CHAPTER 1

INTRODUCTION

1.1 Background Study

A general overview of coolant used by Cameron International Malaysia Systems (CIMS) and the generating process is introduced in this chapter. Problems associated with coolant will be discussed further and lastly, the objective and scope of the study.

Cameron International Malaysia Systems (CIMS) is the leading manufacturer in producing wellheads and subsea trees use for oil exploration. A lot of the manufacturing process involves high precision machining which require coolant as metal working fluid. Coolant, also known as metalworking fluids or metal removing fluids (MWF and MRF) is a complex soup of oil blended into water. Coolant concentrates are typically diluted with anywhere from 80% to 99% water, but are usually used in the 5% range. Machine operations in CIMS involve cutting, drilling, milling and boring. Coolant is used in metalworking to both cool and lubricate tools so that they cut better and last longer. It is required to cool the heat build during the metal contact between the tool drill and the job to be cut. Type of coolant used at CIMS is water miscible, Fuchs Ecocool BF-32X, which composed of highly refined mineral oils, synthetic fatty agents and additives having excellent anti corrosion protection

There are 11 machines in CIMS which require coolant to smooth up their operations. Those machines are Safop, Guruptze, Mazak E-500, Mazak IG-70, GNL RT-1, GNL RT-2, GNL MC-1 and GNL MC-2. All these machines require coolant system. The standard machine uses a coolant sump built into the foundation with coolant capacity of 500 liter. Effluent water (coolant mixed with water) is major hazardous waste generated from production. The total amount of coolant wastes generated until October 2008 is 61 drums (122 000 liter).

1.2 Problem Statement

Current practice in Cameron International Malaysia Systems (CIMS) to manage effluent water is by sending to waste treatment centre. Effluent water is a mixture of coolant and water. The cost of disposing effluent water is increasing every year due to limited treatment services available. The amount is RM 120 per drum and the total amount to dispose 61 drums is RM 7320 which is highly expensive. Furthermore, effluent water is categorized as hazardous waste due to its hazardous ingredients such as alkanoamines and biocides.

Disposing effluent water to ground water will definitely contaminate it even though the percentage of water mix with coolant is higher. Separation technique to remove coolant oil from the effluent water is one of the methods in reducing the coolant disposal cost. Removing coolant by demulsification combined with reverse osmosis technique will be the chosen method in order to have a lower concentration of coolant oil, carbon oxygen demand (COD), total organic carbon (TOC) and turbidity in the effluent water and will be the main objective in this project

1.3 Objectives

The main objectives of the project are as follow:

- To separate coolant from effluent water and obtain lower concentration of coolant in the effluent water.
- To treat effluent water and obtain lower total organic carbon (TOC), chemical oxygen demand (COD), and turbidity in the effluent water
- To purify effluent water

1.4 Scope of Study

- Self study on the separation techniques to be apply for the experiment
- Self study on the parameters influencing separation of coolant from effluent water by demulsification combined with reverse osmosis.
- Develop methodology and parameters to be tested during experiment.
- Implement the separation technique chosen for coolant removal from effluent water.
- Analyze result obtained from the experiment.

CHAPTER 2

LITERATURE REVIEW

2.1 Preface

Before proceeding to the implementation stage of the removing coolant from effluent water by combined demulsification and reverse osmosis, some theoretical knowledge should be learned and well understood. In this chapter the reader is introduced to the glance knowledge on the characteristic of coolant, purpose of demulsifier and how reverses osmosis works.

Quite a number of manufacturing facilities uses significant quantities of metal-working fluids. The fluid which is normally used for instance, is cutting oil or known as coolant which is a typical oil/water emulsion that is commonly used in a variety of high precision machining. It serves the purpose of lubrication, surface cleaning and corrosion prevention. The main problem with this oil is that they become contaminate with use, losing their properties and have to be replaced by new ones, thus yielding an oily wastewater (In-Soung Chang, 2000).

A large volume of waste water in the form of either oil-in-water (o/w) or water-in-oil (w/o) emulsions is generated from various processes industries such as metallurgical, transportation, food processing and petrochemical industries as well as petrochemical industries (B.Chakrabarty, A.K. Ghoshal, M.K. Purkait, 2008). The three broad categories in which industrial oily wastewater exist are free-floating oil, unstable oil/water emulsion and stable oil/water emulsion (B.Chakrabarty, A.K. Ghoshal, M.K. Purkait, 2008). Free-floating oil or unstable oil/water emulsions can be readily removed by using conventional separation process. However, for removing stable oil/water emulsion such as coolant, those conventional processes is not found to be so effective.

Several separation setups have been used for oily water treatment: settlers, deep bed filters, bed coalescers, centrifuges, adsorbes, membranes and others (Radmila Secerov Sokolovic, Slobodan Sokolovic, Snezana Sevic, 2009). Reports say that this conventional method such as biological method can reduce oil concentration to hardly 1% by volume of the total waste water and cannot efficiently remove oil droplets below 1μm size (B.Chakrabarty, A.K. Ghoshal, M.K. Purkait, 2008). Choosing the separation techniques depends on several factor, the most important being oil solubility in water. Oily water with emulsion droplets of higher than 50μm is in unstable state and less than 10 μm considered to be highly stable and is very difficult to separate particularly when oil concentration is in lower range (B.Chakrabarty, A.K. Ghoshal, M.K. Purkait, 2008).

Under such circumstances, the use of membranes offers a potential solution to the problem of micron sized oily water like coolant. The advantages of membrane process such as lower capital cost, the non-requirement of any chemical addition and the capability of generating permeate of acceptable quality (B.Chakrabarty, A.K. Ghoshal, M.K. Purkait, 2008). Membrane-based systems have successfully been applied or considered as possible candidates for post treating kind of liquid wastes in industrial plants (Alireza Rezvanpour, Reza Roostaazad, Mehrdad Hesampour, Marianne Nystrom, Cyrus Gothbi, 2009). The advantages of this system are easy operation, lower cost in some cases and capability of reducing contaminants to below the limits.

2.2 Alternatives in Oily Wastewater Treatment

Lots of methods have been used in treatment of oily wastewater. A research on few methodologies has been done to observe which method is preferred in removing oil from wastewater. Each method mentioned here has its own advantages and disadvantages and every method has its research example.

Reverse Osmosis

Advantages:

- Studies have shown that RO technology is an effective technology to remove organic compounds from water
- It has been reported that microfiltration, ultra filtration and nanofiltration membranes undergo fouling in some circumstances in treatment of oily water whereas RO membrane technology is found to be an effective process to separate organics from water (B.Chakrabarty, A.K. Ghoshal, M.K. Purkait, 2008).
- RO technology is mostly used to separate small fractions (in ppm ranges) of organics from water.

Research example:

An experimental evaluation of reverse osmosis membrane performance in oily water
(Subhi Al-Jeshi, Anne Neville, 2008)

The objective of this paper was to investigate the ability of RO membranes to treat water containing up to 50% (by volume) of oil and to evaluate effect of oil contamination on membrane performance. Two types of membrane used are Osmonics SG and TriSep X20.

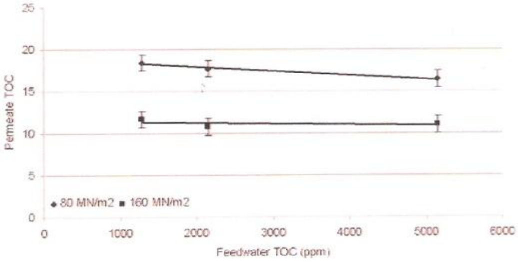


Fig 2.1: TriSep X20 membrane quality at two pressures

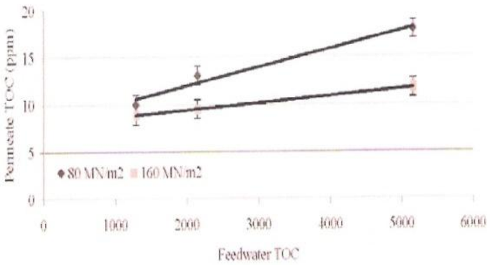


Fig 2.2: Osmonics SG membrane permeate quality at two pressures

The effects of feed water pressure, pH and temperature on membrane performance were investigated. For the effect of feedwater pressure on permeate TOC content, both membranes were tested at 80 and 160 MN/m2.

From the graph, it is observed that increasing feedwater pressure from 80 to 160 MN/m2 leads to better permeate quality from both membranes (Subhi Al-Jeshi, Anne Neville, 2008).That is likely to be due to an increase in water permeation rate, which leads to oil dilution with increased product water flow (Subhi Al-Jeshi, Anne Neville, 2008).

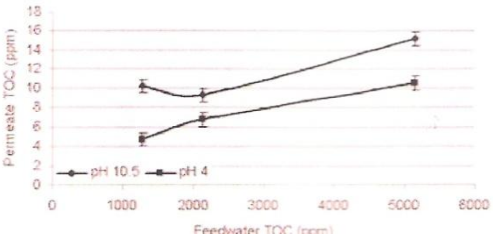


Fig 2.3: TriSep X20 membrane permeate quality at two pH values

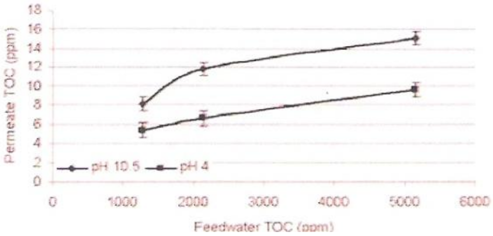


Fig 2.4: Osmonics SG membrane permeate quality at two pH values

For effect of pH on permeate TOC content, it was observed that both membranes were sensitive to pH. Varying feed water pH leads to change in membrane surface charge, which can affect the membrane hydrophibicity and hydrophilicity (Subhi Al-Jeshi, Anne Neville, 2008). At low feed water pH (acidic), the membrane became hydrophilic whereas at high pH (basic) the feed water became hydrophobic. Hydrophobic membrane tends to absorb foulant (oil), which leads to oil attachment on the membrane surface and therefore higher oil passage (Subhi Al-Jeshi, Anne Neville, 2008).

Common problems and troubleshooting of Reverse Osmosis:

All membrane separation processes have the potential for fouling of the upstream membrane surface due to sedimentation of suspended solids. The remedies for fouling are usually one or more of the following:

- Maintain high flow velocities and use turbulence-enhancing spacers in the low clearance flow paths.
- Pretreat feed streams to remove particulates and potentials precipitates. Chemical precipitation before filtration can be used to remove dissolved solids solutes likely to precipitate in membrane modules (Robert B.Long, 1995)
- Clean the membrane modules either chemically or mechanically on a regularly scheduled basis.

Ultrafiltration

Advantages:

- No chemical addition needed to stabilize the emulsion
- High COD removal efficiencies are achieved
- Treatment facilities are quite compact and fully automated.

Research example:

Treatment of oily wastewater by ultrafiltration and ozone.
(In-Soung Chang, 2000)

The aim of this study was to apply membrane technology for the treatment of oily wastewater and to reuse permeate and thus to establish an environmentally benign process. A UF pilot plant equipped with hollow fibers was used for the oily wastewater treatment. In order to reuse the UF permeates as make-up water, partial oxidation using ozone was performed to destroy organic components remaining in the permeates (In-

Soung Chang, 2000). Three kind of oily waste water were used for the experiment; oily wastewater generated from the degreasing process for aluminium casting, oily wastewater generated from the degreasing process for iron casting and coolant. But for this report, only the result obtained from experiment using coolant is emphasized.

Sample	COD (mg/l)	TOC (mg/l)
Cutting oil wastewater	102400	28200
UF permeates	7000	1840

Table 2.1: TOC and COD values for both samples (In-Soung Chang, 2000)

From the result obtained, COD and TOC rejection for coolant is 93.2% and 93.5% (In-Soung Chang, 2000). Generally, most oily wastewater contains only a small concentration of oil. But the ratio of surfactant to oil in coolant should be relatively smaller. In this study, the average emulsion size of the coolant was about 123 nm and the UF membrane readily removed the emulsified coolant in the waste water. It is also found that the UF permeates of coolant cannot be used as make up water for emulsification because they contain large amount of surfactant, the emulsion size and foaming ratio will change (In-Soung Chang, 2000).

Common problems and troubleshooting of Ultrafiltration:

Common problems and troubleshooting of ultrafiltration are the same as reverse osmosis.

Anaerobic Thermophilic Fluidized Bed Reactor (AFBR)

Advantages:

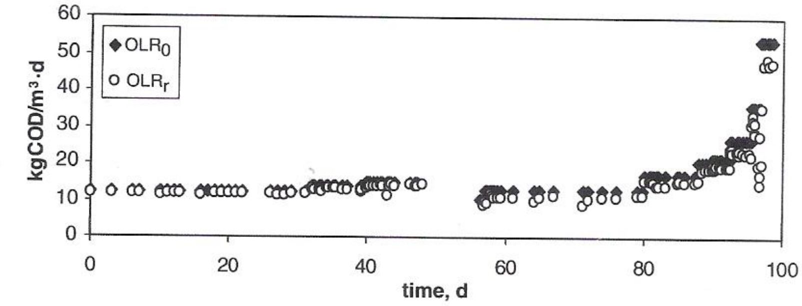
- Low energy consumption
- Low excess sludge production
- Enclosure of odors and aerosols

Research example:

Performance of Anaerobic thermophilic fluidized bed in the treatment of cutting oil wastewater

(M. Perez, 2007)

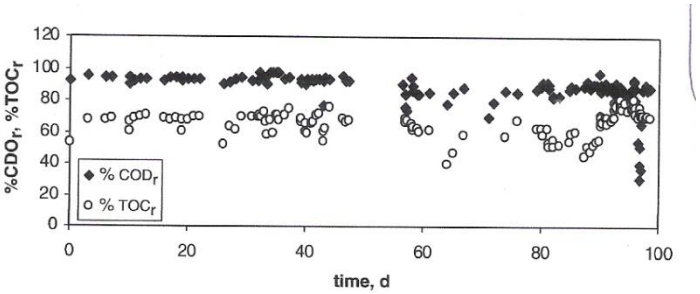
The purpose of this study was to elucidate the treatment effiencency of AFBR that decomposes cutting oil wastewater. The experimental protocol was designed to examine the effect of organic loading rate on the efficiency of COD and TOC removal under different hydraulic retention times, HRT (conditions).



(M. Perez, 2007)

Fig 2.5: Organic loading and removing rate,OLR0 and OLRr as kg COD/m3 d

From the graph, the organic loading rate and removing rate, OLR_0 and $OLR_{r,}$ increased as time increased. This showed that the longer the process time, more kg of COD are removed.



(M. Perez, 2007)

Fig 2.6: Organic removal efficiency (as percentage initial COD and initial TOC)

As for the second graph, AFBR proved that it is efficient in removing organic (as percentage of initial COD and TOC) as the graph is linear.

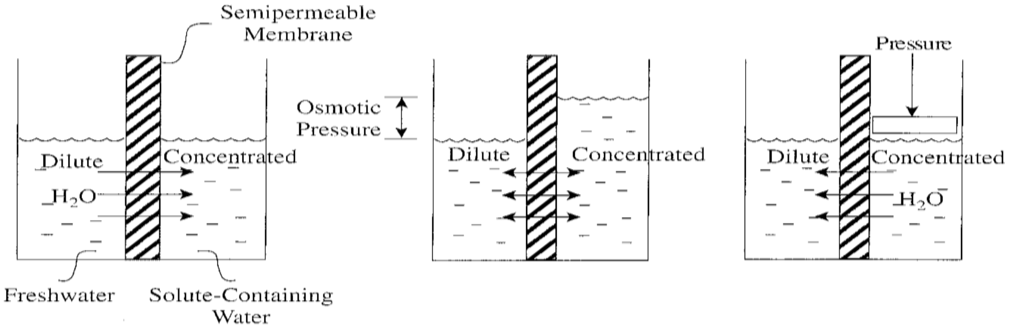
2.3 Reverse Osmosis

2.3.1 Introduction

Reverse osmosis treatment will be conducted as the next polishing step to further lower both chemical oxygen demand (COD) and oil concentration in order to improve the water quality of permeate. The application of RO technology in waste water treatment has been reported since the 1970s (Subhi Al-Jeshi, Anne Neville, 2008). RO membranes have been applied in removing oil pollutants, organic compounds, humic substances, vegetable oil, and pesticides from water to recycle or meet environmental regulations before discharge to sewers (Subhi Al-Jeshi, Anne Neville, 2008). RO membranes were also used successfully in organic environments such as carboxylic acids from ethanol and hexane solutions. Other sources also quoted that membrane filtration is playing a more prominent role in treatment of oily wastes because it provides undeniable advantages: no chemical additives are needed to stabilize the emulsion, high COD removal efficiencies are achieved, and treatment facilities are quite compact and fully automated (In-Soung Chang, 2000). Even though it is effective, results from the tests show that both ultrafiltration (UF) and reverse osmosis (RO) are efficient and economically attractive for the oil wastewater treatment, but these methods cannot be used direct to deal with high strength oil emulsion due to intensive contamination of membrane. (Hongzhong Zhang, Shaoming Fang, Changming Ye, Minghua Wang, Haijun Cheng, Hui Wen, Xianglan Meng, 2008).

2.3.2 Reverse Osmosis

In reverse osmosis, a solvent permeates through a dense asymmetric membrane that is permeable to the solvent but not to the solute. The solvent is usually water and the solutes are usually dissolved salts. The principle of reverse osmosis is illustrated in Figure 2.7(a); a solute dissolved in a solvent in a concentrated form is separated from the same solvent in a dilute form by a dense membrane. Given the difference in concentration across the membrane, a natural process known as osmosis occurs, in which the solvent permeates across the membrane to dilute the more concentrated solution. The osmosis continues until equilibrium is established as in Figure 2.7 (b). At equilibrium, the flow of solvent in both directions is equal and a difference in pressure is established between the two sides of the membrane, the osmotic pressure. Although a separation has occurred as a result of the presence membrane, the osmosis is not useful because the solvent is transferred in the wrong direction, resulting in mixing rather than separation. However, applying a pressure to the concentrated solution, as shown in Figure 2.7(c), can reverse the direction of transfer of solvent through the membrane. This causes the solvent to permeate through the membrane from a concentrated solution to the dilute solution.



(a) Osmotic flow. (b) No flow at equilibrium. (c) Reverse osmosis.

Figure 2.7: Reverse osmosis

The flux of the membrane can be written as

$$N_i = \frac{PM,i}{\delta M} (\Delta P - \Delta \pi) \qquad \text{(Robin Smith, 2005)}$$

Where N_i = solvent (water) flux through the membrane ($kg.m^{-2.}s^{-1}$)

$P_{M,i}$= solvent membrane permeability (kg solvent.m^{-1}.bar^{-1}.s^{-1})

δ_M = membrane thickness (m)

ΔP = pressure difference across the membranes (bar)

$\Delta \pi$ = difference between the osmotic pressures of the feed and permeate solutions (bar)

Hence, as the pressure difference is increased, the solvent flow increases. The pressure difference used varies according to the membrane and the application but is usually in the range 10 to 50 bar but can also be up to 100 bar (Robin Smith, 2005). The osmotic pressure for dilute solutions can be approximated by the Van't Hoff equation:

$$\pi = iRT\frac{Ns}{V}$$ (Robin Smith, 2005)

Where π = osmotic pressure (bar)

i = number of ions formed if solute molecules dissociates

R= universal gas constant (0.083145 $\text{bar.m}^3\text{.K}^{-1}\text{.kmol}^{-1}$)

T = absolute temperature (K)

Ns = number of moles of solute (kmol)

V= volume of pure solvent (m^3)

Applications of reverse osmosis are normally restricted to below 50ºC. When used in practice, the membranes for reverse osmosis must be protected by pretreatment of the feed to reduce membrane fouling and degradation. If necessary, pH should be adjusted to avoid extremes of pHs Also, oxidizing agents such as free chlorine must be removed. Even with elaborate pretreatment, the membrane may still need to be cleaned regularly.

Today, more than 1 000 reverse osmosis desalting plants are producing more than 750, 000, 000 gallons per day of portable water world wide. Other uses of reverse osmosis, usually on a smaller scale than the desalinization of water to produce potable water, include the treatment of industrial wastewater to remove heavy metal ions, non-biodegradable substances and other components of commercial value (Brown LeMay Bursten, 2000), the treatment of rinse water from electroplating processes to obtain a metal ion concentrate and a permeate that can be used as a rinse (Christie John Geankoplis, 2003), the separation of sulfites and bisulfites from effluents in pulp and paper processes , the treatment of wastewater in dying processes (Iwana, Y.Kazuse, 1982), the recovery of constituents having food value from wastewaters in food processingplants for example lactose, lactic acid, sugars and starches (James M.Douglas, 1988), the treatment of municipal water to remove inorganic salts, low-molecular weight organic compounds, viruses and bacteria, and dewatering of certain food products such as coffee, soups, tea, milk, orange juice and tomato juice. (J.D Seader and Earnest J.Henley, 1998). In such applications, membranes must have chemical, mechanical and thermal stability to be competitive with other processes (J.D Seader and Earnest J.Henley, 1998) (James M.Douglas, 1988).

2.4 Demulsifier

Demulsifier is a chemical used to break emulsions (that is, to separate the two phases). The type of demulsifier selected depends on the type of emulsion, either oil-in-water or water-in-oil (Sclumberger). Demulsifying is a pre-step before reverse osmosis to remove coolant oil and to avoid contamination to membrane. Later, the process will be followed by reverse osmosis to lower COD, TOC, turbidity and coolant concentration in the effluent water.

CHAPTER 3

METHODOLOGY

3.1 Project Flow

This project starts when the objectives and scopes of project being defined with the great understanding and feasibility. The project scopes must take into the consideration of time constraint and facility availability. The main interest of this project is to study the suitable type of demulsifier use, the affecting parameters in removing coolant from effluent water and the percentage of oil can be removed by reverse osmosis.

After the goal of this project defined, the appropriate project flow has been designed. The proposed project flow is shown in figure 3.1.

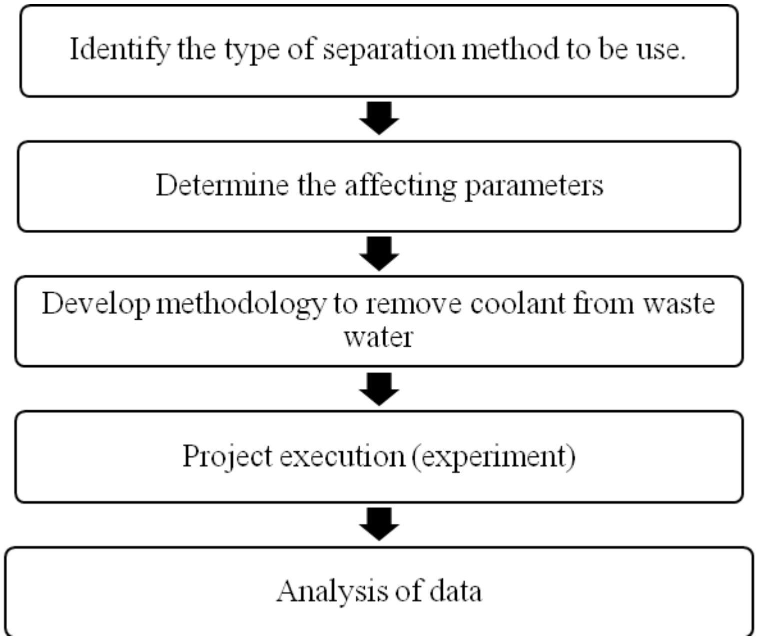


Figure 3.1: Project flow

3.2 Tools and Equipment

Materials	Equipments
Distilled water	1 L beaker
Demulsifier	60 ml container
Coolant	Graduated Cylinder
COD reagent	Water bath
	RO test unit
	Erlenmeyer flask
	Turbidmeter
	DR 5000 Spectrophotometer
	HACH 8000 (Reactor)
	TOC Analyzer
	Syringe

Table 3.1: List of material and equipment use.

3.3 Experimental Procedure

3.3.1 Preparation of coolant solution

The sample of coolant waste for the experimental study will be obtained from an underground store tank of Cameron International Malaysia Systems (CIMS). The sample is characteristic of high-strength oil with a strong milky brown color.

3.3.2 Demulsification Method

Coolant waste was demulsified in a bulk composition. A total of 10 liter of coolant waste was collected. 1 liter of coolant waste was taken to measure the effect of demulsifier dosage, heating temperature and time, whilst the rest are demulsified in bulk composition. 2 drums with volume of 10 liters were used by demulsifying 4.5 liters of effluent water with 3 liters of demulsifier (degreaser).

i) Effect of demulsifier dosage

1. Mix 50 ml of coolant waste with demulsifier (degreaser) with following volumes: 10 ml, 15 ml, 20 ml, 25 ml and 30 ml.
2. Put samples in test bottles without stirring.
3. Determine volume of separated oil after 3 days.

ii) Effect of heating temperature

1. Mix 50 ml of coolant waste with 30 ml of demulsifier (degreaser).
2. Shake the sample and put in water bath for 2 hour at the following temperatures: Room temperature (27°C), 30 ºC, 40ºC, 50ºC, 60ºC and 70ºC.
3. Determine volume of separated oil.

iii) Effect of heating time

1. Mix 50 ml of coolant waste with 30 ml of demulsifier (degreaser)
2. Shake the sample and put in room temperature.
3. Record the amount of separated oil.
4. Take reading at interval of 2 hours for 10 hours.

3.3.3 Reverse Osmosis

(Refer to Appendix V for Process Schematic Diagram of RO Test Unit)

3.3.3.1 Start-up procedure

1. Turn on the main supply.

2. Turn in the “General” button

3. Ensure that all valves are set according to the table below:

Open	Close
V1	DV1
V2	DV4
V3	V4
	V5

Table 3.2: Open and close valves for start-up procedure

4 Fill the tank with effluent water.

5 Turn on the "booster pump". Record the ppm of the effluent water.

6 Switch off the "booster pump".

7 Record the ppm of solution in the tank.

3.3.3.2 RO Membrane

1. Ensure that all valves are set according to the table below:

Open	Close
V1	DV1
V2	DV4
V4	V3
V5	
NV1-Open 20%	
NV2-Open 20%	

Table 3.3: Open and close valves for RO Membrane

2. Set 3/2 way valve "V6" to "RO membrane path".

3. Start up RO by pressing "booster pump on" and wait for 5 minutes for the pump flow to stabilize.

4. Follow by pressing the "high pressure pump on" button in sequence.

5. Slowly regulate the control valves; NV1 and NV2 to obtain desired applied pressures as require in table 3.2 and take readings. Reading should be taken at every 5 minutes interval for 30 minutes.

6. Take the value of total dissolve solids (TDS 1) and total dissolve solids (TDS 2) to calculate percent rejection:

$$\text{Percent rejection} = (1-\frac{Cp}{Cb})\ \text{x}100\%$$

3.3.3.3 Shut-down Procedures

1. Switch off the system by pressing "booster pump off" button. (Both the high pressure pump and the booster pump will be switched off together.)

2. The solution can be drained off from membrane and tanks by opening the drainage valves DV1 when the experiment is completed.

3. Ensure that all valves are set according to the table below:

Open	Close
V1	DV1
V2	DV4
V4	V3
V5	V7

Table 3.4: Open and close valves for shut down procedure

4. Fill the tank with tap water (60 L)
5. Turn on the booster pump. Allow the system to run for 10 minutes.
6. Switch off the system by pressing "Booster pump off" button. (Both the high pressure and the booster pump will be switched off together.)

3.3.4 Turbidity Test

3.3.4.1 Background

Turbidity is a measurement of how cloudy water appears. Technically, it is a measure of how much light passes through water, and it is caused by suspended solid particles that scatter light. These particles may be microscopic plankton, stirred up sediment or organic materials, eroded soil, clay, silt, sand, industrial waste, or sewage. Bottom sediment may be stirred up by such actions as waves or currents, bottom-feeding fish, people swimming, or wading, or storm runoff.

Clear water may appear cleaner than turbid water, but it is not necessarily healthier. Water may be clear because it has too little dissolved oxygen, too much acidity or too many contaminants to support aquatic life.

3.3.4.2 Standard Measures of Turbidity

Turbidity test is measured using turbid meter. Here are the steps on measuring turbidity in the lab:

1. Three samples were taken; Coolant waste before demulsification, coolant waste after demulsification and sample after reverse osmosis.
2. Dilute 2 ml of coolant waste with distilled water.
   Coolant waste before demulsification: 75 ml of distilled water
   Coolant waste after demulsification: 25 ml of water.
3. Samples are poured into the turbid sample bottles and placed in the turbid meter for reading.
4. Reading observed are taken and calculated using following formula to get the exact turbidity:

$$\text{Dilution factor} = \frac{amount\ of\ sample + amount\ of\ diluted\ water}{amount\ of\ sample}$$

$$\text{Turbidity} = \text{reading from turbid meter} \times \text{dilution factor}$$

3.3.5 Total Organic Carbon Analysis

3.3.5.1 Introduction

Total organic carbon (TOC) analysis grew from the need to analyze wastewater and municipal water from organic matter. Measurement of TOC is a much more fast method to determine the organic matter content in water and wastewater, which is directly related to total organic content. TOC analysis is also widely used in monitoring the quality of process water in the semiconductor and pharmaceutical industries. Furthermore, TOC analysis in solids, such as soils and sediments is also important for soil science, etc.

There three main stages involving TOC analysis:

- Oxidizing organic carbon in a sample
- Detecting and quantifying the oxidized product
- Presenting the result in units of mass of carbon per volume of sample (liquids) or per mass of solid (solids)

3.3.5.2 Principles of TOC Analysis

Two types of carbon are present in water: total organic carbon (TOC) and inorganic carbon (IC). Organic carbon bonds with hydrogen or oxygen to form organic compounds. Collectively, the two forms of carbon are referred to as total carbon (TC) and the relationship between them is expressed as:

$$\text{TOC} = \text{TC} - \text{IC}$$

3.3.5.3 TOC Procedure

1. Three samples were taken; Coolant waste before demulsification, coolant waste after demulsification and sample after reverse osmosis.
2. Dilute 27 ml of coolant waste before demulsification and coolant waste after demulsification with distilled water.
   Coolant waste before demulsification: 1500 ml of distilled water
   Coolant waste after demulsification: 1500 ml of water.
3. 40 ml of samples are filtered using shrink filter.
4. Samples are put in TOC analyzer for measurement.
5. Results are taken and calculated using following formula to get the exact turbidity:

$$\text{Dilution factor} = \frac{amount\ of\ sample + amount\ of\ diluted\ water}{amount\ of\ sample}$$

TOC = reading from TOC analyzer x dilution factor

3.3.6 Chemical Oxygen Demand (COD)

3.3.6.1 Introduction

The chemical oxygen demand (COD) is the amount of oxygen consumed to completely chemically oxidize the organic water constituents to inorganic end products. COD is an important, rapidly measured variable for the approximate determination of the organic matter content of water samples. Some water samples may contain substances that are difficult to oxidize. In these cases, because of incomplete oxidation under the given test methods, COD values may be a poor measure of the theoretical oxygen demand. It should also be noted that the significance of the COD value depends on the composition of the water studied.

3.3.6.2 COD Procedure

1. Three samples were taken; Coolant waste before demulsification, coolant waste after demulsification and sample after reverse osmosis.
2. Dilute 27 ml of coolant waste before demulsification and coolant waste after demulsification with distilled water.

   Coolant waste before demulsification: 1500 ml of distilled water

   Coolant waste after demulsification: 1500 ml of water.
3. 40 ml of samples are filtered using shrink filter.
4. 2 ml of filtered samples are put in a small reactor for heating for 120 minutes at 150°C.
5. Samples are cooled and put in DR 5000 Spectrophotometer for measurement.
6. Results are taken and calculated using following formula to get the exact turbidity:

$$\text{Dilution factor} = \frac{amount\ of\ sample + amount\ of\ diluted\ water}{amount\ of\ sample}$$

COD = reading from DR 5000 Spectrophotometer x dilution factor

CHAPTER 4

RESULT AND DISCUSSIONS

4.1 Result

4.1.1 Demulsification process

Three pre-samples were made to see the effectiveness of the demulsifier, IMEC Meczym 579. 3 sets of 50 ml coolant are mixed with 20 ml of demulsifier and leaved for 4 days. Samples were observed on day 4 and oil droplets are accumulated on the coolant surface.

Samples	Sample 1	Sample 2	Sample 3
Amount of oil accumulated (ml)	10 ml	6 ml	5 ml

Table 4.1: Amount of oil collected for three pre-samples

As for bulk demulsifying test, results obtained from drum 1 and 2 are as follow:

	Drum 1	Drum 2
Amount of oil collected (ml)	75 ml	80 ml

Table 4.2: Amount of oil collected for 2 drums after mixing.

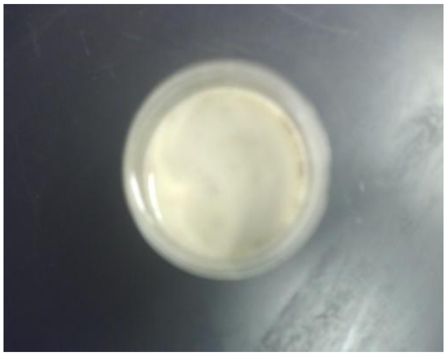


Figure 4.1: Before demulsification process

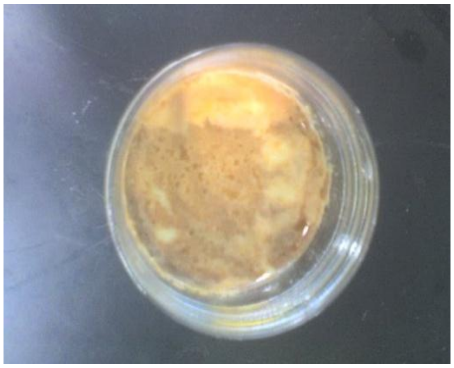


Figure 4.2: After demulsification process

i) Effect of Demulsifier Dosage on Demulsification

Dosage (ml)	Amount of separated oil (ml)
10	3
15	7
20	10
25	12
30	16

Table 4.3: Effect of Demulsifier Dosage on Demulsification

ii) Effect of Heating Temperature on Demulsification

Heating Temperature (°C)	Amount of separated oil (ml)
27	4
30	4
40	4
50	4
60	4
70	4

Table 4.4: Effect of Heating Temperature on Demulsification

iii) Effect of Time on Demulsification

Time (hour)	Amount of separated oil (ml)
2	1
4	5
6	6
8	6
10	9

Table 4.5: Effect of Time on Demulsification

4.1.2 Reverse Osmosis

Time (min)	Pressure (MPa)	TDS 1(ppm)	TDS 2 (ppm)	% rejection
5	3.72	42	24	42.9
10	3.70	40	22	45
15	3.72	40	21	47.5
20	3.72	36	19	47.2
25	3.89	34	18	47
30	3.75	30	18	40

Table 4.6: Permeate Flux, COD and Turbidity of effluent at 30 minutes time interval

4.1.3 Turbidity, TOC and COD tests

Samples	Before Demulsification Process	After Demulsification Process	After Reverse Osmosis Process
Turbidity (NTU)	25294.5	2902.5	0
TOC (ppm)	8910.05	2651.55	17.29
COD (ppm)	32487	24684	206

Table 4.7: Turbidity, TOC and COD tests

4.2 Findings

4.2.1 Demulsification Process

Based on the results obtained as in Table 4.1 and Table 4.2, the amount of oil collected is very minimal compare to the total of coolant waste in each drum which is 5 liters. This is because the major component of coolant waste is water whilst coolant is a minor component. The amount of coolant oil mixed with water is only 5% whilst water is 95%, thus, the amount of oil collected is less.

According to the experimental results obtained from Section 4.1.1, the demulsification for 10 liters of coolant waste was employed with 6 liters of demulsifier (degreaser) at room temperature (27°C) for about 10 hour in order to achieve maximum separation of 1.45 liters oil.

4.2.2 Effect of Demulsifier Dosage on Demulsification Process

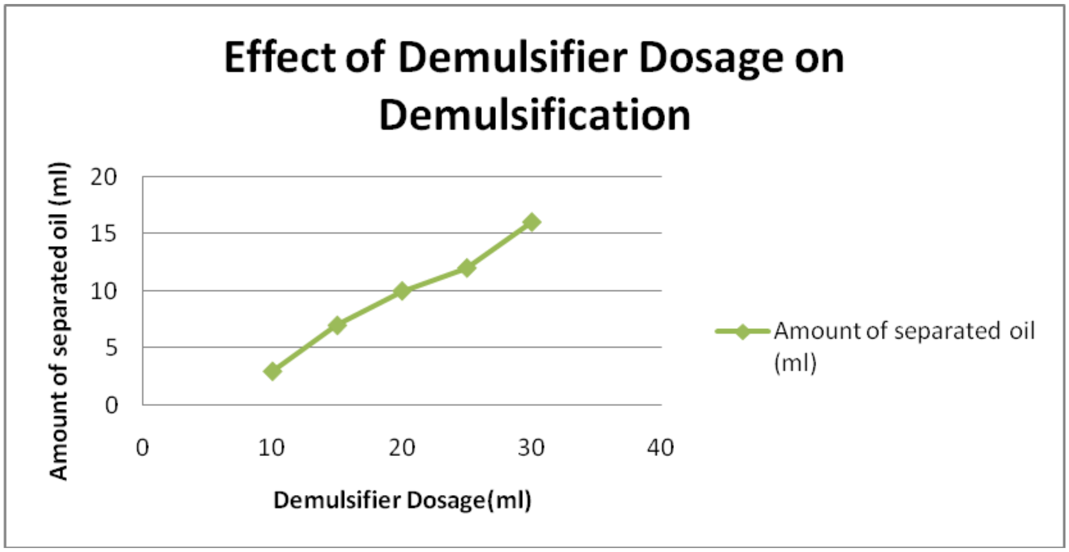


Figure 4.3: Effect of Demulsifier Dosage on Demulsification (T= 27°C, t= 3 days)

Figure 4.3 demonstrates the effect of demulsifier dosage on the volume of oil separated (upper layer). Without stirring, the effect of demulsifier dosage on demulsification is evident at room temperature (27°C) for 3 days. The time is set for 3 days for maximum volume of oil to accumulate. It is noticed that the maximum volume of separated oil is achieved at 30 ml of demulsifier dosage. Hence 30ml of demulsifier can be regarded as a good choice for present demulsification which is to demulsify 50 ml of coolant waste. This is due presumably to the fact that surface charge density of oil emulsion is related with the dosage of demulsifier which has opposite charge to the oil emulsion (Hongzhong Zhang, Shaoming Fang, Changming Ye, Minghua Wang, Haijun Cheng, Hui Wen, Xianglan Meng, 2008).Hence, as the volume of demulsifier used increase, the volume of oil collected also increase.

4.2.3 Effect of Heating Time on Demulsification Process

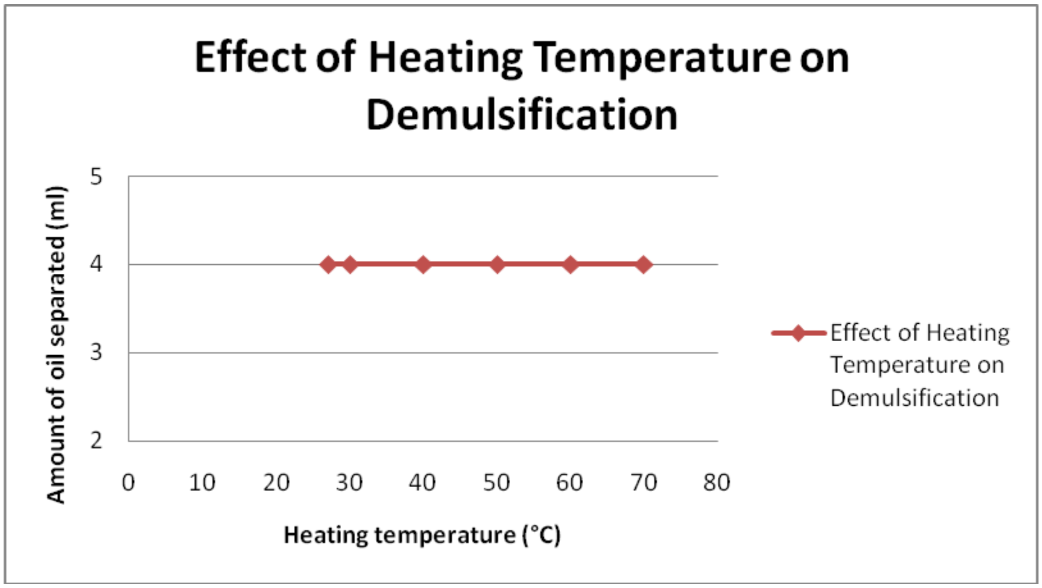


Figure 4.4: Effect of heating temperature on Demulsification

The result shows that the demulsification process is independent of heating temperature. At the beginning, the sample is left for a day for the oil to be cumulated without heating and a total of 4 ml of oil is accumulated. The sample is then put in water bath to observe the effect of heating to demulsification process. The sample is tested for temperature of 30°C, 40° C, 50°C,60°C and 70°C. From the graph, we can see that the heating temperature did not affect the demulsification process. This is due to the type of demulsifier use, which is degreaser (MECZYM 579) contains enzyme.

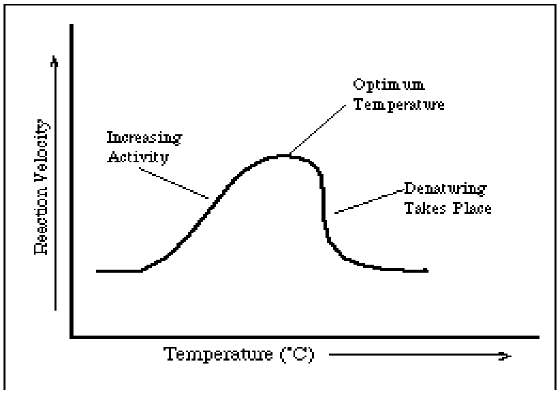


Figure 4.5: Effect of temperature on rate

As shown in Figure 4.5, the reaction rate increases with temperature to a maximum level, then abruptly declines with further increase of temperature. Enzyme in the degreaser has its optimum temperature at 27°C, and even it is subjected to higher temperature, the reaction rate will not increase. Hence, even the sample is heated at temperature above 27° C, no oil is accumulated. Thus, there is minimal reaction at higher temperature and can be assumed that high temperature will deactivated the enzyme. Over a period of time, enzymes will be deactivated at even moderate temperatures.

4.2.4 Effect of Time on Demulsification Process

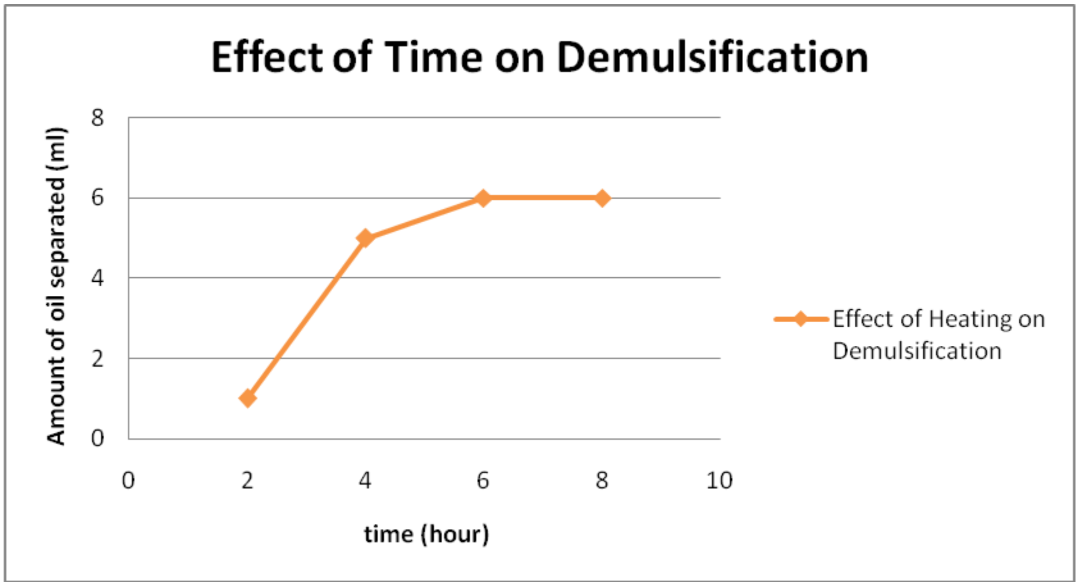


Figure 4.6: Effect of Time on Demulsification Process

The experiment dealing with the influence of time on demulsification was also performed at given demulsifier dosage and heating temperature. It is reasonable as shown in Figure 4.6, that effective demulsification can be achieved at 6 hour and longer. This is due to the enzyme in the demulsifier which require longer time to react. Since the sample is independent of heating temperature, the kinetic energy of the enzyme will not increase. Thus, the only method of increasing of the volume of oil separated is by increasing the time of the sample to be demulsified. As the time increase, it will be sufficient for the enzyme to react and accumulate the oil.

4.2.5 Reverse Osmosis

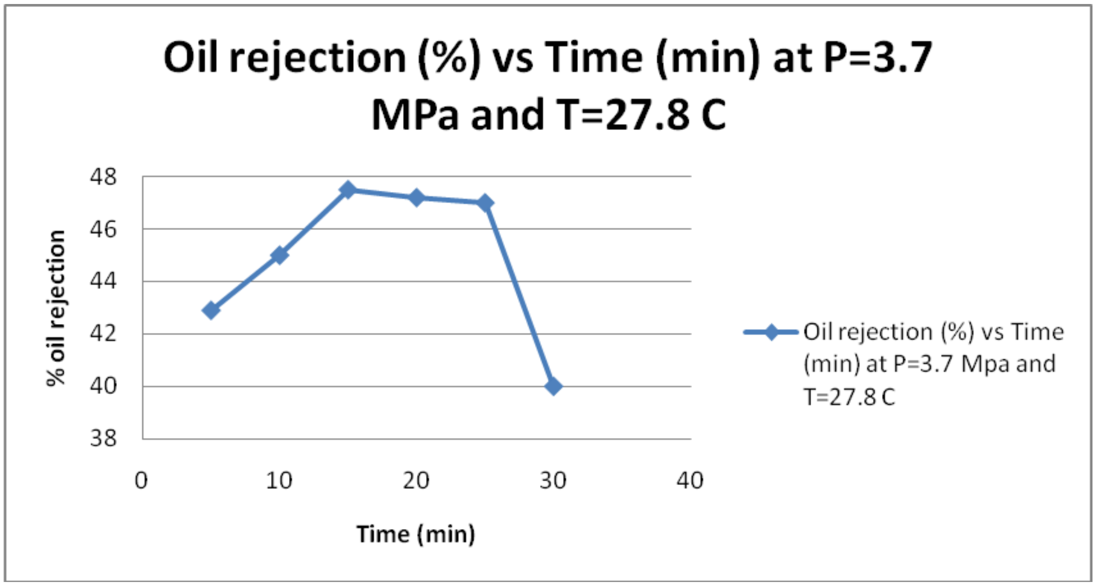


Figure 4.7: Graph of Oil rejection (%) vs Time (min) at P=3.7 MPa and T=27.8 °C

After demulsification, the under effluent water undergo the RO treatment. The batch process was operated for 30 minutes at temperature of 27.8 °C with driving pressure of 3.7 MPa. During the experiment, a small amount of foam could be produced along with the reverse osmosis process due to the mixture of concentrate line with the feed and high pressure.

From the graph, it can be observed that as the time increases, the percentage of oil rejection also decreases. This is because the membrane undergoes fouling after some time after the experiment was conducted. Membrane fouling is defined as the process in which solute or particles deposit onto the membrane surface or into membrane pores such that membrane performance is deteriorated. Membrane fouling can cause severe flux decline and affect the quality of the water produced. At the beginning of the experiment, the water molecule can freely passed through the membrane as there was less oil molecule charges on the surface of the membrane. As time passes, the membrane surface was covered with oil layer resulting in lower amount of water molecule that can pass through. The abrupt decline was observed because the main component of the effluent water is emulsion; a cake layer may build quickly on the membrane surface after some times.

The reason of declining of percentage of oil rejection is due to two parts: membrane pore blocking and concentration polarization (B.Chakrabarty, A.K. Ghoshal, M.K. Purkait, 2008). The pressure applied during this experiment is not constant through time. It fluctuated for after some time after the high pressure pump was on. Thus, the pressure is increasing and decreasing subsequently after the pump was on. It can be observed that the highest pressure reached is at 3.89 MPa at minute 25. The pressure affects the rate of flux decline. The higher the pressure, the greater the flux decline (B.Chakrabarty, A.K. Ghoshal, M.K. Purkait, 2008). The increase of flux declination with higher pressure may be connected with the build-up of the concentration polarization and membrane blocking (B.Chakrabarty, A.K. Ghoshal, M.K. Purkait, 2008). Increasing the feed pressure increases the number of collisions between the emulsion droplets, which in turn break the film between the oil and water causing the oil droplets to coalesce and form large droplets (B.Chakrabarty, A.K. Ghoshal, M.K. Purkait, 2008). As a result, a layer of oil is forming on the surface of the membrane which may be compressed on the surface at higher pressure leading to membrane fouling at a higher rate (B.Chakrabarty, A.K. Ghoshal, M.K. Purkait, 2008).

At the beginning stages of the experiment, most of the oil droplets contribute in jamming the membrane pore by sealing it causing the rejection of flux. Since the pore has been blocked, the amount of water that could pass through is lesser. As time passed, the pore blocking event is slowly stopped and the oil layer, which is formed by settling of oil droplets on membrane surface, begins to control the total membrane resistance. As pore blocking is a very quick process, flux declination also took place at a faster rate.

The accumulation of the oil layer on the membrane surface caused concentration polarization (CP). This phenomenon is happened as the concentration of solute becomes greater at the membrane, the opposing effect of diffusion become greater. The oil layer blocked the pore of the membrane thus preventing the water molecule into the solute and allowing less water molecule passing through. Since this is a batch process, the operation can not be stop immediately after observing lesser percentage of membrane rejection. The operation must be completed within the period, which is 30 minutes before a membrane cleaning process is performed. In this experiment, the membrane cleaning process is done by running tap water through the operation for a few times until a lower concentration of permeate is observed. Presumably, a higher percentage of oil rejection can be obtained if the membrane can be cleaned regularly.

4.2.6 TOC, COD and turbidity analysis

The values of the total organic carbon (TOC), chemical oxygen demand (COD) and turbidity are the important values in determining the success of the experiment. If the values of the three parameters are lower at the end of the experiment, thus it means that the method chosen for this experiment is a success and can be applied. Besides that, the objective of the experiment also has been stated that to obtain lower values of TOC, COD and turbidity.

The percentage of reduction of turbidity after demulsification process is 88.5%. This shows that demulsification process is a prominent step in reducing the volume of coolant oil since the effluent water is contaminated by it. The cloudiness of the effluent water is highly influence by the volume of coolant oil. The effluent water that has undergone demulsification has lower oil volume in it and mainly has more water compared to oil. Since water is the main component of the mixture for this case (after oil has been removed), the cloudiness of the effluent water is lesser compared to before demulsification process. 100% reduction of turbidity was obtained after RO process. This proves that RO process is successful in yielding clearer water that is safe for use. 0 NTU was obtained after the RO process meaning that there is no coolant oil in the effluent water.

For COD analysis, at the beginning, the value of effluent sample before demulsification is higher which 32487 ppm is. This shows that the original effluent water is highly contained with substances that are difficult to oxidize. These substances are mineral oils and fatty acids which are the ingredients of the coolant oil. 24% of reduction was achieved after demulsification process. The value shows that even after demulsification process, there were still substances that is difficult to oxidize left in the effluent water. This may be due to the separation of coolant oil form the effluent sample was not properly done, thus some coolant oil was left in the effluent water.

The samples after RO process was reduced to about 99.3% of the initial COD value of effluent water before demulsification. High percentage reduction of COD values was achieved after RO process because the small pore of the RO membrane has prevented the oil molecules from passing through and retained the oil at high concentration area. Thus, the samples that passed through the membrane have lower concentration of coolant oil in it and mainly distilled water. This samples was taken directly from the permeate line pipe to avoid any contamination of permeate samples from samples in permeate tank.

Besides COD and turbidity values, TOC value was also analyzed. TOC values after demulsification process is 2651.55 ppm which is 70.2% lower than samples before demulsification. This shows that after demulsification process, lesser volume of inorganic carbon and organic carbon in the effluent water after coolant oil has been removed. The inorganic carbons that could be in the effluent water are carbonate, bicarbonate and dissolves carbon dioxide (CO_2) and other matters that derived from non-living sources whereas the organic carbon are materials that can be derived from living sources. After RO process, 99.8% of reduction of TOC values was achieved. This shows that RO process has removed the organic and inorganic carbon in the effluent water sample. The organic and inorganic materials are too large in diameter thus preventing them from passing through the membrane. Besides, the result shows that lesser material derived from decaying vegetation, bacterial growth, and metabolic activities of living organisms or chemicals were in the effluent water. Original result of TOC values are shown in appendix III.

CHAPTER 5

RECOMMENDATIONS AND FUTURE WORKS

5.1 Recommendations

A few recommendations could be suggested in order to improve the results obtained from the experiment. The few recommendations are as follows:

Demulsifier is an important agent in determining the volume of coolant oil that can be collected. This is because the right type of coolant can result in higher volume of coolant oil collected. The type of coolant that is used in this experiment is IMEC Meczym 579 which is enzyme based demulsifier. This demulsifier is a general demulsifier for separating oil from effluent water and is not a specific demulsifier in removing coolant oil. The types of oil that could be separate using this type of demulsifier are cooking oil, food oil and others. Therefore, this oil is not effective in removing high strength and stable oil such as coolant oil. Besides that, IMEC Meczym is enzyme based coolant which has optimum temperature and time dependent. It requires longer time to react and has no effect if varies with temperature. Hence it is suggested that a specific coolant oil demulsifier is used in order to obtain higher volume of oil collected.

Secondly, a better container for demulsification process could be use in order to achieve effective separation. The type of container that is used for this demulsification process does not have an outlet at the bottom, so, the oil cumulated at the top is removed by using a spatula and syringe. This technique may not be efficient because it may cause disturbance to the oil surface. Some of the oil may have been stirred during this process resulting in the mixture of coolant oil with effluent water. The coolant oil also may have dropped to the bottom of the container. Hence it is suggested to use a container with a valve at the bottom so that the bottom effluent water could be discharge slowly into another container by opening the valve slowly so that the oil surface at the top will not be disturbed.

Thirdly, a variation of applied pressure during experiment could be conducted to see the effect of pressure to the percentage of salt rejection. For this experiment, we only applied a single pressure for the reverse osmosis process. Therefore, an effective pressure for the reverse osmosis to perform could not be identified. If the effective pressure could be determined, an effective reverse osmosis process could be performed thus yielding in better results.

Lastly, for RO membrane cleaning process, a cleaning chemical could be use to ensure that no oil is cumulated on the membrane surface. During the membrane cleaning process, tap water is run through the system to ensure that oil has been removed from the membrane. A few runs have to be conducted in order to obtain lower concentration of permeate and feed. Thus, by using a cleaning chemical, minimal runs of cleaning process could be conducted and time saving.

For turbidity test, the amount of distilled water used for dilution should be the same for sample of effluent water before and after demulsification. This is to ensure that the dilution factor is the same and the result is more reliable.

5.2 Future works

Below are the future works that could be conducted for this process:

- Conducted experiments on the discharge parameters of effluent water to analyze if the parameters meet the Department of Environment (DOE) requirement.
- Conduct a cost analysis of implementing the method in a larger scale (plant scale).
- A study on the effect of demulsifier on Chemical Oxygen Demand (COD) should be conducted to ensure that it does not contribute in increasing COD values.

CHAPTER 6

CONCLUSION

Methods for removal of coolant from oily wastewater have been identified. The methods are to conduct experiment starting with demulsification by using demulsifier followed by reverse osmosis. Few affecting parameters such as effect of heating time, heating temperature and demulsifier dosage have been identified to investigate effect of the parameters on the process.

Based on the results obtained, the demulsification process is influence by the amount of demulsisifer dosage and time, whilst, it is independent of heating temperature. The demulsification process can be assumed successful if the amount of oil separated increased by increasing demulsifier dosage and time. Therefore, the first objective which is to separate coolant from effluent water and obtain lower concentration of coolant in the effluent water was achieved.

From the results obtained from reverse osmosis process, the concentration of permeate has been lower. This means that lower concentration of coolant oil in the effluent has been obtained. Based on the TOC, COD and turbidity results, lower values of the three parameters have been obtained after demulsification and reverse osmosis process have been conducted. Thus, the second and third objective of this project has been achieved. However, the COD values that obtained from the experiment is higher than the local's regulation which is 100 ppm. Another process should be followed after reverse osmosis in order to lower the COD values below the limit.

Therefore, it can be said that removal of coolant from effluent water by combined demulsification and reverse osmosis is an effective manner for this project.

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APPENDICES

Appendix I: Calculation of Percentage of Membrane Rejection

Time (min)	Pressure (MPa)	TDS 1(ppm)	TDS 2 (ppm)	% rejection
5	3.72	42	24	42.9
10	3.70	40	22	45
15	3.72	40	21	47.5
20	3.72	36	19	47.2
25	3.89	34	18	47
30	3.75	30	18	40

Percent rejection = $(1-\frac{Cp}{Cb})$ x100%

Percent rejection = $(1-\frac{24}{42})$ x 100%

= 42.9%

Appendix II: Example calculation of TOC, COD and turbidity values

Samples	Before Demulsification Process	After Demulsification Process	After Reverse Osmosis Process
Turbidity (NTU)	25294.5	2902.5	0
TOC (ppm)	8910.05	2651.55	17.29
COD (ppm)	32487	24684	206

a) Turbidity

Example of turbidity calculation for before demulsification sample:

Dilution factor = $\frac{amount\ of\ sample + amount\ of\ diluted\ water}{amount\ of\ sample}$

Dilution factor = $\frac{2\ ml + 75\ ml}{2\ ml}$

= 38.5 NTU

Turbidity = reading from turbid meter x dilution factor

= 657 NTU *38.5

= 25294.5 NTU

Example of turbidity calculation for after demulsification sample:

Dilution factor = $\frac{2\ ml + 25\ ml}{2\ ml}$

= 13.5 NTU

Turbidity = reading from turbid meter x dilution factor

= 215 NTU *13.5

= 2902.5 NTU

Percent reduction after demulsifcation = $\frac{25294.5 - 2902.5}{25294.5}$ x 100% = 88.5%

Percent reduction after RO = $\frac{25294.5 - 0}{25294.5}$ x 100% = 100%

b) TOC

Example of TOC calculation for before demulsification sample:

Dilution factor = $\frac{amount\ of\ sample + amount\ of\ diluted\ water}{amount\ of\ sample}$

Dilution factor = $\frac{27\ ml + 1500\ ml}{27\ ml}$

= 56.5 ppm

TOC = reading from TOC analyzer x dilution factor

= 157.7 ppm *56.5

= 8910.05 ppm

Example of TOC for after demulsification sample:

Dilution factor = $\frac{27\ ml + 1500\ ml}{27\ ml}$

= 56.5 ppm

TOC = reading from turbid meter x dilution factor

= 46.93 ppm *56.5

= 2651.55 ppm.

Percent reduction after demulsifcation = $\frac{8910.05 - 2651.55}{8910.05}$ x 100% = 70.2%

Percent reduction after RO = $\frac{8910.05 - 17.29}{8910.05}$ x 100% = 99.8%

c) COD

Example of COD calculation for before demulsification sample:

Dilution factor = $\frac{amount\ of\ sample + amount\ of\ diluted\ water}{amount\ of\ sample}$

Dilution factor = $\frac{30\ ml + 1500\ ml}{30\ ml}$

= 51 ppm

COD = reading from COD analyzer x dilution factor

= 637 ppm *51

= 32487 ppm

Example of COD for after demulsification sample:

Dilution factor = $\frac{30\ ml+1500\ ml}{30\ ml}$

= 51 ppm

COD = reading from turbid meter x dilution factor

= 484 ppm *51

= 24684 ppm.

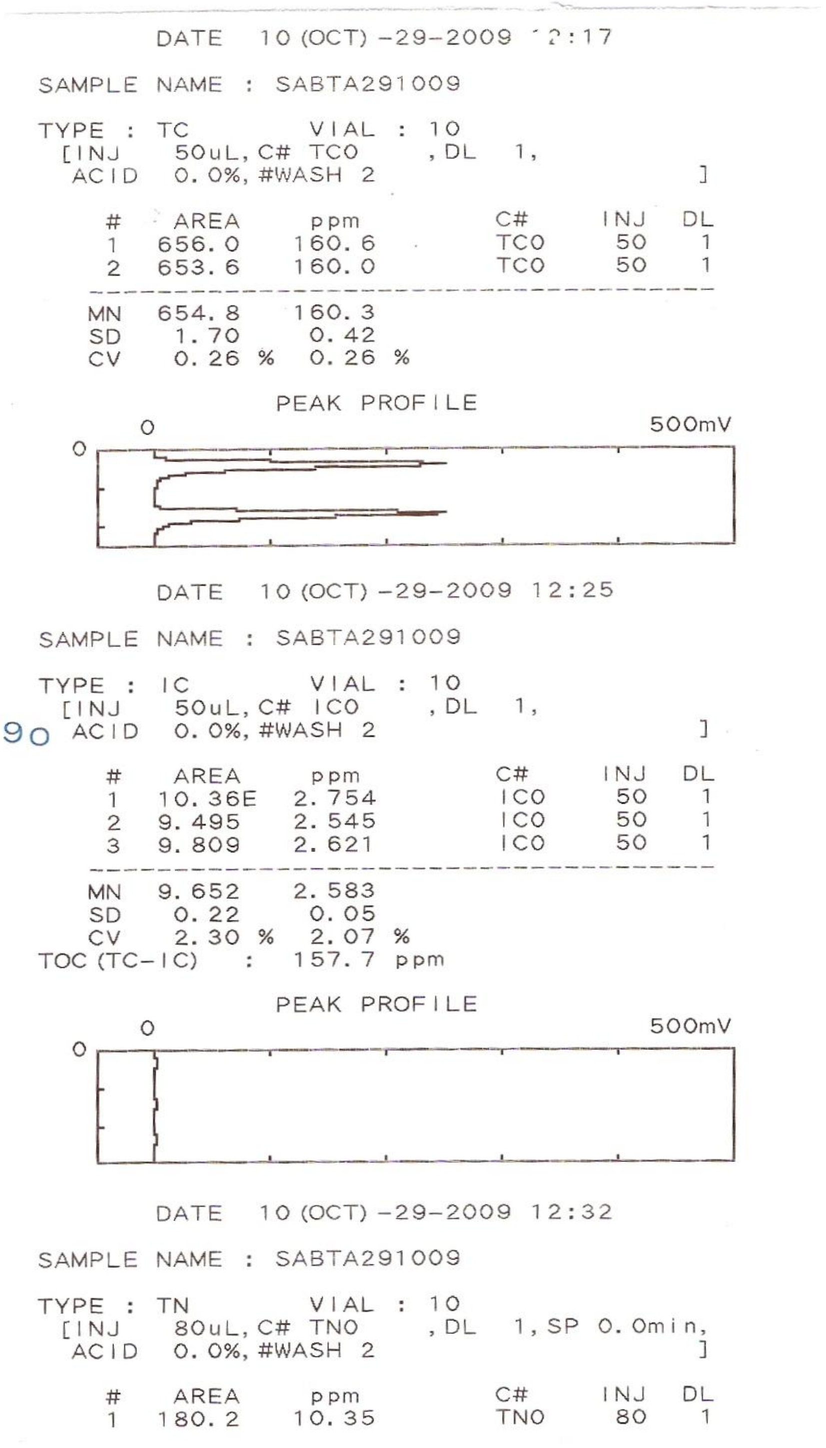
Percent reduction after demulsifcation = $\frac{32487-24684}{32487}$ x 100% = 24%

Percent reduction after RO = $\frac{32487-206}{32487}$ x 100% = 99.3%

Environmental Quality (Sewage And Industrial Effluents) Regulations 1978

NO	PARAMETERS	UNIT	VALUE
1	Temperature	^{o}C	40
2	pH	-	5.5 – 9.0
3	BOD at 20^{0}c	mg/l	50
4	COD	mg/l	100
5	Suspended Solid	mg/l	100
6	Oil & grease	mg/l	100

Appendix III: Original results from TOC Analyzer



```
         DATE   10(OCT)-29-2009 12:17

SAMPLE NAME : SABTA291009

TYPE : TC         VIAL : 10
 [INJ   50uL,C# TCO     ,DL   1,
  ACID  0.0%,#WASH 2                    ]

    #    AREA      ppm          C#     INJ   DL
    1   656.0     160.6         TCO     50    1
    2   653.6     160.0         TCO     50    1
   -------------------------------------------
   MN   654.8     160.3
   SD     1.70      0.42
   CV     0.26 %    0.26 %

              PEAK PROFILE
      0                                   500mV
  0

         DATE   10(OCT)-29-2009 12:25

SAMPLE NAME : SABTA291009

TYPE : IC         VIAL : 10
 [INJ   50uL,C# ICO     ,DL   1,
90 ACID  0.0%,#WASH 2                   ]

    #    AREA      ppm          C#     INJ   DL
    1   10.36E    2.754         ICO     50    1
    2   9.495     2.545         ICO     50    1
    3   9.809     2.621         ICO     50    1
   -------------------------------------------
   MN   9.652     2.583
   SD     0.22      0.05
   CV     2.30 %    2.07 %
TOC(TC-IC)   :    157.7 ppm

              PEAK PROFILE
      0                                   500mV
  0

         DATE   10(OCT)-29-2009 12:32

SAMPLE NAME : SABTA291009

TYPE : TN         VIAL : 10
 [INJ   80uL,C# TNO     ,DL   1,SP 0.0min,
  ACID  0.0%,#WASH 2                    ]

    #    AREA      ppm          C#     INJ   DL
    1   180.2     10.35         TNO     80    1
```

Figure 1: TOC value for sample of effluent water before demulsification

```
              PEAK PROFILE
```

```
       DATE   10 (OCT) -29-2009 12:36

SAMPLE NAME  :  SABTA291009

TYPE  :  TC          VIAL  :  11
 [INJ    50uL, C#  TCO      , DL   1,
  ACID   0. 0%, #WASH  2                    ]

    #    AREA      ppm          C#     INJ   DL
    1   200. 7    48. 89        TCO     50    1
    2   199. 2    48. 52        TCO     50    1
   ------------------------------------------------
   MN   200. 0    48. 71
   SD     1. 06     0. 26
   CV     0. 53 %   0. 53 %
              PEAK PROFILE
```

```
       DATE   10 (OCT) -29-2009 12:44

SAMPLE NAME  :  SABTA291009

TYPE  :  IC          VIAL  :  11
 [INJ    50uL, C#  ICO      , DL   1,
  ACID   0. 0%, #WASH  2                    ]

    #    AREA      ppm          C#     INJ   DL
    1   6. 340    1. 784        ICO     50    1
    2   6. 255    1. 763        ICO     50    1
   ------------------------------------------------
   MN   6. 298    1. 774
   SD     0. 06     0. 01
   CV     0. 95 %   0. 82 %
TOC (TC-IC)    :  46. 93 ppm
              PEAK PROFILE
```

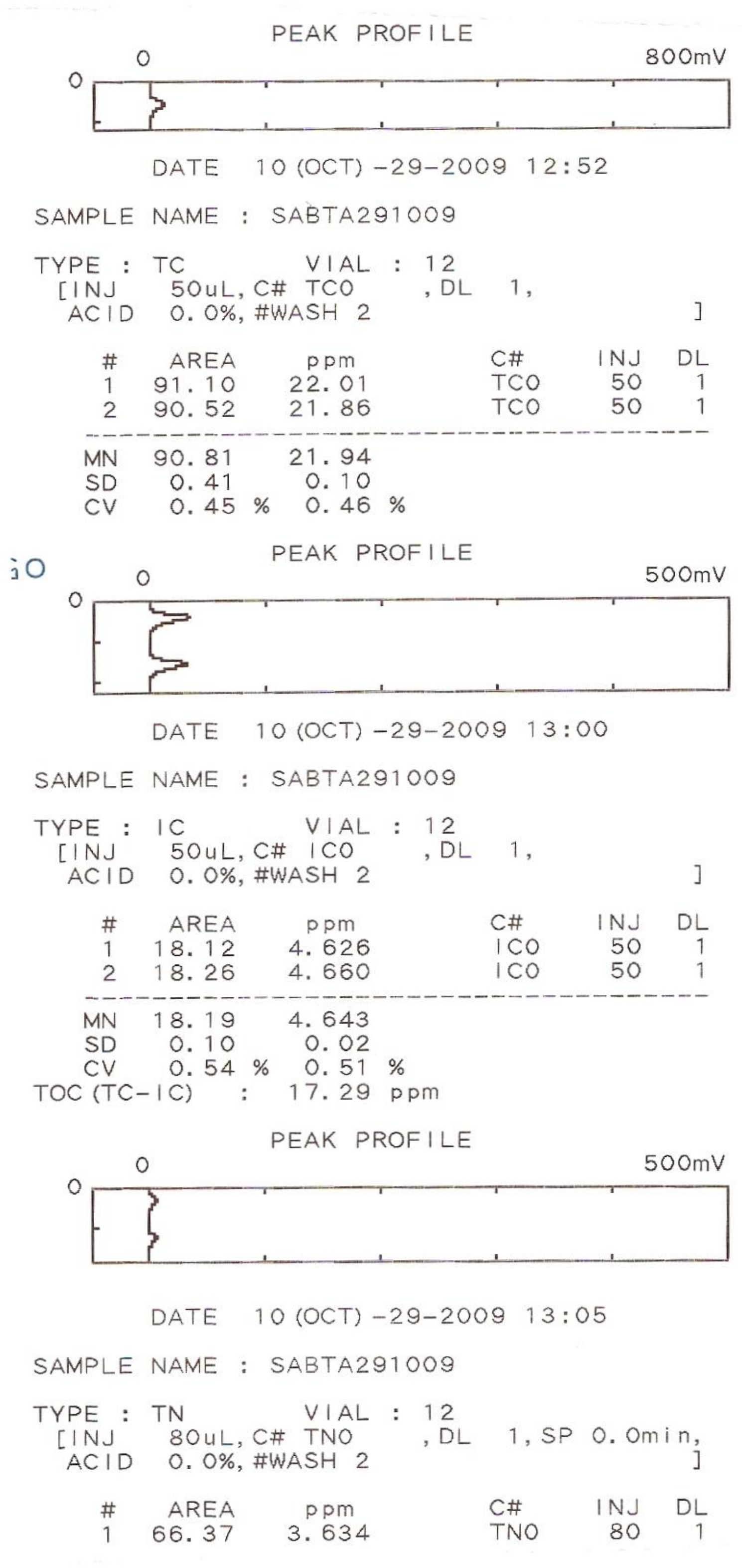
```
       DATE   10 (OCT) -29-2009 12:49

SAMPLE NAME  :  SABTA291009

TYPE  :  TN          VIAL  :  11
 [INJ    80uL, C#  TN0      , DL   1, SP  0. 0min,
  ACID   0. 0%, #WASH  2                    ]

    #    AREA      ppm          C#     INJ   DL
    1   [illegible] 86    3. 309        TN0     80    1
```

Figure 2: TOC value for sample of effluent water after demulsification



```
                      PEAK  PROFILE
        0                                        800mV
   0
              DATE    10 (OCT) -29-2009  12:52

SAMPLE  NAME  :  SABTA291009

TYPE  :  TC          VIAL  :  12
 [INJ    50uL, C#  TC0      , DL    1,
  ACID   0. 0%, #WASH  2                           ]

     #    AREA      ppm           C#       INJ   DL
     1   91. 10    22. 01         TC0       50    1
     2   90. 52    21. 86         TC0       50    1
    ---------------------------------------------------
    MN   90. 81    21. 94
    SD    0. 41     0. 10
    CV    0. 45  %   0. 46  %

                      PEAK  PROFILE
        0                                        500mV
   0
              DATE    10 (OCT) -29-2009  13:00

SAMPLE  NAME  :  SABTA291009

TYPE  :  IC          VIAL  :  12
 [INJ    50uL, C#  IC0      , DL    1,
  ACID   0. 0%, #WASH  2                           ]

     #    AREA      ppm           C#       INJ   DL
     1   18. 12    4. 626         IC0       50    1
     2   18. 26    4. 660         IC0       50    1
    ---------------------------------------------------
    MN   18. 19    4. 643
    SD    0. 10     0. 02
    CV    0. 54  %   0. 51  %
TOC (TC-IC)     :   17. 29  ppm
                      PEAK  PROFILE
        0                                        500mV
   0
              DATE    10 (OCT) -29-2009  13:05

SAMPLE  NAME  :  SABTA291009

TYPE  :  TN          VIAL  :  12
 [INJ    80uL, C#  TN0      , DL    1, SP  0. 0min,
  ACID   0. 0%, #WASH  2                           ]

     #    AREA      ppm           C#       INJ   DL
     1   66. 37    3. 634         TN0       80    1
```

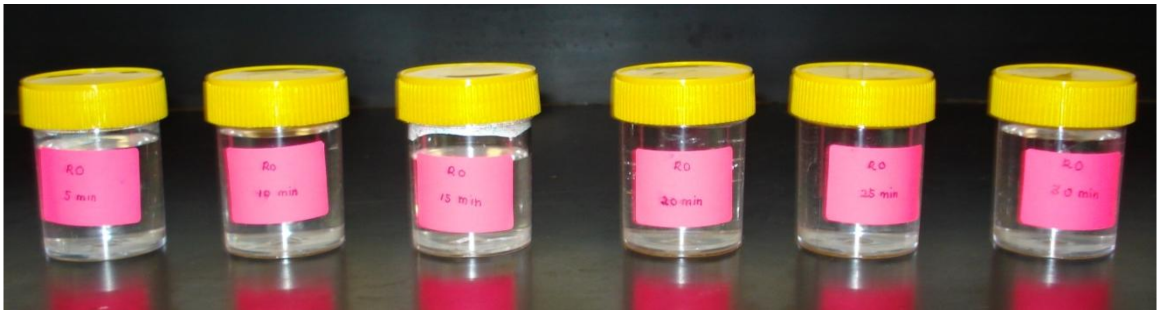
Figure 3: TOC value for sample of effluent water after RO process

Appendix IV: Effluent water samples after demulsification and after RO process

Figure 4: Samples of effluent water after demulsification

Figure 5: Oil accumulated on the effluent water surface

Figure 6: Effluent sample after RO process



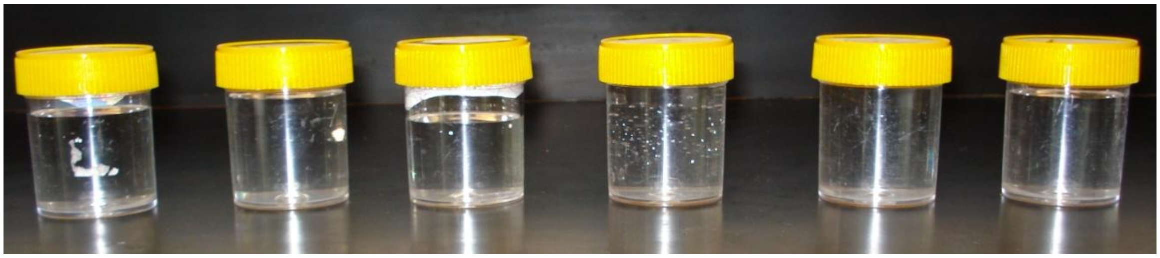


Figure 7: Effluent samples taken for every 5 minutes intervals for 30 minutes after RO process

Figure 8: TOC Analyzer

Appendix V: RO Schematic diagram

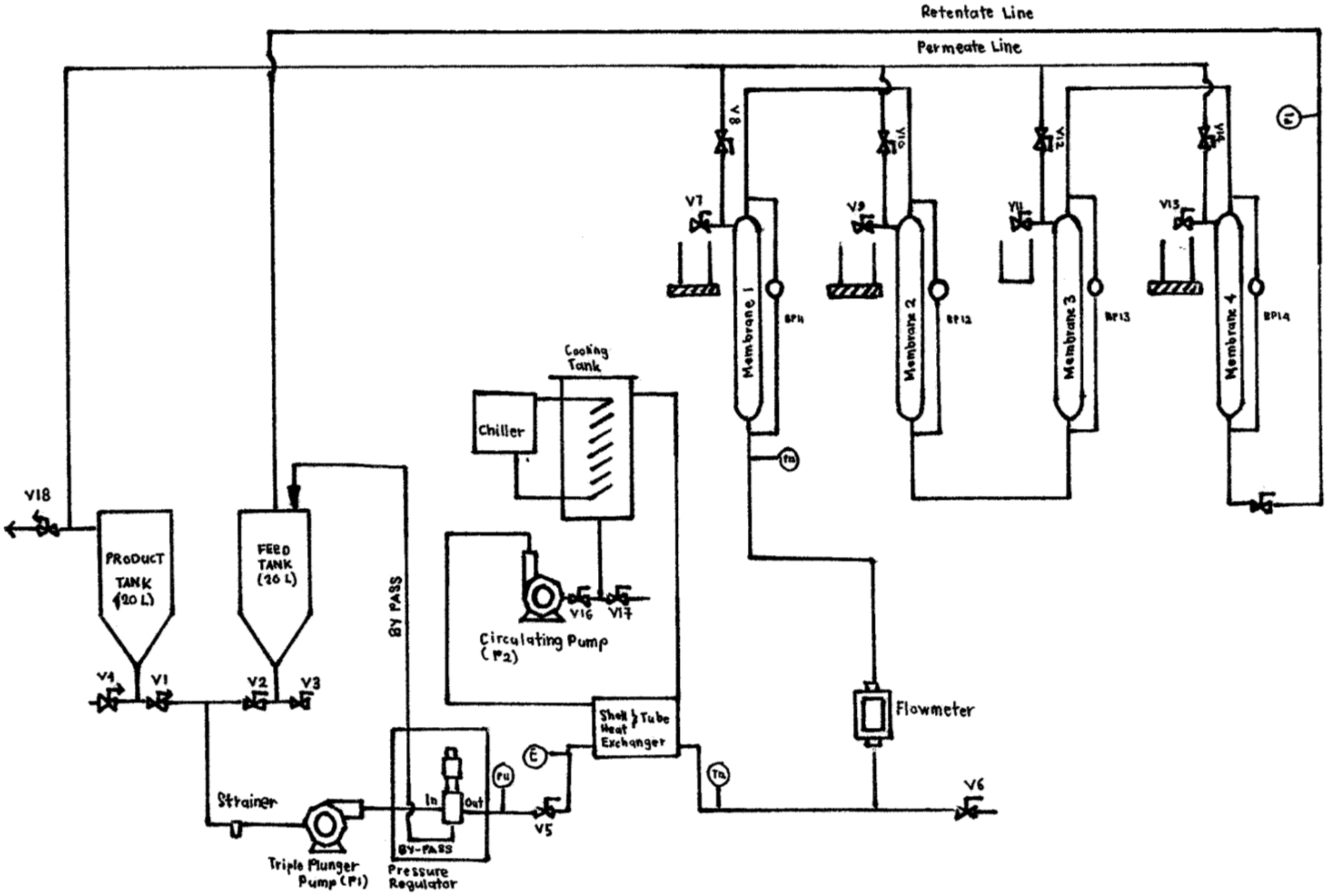


Figure 9: Process Schematic Diagram of RO Test Unit