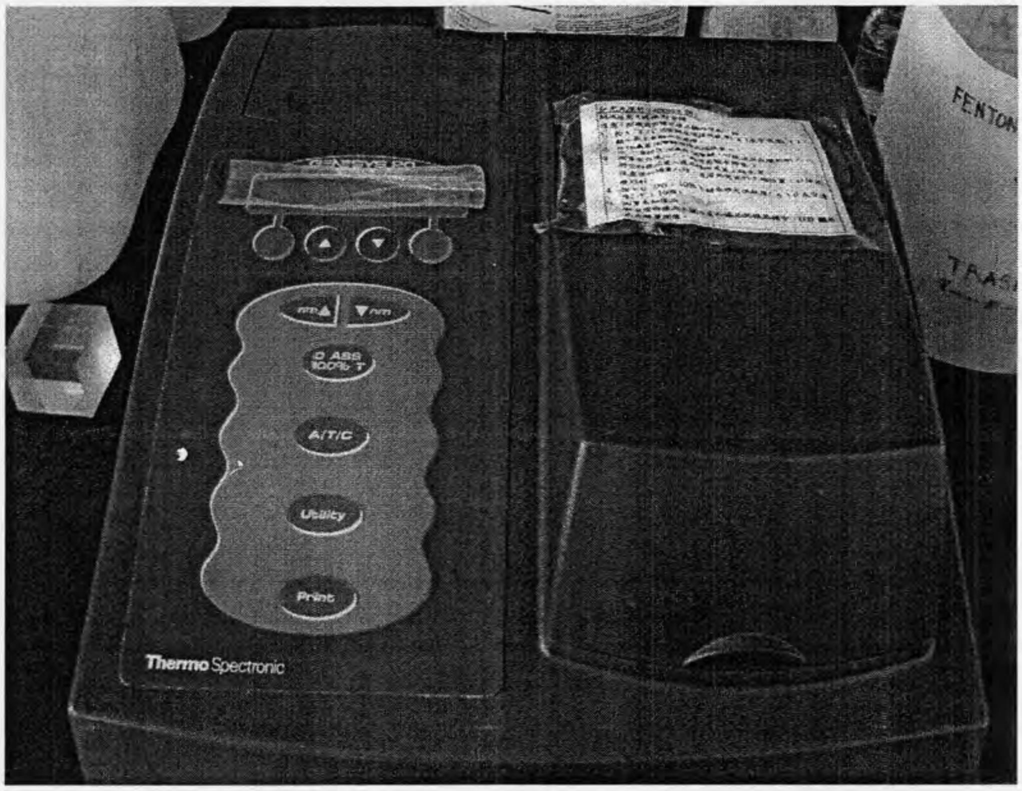

Figure B.11: Spectrophotometer

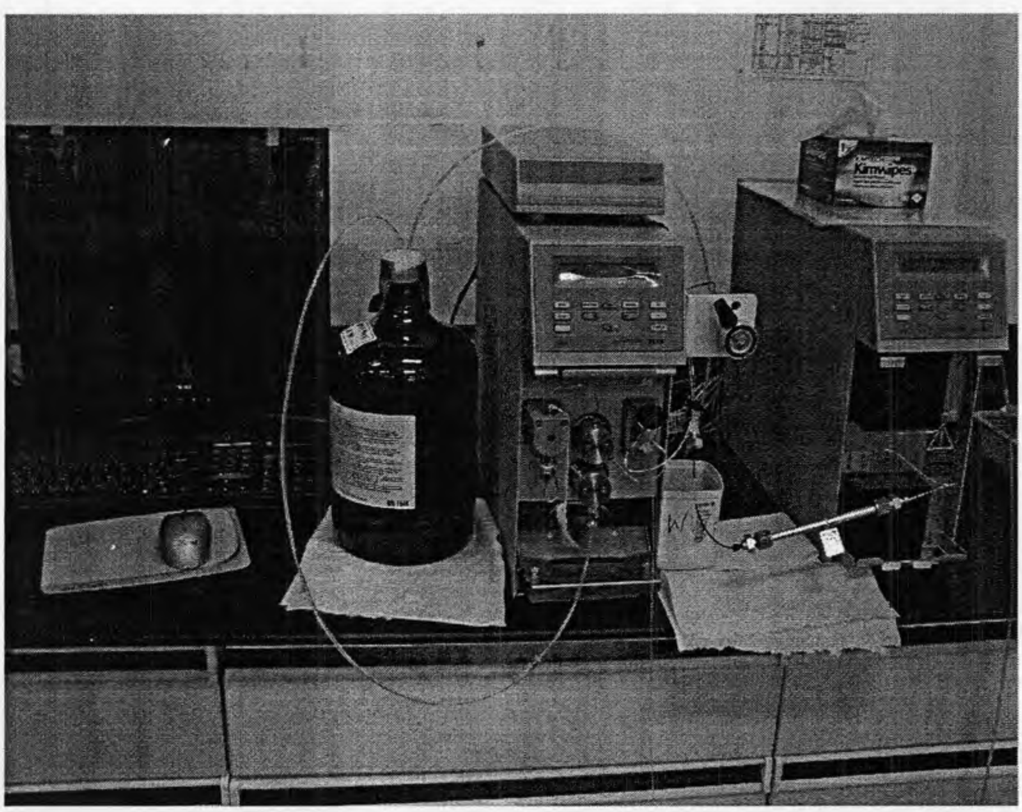

Figure B.12: HPLC

Figure B.13: IC

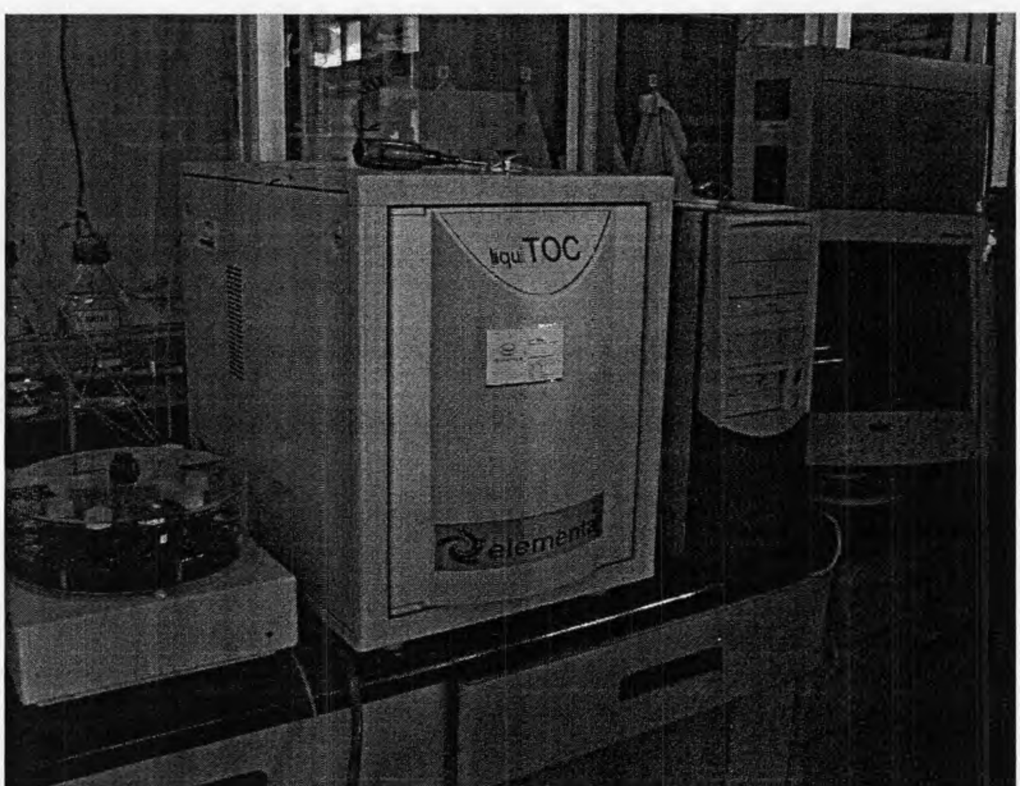

Figure B.14: TOC

APPENDIX C
Experimental Data

Table C.1: Effect of processes on the OT oxidation

Time (min)	C/Co							
	EL	PL	PEL	H_2O_2	H_2O_2 + UV	H_2O_2 + current	Fe^{2+} + current	Fe^{2+} + UV
0	1	1	1	1	1	1	1	1
1	0.9950	0.9804	0.9439	0.9987	0.9968	0.989	0.9967	0.994
2	0.9884	0.9558	0.9715	0.9962	0.9839	0.992	0.9978	0.9947
5	0.9979	0.9659	0.9643	0.9864	0.9746	0.9912	0.9955	0.9941
10	0.9965	0.9510	0.9830	0.9903	0.9058	0.9941	0.9941	0.9955
15	0.9999	0.9533	0.9397	0.9998	0.9952	0.9947	0.9949	0.9887
30	0.9970	0.9428	0.9466	0.9871	0.9762	0.9928	0.9995	1.0017
45	0.989	0.9785	0.9460	1.0125	0.9061	0.9936	0.9937	0.9962
60	1.0015	0.9928	0.9558	0.9997	0.9320	0.9955	0.9956	0.997
90	0.9966	0.9977	0.9913	0.9973	0.9317	0.9940	0.9964	0.9939
120	1.0023	0.9816	0.9619	0.9982	0.9319	0.9916	0.9933	0.9989

EL = Electrolysis (current alone)
PL = Photolysis (UV alone)
PEL = Photo-electrolysis (current and UV)

Note: At condition pH 2, $[Fe^{2+}]$ = 1.0 mM, $[H_2O_2]$ = 4.85 mM, current = 1 A

Table C.2: Effect of processes on the COD removal

Time (min)	C/Co							
	EL	PL	PEL	H_2O_2	H_2O_2 + UV	H_2O_2 + current	Fe^{2+} + current	Fe^{2+} + UV
0	1	1	1	1	1	1	1	1
1	1	1.0412	1.0340	1.0185	1.0526	1.0869	1	1
2	09813	1.0618	1.0454	1.0740	1.0877	1.0434	1	1.025
5	1.0186	1.0618	1.0454	1.0740	1.0526	1.0869	1.0348	1.0375
10	1	1	1.0454	1.0925	1.0175	1	1.0232	1.025
15	1	1.0412	1.0113	1.0370	0.9473	0.9565	1.0348	1.05
30	1.0373	1.0103	1.0113	1.1481	1.0877	1.0434	1.0465	0.975
45	1.0280	1.0103	1.0340	1.1111	1.0526	1.0434	1.0465	1.025
60	1.0079	1.0412	1.0340	1.0555	0.9824	1.0869	1	1.05
90	1.0373	1.0412	1.0227	1.1481	1.0877	1.0869	0.9767	0.9875
120	1.0373	1	1.0113	1.1296	0.9824	1.0434	1	1.0375

EL = Electrolysis (current alone)
PL = Photolysis (UV alone)
PEL = Photo-electrolysis (current and UV)

Note: At condition pH 2, $[Fe^{2+}]$ = 1.0 mM, $[H_2O_2]$ = 4.85 mM, current = 1 A

Table C.3: OT degradation by several Fenton processes

Time (min)	C/Co		
	Fenton	EF	PEF
0	1	1	1
1	0.607222	0.653625	0.591039
2	0.595057	0.558091	0.570061
5	0.580253	0.494105	0.512885
10	0.571675	0.342679	0.360843
15	0.554022	0.26999	0.261471
30	0.520137	0.226299	0.129119
45	0.501662	0.117185	0.090787
60	0.435451	0.092211	0.007399
90	0.301164	0.019231	0.005347
120	0.100744	0	0

EF = electro-Fenton
PEF = photoelectro-Fenton

Note: At pH 2, $[Fe^{2+}]$ = 1.0 mM, $[H_2O_2]$ = 4.85 mM, current = 1 A

Table C.4: COD removal by several Fenton processes

Time (min)	C/Co		
	Fenton	EF	PEF
0	1	1	1
1	0.963636	0.909091	0.861386
2	0.872727	0.863636	0.821782
5	0.854545	0.772727	0.772277
10	0.818182	0.712121	0.722772
15	0.80000	0.712121	0.722772
30	0.781818	0.666667	0.673267
45	0.763636	0.606061	0.633663
60	0.727273	0.545455	0.574257
90	0.690909	0.5	0.544554
120	0.636364	0.5	0.475248

EF = electro-Fenton
PEF = photoelectro-Fenton

Note: At condition pH 2, $[Fe^{2+}]$ = 1.0 mM, $[H_2O_2]$ = 4.85 mM, current = 1 A

Table C.5: The oxidation of maleic acid by various Fenton processes

Time (min)	Maleic acid (mM)		
	Fenton	EF	PEF
0	0	0	0
1	0	0.28033	0.1545
2	0	0.3112	0.23491
5	0	0.35618	0.23579
10	0.05924	0.40028	0.27239
15	0.07644	0.41924	0.27063
30	0.11069	0.44056	0.25946
45	0.11422	0.47378	0.22594
60	0.11451	0.35736	0.21903
90	0.13524	0.29753	0.20286
120	0.1545	0.28033	0.1986

EF = electro-Fenton
PEF = photoelectro-Fenton

Note: At condition pH 2, $[Fe^{2+}]$ = 1.0 mM, $[H_2O_2]$ = 4.85 mM, current = 1 A

Table C.6: The oxidation of oxalic acid by various Fenton processes

Time (min)	Oxalic acid (mM)		
	Fenton	EF	PEF
0	0	0	0
1	0	0.03591	0.02282
2	0	0.05334	0.0238
5	0	0.07217	0.02702
10	0.02016	0.1477	0.0441
15	0.04501	0.16205	0.08722
30	0.04613	0.18774	0.11151
45	0.06384	0.13202	0.11641
60	0.07266	0.11753	0.10234
90	0.16877	0.10822	0.08001
120	0.35028	0.11032	0.06965

EF = electro-Fenton
PEF = photoelectro-Fenton

Note: At condition pH 2, $[Fe^{2+}]$ = 1.0 mM, $[H_2O_2]$ = 4.85 mM, current = 1 A

Table C.7: TOC removal of OT solution by electro-Fenton and photoelectro-Fenton processes

Time (min)	Concentration	
	EF	PEF
0	1	1
5	0.976982	0.99449
10	0.943734	0.958678
15	0.914322	0.947658
30	0.883632	0.849862
45	0.883632	0.847107
60	0.882353	0.816804
90	0.882353	0.706612
120	0.882353	0.693837

EF = electro-Fenton
PEF = photoelectro-Fenton

Note: At condition pH 2, $[Fe^{2+}]$ = 1.0 mM, $[H_2O_2]$ = 4.85 mM, current = 1 A

APPENDIX D
Reaction rate Determination

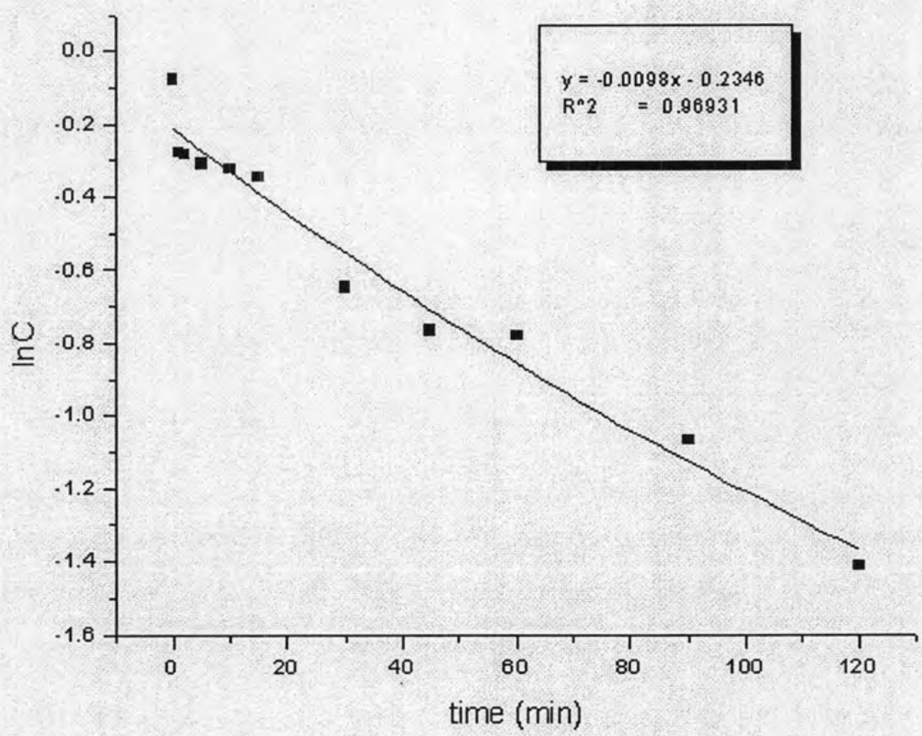

Figure D.1: First-order kinetic reaction determination for the whole reaction of an example from the results of Design-Expert software by electro-Fenton process at pH 2, $[Fe^{2+}]$ = 0.2 mM, $[H_2O_2]$ = 5 mM and 4 A of current

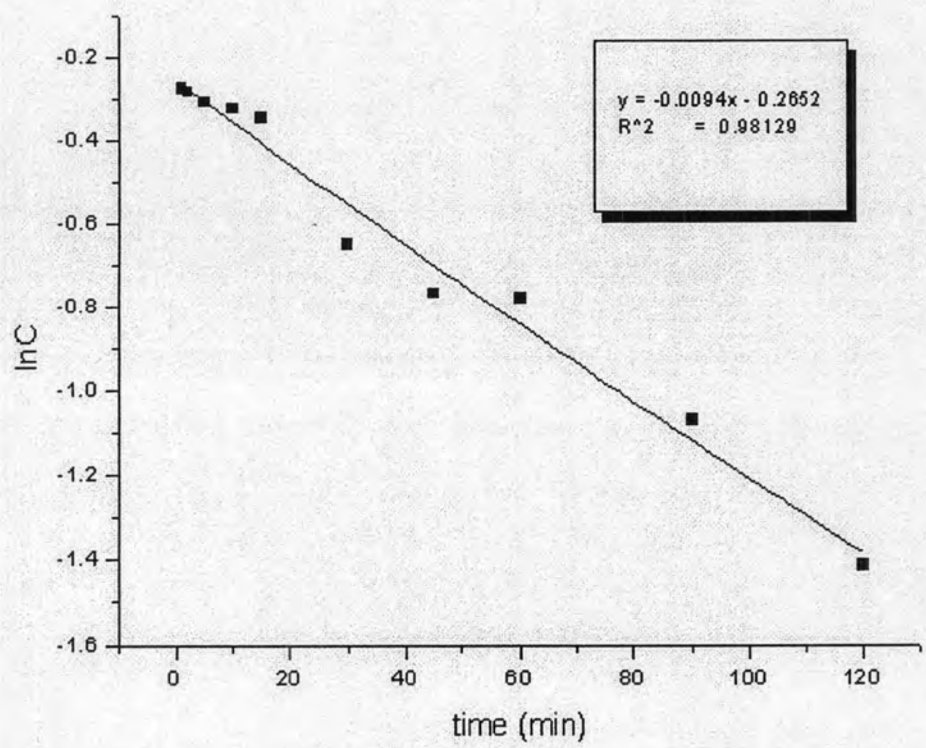

Figure D.2: First-order kinetic reaction determination for no initial reaction of an example from the results of Design-Expert software by electro-Fenton process at pH 2, $[Fe^{2+}]$ = 0.2 mM, $[H_2O_2]$ = 5 mM and 4 A of current

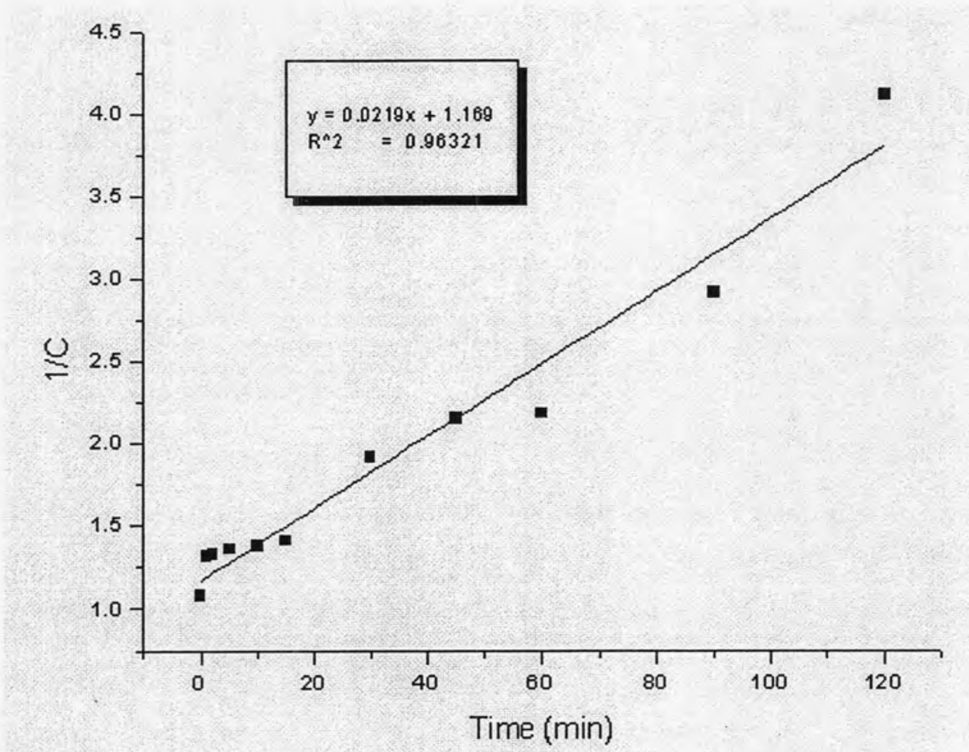

Figure D.3: Second-order kinetic reaction determination for the whole reaction of an example from the results of Design-Expert software by electro-Fenton process at pH 2, $[Fe^{2+}]$ = 0.2 mM, $[H_2O_2]$ = 5 mM and 4 A of current

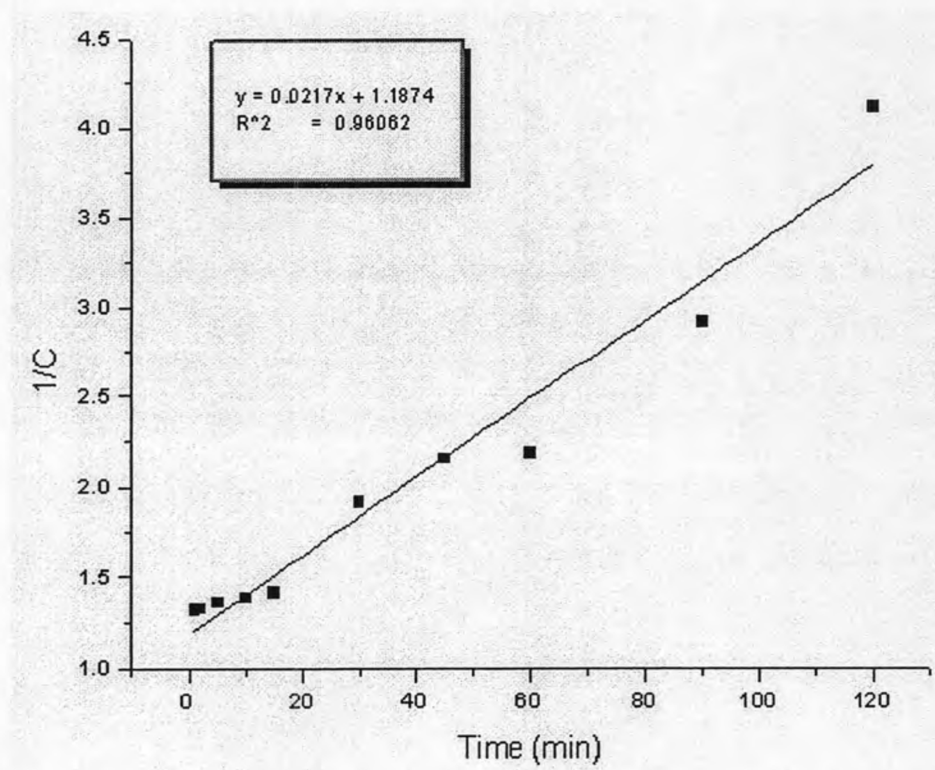

Figure D.4: Second-order kinetic reaction determination for no initial reaction of an example from the results of Design-Expert software by electro-Fenton process at pH 2, $[Fe^{2+}]$ = 0.2 mM, $[H_2O_2]$ = 5 mM and 4 A of current

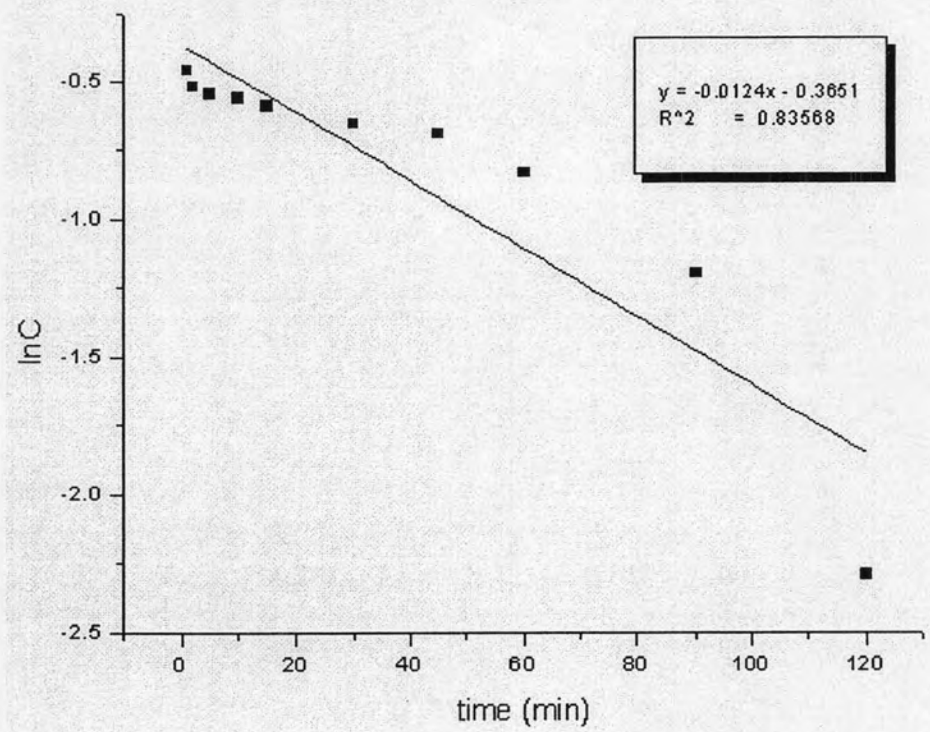

Figure D.5: First-order kinetic reaction for Fenton process at the optimum condition; pH 2, $[Fe^{2+}]$ = 1 mM, $[H_2O_2]$ = 4.85 mM and 1 A of current

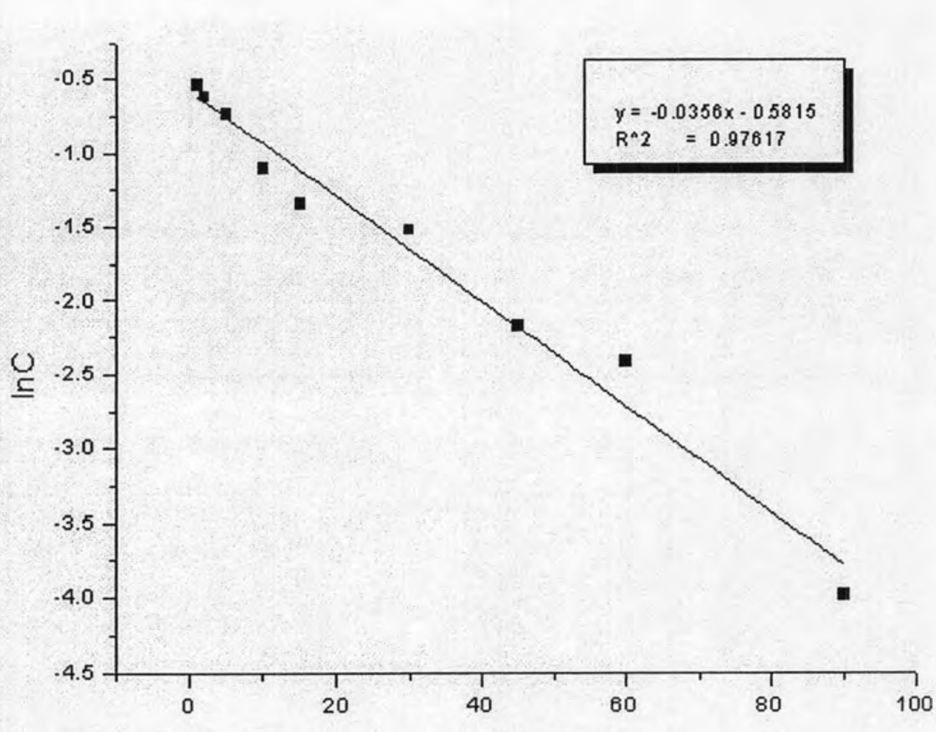

Figure D.6: First-order kinetic reaction for electro-Fenton process at the optimum condition; pH 2, $[Fe^{2+}]$ = 1 mM, $[H_2O_2]$ = 4.85 mM and 1 A of current

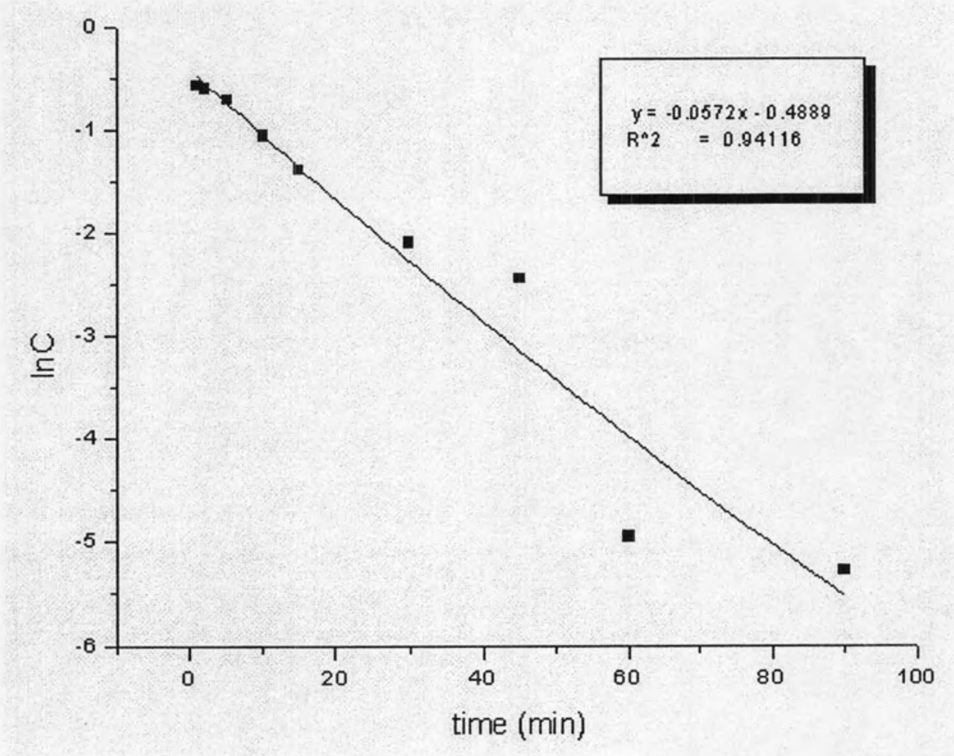

Figure D.7: First-order kinetic reaction for photoelectro-Fenton process at the optimum condition; pH 2, $[Fe^{2+}]$ = 1 mM, $[H_2O_2]$ = 4.85 mM and 1 A of current

BIOGRAPHY

Miss Somporn Singhadech was born on December 16, 1985 in Bangkok, Thailand. She received her Bachelor's degree with honor in Department of Environmental Science from Faculty of Science, Silpakorn University, Nakhon Pathom, Thailand in 2007. She pursued her Master's degree study in the National Center of Excellence for Environmental and Hazardous Waste Management, Inter-Department of Environmental Management, Graduate School, Chulalongkorn University, Bangkok, Thailand on May, 2007. She finished her Master's degree on March, 2009. She had published part of her works entitled "Degradation of o-Toluidine by electro-Fenton Process" in the 8^{th} National Environmental Conference on Wastewater and Wastewater Treatment Technology on March 25-27, 2009 in Nakohnratchasima.