DECOLOURISATION OF DYE SOLUTION CONTAINING AZO ACID ORANGE 7 BY ELECTRICITY



BUR SHAYLINDA BINTI MOHD ZIN

UNIVERSITI TEKNOLOGI MALAYSIA

PERPUSTAKAAN UTHM



UNIVERSITI TEKNOLOGI MALAYSIA

UDUL:				ION CONTAINING
•	- · · · · · · · · · · · · · · · · · · ·	AZO ACID ORAN	NGE 7 BY E	LECTRICITY
		SESI PENGAJIAN: _	2005/2006	
aya		NUR SHAYLIND	A BINTI M	OHD ZIN
		tesis (PSM/Sarj		-Falsafah)* ini disimpan syarat-syarat kegunaan sepe
. Tesis a	dalah hakmilik V	Universiti Teknolog	gi Malaysia.	
_		i Teknologi Malay	sia dibenarka	an membuat salinan untuk tuju
. Perpusi	an sahaja. takaan dibenarka i pengajian tingg a tandakan (✔)		tesis ini seb	agai bahan pertukaran antara
	SULIT TERHAD ERPUS	kepentingan M AKTA RAHSIA (Mengandungi	alaysia sepe A RASMI 19 makluma	1KO LOL
✓	TIDAK TEI	RHAD		
				Disahkan oleh
	My	2	F	Ta any
(T.A	NDATANGAN	PENULIS)	(TANDAT	ANGAN PENYELIA)
Alamat tet	ap:			
41, JA	LAN INDAH 1	/1,	ACCOCT	TE DOOFFEEOOD DD EAD
TAMA	N AMPANG I	NDAH,	OTHMAI	ATE PROFESSOR DR. FADI N
	SELANGOR D	ARUL EHSAN	Nama	Penyelia
68000				

"I hereby declare that I have read this project report and in my opinion this project report is sufficient in terms of scope and quality for the award of the degree of Master of Engineering (Civil-Environmental Management)

Signature: Jakumaka Tunku Tun Aminah

Name of Supervisor: Associate Professor Dr. Fadil Othman

Date: 31 OLTOBER 2005

DECOLOURISATION OF DYE SOLUTION CONTAINING AZO ACID ORANGE 7 BY ELECTRICITY

NUR SHAYLINDA BINTI MOHD ZIN



Faculty of Civil Engineering Universiti Teknologi Malaysia AN TUNKU TUN AMINAH

I declare that this project report entitled "Decolourisation of dye solution containing Azo Acid Orange 7 by electricity" is the result of my own research except as cited in the references. The project report has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature:

Name: Nur Shaylinda Binti Mohd Zin

Date: 31 OCTOBER 2005



ACKNOWLEDGEMENT

In preparing this project, I was in contact with many people, researchers, academicians, and practitioners. They have contributed towards my understanding and thoughts. In particular, I wish to express my sincere appreciation to my project supervisor, Associate Professor Dr. Fadil Othman, for encouragement, guidance, critics and friendship. I am also very thankful to Encik Fadil Din for his guidance, advices and motivation. Without their continued support and interest, this thesis would not have been the same as presented here.

I am also indebted to Universiti Teknologi Malaysia (UTM) for the facilities and services. Librarians at UTM also deserve special thanks for their assistance in supplying the relevant literatures.

My fellow postgraduate students should also be recognized for their support. My sincere appreciation also extends to all my colleagues and others who have provided assistance at various occasions. Their views and tips are useful indeed. Unfortunately, it is not possible to list all of them in this limited space. I am grateful to all my family members.

ABSTRAK

Sisa berwarna daripada aktiviti industri memberi kesan kepada persekitaran dan kesihatan manusia. Pelbagai kaedah telah digunakan untuk menyahwarna sisa ini termaksuklah kaedah yang menggunakan aplikasi elektrik. Kajian ini dilaksanakan untuk menyelidik kelakuan larutan yang mengandungi bahan pewarna Azo asid oren 7 oleh elektrik. Fokus kajian ini menjurus kepada kesan penyahwarnaan oleh faktor ketumpatan arus elektirk, kepekatan pewarna dan tempoh aliran arus elektrik. Sel kimia yang mudah disediakan dengan menggunakan dua kepingan diperbuat daripada besi tulen dan besi campuran. Keberkesanan kaedah ini ditentukan melalui pengukuran peratus penyingkiran warna dan penyerapan. Peratus penyingkiran warna adalah lebih tinggi daripada peratus penyingkiran penyerapan. Semasa proses dijalankan apabila jumlah ketumpatan arus elektrik dan tempoh aliran arus elektrik semakin bertambah, penyahwarnaan turut meningkat. Berdasarkan analisis MINITABTM, ketumpatan arus elektrik, kepekatan pewarna dan tempoh aliran arus elektrik memang mempengaruhi peratus penyingkiran warna.. Kecekapan penyahwarnaan bagi kepekatan antara 100 mg/l dan 200 mg/l boleh ditingkatkan kepada 95% penyingkiran warna dan 88% penyingkiran penyerapan pada tempoh aliran arus elektrik 18 minit dan ketumpatan arus elektrik pada nilai 120 A/m².

ABSTRACT

Colouring effluent from industrial activities may affect environment and human health. Many methods have been used to decolourise such effluent including using electricity. This study was performed to investigate the behaviour of decolourisation of solution containing Azo Acid Orange 7. This study was focusing on the effect of decolourisation due to current density, dye concentration and duration of current flow. Simple electrochemical cell was prepared by using iron and steel plate electrode. The effectiveness of the method was determined by measuring percentage of colour and absorbance removal. The percentage of colour removal was higher then the percentage of absorbance removal. It is found that decolourisation was directly proportional to current density, duration of electric current flow and concentration of the dye. Based on MINITABTM analysis current density, duration and concentration does affecting the percentage of colour removal. The efficiency of decolourisation for 100 mg/l to 200 mg/l concentration was able to increase up to more 95% of colour removal and more than 88% of absorbance removal when duration was 18 minutes and current density was 120 A/m².

		2.5	Colour removal technique	13
		2.5.1	Colour removal technique using electricity	16
3		METI	HODOLOGY	19
		3.1	Introduction	19
		3.2	Preparation of apparatus	20
		3.3	UV/VIS spectrophotometer Jasco model 7800	24
		3.4	DR/4000 (Hach) spectrophotometer	24
		3.5	Preparation of electrode	24
		3.6	Preparation of artificial wastewater	25
		3.7	Preliminary laboratory test	25
		3.7.1	Decolourisation under the influence of different	
			current density	27
		3.7.2	Decolourisation under the influence of different	
1	D		duration	27
		3.7.3	Decolourisation under the influence of different	AI
			concentration.	28
	PER	3.7.4	Decolourisation with fixed concentration.	28
	1 2	3.8	Final laboratory test	29
4		RESU 4.1	ULT AND DISCUSSION Introduction	31 31
		4.1	Preliminary laboratory test	31
		4.3	Final laboratory test	38
		4.4	Response surface method (RSM)	39
		4.4.1	Response Surface Method (RSM) for	37
		7.7.1	decolourisation	39
		4.4.2	Response Surface Method (RSM) for	ري
		ਜ .ਜ. ∠	absorbance removal	39
		4.5	Behaviour of decolourisation	41
		T.J	Dona rour or accordangument	• •

		2.5	Colour removal technique	13
		2.5.1	Colour removal technique using electricity	16
3		MET	HODOLOGY	19
5		3.1	Introduction	19
		3.2	Preparation of apparatus	20
		3.3	UV/VIS spectrophotometer Jasco model 7800	24
		3.4	DR/4000 (Hach) spectrophotometer	24
		3.5	Preparation of electrode	24
		3.6	Preparation of artificial wastewater	25
		3.7	•	25
			Preliminary laboratory test Decolourisation under the influence of different	23
		3.7.1		27
		2.7.0	current density	27
		3.7.2	Decolourisation under the influence of different	27
		0.50	duration	27
		3.7.3	Decolourisation under the influence of different	20
	DF	RP	concentration.	28
	7 -	3.7.4	Decolourisation with fixed concentration.	28
		3.8	Final laboratory test	29
		DECL	II TE AND DIOCUICCION	2.1
4		4.1	JLT AND DISCUSSION Introduction	31 31
		4.2	Preliminary laboratory test	31
		4.3	Final laboratory test	38
		4.4	Response surface method (RSM)	39
		4.4.1	Response Surface Method (RSM) for	
			decolourisation	39
		4.4.2	Response Surface Method (RSM) for	
		2	absorbance removal	39
		45	Rehaviour of decolourisation	41

PAGE

LIST OF TABLES

TITLE

TABLE NO.

Table 2.1	Industrial sources of water pollution	9
Table 2.2	Typical characteristic of dyes used in textile industry	12
Table 4.6	CCRD experimental results using MINITAB TM	40
P	RPUSTAKAAN TUNKU T	UN AMINAH

LIST OF FIGURES

FIGURE NO	. TITLE	PAGE
Figure 2.1	Structure of Azo Acid Orange 7 (CI No. 15510)	11
Figure 3.1	Schematic diagram arrangement of apparatus	21
Figure 3.2	Detailed schematic diagram of electrode location	22
Figure 3.3	UV/VIS spectrophotometer Jasco model 7800 at 480 nm	23
Figure 3.4	DR/4000 (Hach) spectrophotometer	23
Figure 3.5	DR/4000 (Hach) spectrophotometer UHQ-Elgastat 120	26
Figure 4.1	Percentage changes of decolourisation and removal to	
PE	current density.	35
Figure 4.2	Percentage changes of decolourisation and removal to	
	duration	36
Figure 4.3	Percentage changes of decolourisation and removal to	
	concentration	36
Figure 4.4	Comparison between absorbance removal and colour	
	removal current density	37
Figure 4.5	Relationship between absorbance and concentration	37
Figure 4.6	Percentage changes of decolourisation and removal to	
	current density	37
Figure 4.7	Percentage changes of decolourisation and removal to	
	duration	38

Figure 4.8 Surface plot of percentage decolourisation under the influence of current density and duration 42 Figure 4.9 Contour plot of percentage decolourisation under the influence of current density and duration 43 Figure 4.10 Surface plot of percentage decolourisation under the influence of current density and concentration 43 Figure 4.11 Contour plot of percentage decolourisation under the 44 influence of current density and concentration Figure 4.12 Surface plot of percentage decolourisation under the influence of duration and concentration 44 Figure 4.13 Contour plot of percentage decolourisation under the influence of duration and concentration 45 Figure 4.14 Surface plot of percentage removal under the influence of duration and current density 46 Contour plot of percentage removal under the Figure 4.15 influence of duration and current density Figure 4.16 Surface plot of percentage removal under the 47 influence of concentration and current density Contour plot of percentage removal under the Figure 4.17 influence of concentration and current density 48 Figure 4.18 Surface plot of percentage removal under the 48 influence of concentration and duration Figure 4.19 Contour plot of percentage removal under the

influence of concentration and duration

49

LIST OF SYMBOLS

Initial absorbance Ao

A Final absorbance

Initial color (ADMI) Co



LIST OF APPENDICES

APPENDIX TITLE

A

Result of experiment with 10 minutes duration, 50 mg/l concentration of azo acid orange 7, 500 ml volume of sample, 100 rpm rate of stirrer, 3 cm distance between electrodes, plate steel cathodes and plate iron anode with size 50 mm x 50 mm x 1mm.

В

Result of experiment with 80 a/m^2 of current density, 50 mg/l concentration of Azo acid orange 7, 500 ml volume of sample, 100 rpm rate of stirrer, 3 cm distance between electrodes, plate steel cathodes and plate iron anode with size $50 \text{ mm} \times 50 \text{ mm} \times 1 \text{mm}$.

PER

Result of experiment with 10 minutes duration, 80 A/m² of current density, 500 ml volume of sample, 100 rpm rate of stirrer, 3 cm distance between electrodes, plate steel cathodes and plate iron anode with size 50 mm x 50 mm x 1mm.

D

Result of experiment with 18 minutes duration, 100 mg/l of concentration, 500 ml volume of sample, 100 rpm rate of stirrer, 3 cm distance between electrodes, plate steel cathodes and plate iron anode with size 50 mm x 50 mm x 1mm.

Е

Result of experiment with 160 A/m² of current density, 100 mg/l of concentration, 500 ml volume of sample, 100 rpm rate of stirrer, 3 cm distance between electrodes, plate steel cathodes and plate iron anode with size 50 mm x 50 mm x 1mm.

F

Output of RSM analysis color removal

v	17
\sim	. v

G	Output of RSM analysis for absorbance removal	63
Н	Figures of laboratory test	64



CHAPTER 1

INTRODUCTION

1.1 Background

Colour is a visible pollutant and its presence not only hampers the aesthetic quality of surface waters but also affects and alters the aquatic ecosystem by reducing the penetration of light (Prabhakara et.al, 1990). The development of industry and improvement of human life, cause more and more dyes are used and needed. Dyes are coloured, ionising, aromatic organic compounds (Fessenden et.al, 1990). A wide variety of dyes are used by industry and released into the environment as industrial effluents. Textile are the industry that largely using this product. These dyes have to be highly stable in everyday use and resistant to microbial degradation. Azo dyes are the largest class of dyes used in industry. In general, bacteria are not able to degrade Azo dyes. However, some anaerobic bacteria in intestinal micro flora have been demonstrated to degrade a few Azo dyes. Under these conditions the Azo can be toxic and potentially carcinogenic (Maarit et.al, 2000).

Dyes caused serious environmental pollution and health problem in many ways. Highly colour dye wastewater, contain a large amount of chemical (Qian and Gu., 1994). That's make the treatment of dye wastewater are important. The treatments of dye wastewater are expensive, so water –reused is attractive practice that able to give and operation and cost effective treatment system (Eroglu et.al, 1991). The treatments of textile wastewater are based on chemical and biological treatment (Nicolaou et.al, 1992).

One of alternative way that has been used to treat dye wastewater is by using system that used electric energy (Daneshvar et.al, 2004). Example of method that used electric energy is electrocoagulation, electrolysis and many more. This study was using the application of electrolchemical to treat coloured wastewater. This system has been in existence for many years. The process is based on principles involving responses of water contaminants to strong electric fields. According to Daneshvar et.al (2004), the chemistry, pH, particle size and chemical constituent of wastewater are influencing this process. The range of current density, duration of current flow and dye concentration was used for this study was based on study done by Daneshvar .et.al. But the study done by Daneshvar was using Acid Red 14, while for this study Azo Acid Orange 7 was used. Azo Acid orange 7 was used to determined the applicability of the process to others type of colours. Azo dye was chosen because almost half of the dyes used in textile industry are Azo type and it caused environmental problem when 15% of it discharge into the environment without proper treatment (Mat Daud, 2003)

The test was conducted by using plate steel cathodes and plate iron anode with size 50 mm x 50 mm x 1 mm and distance between electrode is 3 cm. Artificial wastewater was prepared by mixing Azo acid orange 7 dye with discharged water. The concentration of the artificial wastewater was between 25 mg/l to 200 mg/l. Volumes of artificial wastewater that was used for each test was 500 ml. Current density that was used between 40 A/m² to 240 A/m². The duration of current flow for each test were with in 5 to 25 minutes, each stage required different sample. The initial and final absorbance

and colour for each sample were determined in order to determine the behaviour of decolourisation of the sample. The initial and final absorbance was determined by using UV/VIS spectrophotometer Jasco model 7800 to calculate the percentage of absorbance removal. Colour was measured to determined the physical condition of the sample in term of it colour. Absorbance was measured to determine the concentration of sample. Value of colour must equal to value of absorbance. By measuring the absorbance at the same time it able to conform value of colour that has been obtained. The percentage of removal was determined by using formula below:

Absorbance removal (%) =
$$\underline{Ao - A} \times 100$$

Ao = initial absorbance(1)

The initial and final colour was determined by using DR/4000 (Hach) spectrophotometer. The percentage of colour removal was determined by using formula below:

A = final absorbance

Colour removal (%) =
$$\underline{Co} - \underline{C} \times 100$$

$$Co$$

$$.....(2)$$

$$Co = \text{initial colour (ADMI)}$$

$$C = \text{final colour (ADMI)}$$

Laboratory works were divided into two stages, which was preliminary laboratory test and final laboratory test. Results obtain in preliminary laboratory test and final laboratory test was used in the MINITABTM analysis. Based on the result of the

MINITABTM analysis, the most significance factor that influencing decolourisation of Azo acid orange 7 was able to obtain.

1.2 Problem statement

Fabrics are important to all humankind. People used fabric to make clothes and others household equipment. Due to high demand of fabric a lot of fabrics are made to fulfil this demand. In the process of making fabric a lot of colours were used in dyeing process. In the dyeing process highly coloured wastewater were produced as the process involved a lot of water. According to Jabatan Alam Sekitar, Malaysia is experiencing rapid economic growth of textile industries and this caused a lot of highly coloured textile effluent produced.

The wastewater effluents from the dyeing process are colourful and their decolourisations are very important before discharge. Dyes need to be treat before discharge because it affects the environment and human health (Zee et.al 2002). In order to have sustainable development this effluent must be treated, as the effluent is aesthetically unpleasant when discharge to receiving water and can be polluted.

Various physical and biological technique such as membrane filtration, electrolysis, flocculation, ion exchange, oxidation, aerobic, anaerobic, anoxic and biodegradation were used to removes the dye from the effluent. According to Daneshvar et.al, (2004), the effective methods are by using activated carbon or oxidation process but the cost are really high. As an alternative method which using electric energy in the process was used. This process has a fast rate of pollutant removal, simplicity in

operation, low operating and equipment cost (Daneshvar et.al, 2004). This process has been tested successfully to treat many kind of wastewater such as restaurant wastewater, urban wastewater, defluoridation of water and many more. It is expected that this method would be an ideal choice for decolourisation of dye solution. But the performances of this method are not well defined as it is considered to be new method in treating textile wastewater. By doing this study, hopefully it able to give more information about it.

This study investigates the effectiveness of using electricity in treating textile effluent. But this study was focusing in Azo dye, since Azo are the largest class of dye used in textile industry (Zee, 2002) and it's constitute a major class of environmental pollutant (Tan and Gu, 2001). Furthermore, Azo dye can be toxic and potentially carcinogenic (Maarit et.al, 2000). This study were focusing in determined the behaviour of decolourisation of Azo Acid Orange 7 by using electricity under the influence of current density, duration of current flow and concentration of dye.

The entire test was done by using plate steel cathodes and plate iron anode. Anode, withdraw electrons from the electrode material, which result in release of Fe(II)-ions produced iron hydroxide. Then cathode produces H₂ gas from water. Iron hydroxide that remains in aqueous stream as a gelatinous suspensions removed the pollutant by complexation or electrostatic attraction. H₂ gas produced in cathode form bubble and caused the floc produced to be floating on the surface of water. Organic compound from dyes react through a combination of electrochemical reduction, electrocoagulation and electrofloatation reactions (Ching et.al, 2005).

1.3 Objective of study

To determine the behaviour of decolourisation of Azo acid orange 7 by using electricity under the influence of current density, duration of current flow and dye concentration.

1.4 Scope of study

There were some matters to be considered, under the laboratory work which were:

- 1. The test conducted by using plate steel cathodes and plate iron anode with size 50 mm x 50 mm x 1mm and distance between electrodes is 3 cm.
- 2. Artificial wastewater was made from Azo acid orange 7 dyes, the concentration was in the range of between 25 mg/l to 200 mg/l.
- 3. Volumes of artificial wastewater used, 500 ml.
- 4. The current density used between 40 A/m² and 240 A/m²
- 5. The duration of current flow for each test were with in 5 to 25 minutes, each duration stage required different sample
- 6. The laboratory works were focusing in obtaining initial and final colour and absorbance change for each test.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Surface water becomes coloured by pollution caused by highly coloured wastewater (Sawyer et.al, 1994). Coloured wastewater was waste from dyeing operation in paper, leather, and textile industry. In Malaysia textile industry is the largest industry that discharged highly coloured wastewater (Japan Consulting Institute, 1993). Coloured wastewater affecting environment and human health if directly discharge without treatment. That's make the treatment of dye wastewater are important. There are many treatment used to treat dyes effluent including using electricity. However, the performances of this process are not well defined

2.2 Water pollution by textile industry

Water is so common that we take it for granted. Moreover, its covers nearly three-fourth of the surface of the earth. Water pollution problems in any part of world

are far worse from day to day. In 1998, it has been estimated that in year 2000, 2.2 billion people in the developing countries will lack access to safe drinking water (Gilbert, 1998). What are the causes of this problem? The answer is due to human activity and unbalanced development. Water that has been withdrawn, used for some purposed and returned will be polluted in one-way or another. Agricultural return water containing pesticides, fertilizer, and salt, municipal return water containing human sewage, industry returned water-containing chemical. All of this is due to human activity. When water was polluted, the water become unsuitable for drinking water and habitat for aquatic live.

Wastewater discharged from textile industry characterized by high chemical demand (COD), low biodegradability, high salt content and is the source aesthetic pollution related to colour (Alinsafi et.al, 2004). Dyes formula contain numerous auxiliary ingredients for desizing, scouring and mercerising (Wu et.al,1998). The salt and heavy metal from highly coloured wastewater are toxic to aquatic live (Wu et.al, 1998). While some of dye such as Azo dye is carcinogenic, this can cause serious health problem such as cancer (Maarit et.al, 2000). This caused the treatment of dye before discharged are important in order to ensure sustainable development able to achieve.

Based on report wrote by Japan Consulting Institute (1994), textile industry is the fifth major industry that become source of environmental problem (table 2.1). However, in term of colouring effluent textile industry is the largest industry discharging colouring effluent. So it is important to studies treatment process that is efficient to reduce the colour in the effluent. In order to ensure our water are safe for future generation.

Japan Consulting Institute (1994) report wrote that, in Malaysia, textile industry is the second largest industry following the electric appliance industry in term of export. Since the domestic market for the product is small, so most of products are exported.

The export amount reached 6433 million in 1992. Due to growth of the industry, textile companies has formed Malaysia Textile Manufacturer Association (MTMA). In 1994 number of member registered in MTMA was 290. All of this factories scattered around Selangor, Johor, Pulau Pinang, Terengganu, Kedah and Kelantan

 Table 2.1
 Industrial sources of water pollution

Type of industry	Percentage, %
Palm oil	11.6
Raw natural rubber	8.6
Rubber and product	14.1
Food and beverage	40.5
Textile and leather	9.0
Paper	4.4
Chemical	11.8
Total	100

(source: Environmental \overline{Q} uality report 1991)

In accordance with the development of textile industry, the pollution of environment by the industry has become apparent. Especially coloured wastewater discharge from dyeing factory. Coloured wastewater caused serious environmental problems in various locations. As example in Penang, visitor enjoying diving but at the same time wastewater from dyeing factories was flows into the diving area (DOE, 1997). Previously DOE has conducted investigation in the bigger textile company. Based on their report, bigger textile industry does equipped with treatment facilities. However, for coloured problem the factories not able to solve the problem of decolourisation. Many of factories discharge coloured wastewater without any treatment because colour is outside the scope of regulation. For small to middle size factory they don't event have treatment facilities to reduced pH, TSS, COD,BOD, temperature and all the hazardous chemical. This caused the pollution caused by textile industry become

worst. To reduce water pollution caused by textile industry, study must be done to treat the textile effluent efficiently.

2.3 Dyes

According to Allen (1971) dye is a coloured substance that can be applied in solution or dispersion to a substrate, thus giving it a coloured appearance. Usually a substrate is a textile fibre, but it may be paper, leather, hair, fur, plastic material, wax, a cosmetic base or a foodstuff. Dyes may be classified in several ways, according to it's chemical constitutions, application class, or end-use (DOE,1997). The primary classification of dyes is based on the fibres to which they can be applied and the chemical nature of dye. Each different dye is given colour index (C.I) generic name determined by its application characteristic and colour (Zee, 2002). Society of Dyers and Colourist and the American Association of Textile Chemist and Colourist edited C.I every three month. Previously, 28000 commercial dyes name was list by C.I. *Table 2.2* list the major class of dyes used by the textile industry.

However, this study only focusing in one type of dyes which was Azo dye. Azo dye is the largest class of dye used by textile industry (Zee, 2002). Moreover, according to Allen (1971), Azo dyes account for over 60% of total number of dye manufactured. That is why Azo acid orange 7 was used to represent Azo class of dye for this study.

2.4 Azo dye

Azo dyes are the most widely used dyes in industry with a world market share 60-70% (Sen and Demirer, 2003). Approximately 10000 Azo dye are currently manufactured and it is estimated that at least 15% of these are released into the environment (Donlon et.al, 1997). They occur in industrial effluent, groundwater, contaminated soils and sediments. These organic solution will degrade under anaerobic condition naturally (Sen and Demirer, 2003). This process generated aromatic amines. Aromatic amines are suspected to be mutagenic and carcinogenic. Moreover, it can cause serious danger to aquatic life and human life.

The chemistry of this dyes ranges from simple monoazo compounds to complex polyazo structure and their property varied (Allen, 1971). This dyes are characterized by nitrogen to nitrogen double bond (N=N) (Donlon, B. et.al, 1997). There are various class of dye that contain Azo compound including direct dye, acid dye, reactive dye, dispersed dye, pigment dye, basic dye and mordant dye (Zee, 2002). The colour in Azo dyes is due to the azo bond and associated chromophres (Sen and Demirer, 2003). Azo acid orange 7 was the type of dye that used in this study. It is also kwon as naphthalene orange G and equivalent to Raussin's orange II. Maximum wavelength of Azo acid orange 7 is 480 nm. It is in monoazo groups. *Figure 2.1* shows molecule structure of Azo acid orange 7.

Figure 2.1 Structure of Azo Acid Orange 7 (CI No. 15510)

 Table 2.2
 Typical characteristic of dyes used in textile industry.

Dve class	Description	Fibers typically applied to	Typical pollutant associated with
again of a	P		various dyes
Acid	Water-soluble anionic compounds	wool, nylon	Colour, organic acids, unfixed dyes
Basic	Water-soluble, applied in weakly acidic dye baths, very bright dyes	Acrylic, some polyesters	N/A
	Water-soluble. Anionic compounds, can be	Cotton, rayon, other	Colour, salt, unfixed dye, cationic fising agents, surfactant, defoamer,
Direct	applied directly to cellulosics without mordant (or metals like chromium and copper)	cellulosics	levelling and retarding agents, finish, diluents
Disperse	Not water-soluble	Polyester acetate, other synthetics	Colour, organic acids, phosphate, carriers, levelling, defoamers, lubricants, diluents
Reactive	Water-soluble, anionic compounds, largest dyes class	Cotton, other cellulosics, wool	Colour, salt, alkali, unfixed dye,, surfactant, defoamer, diluents, finish
Sulfur	Organic compounds containing sulphur or sodium sulphide	Cotton, other cellulosics	Colour, alkali, oxidizing agents, reducing agent, unfixed dye
Vat	Oldest dyes, more chemically complex, water-insoluble	Cotton, other cellulosics	Colour, alkali, oxidizing agent, reducing agent
			(2001 100 :::007)

(Sources: DOE, 1997)

2.5 Colour removal technique

Various physical and biological pre-treatment and post-treatment can be used to decolourise dye in coloured wastewater. Physicochemical techniques including membrane filtration, coagulation, flocculation, precipitation, floatation, adsorption, ion exchange, ultrasonic mineralisation, electrolysis, advanced oxidation, and chemical reduction. While biological technique including bacterial and fungal biosorption and biodegradation in aerobic, anaerobic, anoxic or combined anaerobic/aerobic treatment processes. In choosing type of treatment several factor need to be consider such as type of dye to be treat, composition of wastewater, cost of required chemical, cost of operation, handling cost of waste product generated. It is up to the industry to choose which treatment is suitable for their factory. (Zee, 2002)

Membrane filtration is usually used to treat reactive dye bath effluent, because it could potentially reduced waste volume while simultaneously recovering salt (Sen and Demirer, 2003). Moreover, it can separate two or more component from liquid stream by their molecular size. The advantages of membrane filtration is it is a quick method with low spatial requirement and the permeate can be reused. Disadvantages of membrane filtration are flux decline and membrane fouling, necessitating frequent cleaning and regular replacement of modules, concentrate generates required further treatment and the capital cost is high (Zee, 2002).

Adsorption and ion exchange can be used to remove dye by using activated carbon (Zee, 2002). Activated carbon adsorption has been extensively studied as a waste treatment method for the removal of different classes of dyes from wastewater. Generally, carbon adsorption of dye in neither very efficient nor economical when used alone. However, when used in tandem with polymer flocculation, chemical reduction or biodegradation, it becomes a very useful polishing step for efficient dye removal

(Abraham and Freeman, 1996). Several factors such as pH, contact time, carbon dosage and choice of activated carbon must be taken into consideration for optimum removal of dyes from wastewater (Abraham and Freeman, 1996). Commercial activated carbon can be made from lignite and bituminous coal, wood, pulp mill residue, coconut shell, and blood (Abraham and Freeman, 1996). However, many other type of absorbance material can be used such as palm fruit bunch particle, rice husk wheat straw and many more (Zee, 2002). Study done by Othman et.al (2005) proved that carbon made from oil palm kernel shell (MOPKES) has potential ability to be used as adsorbent material. Multiple choice of absorbance material gives advantages to the process. Since the material are easily obtain in the market and most of it are material that are un-wanted by the manufacturer. This process able to reduced amount of solid waste produced by the manufacturer since it used material that will be disposed. Disadvantages of this treatment are it produced waste sludge that required to be treat and disposed off (Zee, 2002).

Advanced oxidation process can also be used to remove dye. It can be defined as oxidation of compounds with an oxidation potential higher than that of oxygen. Advanced oxidation processes based on generation of highly reactive radical species that can react with wide range of compound that is difficult to degrade such as dye molecules. Advanced oxidation process including chlorination, bleaching, ozonation, fenton oxidation and photocatalytic oxidation. But ozonation, UV/H₂O₂, Fenton's reagent (Fe²/H₂O₂) and UV/TiO₂¹³ are the most widely advanced oxidation processes that have been widely studied. (Zee, 2002)

Chemical oxygen demand (COD) and colour can be removed from coloured wastewater by flocculation and coagulation. Usually this process used during pretreatment or post treatment. The process required addition of coagulant that associate with the pollutant then form flocs that remove from wastewater. Usually lime, magnesium, iron and aluminium salt is used as coagulant. Disadvantages of this process

it produce toxic sludge that must be properly disposed. Recently, organic polymer coagulant is added to reduce the volume of sludge produce. However most of the polymer used in colour removal is potentially toxic to aquatic life event at very low concentration. (Zee, 2002)

Biological dye removals are based on microbial biotransformation of dyes. Dyes are not easily biodegrade. Generally, Azo dyes are resisting to aerobic bacterial biodegradation. Only bacteria with specialized Azo dye reducing enzymes were found to degrade Azo dyes under fully aerobic condition. Breakdown of Azo linkage by reduction under anaerobic reduction implies decolourisation as the Azo dyes are converted to colourless but at the same time potentially harmful aromatic amines. Then this aromatic amines will go further conversion under aerobic condition. Usually it implies combined anaerobic and aerobic treatment to ensure efficient dye removal treatment. Disadvantage of this treatment the anaerobic of Azo dye reductions are relatively slow so it required long time for treatment process. (Tan, 2001)

Based on all the process that has been discussed, none of the process is perfectly able to remove colour from the coloured wastewater. Since there will always be a pro and contrast in term of cost, efficiency, and by product produced. It proved that each technique has its limitations. Most of treatments nowadays consist of combination of different technique such as electrocoagulation. This study was based on electrochemical process including the principle and reaction involved throughout the decolourisation of the dye. As been explained before this process involved the application of electricity. This treatment is considered new in textile industry. Not much was know about the application of the process in treating coloured wastewater from textile industry. By doing this study it able to give more information about the behaviour of the process in removing colour from dye solution.

2.5.1 Colour removal technique using electricity

Treatment that used electricity has been tested successfully to treat others wastewater such as restaurant wastewater and urban wastewater. Basically when electricity was used in the treatment, electrolysis process occurred. It is based on applying an electric current through the wastewater to be treated by using electrodes. Anode is a sacrificial metal (usually iron) electrode that withdraws electron from the electrode material, which result in release of Fe(II)-ions into the bulk solution. Iron upon oxidation produced iron hydroxides that remains in aqueous stream as a gelatinous suspensions removed the pollutant by complexation or electrostatic attraction (Daneshvar et.al, 2004). Then cathode produces H₂ gas from water and cause the floc form to be floated at the surface (Zee, 2002). Organic compound from dyes react through a combination of electrochemical reduction, electrocoagulation and electrofloatation reactions (Ching et.al, 2005).

The used of electricity in treating wastewater have been in existence for many years. It has been practiced since 20th century to treat wastewater but with limited success (Daneshvar et.al, 2004). At that time, the technology was primitive, electric power was expensive, and wastewater does not appear to be a major treat to the environment (Abraham and Freeman, 1996). Numerous methods for electrolytic treatment have been developed since then. However, in the last decade it has been increasing used in South America and Europe.

This process has been tested successfully for treating variety of wastewaters such as restaurant wastewater, urban wastewater, defluoridation of water, separation of aqueous, suspension of ultrafine particle, and removal of nitrate from water (Alinsafi et.al, 2004). According to Abraham and Freeman (1996), this process able to removed BOD, COD, total organic carbon, total dissolved solid, total suspended solids, and heavy

metal such as chromium, copper, molybdenum and zinc. However, the performances of this approach in treating textile wastewater are not well defined.

This process is based on principles involving responses of water contaminants to strong electric fields. Basically it is the combination of electrolysis and coagulation method. Compare to convectional coagulation this process has advantages in removing small colloidal particles, have lager probability of being coagulated because of electric field that set them in motion and produce low sludge. Generally, treatment of coloured wastewater is highly in cost. However, this process offers low cost for operating and maintenance (Ching et.al, 2005). Furthermore, the equipment is simple and easy to be operated (Alinsafi et.al, 2004).

This process involved the formation of flocs of metal hydroxides within the effluent to be cleaned by electro-dissolution of soluble anodes. This process will destabilizing and aggregating fine particle (Chen et.al, 2000). In the mean time, tiny hydrogen bubble produced at the cathode induce the floatation of most flocs, and helping successfully separate particle from wastewater (Alinsafi et.al, 2004). Three main processes occur during the process, electrolytic reactions at the surface of electrodes, formation of coagulant in aqueous phase, adsorption of soluble or colloidal pollutants on coagulants, and removal by sedimentation or floatation (Alinsafi et.al, 2004).

In order to optimise the treatment process several parameter such as size, shape, and distance between electrode, current density, pH, reaction time must be properly selected (Alinsafi et.al, 2004). It is important to select properly the condition of parameter to ensure the cost for treatment and removal efficiency according to needs.

There were many type of electrode in term of material, size, shape and arrangement. Each different type of electrode affecting the process differently. Study done by Daneshvar (2004), showed that electrocoagulation with several electrodes was more effective compare to simple cell. The shape of electrode such as plate and rod gave different affect to the process. Plate electrode has more surface area compare to rod electrode. Distance between electrodes is important. The distance of electrode used by Ching et.al (2005) was 1 mm distance, while Alinsafi et., al (2004) used 2 mm and Chen et.al (2000) used 3mm. Basically the most suitable condition of parameter for the process are depending on the wastewater property that required to be treated.

Treatment of textile wastewater by using electricity is a proven technology for removing colour. However only limited research has been conducted about this method.

These cause it is hard to obtain the range of current density, pH, duration and concentration that suitable for the treatment. But when utilised properly the process able to gave cost-effective method to treat textile effluents. (Abraham and Freeman, 1996)

CHAPTER 3

METHODOLOGY

3.1 Introduction

Method of the study was based on laboratory work and MINITABTM analysis. MINITABTM analysis was used to reduce amount of experiment that required to be carried out. More than 80% amount of experiment was able to reduced. However in order to used MINITABTM analysis, the laboratory works were divided into two stages. Which was preliminary laboratory test and final laboratory test.

The range of current density, duration of current flow and concentration was a prerequisite for MINITABTM analysis. Therefore, preliminary laboratory test was carried out, so that range of factor that give more then 80% decolourisation and absorbance removal was determined. While final laboratory test was carried out based on design of experiment (DOE). DOE is one of application in MINITABTM analysis. DOE analysis was done based on the range obtained in the preliminary laboratory test. DOE analysis was use to obtained the value of current density, duration and concentration for each

experiment. The result obtained from DOE was analysed by using respond surface method (RSM). The most significance factor that influence the decolourisation of Azo acid orange 7 were determined by using RSM analysis. Finally, two types of graph were obtain which was surface plot and contour plot that are useful in giving a better understanding of the behaviours of decolourisation of Azo acid orange 7 under the influence of current density, duration and concentration of dye.

3.2. Preparation of the apparatus

The apparatus used for the test was:

- 1. DC power supply (range between 40 A/m^2 to 240 A/m^2)
- 2. Milliamp meter
- 3. Circuit board with positive and negative connection.
- 4. Anode from iron and cathode from steel with size of 50 mm x 50 mm x 1mm.
- 5. Glass stirrer
- 6. Electrochemical reactor
- 7. Timer to measured the duration
- 8. Vacuum filtration equipment

During the process, flocs were form and floating at the surface of sample (refer appendix H). To remove the flocs, sample was filtered by using vacuum filtration equipment. Absorbance and colour was measured before the process started and after sample was filtered. The equipment required to measured absorbance was UV/VIS spectrophotometer Jasco model 7800 at 480 nm (figure 3.3). While, DR/4000 (Hach) spectrophotometer was used to determine percentage of decolourisation (figure 3.4)



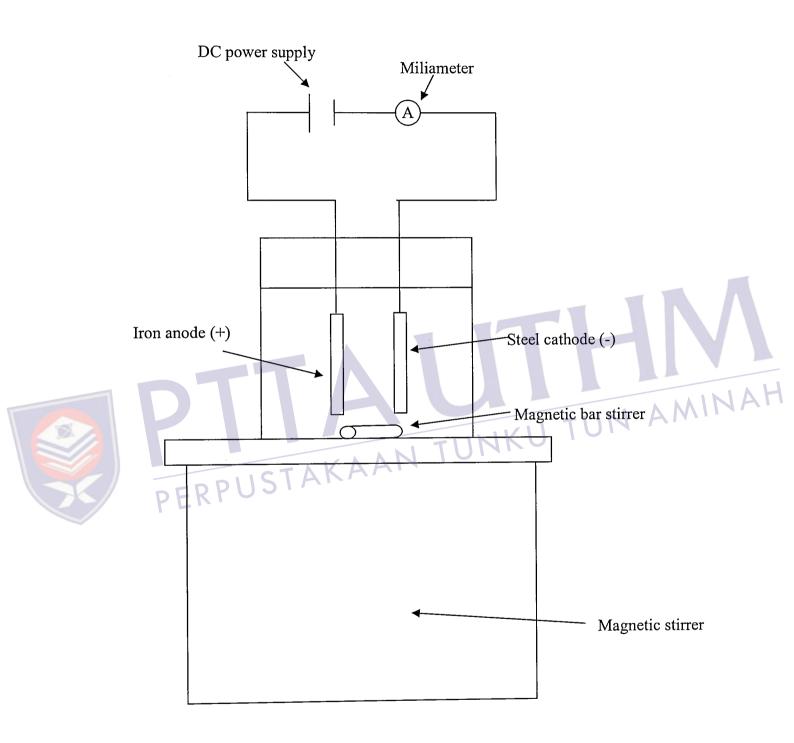


Figure 3.1 Schematic diagram arrangement of apparatus

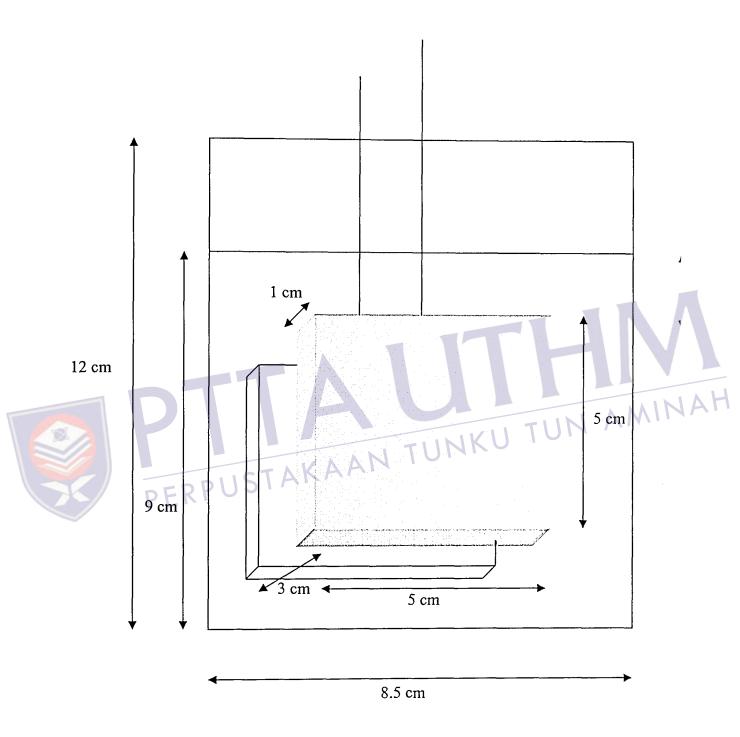


Figure 3.2 Detailed schematic diagram of electrode location

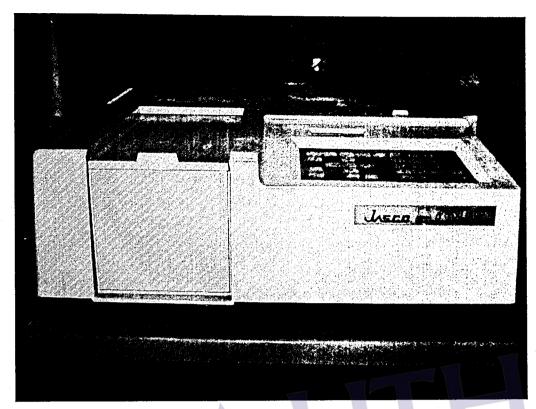


Figure 3.3 UV/VIS spectrophotometer Jasco model 7800 at 480 nm

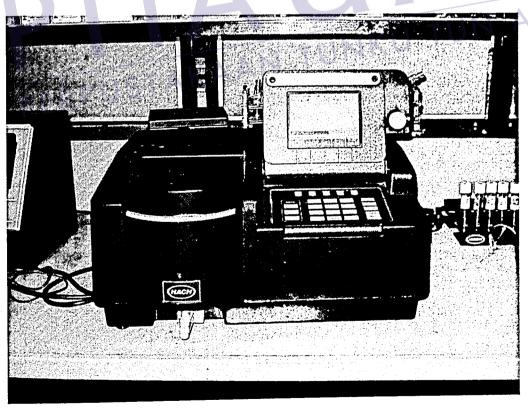


Figure 3.4 DR/4000 (Hach) spectrophotometer



3.3 UV/VIS spectrophotometer Jasco model 7800

This instrument measured the absorption of a sample in the ultraviolet region of 200 to 1100 nm wavelength to make quantitative analysis. It uses a deuterium lamp in the ultraviolet region, a tungsten iodine lamp in the ultraviolet region and a tungsten iodine lamp in the visible to near infrared region as the light source. The light from the light source passing through a high order light source, after passing through a high order light cut-off filter, goes to the monochromator that uses a concave diffraction grating. The light is monohromated by the monochromator and is split into pass through the sample. The unit of measurement are expressed in term of absorption.

3.4 DR/4000 (Hach) spectrophotometer

Colour removal was measured at 480 nm wavelength. Process of measuring colour was same as the Jusco spectrophotometer. True colour was measured because the sample was filtered before measurement. The unit of colours are in ADMI (American Dye Manufactured Institute).

3.5 Preparation of electrode

The electrodes that were used made from iron and steel. The size was 50 mm x 50 mm x 1mm. The height of the plate was 50 mm while the width was 50 mm. The

REFERENCES

- Abraham, R. and Freeman, H.S. (1996). Environmental chemistry of dyes and pigments. Canada: John Wiley & Sons. Inc.
- Alinsafi, A., Khemis, M., Pons, M.N., leclerc, J.P, Yaacoubi, A., Benhammou, A., and Nejmeddine, A. (2004). Electrocoagulation of reactive textile dyes and textile wastewater. *Journal of Chemical Engineering* (Elsevier). 44. 461-470
- Allen, R.L.M. (1971). Colour Chemistry. Britain: Pitman Press.
- Chen, G., Chen, X., and Yue, P.L. (2000). Electrocoagulation and Electrofloatation of Restaurant Wastewater. *Journal of Environmental Engineering* (ASCE). 126. 858-863.
- Ching, J. L., Shang, L.L., Chao, Y.K., and Chung, H.W.(2005). Pilot-Scale electrocoagulation with bipolar Aluminum Electrodes for On-site Domestic Greywater Reuse. *Journal of Environmental Engineering* (ASCE). 131. 491-495
- Daneshvar, N., Sorkhabi, H.A. and Kasiri, M.B. (2004). Decolourisation of dye containing acid red 14 by electrocoagulation with comparative investigation of different electrode connections. *Journal of Hazardous Material*. B112. 55-62.
- Donlon, B., Razo-Florez, E., Luijten, M., Swarts, H., Lettinga, G., and Field, J. (1997).

 Detoxification and partial mineralization of the azo dye mordant orange 1 in a continuous up flow anaerobic sludge-blanket reactor. *Journal of Microbiological Biotechnology*. 47. 83-90

- Eroglu, V.T., Ozturk, T. T. and Kor, N.(1991). Cost effective treatment of textile mill effluent by water reuse. *New Developments in Industrial Wastewater Treatment*.32. 127-131.
- Department of Environmental (1991). Environmental Quality Report. Malaysia. Unpublished
- Fessenden, R. J. and Fessenden, J.S. (1990). *Organic Chemistry*. 4th ED. California: Brooks Publishing Company.
- Holt, P., Hutchin, R. A, and DeJohn, P.B. (1999). Electrocoagulation as a wastewater treatment. *The Third Annual Australian Environmental Engineering Research Event*. 23-26 November. AEER, Victoria
- Japan Consulting Institute (1993). Report on Malaysian Textile Industry. Volume III.

 Japan. Unpublished
- Maarit, H., Ollikka P., and Suominen, I. (2000). Decolourisation of Azo dye Crocerien

 Orange by phanerochate chrysosporium ligning peroxidases. Doctorial thesis.

 Department of Biological and Environmental Science of University of Jyvaskyl.

 Findland
- Azra MunirahMat Daud (2003). Pengolahan airsisa industri menggunakan TiO₂ dengan tenaga elektrik voltan rendah. Master thesis. Faculty Civil Engineering Universiti Teknologi Malaysia. Malaysia.
- Myer, H.R. and Montogomery, C.D. (2002). Response Surface Mehodology: Process and product optimisation using design experiments. United States of America: John Wiley & Sons.Inc.

- Nicolaou, M. and Hadjivassilis, I. (1992). Treatment of wastewater from textile industry. Industrial Wastewater Treatment and Disposal. 25. 31-35.
- Fadil Othman, Kenzo Iwao, Mohd Razman Salim, Mohamed Rusop and Johan Sohaili (2005). Typical application of Malaysian oil palm biomass for improving environmental quality: A case of carbon made of oil palm kernel shell. *Brunei International Conference on Engineering and Technology*. 4–7 July. BICET, Brunei.
- Prabhakara, A.V.S, Iyengar, L. and Karthikeyan, J. (1990). Removal of colour from distillery wastewater. *Purdue Industrilal Conference Proceeding*. 44, 787-794.
- Qian, Y. and Gu, X. (1994). A way for water pollution control in Dye Manufacturing Industry. *Purdue industrial Waste Conference*.49.771-775.
- Sawyer, C.N., McCarty, P.L, and Parkin, G.F. (1994). Chemistry for Environmental Engineering. 4th Edition. Singapore: McGraw-Hill . Inc.
- Sen, S. and Demirer, G.N. (2003) Anaerobic Treatment of synthetic textile wastewater containing a reactive Azo dye. *Journal of Environmental Engineering* (ASCE).129.595-601
- Tan, C.C. (2001). Integrated and sequential anaerobic/aerobic biodegradation of Azo dyes, Doctoral thesis, Wageningen University, Netherlands.
- Tan, G.Y. and Gu, N.C. (2001), Intergrated and sequential anaerobic/aerobic biodegradation of azo dyes, Doctorial thesis, Wageningen University, Netherlands
- Vlyssides, A.G et.al (2002). Electrochemical treatment in relation to pH of domestic wastewater using Ti/Pt electrodes. *Journal of Hazardous Material* (Elsevier). 215-226

- Wu, J., Eiteman, M.A., and Law, S.E. (1998). Evaluation of membrane filtration and azonation process for treatment of reactive-dye wastewater. *Journal of Environmental Engineering* (ASCE). 124. 272-277
- Zee, J.J. (2002). *Anaerobic azo dye reduction*, Doctoral thesis, Wageningen University, Netherlands.

