REMOVING HEAVY METAL IONS FROM INDUSTRIAL WASTE WATER THROUGH AFC99, AFC40 & CA202 MEMBRANE

By

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Dissertation submitted in partial fulfillment of
the requirement for the
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(CHEMICAL ENGINEERING)

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CERTIFICATION OF APPROVAL

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Approved by,

Supervisor
(Dr. AzryBorhan)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
May 2013

CERTIFICATION OF ORIGINALITY

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

(CHIN YONG BAO)

ABSTRACT

Environment pollution has been found to be present widely in the environment. Among all pollutions, heavy metal pollution has become one of the most serious environmental problems today.. With the rapid development of industries such as semiconductors industries, fertilizer industries, metal plating facilities etc, the amount of heavy metal ions being discarded into their wastewater also increase tremendously. Unlike organic contaminants, heavy metals are not biodegradable and tend to accumulate in living organisms and many heavy metal ions are known to be toxic or carcinogenic. Toxic heavy metals of particular concern in treatment of industrial wastewaters include zinc, copper, nickel, mercury, cadmium, lead and chromium. This is why the treatmentof heavy metals is of special concern due to their recalcitrance and persistence in the environment.In this project, we are looking specifically at semiconductors industries and the heavy metals which are present in the wastewater are copper, zinc and aluminum.

In recent years, various methods for heavy metal removal from wastewater have been extensivelystudied. This paper reviews the current methods that have been used to treat heavy metal wastewaterand evaluates these techniques as well as looking into the new technique which is the Reverse Osmosis method in semiconductor industries.This proposal outlines the experimental study to determine the efficiency of reverse osmosis membrane in removing heavy metal ions from wastewater at semiconductor industry. Three operating condition will be varied in the experiment: Pressure, pH value and concentration of the feed versus the flux. At the same time, the total dissolved solids (TDS), conductivity, turbidity and Trans-membrane pressure (TMP) will be tested for each parameters with four different types of membrane which are AFC99, AFC40 and CA202 to determine the membrane's efficiency. Low value of total dissolved solids (TDS), conductivity, turbidity and trans-membrane pressure (TMP) concludes to high efficiency of the membrane in removing the heavy metal ions from wastewater treatment.

ACKNOWLEDGEMENT

The author strongly understands that the success of this project would not be possible without the involvement and contributions of certain parties, organizations and individuals. Therefore, the author would like to take this opportunity to express his deepest gratitude to them. First and foremost, the author would like to thank his supervisor, Dr. AzryBorhan for the continuous assistance, supervision and advise that he has given throughout the entire Final Year Project implementation. He has constantly provided guidance and valuable knowledge for every stage since the planning phase of the project until this dissertation has been completed.

The author would also like to thank Dr. Nurhayati Mellon, the FYP coordinator, for conducting several seminars and briefings to assist the FYP students as well as helping to arrange the schedule for Pre-SEDEX and Viva for students by inviting external examiners to evaluate the students. Besides that, the author wishes to express his gratitude to his colleagues and beloved family for their support and encouragement throughout the duration of the project.

In conclusion, the author would like to thank all parties involved once again for making this Final Year Project a success.

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CHAPTER 1

INTRODUCTION

1. Introduction

1.1 Background of Study

The semiconductor industry is a fast paced and highly competitive business. Due to the fierce competition and continuation of fast developing new technology in this sector, the cost to produce a new chip can fall as much as 50% within few months. As much as the technology is improving, the amount of heavy metal ions being discard into their wastewater also increase tremendously due to the large production of semiconductors for global demand.Thanks to the advancement of technology and the heavy awareness of environmental policies, there are more and more methods being invented and optimized from conventional treatment such as chemical precipitation to membrane separation. Membrane separation is a technology which selectively separates materials via pores and minute gaps in the molecular arrangement of a continuous structure. The membrane separations are categorized by the separation driving force and pore size. Among the types of membrane separations are Ultra-filtration (UF), Microfiltration (MF),Reverse Osmosis (RO) and Ion Exchange (IE). In this context, Ultra-filtration (UF) and Reverse Osmosis (RO) are being prioritized.

There are three different types of reverse osmosis modules which are tubular, spiral and hollow fiber modules. Tubular membranes are not self- supporting membranes. They are located on the inside of a tube, made of special kind of material. The location of tubular membranes is inside a tube, so the flow of the membrane is usually inside out. Tubular membrane has a diameter of about 5 to 15 mm (Membrane Technology).

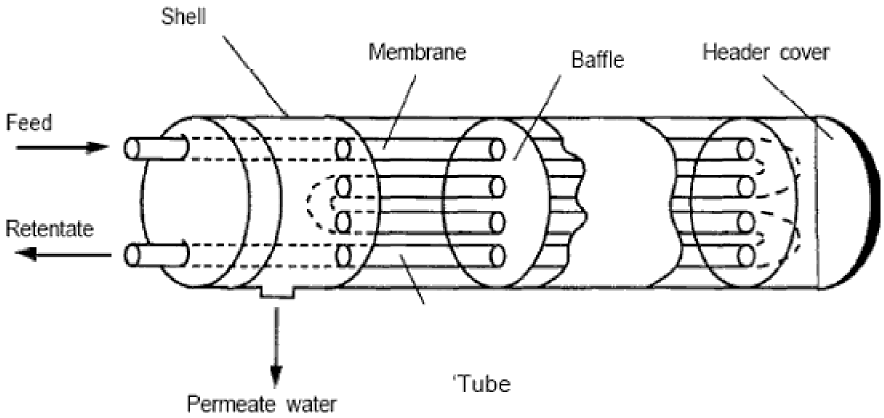


Figure 1.1: Cross section of tubular membrane

Spiral membranes consist of two layers of membrane, placed onto a permeate collector fabric. This envelope is wrapped around a centrally places permeate drain. This causes the packing density of the membranes to be higher. The feed channel is placed at moderate height, to prevent plugging of the membrane unit (Membrane Technology). Figure 1.3 shows the cross section of spiral membranes:

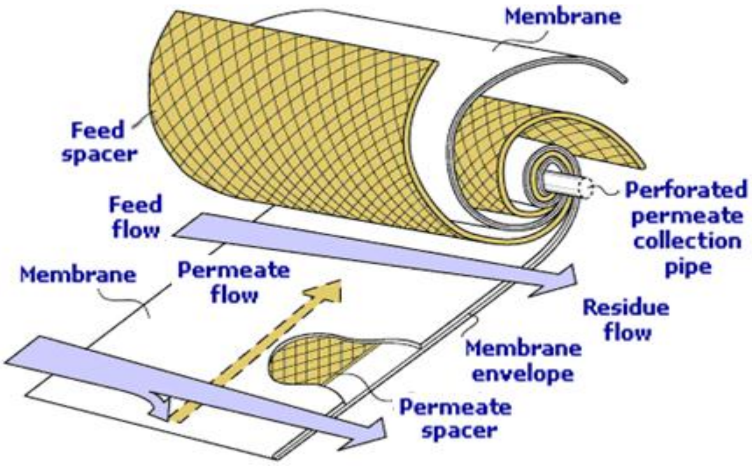


Figure 1.2 Cross section of spiral membrane

Hollow fiber membranes are membranes with a diameter of below 0.1 micrometer. The chances of plugging of a hollow fiber membrane are very high. The membranes can only be used for the treatment of water with low suspended solids content (Membrane Technology). For this project, tubular modules are chosen for reverse osmosis system. Figure 1.4 shows the cross section of hollow fiber membranes:

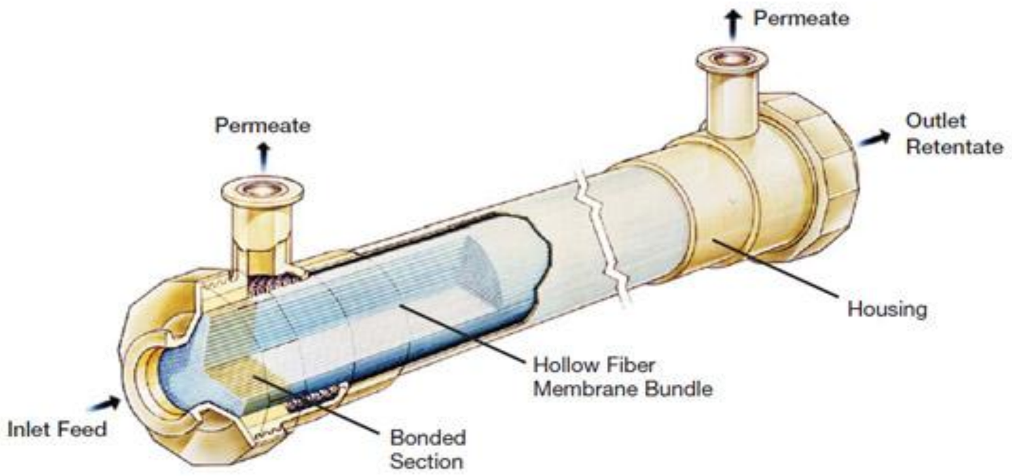


Figure 1.3 Cross Section of hollow fiber membrane

1.2 Problem Statement

1.2.1 Problem Identification

When a concentrated feed solution is passed through the membrane continuously with high pressure and concentration, the flux rate/permeate rate will decrease resulting a low value of GSFD (gallons per square foot per day) is obtained. This is due to the formation of membrane fouling and concentration polarization. Besides that, extensive pH value and high temperature of the feed will also affect the efficiency and the lifespan of the membrane and hence affect the overall process system.

1.2.2 Significance of the Project

The significance of this project is that at the end of this experiment, if all the objectives and aims are able to achieve, the current semiconductor industry will be able to shift their conventional wastewater treatment of chemical precipitation to RO system. This will actually bring the whole industry to a different paradigm as RO system will require less space than compare to conventional wastewater treatment and RO system is much more environmental friendly due to the less usage of chemicals to remove the sludge.

1.3 Objective

The objective of this research is toinvestigate the feasibility of membrane in removing heavy metal ions from industrial waste water by using reverse osmosis membrane. The objectives of this study as listed as below:

i. To study possibility of removing heavy metal ions using composite reverse osmosis membrane
ii. To study the variation of data collected during process: Pressure vs flux, pH value versus flux and also concentration vs flux.
iii. To simplify the conventional wastewater treatment into RO system

1.4 Scope of Study

As stated earlier, this study will be focusing on heavy metal ions removal using reverse osmosis membrane. Below are the scopes of study in this project;

i. Pretreatment of the wastewater : ultra-filtration (UF)
ii. To conduct the experiment of heavy metal ions removal using reverse osmosis membrane (RO)
iii. To study the effect of concentration against flux
iv. To study the effect of pH value against flux
v. To study the effect of pressure and flux
vi. To study the composition of wastewater before and after the experiment.

1.5 The Relevancy of the Project

This project is entitled “Removal of heavy metal ions from industrial wastewater through membrane separation” is relevant to the students as it increases the knowledge on current technology used in separation system. Other than that, this project also gives an overview on different types of membranes. As mentioned in previous section, membranes are vastly used in wastewater treatment plant. There are three types of module used for reverse osmosis which are tubular, spiral and hollow fiber. Every module has their advantageous and disadvantageous. All these types of membraneshave been used in removing heavy metals in semiconductor industry.Besides that, this project can serves as guidelineon reverse osmosis membrane which in this case we are talking about membrane type AFC99, AFC40 and CA202.

Apart of that, although reverse osmosis is widely used and theoretically it is possible to separate heavy metal ions using reverse osmosis membrane based on their molecular weight but still, they are no experiment yet to prove which type of membrane is the best in the removal of heavy metal ions which in this case, we are focus on aluminum, copper and zinc metal ions. Hence, there is a need for this experiment to carry out and proves its efficiency. Furthermore, by varying the operating conditions of this experiment which are feed concentration, feed pH and feed pressure against the permeate flux will give a better understanding on the effect of these parameters on the wastewater with respect to the before and after process. In addition, this project is also relevant because each parameter will be tested three times to get three sets of data in order to plot a graph for better understanding and the result of this experiment is further strengthen by measuring the total dissolved solids (TDS), conductivity, turbidity and trans-membrane pressure (TMP) of the wastewater before and after treatment.

1.6 Feasibility of the Project within Scope and Time Frame

This project is expected to be finished in about 7 months. During this time, the project will consists of pretreatment process, experiment on heavy metal ions removal using reverse osmosis membrane and chemical analysis after the experiment to study the composition of the components in the product mixture. From given allocation of time and operating conditions that will be varied in the experiment, this project is expected to be completed in the time given. All the scope of study that has been mentioned also will be done during the allocation of time. The feasibility of this project is shown in the Gantt chart that will be shown in later section. From there, it shown that this project will be completed and feasible within the allocated scope and time frame.

CHAPTER 2

LITERATURE REVIEW

2. Literature Review

Membrane separation technology is vastly used in manufacturing industries nowadays especially in semiconductor sector. Among the types of membrane separations are Ultra-filtration (UF), Microfiltration (MF), Reverse Osmosis (RO) and Ion Exchange (IE). Generally speaking, reverse osmosis is not a filtration technique like ultra-filtration or microfiltration, but a true membrane process. Y.C Huang and S.S Koseoglu (1993) mentioned that operational mechanism in reverse osmosis is the interaction between membrane and solution where the effectiveness of the removal or concentration or target compounds are largely determined. The typical salt rejection for reverse osmosis is 95%-99% and the corresponding operating pressure is 5.6MPa to 10.5MPa. Among the membranes for reverse osmosis which commonly available in market are aromatic polyamide (PA), thin-film composite (TPS) and cellulose acetate (CA).

Besides semiconductor sector, reverse osmosis also has the capability for treating industrial wastewater from textile, chemical, petroleum refineries etc. An overview discussion for applications of RO to complex industrial wastewater treatment had been presented by Slater and Ahlert (1983). The article was further backed up by Chem et al. (1985), Bhattacharyya et al. (1992) and Lloyd (1985) where the ultimate goal for RO is to achieve zero discharge with pure water and concentrate that can be recycled in the process. Table 1 shows the comparison between Reverse Osmosis and other membranes.

Table 1:Comparison between Reverse Osmosis and other membranes

Properties	Size of pores (microns)	Applications
Microfiltration	0.1	- Cold sterilisation of beverages - Pharmaceuticals - Separation of oil/ water emulsions
Ultrafiltration	0.01	-Food processing -Dialysis -Textile Industry
Nanofiltration	0.001	- Drinking water purification - Food industry
Reverse Osmosis	0.0001	- Drinking water purification - Wastewater purification - Desalination

Although minute amount of heavy metal is essential for human health but more than the amount requirement by the body will cause intolerant to our human organs and serious side effects will occur. This is the reason why removals of heavy metal ions are very crucial to mankind. The heavy metals of particular concern in treatment of industrial wastewaters are normally cadmium, nickel, copper, zinc, mercury and lead. Chronic exposure of cadmium results in kidney dysfunction and high levels of exposure will result in death. Excess amount of nickel which is human carcinogen, will bring serious kidney and lung problems aside from pulmonary fibrosis, skin dermatitis and gastrointestinal distress (Borba et al., 2006). Copper does essential work in animal metabolism but the excessive intake of copper will cause serious toxicological concerns such as convulsions; cramp, puking or even worst death (Paulino et al., 2006). Table 2 shows the heavy metal removal by RO system.

Table 2:Heavy metal removal by RO system

**Data obtained from Mohsen-Nia et al 2007; Zhang et al 2009; Chan &Dudeney 2008; Ipek, 2005*

Membrane	Heavy Metals	Initial Concentration	Removal efficiency (%)	Condition
RO	Cu2+, Ni2+	500mg/L	99.5	Operation pressure 5atm
RO	Cu2+	20-100mg/L	70-95	Low pressure reverse osmosis combined with electro-winning
RO	As	<500µg/L	As(V) 91-99, As(III) 20-55	N/A
RO	Ni2+, Zn2+	Ni2+: 44-49 ZN2+: 64-170mg/L	99.3 98.9	Operational pressure 1100kPa

On the other hand, zinc is a trace element that is essential for human health. It is important for the physiological functions of living tissue and regulates many biochemical processes. However, zinc overdosewillcause eminent health problems such as skin irritations, stomach cramps, anemia and nausea (Oyaro et al., 2007).Mercury is another heavy metal which consider as neurotoxin, it can cause damage to the central nervous system and high concentrations of mercury cause impairment of pulmonary and kidney function, chest pain and dyspnoea (Namasivayam and Kadirvelu, 1999). The Minamata incident as mentioned earlier is the worst mercury poisoning disaster in mankind. Lastly, lead will cause damage to the central nervous system, liver, reproductive system, kidney and brain function. The toxic symptoms are anemia, insomnia, headache, dizziness, irritability, weakness of muscles, hallucination and renal damages (Naseem and Tahir, 2001).

Table 3 summarized all the types of heavy metals and its side effects.

**Data obtained from Borba et al., 2006; Paulino et al., 2006; Oyaro et al., 2007; Namasivayam and Kadirvelu, 1999; Naseem and Tahir, 2001*

Types of Heavy Metal	Side Effects
Cadmium	Carcinogen, accumulates in liver and kidney
Nickel	Kidney, lung problems, pulmonary fibrosis, skin dermatitis and gastrointestinal distress
Copper	Convulsions; cramp, puking, death
Zinc	Skin irritations, stomach cramps and nausea
Mercury	Highly toxic, damage to nervous system
Lead	Brain and kidney damage

Furthermore, Liu Feini et al. (2008) mentioned that good consistency was observed after the characteristic and filtration behavior of RO process including the salt rejection, ion rejection and water flux versus operating pressure were evaluated and compared with theoretical calculation using mass transfer models. In addition, it was also found that the rejection of salt could be more than 95% and the Chemical Oxygen Demand (COD) value in permeate was 10mg/L using the RO composite membrane. Although that removal efficiency of RO membrane is high but due to its performance and its life-time are highly sensitive to the wastewater properties where the drawback of RO membrane should also taken into account which is the fouling and concentration polarization effect. Therefore, Jae-Wook Lee et all. (2005) suggested that pre-treatment units are required depending on the nature of wastewater. This can increase the lifespan of the RO membrane significantly as most of the bigger particles are removed by the pre-treatment units and indirectly reduce the burden of particles removal on RO membranes.Table 4 shows the four types of membrane info used in the experiment.

Table 4: Reverse Osmosis membrane information

Membrane Type	AFC99	AFC40	CA202
Material	Polyamide Film	Polyamide Film	Cellulose Acetate
Max pH Range	1.5 – 12	1.5 – 9.5	2 – 7.25
Recommended Maximum Pressure (bar)	64	60	25
Maximum Temperature (℃)	80	60	30
Apparent Retention Character	99% NaCI	60% CaCl2	2,000MW
Hydro-phyllicity	3	4	5
Solvent Resistance	++	++	+

The study which determine the efficiency of reverse osmosis as separation technique by A.Borhan et al. (2012) where the efficiency of reverse osmosis in removing salts from produced water from oil refinery desalting unit is studied. The wastewater feed was taken from PETRONAS Penapisan (Melaka) SdnBhd where real-time wastewater is being used. The experiment was carried out in three different operating conditions where the Trans-membrane pressure (TMP), feed concentration and feed pH where being tested towards permeate flux and percentage of total dissolved solid rejection. The value used for feed pH are 4, 7, and 10; Trans-membrane pressure (TMP) are 8, 12, 16 and 20 bars respectively; feed concentration of 450mg/L, 550mg/L and 900 mg/L. It was observed that permeate flux increase with increasing TMP while increasing feed concentration and feed pH caused permeate flux and percentage rejection to decrease. Furthermore, the pH is found to effect the permeate flux and percentage rejection of produced water of desalting unit because the property of surface material of membranes changes with pH. In addition, the optimum range of pH for higher percentage rejection and permeate flux was found to be at acidic condition.Figure1 shows the typical chemical precipitate method being used by semiconductor industry.

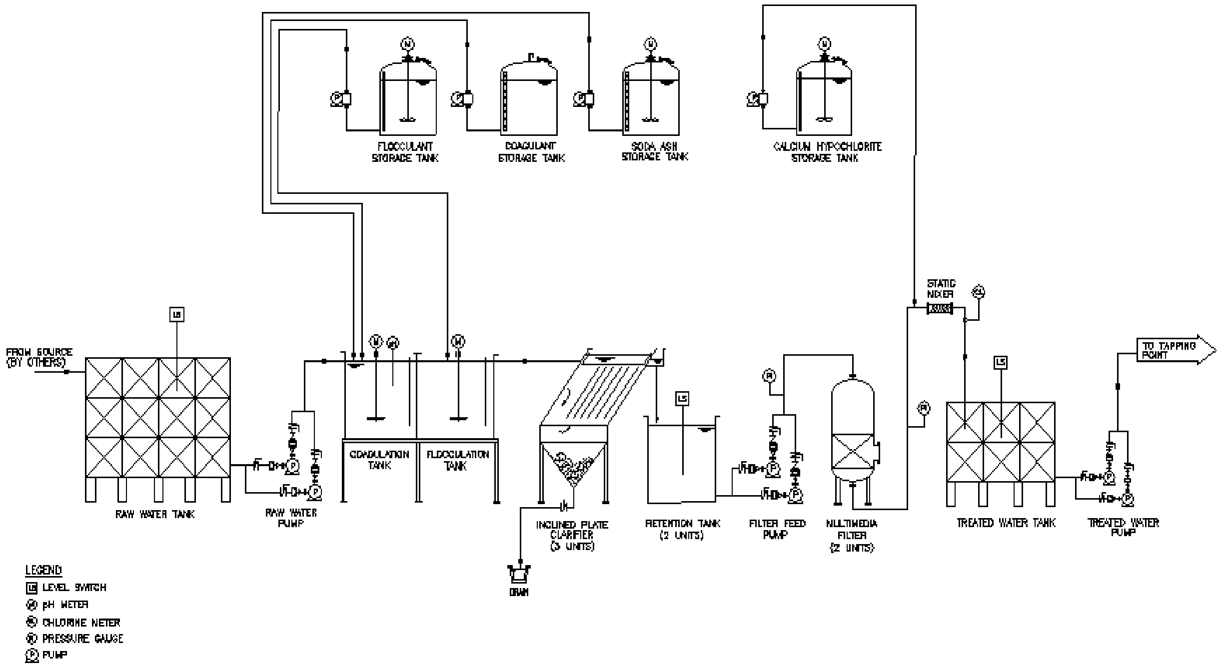
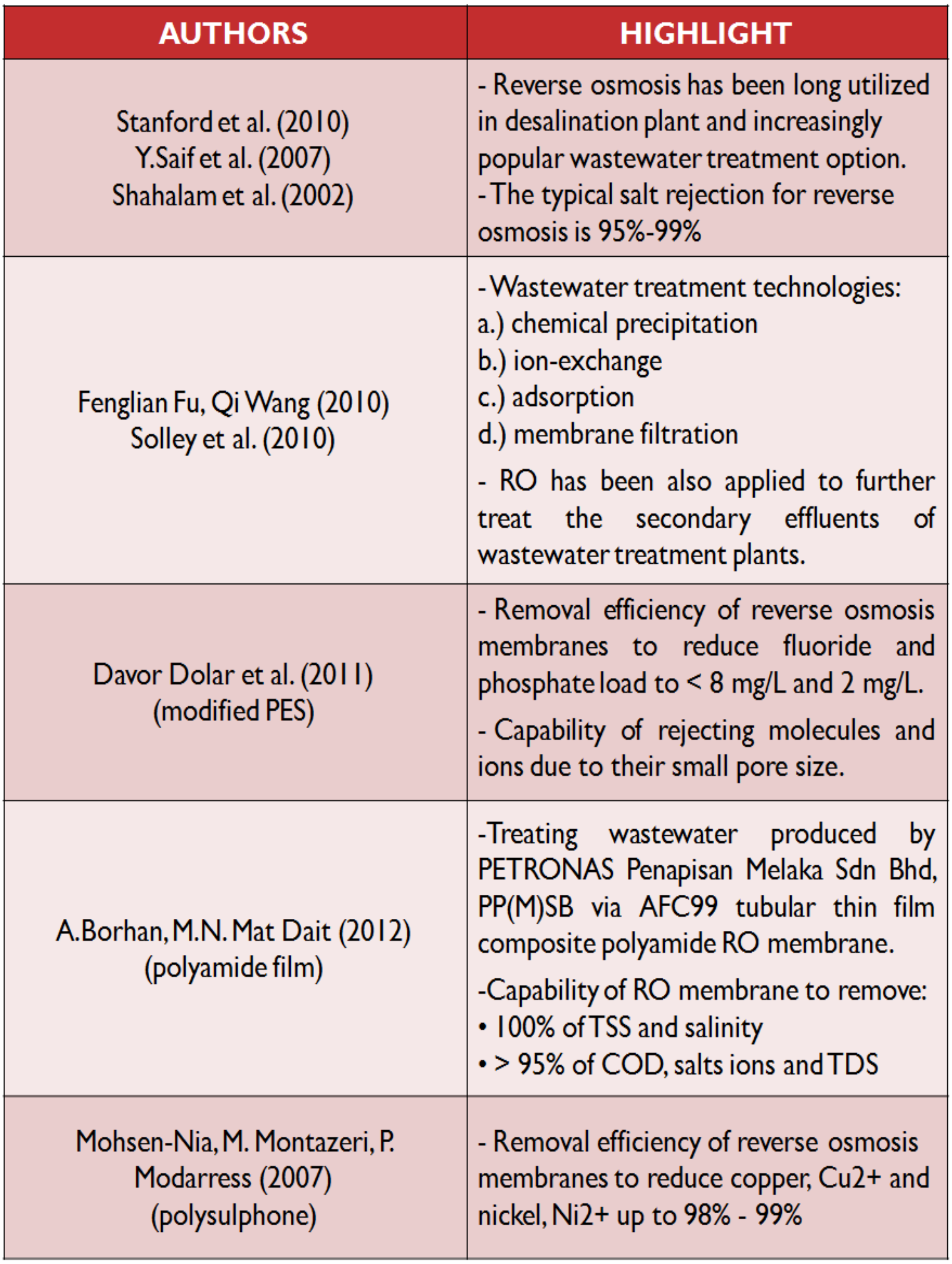


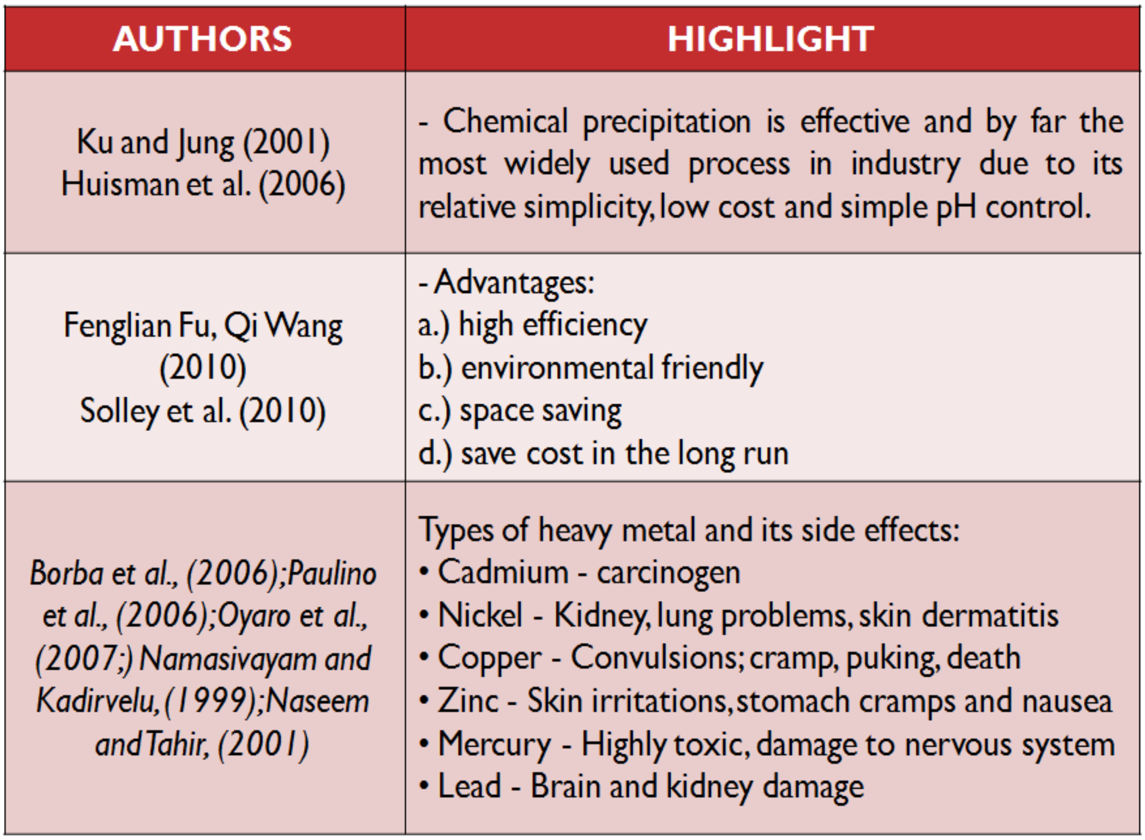
Figure2.1: Wastewater Treatment Plant (chemical precipitate)

Fenglian Fu & Qi Wang (2011) discussed on the current methods that have been used to treat heavy metal wastewater and evaluates these techniques. Among the technologies is membrane filtration, chemical precipitation, floatation, electrochemical, ion-exchange, coagulation-flocculation and adsorption methods. About 185 published studies (1988-2010) are reviewed in their paper and it is proven that the most frequently methods to be used for the treatment of heavy metal wastewater are membrane filtration, adsorption and ion exchange. From Table 1 below, a survey result from their research had been investigated and this shows that RO is an increasingly popular wastewater treatment method in chemical end environmental engineering. On top of this, Mohsen-Nia et al. (2007), Dialynas and Diamadopoulos (2009) tested on a pilot-scale membrane bioreactor system in combination with RO system and they found that the efficiency in removing heavy metal were indeed very high. Overall, the results shows that reverse osmosis membrane provide high rejection and also produces good water flux at optimum operating conditions.

Below are the summaries of some of the important papers that had been reviewed and the authors and highlights are being display as below:-



AUTHORS	HIGHLIGHT
Stanford et al. (2010) Y.Saif et al. (2007) Shahalam et al. (2002)	- Reverse osmosis has been long utilized in desalination plant and increasingly popular wastewater treatment option. - The typical salt rejection for reverse osmosis is 95%-99%
Fenglian Fu, Qi Wang (2010) Solley et al. (2010)	- Wastewater treatment technologies: a.) chemical precipitation b.) ion-exchange c.) adsorption d.) membrane filtration - RO has been also applied to further treat the secondary effluents of wastewater treatment plants.
Davor Dolar et al. (2011) (modified PES)	- Removal efficiency of reverse osmosis membranes to reduce fluoride and phosphate load to < 8 mg/L and 2 mg/L. - Capability of rejecting molecules and ions due to their small pore size.
A.Borhan, M.N. Mat Dait (2012) (polyamide film)	-Treating wastewater produced by PETRONAS Penapisan Melaka Sdn Bhd, PP(M)SB via AFC99 tubular thin film composite polyamide RO membrane. -Capability of RO membrane to remove: • 100% of TSS and salinity • > 95% of COD, salts ions and TDS
Mohsen-Nia, M. Montazeri, P. Modarress (2007) (polysulphone)	- Removal efficiency of reverse osmosis membranes to reduce copper, Cu2+ and nickel, Ni2+ up to 98% - 99%



AUTHORS	HIGHLIGHT
Ku and Jung (2001) Huisman et al. (2006)	- Chemical precipitation is effective and by far the most widely used process in industry due to its relative simplicity, low cost and simple pH control.
Fenglian Fu, Qi Wang (2010) Solley et al. (2010)	- Advantages: a.) high efficiency b.) environmental friendly c.) space saving d.) save cost in the long run
Borba et al., (2006);Paulino et al., (2006);Oyaro et al., (2007;) Namasivayam and Kadirvelu, (1999);Naseem and Tahir, (2001)	Types of heavy metal and its side effects: • Cadmium - carcinogen • Nickel - Kidney, lung problems, skin dermatitis • Copper - Convulsions; cramp, puking, death • Zinc - Skin irritations, stomach cramps and nausea • Mercury - Highly toxic, damage to nervous system • Lead - Brain and kidney damage

CHAPTER 3

METHODOLOGY

3. Methodology

3.1 Research method

Methodologies are divided into two main phases which are literature and experimental study. Both phases are briefly described below:

3.1.1 Literature study

i. Define study objectives
ii. Study on published journals of heavy metal ions removal in semiconductor industry as well as the efficiency of reverse osmosis as separation method.
iii. Study of parameters that will be used for experimental study
iv. List all chemicals and equipment required for experimental study

3.1.2 Experimental study

i. Prepare experimental study procedure
ii. Preparation of sample of wastewaters for experimental study
iii. Preparation of membrane and equipment used.
iv. Analysis of heavy metal ions rejection using reverse osmosis membrane by varying their operating conditions such as Trans-membrane pressure, pH value and feed concentration.

3.2 Tools required

Equipment, chemicals and apparatus required for this study are listed as below:

Table 5: List of Chemicals

No.	Chemical(s)	Function
1.	Wastewaters from semiconductor industry **INFINEON TECHNOLOGIES (M) SDN BHD**	Feed

Table 6: List of Apparatus

No.	Apparatus	Size	Function
1.	Beaker	30 mL – 1L	Sample preparation
2.	Conical Flask	50 mL	Sample preparation
3.	Volumetric Flask	10 mL – 250 mL	Sample preparation
4.	Finepipette	1 µL – 50 mL	Separate top & bottom phase
5.	Measuring Cylinder	10 mL – 50 mL	Sample preparation
6.	Test Tube	10mL	Sample preparation

Table 7: List of Equipment

No.	Equipment/machine	Function
1.	Membrane Test Unit	Removal of heavy metal ions from the sample
2.	Atomic Absorption Spectroscopy (AAS)	Quantitative determination of heavy metal ions (aluminium, zinc & copper)

3.3 Detailed activities of this project as listed in Table below:

Phase	Activities
Pre-experiment	Studying on related journal: heavy metal ions removal in semiconductor industry as well as the efficiency of reverse osmosis as separation method.
	Study of parameters that will be used in the experiments
	Prepare laboratory documentations: • Job Safety Analysis • Material Safety Data Sheet • Experimental procedures
Experiment	Prepare sample from wastewater treatment
	Pre-treatment of the sample to reduce the solids in the sample so that fouling will not happened during experiment: • Sedimentation • Vacuum pump
	Prepare the membrane that will be used for the experiment. Then run the experiment by using tap water to identify the fouling occurrence. This serve as a guidelines to the experiment when varies operating conditions are carry out later.
	Run the experiment by varying the operating condition
	Determine the composition in the sample before and after the experiment by utilizing chromatography
	Study the effect of operating condition to the efficiency of reverse osmosis as separation membrane
Post-experiment	Calculate the rejection factor for heavy metal ions after using reverse osmosis membrane.

Flow chart of experimental procedures is further explained as below:

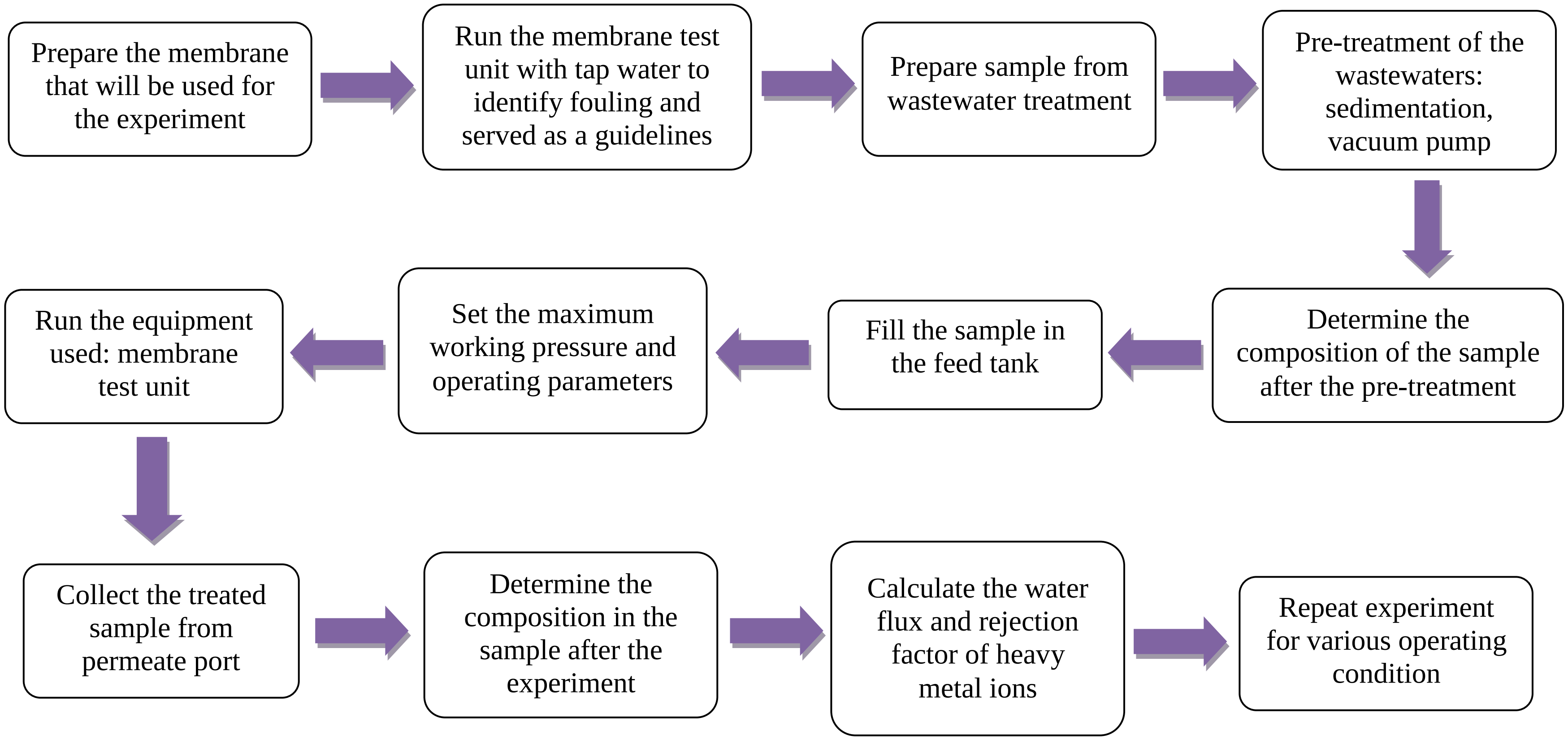


Figure 3.1: Detailed Experimental Procedures

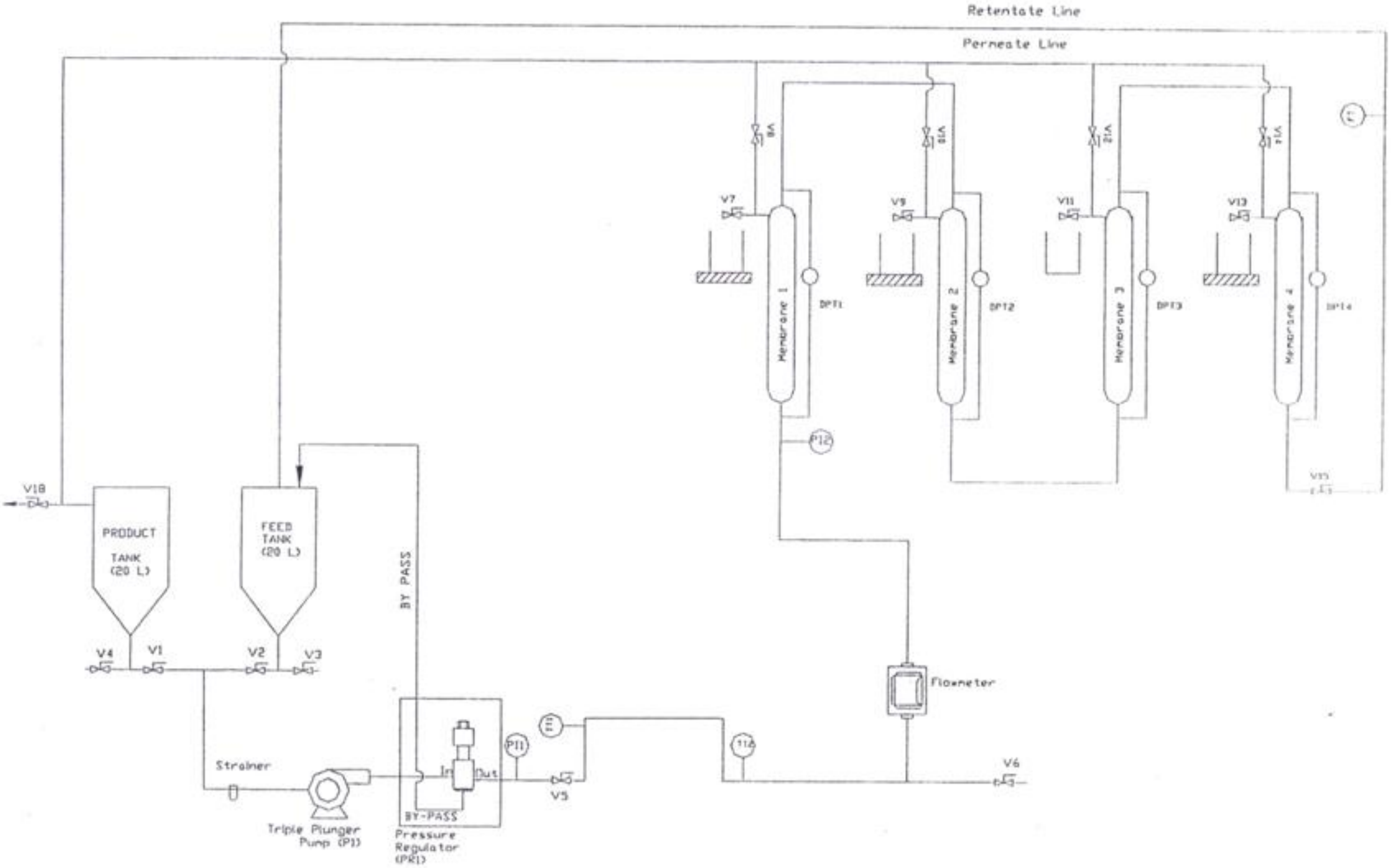


Figure3.2**:** Process Schematic Diagram

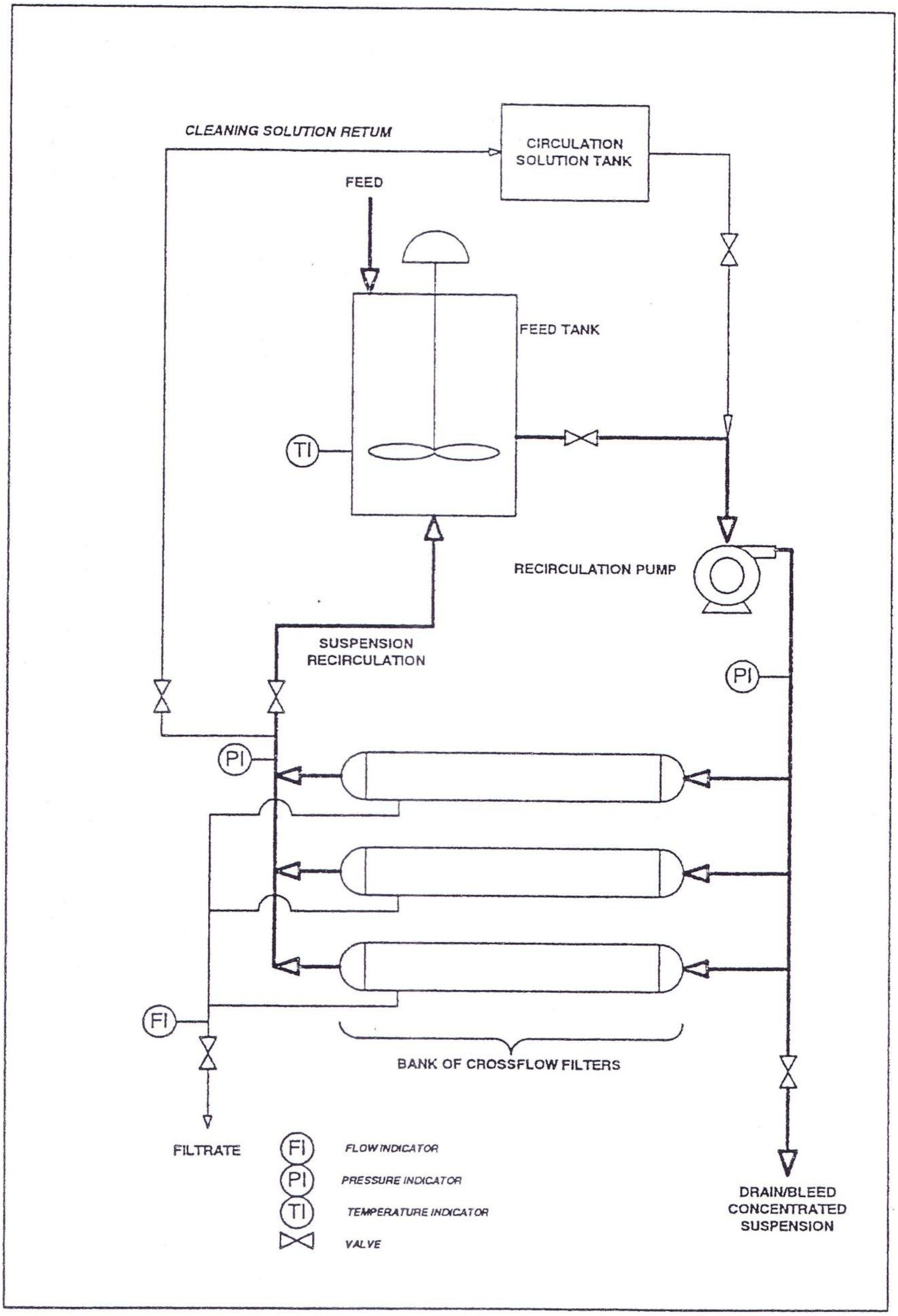
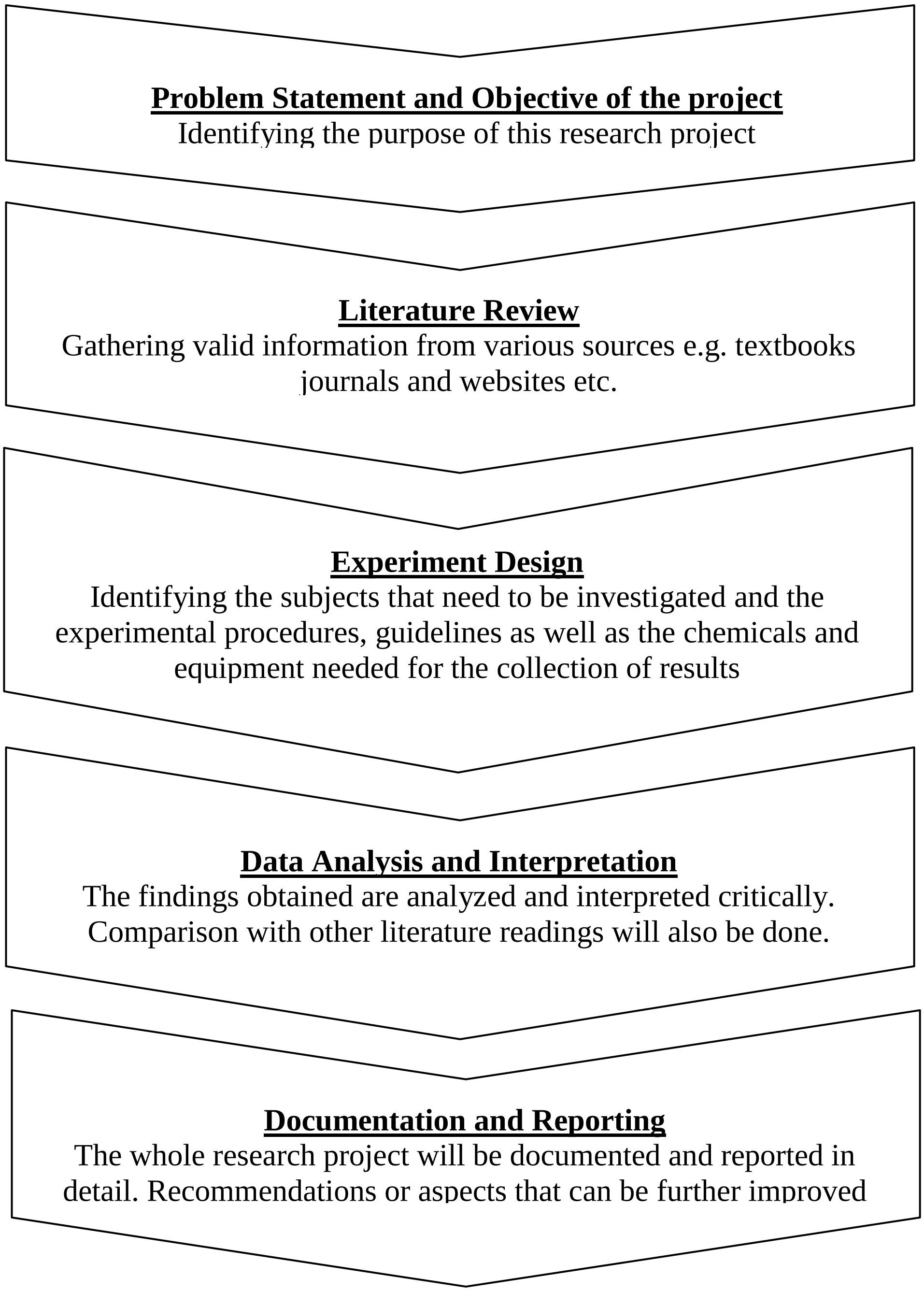


Figure 3: A typical crossflow operation includes recirculation loop

3.4 Key Milestones

Several key milestones for this research project must be achieved in order to meet the objective of this project



Problem Statement and Objective of the project
Identifying the purpose of this research project

Literature Review
Gathering valid information from various sources e.g. textbooks journals and websites etc.

Experiment Design
Identifying the subjects that need to be investigated and the experimental procedures, guidelines as well as the chemicals and equipment needed for the collection of results

Data Analysis and Interpretation
The findings obtained are analyzed and interpreted critically. Comparison with other literature readings will also be done.

Documentation and Reporting
The whole research project will be documented and reported in detail. Recommendations or aspects that can be further improved

3.5 Gantt Chart (FYP I)

No.	Activity/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1.	Literature study: Heavy metal removal in semiconductor industry as well as the efficiency of reverse osmosis as separation method	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦
2.	Project Extended Proposal		🟦	🟦	🟦	🟦	●								
4.	Prepare Experimental Procedures							🟦	🟦						
5.	Proposal defence presentation									🟦					
6.	Prepare sample for experimental study							🟦	🟦	🟦	🟦				
7.	Familiarize with analytical equipment										🟦	🟦	🟦	🟦	🟦
8.	Pre-treatment of the sample												🟦	🟦	🟦
9.	Interim Report														●

● Suggested milestone

🟦 Process

Gantt Chart (FYP II)

No.	Activity/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1.	Prepare sample for heavy metal ions removal	🟦	🟦												
2.	Determine composition of sample before and after the experiment	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦			
3.	Varying operating condition for the experiment	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦			
5.	Calculate the permeate flux for each experiment	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦			
6.	FYP II progress report	🟦	🟦	🟦	🟦	🟦	🟦	🟦	●						
7.	Pre-EDX poster						🟦	🟦	🟦	🟦	●				
8.	Technical Paper		🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	●				
9.	Dissertation soft copy		🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	●				
10.	Viva									🟦	●				
11.	Dissertation (finalized)									🟦	🟦	🟦	🟦	🟦	●

● Suggested milestone

🟦 Process

CHAPTER 4

RESULT AND DISCUSSION

4. Result and Discussion

4.1 Result

FLUX - Pressure at 10bar

Table 8: AFC99, AFC40 and CA202 at 10bar

AFC99 Membrane			AFC40 Membrane			CA202 Membrane		
Time (min)	Volume (L)	Water Flux (L/m2h)	Time (min)	Volume (L)	Water Flux (L/m2h)	Time (min)	Volume (L)	Water Flux (L/m2h)
0	0.0	0.0000	0	0	0.00	0	0	0.00
2	20.4	0.0136	2	32.7	0.02	2	50.5	0.03
4	25.7	0.0343	4	38.4	0.05	4	57.8	0.08
6	29.9	0.0598	6	43.9	0.09	6	60.2	0.12
8	34.2	0.0912	8	48.1	0.13	8	65.6	0.17
10	42.5	0.1417	10	54.5	0.18	10	72.5	0.24
12	49.2	0.1968	12	59.2	0.24	12	78.2	0.31
14	53.7	0.2506	14	63.8	0.30	14	83.8	0.39
16	57.5	0.3067	16	69.6	0.37	16	88.6	0.47
18	59.3	0.3558	18	70.7	0.42	18	89.9	0.54
20	59.4	0.3960	20	71.3	0.48	20	90.2	0.60

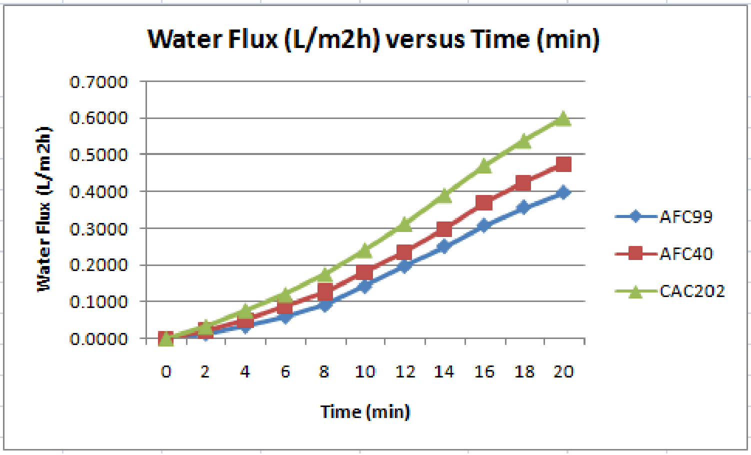


Figure 4.0: AFC99, AFC40 and CA202 at 10bar

<u>Pressure at 10bar, 15bar & 20bar – ZINC</u>

Table 9: AFC99, AFC40 and CA202 at 10bar, 15bar & 20bar for Zinc

Time (min)	AFC99 Membrane		
	10 bar	15 bar	20 bar
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	135	135	135
2	108	120	60
4	102	92	45
6	94	75	31
8	87	32	22
10	74	28	18
12	69	17	13
14	57	20	10
16	45	24	15
18	38	32	18
20	33	40	22

Time (min)	AFC40 Membrane		
	10 bar	15 bar	20 bar
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	135	135	135
2	131	130	125
4	127	116	98
6	118	108	83
8	102	93	71
10	84	84	60
12	77	65	52
14	68	58	44
16	53	44	31
18	47	40	26
20	38	32	21

Time (min)	CA202 Membrane		
	10 bar	15 bar	20 bar
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	135	135	135
2	135	133	124
4	134	128	120
6	130	114	107
8	123	109	99
10	110	100	81
12	98	95	73
14	87	82	68
16	74	71	62
18	69	63	52
20	61	57	40

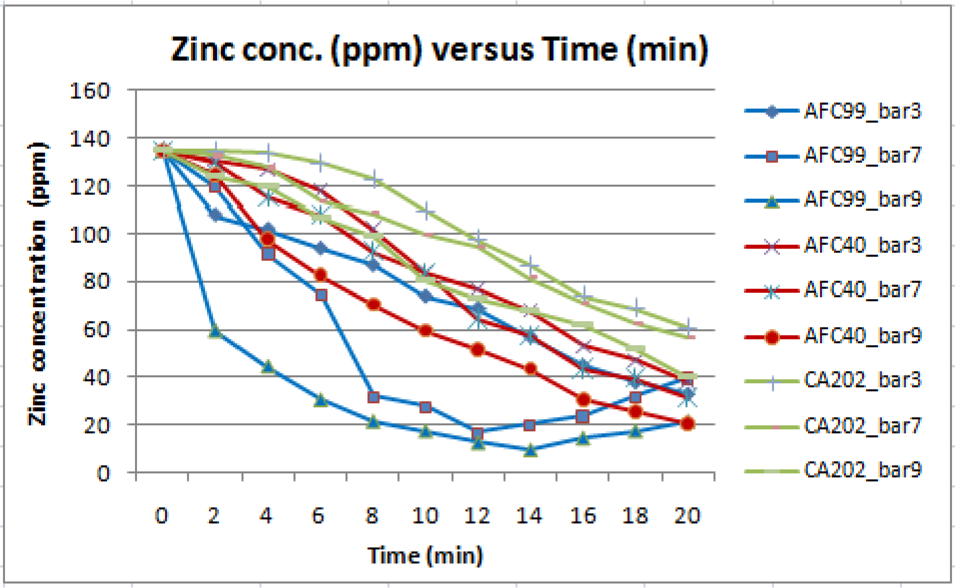


Figure 4.1: AFC99, AFC40 and CA202 at 10bar, 15bar& 20bar for Zinc

Pressure at 10bar, 15bar & 20bar –ALUMINIUM

Table 10: AFC99, AFC40 and CA202 at 10bar, 15bar & 20bar for Aluminum

Time (min)	AFC99 Membrane		
	10 bar	15 bar	20 bar
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	87	87	87
2	85	80	74
4	79	72	50
6	73	60	43
8	64	58	38
10	58	46	25
12	51	32	17
14	43	27	15
16	32	23	10
18	27	20	8
20	25	18	7

Time (min)	AFC40 Membrane		
	10 bar	15 bar	20 bar
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	87	87	87
2	86	85	80
4	82	81	72
6	78	78	68
8	71	69	61
10	65	54	48
12	59	47	42
14	54	39	35
16	47	31	28
18	41	26	16
20	38	23	15

Time (min)	CA202 Membrane		
	10 bar	15 bar	20 bar
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	87	87	87
2	86	86	83
4	86	84	77
6	81	80	71
8	79	72	65
10	72	68	59
12	68	61	53
14	62	58	46
16	57	53	38
18	53	47	29
20	46	40	23

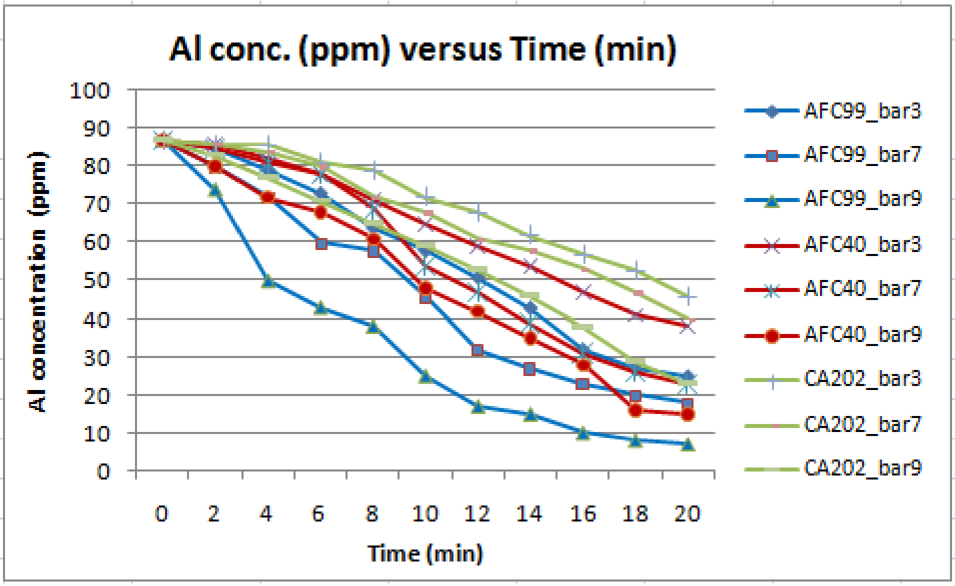


Figure 4.2: AFC99, AFC40 and CA202 at 10bar, 15bar & 20bar for Aluminum

Pressure at 10bar, 15bar & 20bar – COPPER

Table 11: AFC99, AFC40 and CA202 at 10bar, 15bar & 20bar for Copper

Time (min)	AFC99 Membrane		
	10 bar	15 bar	20 bar
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	120	120	120
2	110	98	83
4	83	70	65
6	65	57	43
8	59	41	25
10	43	37	14
12	37	13	9
14	25	10	4
16	19	5	2
18	18	5	2
20	15	5	2

Time (min)	AFC40 Membrane		
	10 bar	15 bar	20 bar
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	120	120	120
2	110	98	83
4	83	70	65
6	65	60	58
8	62	55	42
10	40	32	21
12	33	26	13
14	24	15	7
16	19	11	5
18	15	8	3
20	13	6	2

Time (min)	CA202 Membrane		
	10 bar	15 bar	20 bar
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	120	120	120
2	117	110	98
4	109	83	70
6	100	65	43
8	94	59	25
10	75	37	14
12	69	13	9
14	54	10	4
16	48	5	2
18	32	5	2
20	24	5	2

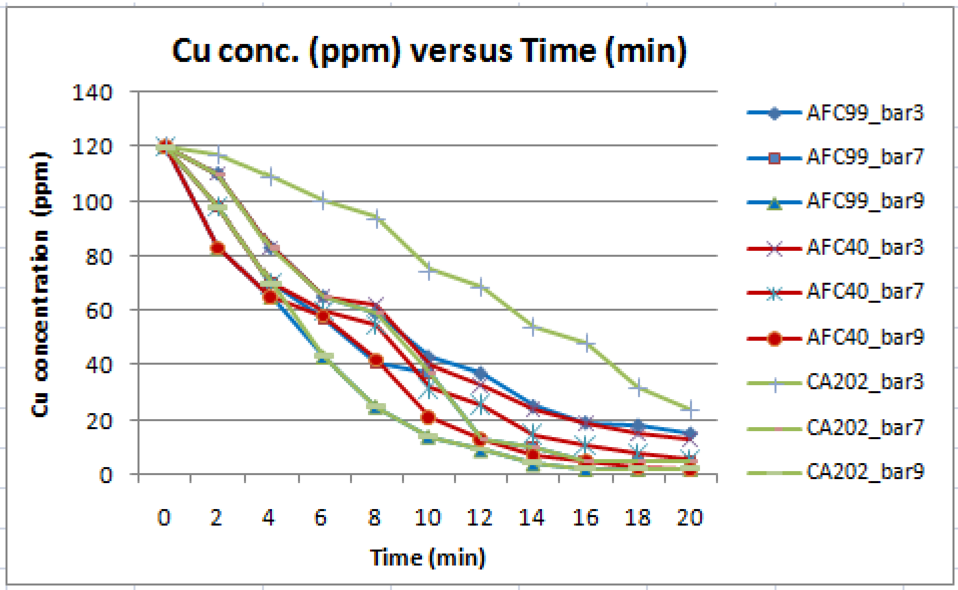
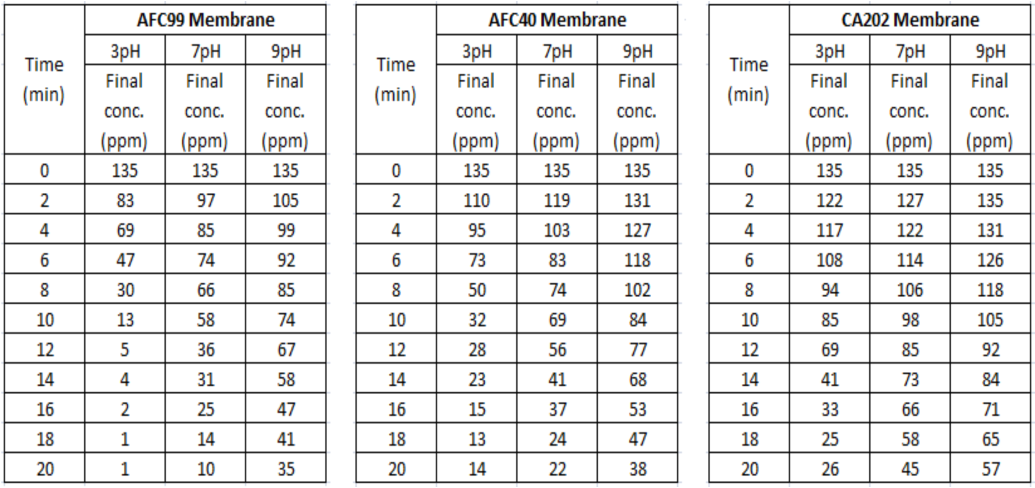


Figure 4.3: AFC99, AFC40 and CA202 at 10bar, 15bar & 20bar for Copper

pHat pH3, pH7 & pH9 – ZINC

Table 12: AFC99, AFC40 and CA202 at pH3, pH7 & pH9 for Zinc



Time (min)	AFC99 Membrane			Time (min)	AFC40 Membrane			Time (min)	CA202 Membrane		
	3pH	7pH	9pH		3pH	7pH	9pH		3pH	7pH	9pH
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)		Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)		Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	135	135	135	0	135	135	135	0	135	135	135
2	83	97	105	2	110	119	131	2	122	127	135
4	69	85	99	4	95	103	127	4	117	122	131
6	47	74	92	6	73	83	118	6	108	114	126
8	30	66	85	8	50	74	102	8	94	106	118
10	13	58	74	10	32	69	84	10	85	98	105
12	5	36	67	12	28	56	77	12	69	85	92
14	4	31	58	14	23	41	68	14	41	73	84
16	2	25	47	16	15	37	53	16	33	66	71
18	1	14	41	18	13	24	47	18	25	58	65
20	1	10	35	20	14	22	38	20	26	45	57

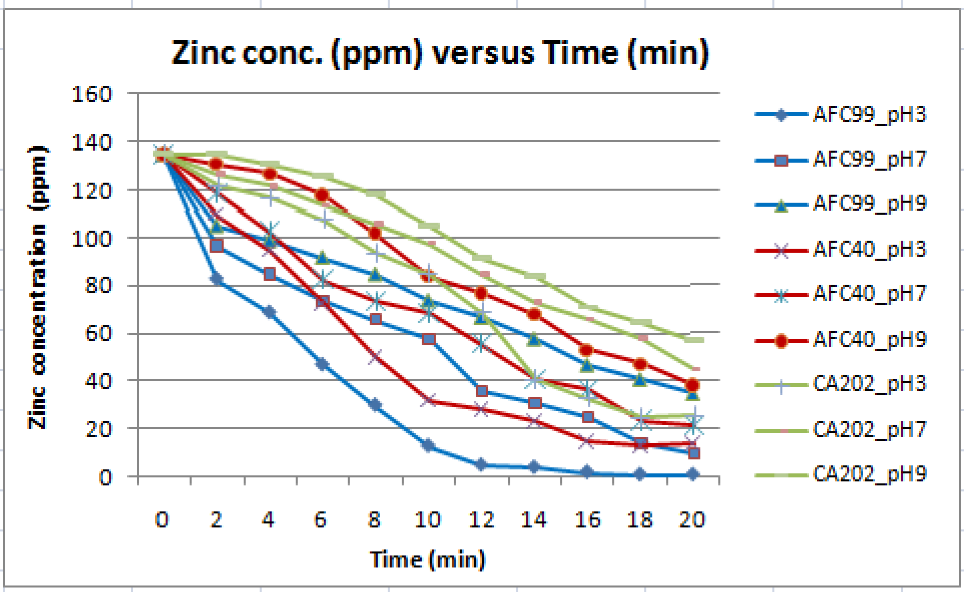


Figure 4.4: AFC99, AFC40 and CA202 at pH3, pH7 & pH9 for Zinc

<u>pHat pH3, pH7 & pH9 – ALUMINIUM</u>

Table 13: AFC99, AFC40 and CA202 at pH3, pH7 & pH9 for Aluminum

Time (min)	AFC99 Membrane		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	87	87	87
2	74	78	87
4	50	57	82
6	43	49	74
8	38	41	67
10	15	24	61
12	13	18	53
14	8	15	44
16	5	13	35
18	5	14	30
20	5	11	28

Time (min)	AFC40 Membrane		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	87	87	87
2	74	78	86
4	50	65	80
6	43	53	75
8	38	46	67
10	31	41	62
12	27	35	56
14	15	28	51
16	8	21	46
18	8	15	39
20	7	14	33

Time (min)	CA202 Membrane		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	87	87	87
2	74	78	86
4	50	69	85
6	43	55	84
8	38	48	77
10	30	41	73
12	26	37	70
14	19	28	64
16	14	22	61
18	15	18	57
20	12	17	50

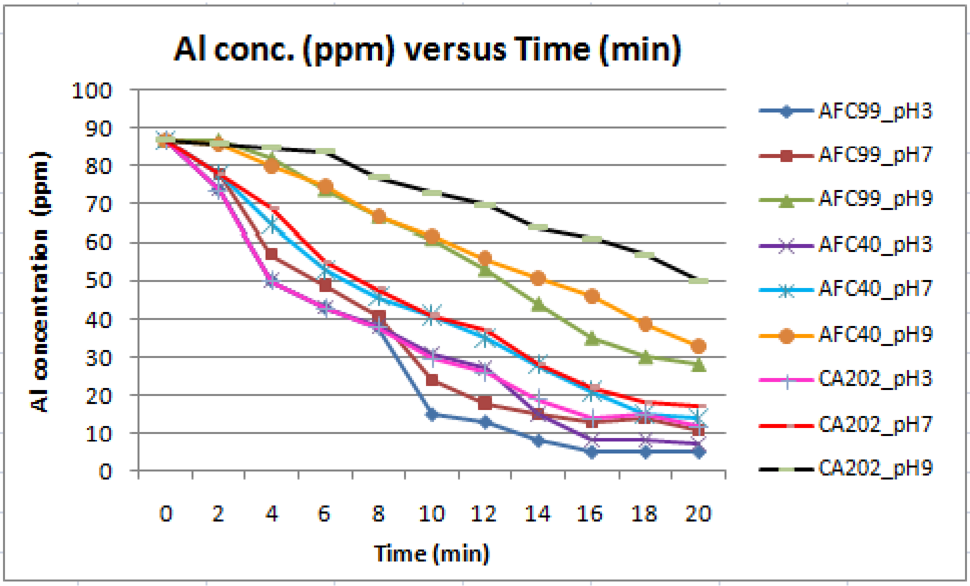


Figure 4.5: AFC99, AFC40 and CA202 at pH3, pH7 & pH9 for Aluminum

pHat pH3, pH7 & pH9 – COPPER

Table 14: AFC99, AFC40 and CA202 at pH3, pH7 & pH9 for Copper

Time (min)	AFC99 Membrane		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	120	120	120
2	110	112	115
4	83	85	95
6	62	71	78
8	51	58	65
10	33	43	50
12	11	22	41
14	5	15	33
16	3	11	25
18	1	8	19
20	1	5	12

Time (min)	AFC40 Membrane		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	120	120	120
2	98	110	115
4	65	77	85
6	39	65	70
8	21	56	65
10	12	35	43
12	9	17	35
14	4	10	22
16	2	9	15
18	2	9	12
20	2	8	10

Time (min)	CA202 Membrane		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	120	120	120
2	85	98	115
4	73	83	104
6	51	65	97
8	29	59	88
10	18	52	81
12	10	43	72
14	8	31	61
16	6	24	52
18	5	16	38
20	5	12	29

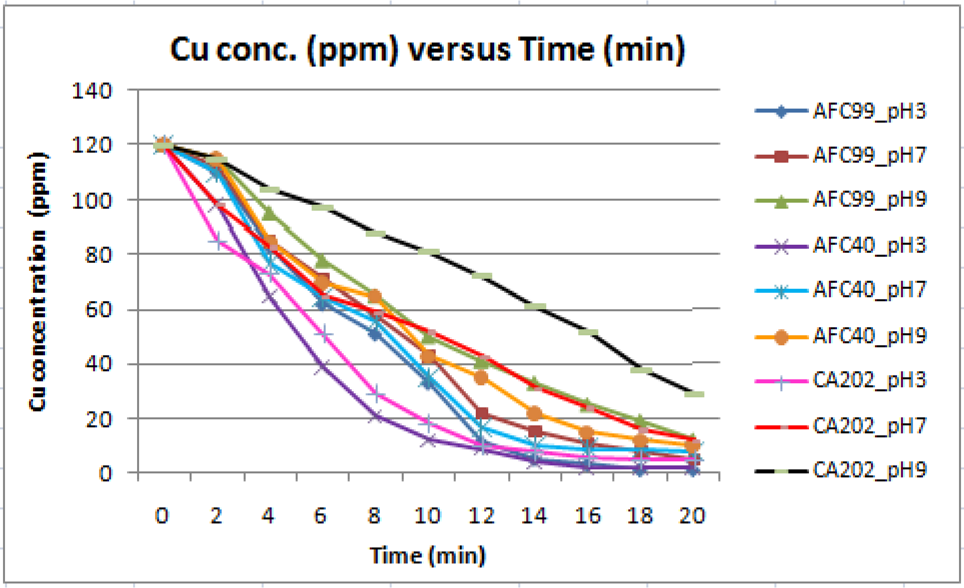


Figure 4.6: AFC99, AFC40 and CA202 at pH3, pH7 & pH9 for Copper

Concentrationat 75% Volume, 50% Volume & 25% Volume – ZINC

Table 15: AFC99, AFC40 and CA202 at 75% Volume, 50% Volume & 25% Volume for Zinc

Time (min)	AFC99	AFC40	CA202
	75%		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	101	101	101
2	93	97	100
4	85	92	97
6	69	87	91
8	54	74	88
10	48	65	79
12	35	58	71
14	27	49	65
16	21	35	58
18	16	27	51
20	15	21	46

Time (min)	AFC99	AFC40	CA202
	50%		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	68	68	68
2	60	64	67
4	52	61	65
6	44	56	60
8	38	51	54
10	31	47	51
12	26	40	46
14	23	35	42
16	18	29	37
18	16	23	30
20	13	19	24

Time (min)	AFC99	AFC40	CA202
	25%		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	34	34	34
2	29	31	31
4	24	26	29
6	20	22	26
8	17	19	25
10	14	18	23
12	13	16	19
14	10	14	16
16	7	10	14
18	6	9	11
20	5	8	10

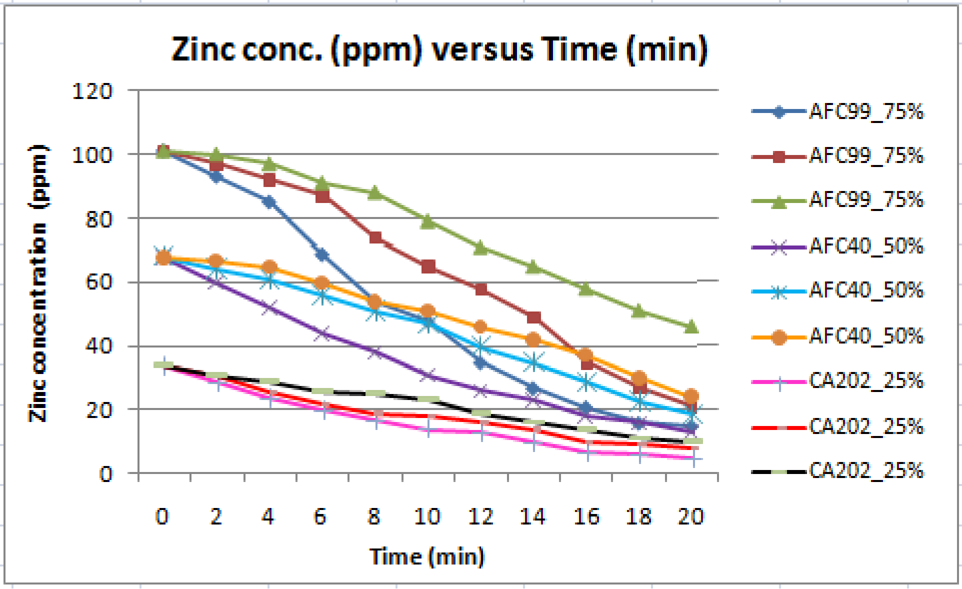


Figure 4.7: AFC99, AFC40 and CA202 at 75% Volume, 50% Volume & 25% Volume for Zinc

Concentrationat 75% Volume, 50% Volume & 25% Volume – ALUMINIUM

Table 16: AFC99, AFC40 and CA202 at
75% Volume, 50% Volume & 25% Volume for Aluminum

Time (min)	AFC99	AFC40	CA202
	75%		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	65	65	65
2	60	62	64
4	54	57	60
6	49	51	56
8	42	45	52
10	36	40	48
12	28	37	45
14	23	33	41
16	19	28	37
18	15	25	34
20	9	20	28

Time (min)	AFC99	AFC40	CA202
	50%		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	44	44	44
2	39	41	43
4	32	35	42
6	27	31	39
8	23	25	35
10	18	22	29
12	16	19	25
14	12	17	21
16	9	15	18
18	8	14	17
20	6	12	16

Time (min)	AFC99	AFC40	CA202
	25%		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	22	22	22
2	17	19	20
4	17	17	19
6	16	17	18
8	15	16	18
10	13	14	18
12	12	13	17
14	11	12	15
16	11	11	14
18	8	10	13
20	6	10	12

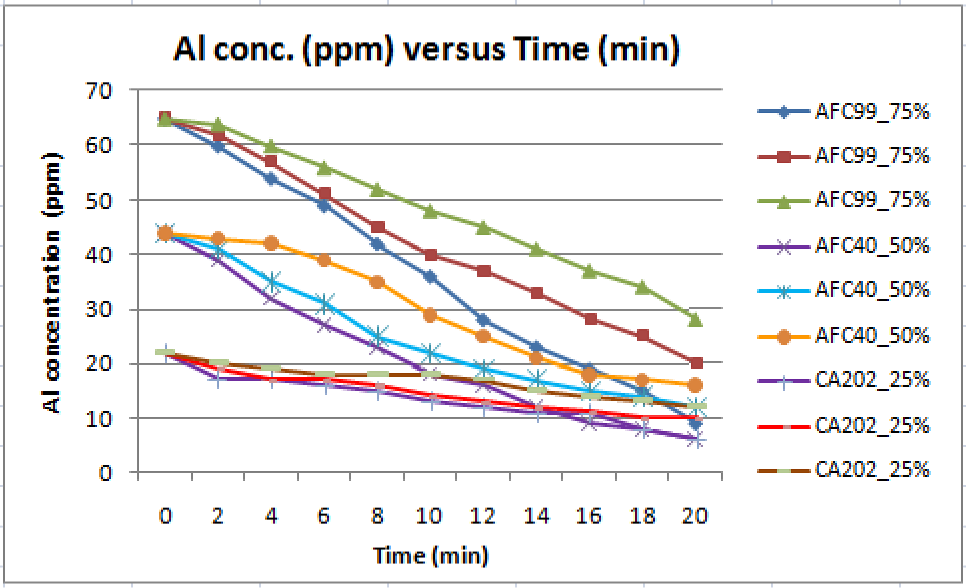


Figure 4.8: AFC99, AFC40 and CA202 at
75% Volume, 50% Volume & 25% Volume for Aluminum

Concentrationat 75% Volume, 50% Volume & 25% Volume – COPPER

Table 17: AFC99, AFC40 and CA202 at
75% Volume, 50% Volume & 25% Volume for Copper

Time (min)	AFC99	AFC40	CA202
	75%		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	90	90	90
2	81	84	88
4	75	79	83
6	69	74	78
8	63	68	75
10	56	66	70
12	49	59	63
14	37	47	56
16	25	35	48
18	14	22	38
20	10	15	26

Time (min)	AFC99	AFC40	CA202
	50%		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	60	60	60
2	53	56	58
4	48	52	55
6	45	48	51
8	41	44	46
10	37	39	42
12	33	35	38
14	24	28	33
16	17	24	29
18	12	17	25
20	9	13	20

Time (min)	AFC99	AFC40	CA202
	25%		
	Final conc. (ppm)	Final conc. (ppm)	Final conc. (ppm)
0	30	30	30
2	24	27	29
4	21	26	27
6	19	24	25
8	19	22	25
10	16	19	23
12	15	17	21
14	14	16	18
16	12	15	17
18	7	12	16
20	5	9	15

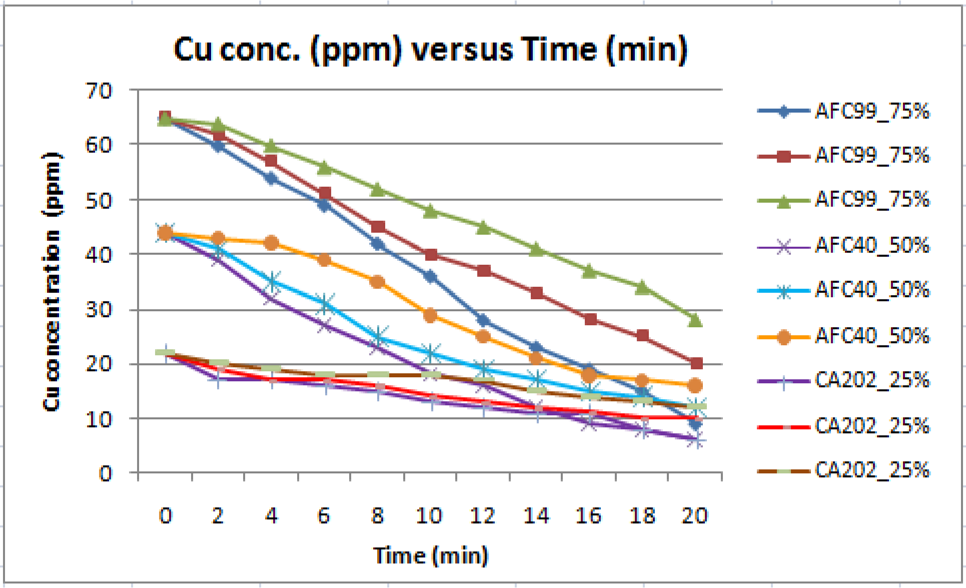


Figure 4.9: AFC99, AFC40 and CA202 at
75% Volume, 50% Volume & 25% Volume for Copper

4.2 Discussion

During the first half of the experiment, pressure of 10bar, 15bar and 20bar are used respectively as the manipulating variables and run for 20 minutes to observe the changes in the system. It is observed that permeate collected increased as time goes and the calculated water flux increased together with the increment of permeate volume. The filtered permeate is collected for every 2 minutes and each collected permeate are tested differently by utilizing Atomic Absorption Spectroscopy (AAS). Taking all the variables in comparison, we can observed that the volume of permeate collected increases as higher pressure is being input. At 10bar pressure, the volume of permeate continue to increase even after 20 minutes; at 15 bar pressure, the volume of permeate remained at 85ml starting from 16th minute onwards; at 20 bar pressure, the volume of permeate came to a steady volume at 87.9ml from 14th minute onwards which is 2 minutes earlier that 15 bar pressure. This is because at 10bar, the pores of the membrane are not fully clogged and hence permeate will continue to flow but for 15bar pressure it started to clogged at time 16th minute while at 20 bar pressure clogged at time 14th minute due to high pressure.

Taking a look at the initial and final concentration of the heavy metals, indeed the final concentrations of heavy metals in permeate decreased. The initial concentration of zinc metal is 135ppm and after treatment, the concentration is at 15ppm for 10 bar, 8ppm for 15 bar and 22ppm for 20 bar; the initial concentration of aluminum metal is 87ppm and after treatment, the concentration is at 25ppm for 10 bar, 18ppm for 5 bar and 22ppm for 20 bar; the initial concentration of copper metal is 120ppm and after treatment, the concentration is at 5ppm for 10 bar, 2ppm for 15 bar and 26ppm for 20 bar. For zinc removal at 20bar, the concentration increased at the 15th minute. This is because the high pressure somehow pushes the particles and ions though the membrane pores and causes the increment of zinc concentration. This happened for copper concentration at 20bar as well due to high pressure. Overall, the rejection rate is more than 90% and the objective is achieved.

The second parameter will be the pH value. In this project, pH3, pH7 and pH9 are being used as three different variables. The initial concentration of zinc is at 135ppm. AFC99 membrane is being test with three different pH values and the result shows that the final concentration increased as the pH goes higher. The tread goes the same as AFC40 and CA202 membrane. This can be concluded that all three membranes favor acidic pH value as its optimum pH to operate. Another reason is the material of the membrane itself, AFC99 and AFC40 are made up from polyamide film while CA202 is made up from cellulose acetate. These materials can operate better in acidic environment rather than in alkali environment. This followed by observing the final concentration of aluminum and copper ions. The final concentration is the best at AFC99 at pH3 which is 5ppm and the membrane managed to remove >95% of the heavy metals and once again, all these membrane achieved lower final concentration in acidic environment.

The third parameter is to study on the concentration of the wastewater in termed of volume percentage. Three different volume percentages which are 75%, 50% and 25% were being used. 75% volume indicates that out of the total volume of wastewater, 25% of the overall is consisted of distilled water and same goes to 50% and 25% volume. Due to the dilution, the initial concentration of each heavy metal is different. For zinc metal, the initial concentration for 75% volume is 101ppm, 50% volume is 68ppm and 25% volume is 34ppm. For aluminum metal, the initial concentration for 75% volume is 65ppm, 50% volume is 44ppm and 25% volume is 22ppm. For copper metal, the initial concentration for 75% volume is 90ppm, 50% volume is 60ppm and 25% volume is 30ppm. The experiment proved that even with 100% concentration, the final concentration for all heavy metal tested is passed under Standard A and Standard B under the Discharge of Industrial Effluent in Department of Environment.

Take a look at the cost between the conventional method and membrane separation. At constant flow rate, the conventional method which is the chemical precipitate require a constant supply of chemical dosing and other chemicals in order to support the whole system. On the other hand, membrane separation does not require any chemicals and no other cost; it only need to purchase the whole equipment in once and for all. The only thing is just to change the membrane tubes after a period of time which is far more convenience than the chemical precipitate. Furthermore, the conventional method require a large amount of space in order to place its equipments like settling tanks, clarifiers, dosing tanks and many more while membrane separation only need a small space to place its membrane tubes and a place to collect the permeate and retentate. In a nutshell, membrane separation is a very potential way in removing heavy metal ions and the result in removing heavy metal ions are on par and even better than the conventional method which is the chemical precipitate.

CHAPTER 5

CONCLUSION

5. Conclusion

At the end of this project, this experiment is relevant to the objectives because it fulfilled all the objectives which are firstly, to study possibility of removing heavy metal ions using composite reverse osmosis membrane. The types of membranes we are using inthis experiment are AFC99, AFC40 and CA202. Secondly is to study the variation of data collected during process: Pressure vs flux, pH value versus flux and also concentration vs flux. Graphs and results data will be obtained and comparisons will be made to determine its efficiency in removing heavy metal ions which in this care are aluminum, zinc and copper. Lastly is to simplify the conventional wastewater treatment into RO system when the results proves that the RO system

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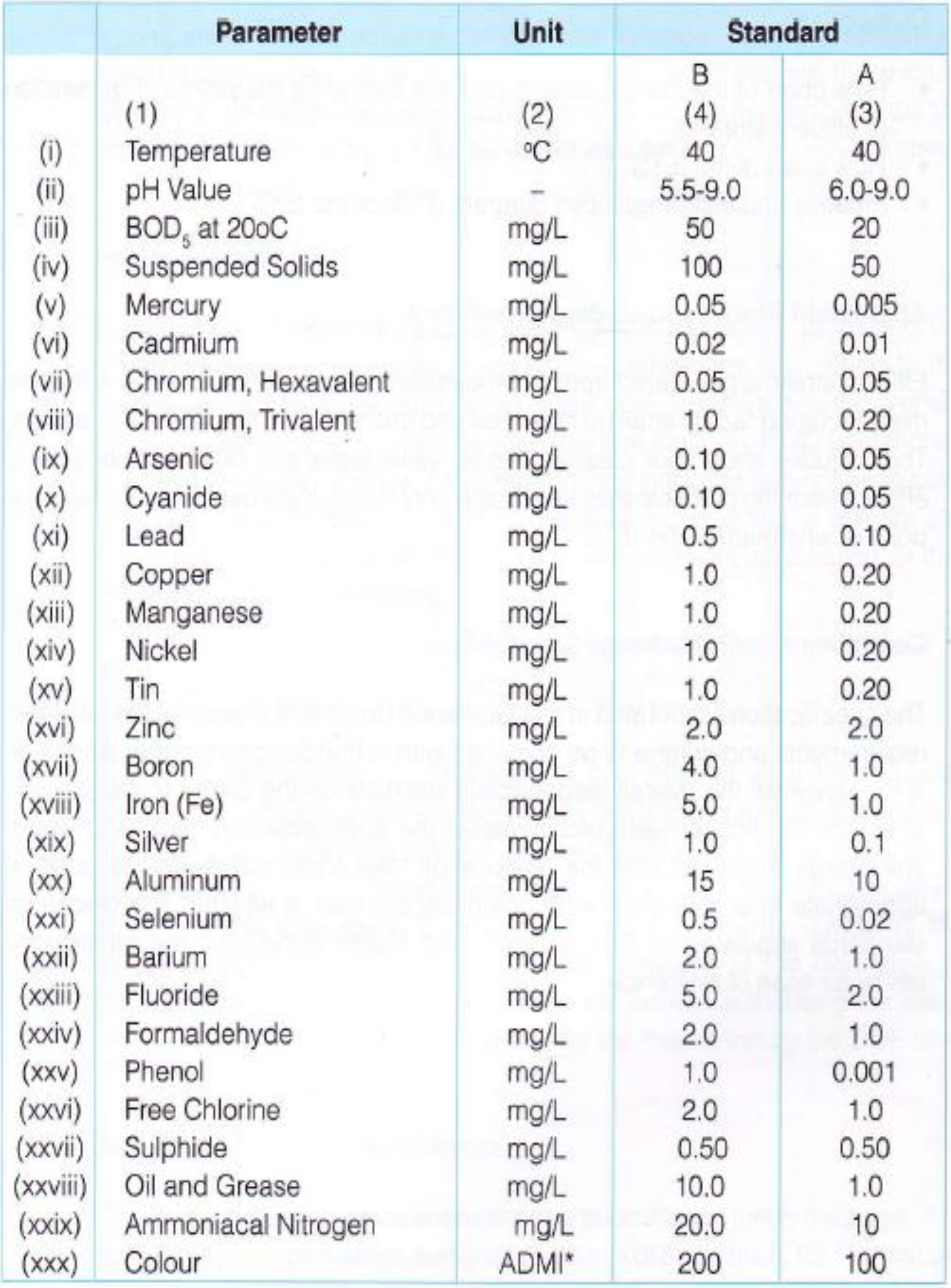
APPENDIX

Fifth Schedule to the Industrial Effluent Regulations, 2009

Table 19 Acceptable Conditions for Discharge of Industrial Effluent or Mix Effluent of Standard A & Standard B



	Parameter	Unit	Standard	
			B	A
	(1)	(2)	(4)	(3)
(i)	Temperature	°C	40	40
(ii)	pH Value	–	5.5-9.0	6.0-9.0
(iii)	BOD_5 at 20oC	mg/L	50	20
(iv)	Suspended Solids	mg/L	100	50
(v)	Mercury	mg/L	0.05	0.005
(vi)	Cadmium	mg/L	0.02	0.01
(vii)	Chromium, Hexavalent	mg/L	0.05	0.05
(viii)	Chromium, Trivalent	mg/L	1.0	0.20
(ix)	Arsenic	mg/L	0.10	0.05
(x)	Cyanide	mg/L	0.10	0.05
(xi)	Lead	mg/L	0.5	0.10
(xii)	Copper	mg/L	1.0	0.20
(xiii)	Manganese	mg/L	1.0	0.20
(xiv)	Nickel	mg/L	1.0	0.20
(xv)	Tin	mg/L	1.0	0.20
(xvi)	Zinc	mg/L	2.0	2.0
(xvii)	Boron	mg/L	4.0	1.0
(xviii)	Iron (Fe)	mg/L	5.0	1.0
(xix)	Silver	mg/L	1.0	0.1
(xx)	Aluminum	mg/L	15	10
(xxi)	Selenium	mg/L	0.5	0.02
(xxii)	Barium	mg/L	2.0	1.0
(xxiii)	Fluoride	mg/L	5.0	2.0
(xxiv)	Formaldehyde	mg/L	2.0	1.0
(xxv)	Phenol	mg/L	1.0	0.001
(xxvi)	Free Chlorine	mg/L	2.0	1.0
(xxvii)	Sulphide	mg/L	0.50	0.50
(xxviii)	Oil and Grease	mg/L	10.0	1.0
(xxix)	Ammoniacal Nitrogen	mg/L	20.0	10
(xxx)	Colour	ADMI*	200	100