

CERTIFICATION OF APPROVAL

Amine Removal from Water

by

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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.



AMIZA SURMI

ABSTRACT

CO₂ removal using amines is well understood because of it has been widely used for acid gas removal. One way to reduce the cost of amine is by recovered and recirculated back into the process stream. Few methods have been developed to optimize the recovery of the amine. The used of membrane to remove amine from the effluent water is still devoid. Therefore, this unprecedented study will highlight the feasibility of using membrane process to separate amine from effluent water prior to discharge.

It is found that the reverse osmosis method gives better performance and more reliable results than the ultra filtration membrane for amine removal from water, with salt rejection more than 90%. This is due to the smaller pore size of the RO membrane, which is less than 1nm to 10nm while the UF membrane can only reject contaminants no smaller than 0.01 μm with 10nm to 100 nm pore size.

Other factors affecting the membrane performance are feedwater pressure, concentration, temperature, pH, concentration polarization, and the membrane recovery. However, in this project, the parameter that had been studied in this experiment is the effect of the feed pressure and the feed concentration. From the result for the separation of amines from water, reverse osmosis give better performance in increasing the pressure as well as the feed concentration in order to get more amines at the rententate side.

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ABBREVIATIONS

MEA	Monoethanolamine
DGA	Diglycolamine
DEA	Diethanolamine
DIPA	Disopropanolamine
MDEA	Methyl Diethanolamine
TEA	Triethylamine
RO	Reverse Osmosis
UF	Ultrafiltration
COD	Chemical Oxygen Demand

NOMENCLATURES

Terminology

Permeate	the substance that passes through the membrane
Reject	the substance that is retained by the membrane

Symbols

J	Flux: volumetric flow of material through the membrane per unit area ($\text{m}^3/\text{m}^2\text{s}$)
P	Pressure (Pa)
ΔP	Trans-membrane pressure (TMP)
R_m	Membrane résistance
N_w	solvent (water) flux ($\text{kg}/\text{m}^2\text{s}$)
P_w	solvent membrane permeability ($\text{kg solvent}/\text{s.m.atm}$)
Lm	membrane thickness (m)
ΔP	$P_1 - P_2$ (hydrostatic pressure difference with P_1 pressure exerted on feed and P_2 on product solution), (atm)
$\Delta\pi$	$\pi_1 - \pi_2$ (osmotic pressure of feed solution – osmotic pressure of product solution), (atm)
n	number of kg mol of solute
V_m	volume of pure solvent (m^3)
R	gas law constant
T	operating temperature, (K)
J_i	membrane flux
k_{ov}	overall mass transfer coefficient
k_m	mass transfer coefficient of the membrane
k_{bt}	mass transfer coefficient of the fluid boundary layer
c_{ib}	concentration of component i in bulk feed solution
c_{ip}	concentration of component i in bulk permeate solution

CHAPTER 1

INTRODUCTION

1.1 Background of study

Solutions of alkanamines are an industry important class of compounds used in the natural gas, petroleum chemical plants, and ammonia industries for the removal of carbon dioxide (CO₂) and hydrogen sulfide (H₂S) from the gas streams. A wide variety of alkanamines such as Monoethanolamine (MEA), diethanolamine (DEA) diisopropanolamine (DIPA), N-methyldiethanolamine (MDEA) have been used industrially for a number of years(Kohl & Riesenfeld,1985). These processes use a solvent either an alkanamine or an alkali-salt (hot carbonate processes) in an aqueous solution, which reacts with the acid gas (H₂S and CO₂) to form complex or bond. This complex is subsequently reversed in the regenerator at elevated temperature and reduced acid gas partial pressure releasing the acid gas and regenerating the solvent for reuse. [22]

The alkanamines are classified by the degree of substitution denoting primary amine, a double substitution, a secondary amine and triple substitution, a tertiary amine. Each of the alkanamines has at least one of hydroxyl group and one amino group. In general, the hydroxyl group serves to reduce the vapor pressure and increase water solubility, while the amino group provides the necessary alkalinity in water solutions to promote the reaction with acid gases. It is readily apparent looking at the molecular structure that the non-fully substituted alkanamines have hydrogen atoms at the non-substituted valent sites on the central nitrogen. This structural characteristic plays an important role in the acid gas removal capabilities of the various treating solvents.

A sour gas containing H₂S and/or CO₂ is introduced at the bottom of a high-pressure absorber where it rises and counter currently contacts an aqueous alkanamine solution that is introduced at the top of the absorber. The CO₂-rich amine solution that results is then introduced at the top of a stripper where it countercurrent contacts steam at an

elevated temperature and reduced pressure. The steam strips the CO₂ and H₂S from the solution and the lean alkanoamine solution is pumped through the heat exchanger where it is cooled and reintroduced at the top of absorber.

For example, in the Petronas Fertilizer Kedah (PFK) Sdn. Bhd where the only Petronas' company in Peninsular Malaysia that well known with the urea and ammonia production, also using CO₂ Removal System. The system is known as Benfield System. The main objective in the system is to remove CO₂ from natural gas by using diethanolamine (DEA). DEA act as an activator for the absorption and contains about 3wt percentage of DEA, which will increases the mass transfer rate of CO₂ from gas phase to the liquid phase. DEA will also decrease the CO₂ vapor pressure. The absorption of CO₂ from natural gas take places in the CO₂ Absorber, meanwhile the CO₂ will be extracted from the solution in the regenerator. After that, the lean solution will be recycled to the absorber for the absorption process again. ^[1]*(Refer appendix A)*

However, amine carry over is commonly discussed problem in gas plant utilizing amine as a medium to obliterate acid gases from the incoming gas stream. Amine carry over will potentially end up in the effluent water thus, if improperly discharge will pollute the environment. Mixture of water with high amine concentration will resort to high COD value, which is a direct measurement of organic contaminants level. As mandated by laws, waste water to environment need to have a COD value of <100mg/L, hence leave most of the gas treating plants with no option but to look for ways to alleviate this potential problem.^[8]

The carry over of amine into the effluent system of liquefied natural gas (LNG) plant is a concerned matter. Despite other sources such as presence of salts and organic compounds, amine is also known to be one of the main contributors to increase the chemical oxygen demand (COD) of the effluent. Due to its ability to increase COD level of the effluent, separation of amine from the discharge water is must be done task. Gas treating plants around the world have installed various conventional methods to mitigate

this potential problem. The use of membrane to remove amine from the effluent water is still devoid. The amine from the effluent would be separated and concentrated to the highest level possible so that amount of used amine accrued could be reused as a top up to the existing inventory. The study of using membrane to separate amine compound from water is still new and unprecedented. This is due to the tenacious nature of the mixture of amine and water. This make the intricate separation process is a formidable challenge.

1.2 Problem Statement

CO₂ removal using amines is well understood because of it has been widely used for acid gas removal. One way to reduce the cost of amine is by recovered and recirculated back into the process stream. Few methods have been developed to optimize the recovery of the amine. Membranes are rated for suitability as an application filter based on the criteria such as pore size and morphology, hydrophilicity, chlorine resistance, chemical resistance, pH range tolerance, temperature and pressure tolerance, permeability, stability of the pore structure, clean ability, fouling resistance and consistency and quality of the membrane.

Nowadays, apart from other separation industries, membrane process is widely used in water purification industry or waste water treatment plant to obviate suspended and dissolved solids, heavy metals and other kind of impurities from the water stream. The used of membrane to remove amine from the effluent water is still devoid. Therefore, this unprecedented study will highlight the feasibility of using membrane process to separate amine from effluent water prior to discharge. The objective is rather simple to reduce the COD value of the effluent to meet the target set by the Department of Environment (DOE) of Malaysia, which is <100mg/L. ^[8]

1.3 Objective and Scope of study

The main objectives of the project are:

- 1.3.1 To demonstrate the separation of amines through two different type of membrane system which are Reverse Osmosis (RO) for high pressure driven and Ultrafiltration (UF) for low pressure driven.
- 1.3.2 To study and investigate the effects of different operating parameters like pressure and concentration on the separation process by using membrane system.
- 1.3.3 To investigate the effect of amine concentration on the rate of filtration
- 1.3.4 To compare the finding of the research with the other literatures.

The scope of the study will be limited for:

- 1.3.5 Conducting literatures review on the usage of the different type of membranes in the separation mixture of amines and water.
- 1.3.6 Conducting the experiments for different parameters that affect the separation processes like pressures and concentrations of amines passing through the different types of membrane.
- 1.3.7 Since this project will be in the form of laboratory experiments and data analysis, student is to explore research problems and build research objectives, apply appropriate methodology, analyze the outcomes and report the findings

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1 Overview of Membrane

Membranology has developed progressively during the last two decades, and has been applied in various process such as reverse osmosis of desalination and water purification, electrodialysis in a chlorine-caustic cell, Ultrafiltration, pervaporation, gas separation, hemodialysis, controlled released of drugs, genetic engineering and others. Many kinds of membrane are known, differing in structure and function. A comprehensive representation of the relationship between pore diameter, membrane separation process, and penetrant size as shown figure below. it is possible to classify membranes according to their structure^[4]

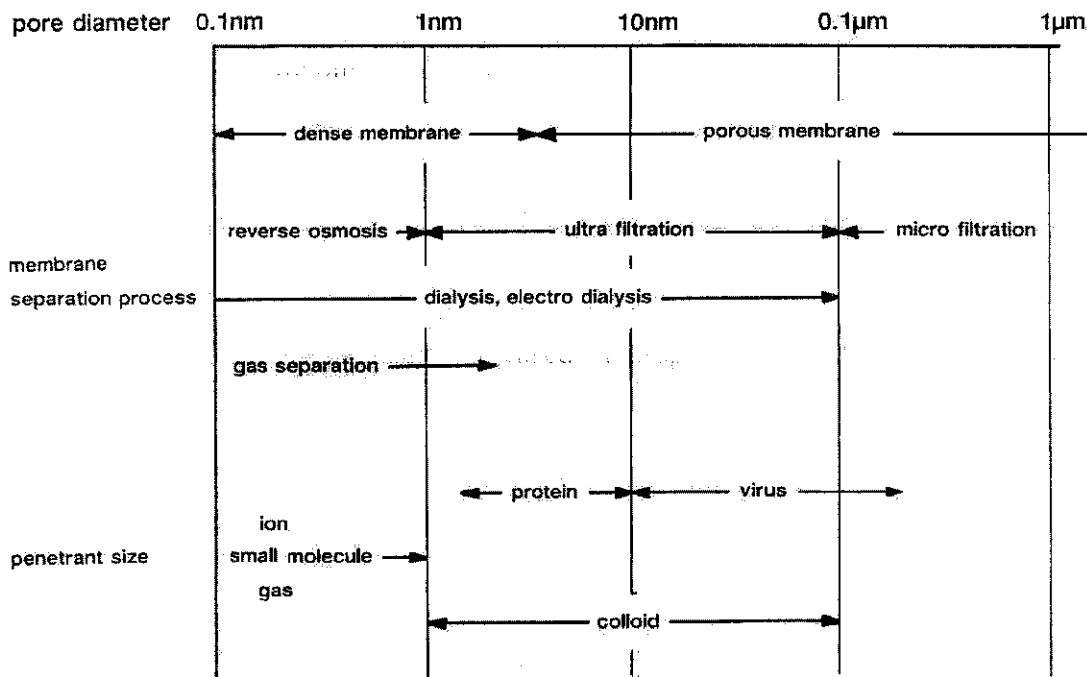
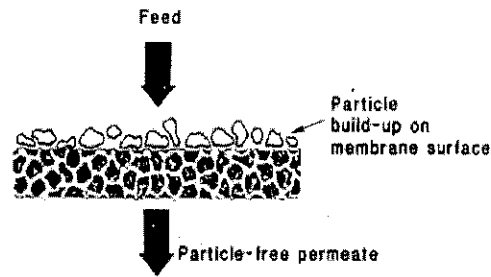


Figure 2.1.1 Application of membrane for different pore size

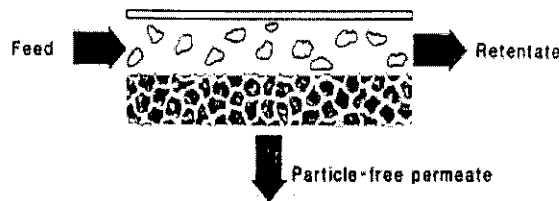
There are two types of pressure driven filtration, dead-end and cross flow. In dead-end filtration, the feed solvent to the system passes through the membrane, which is the only exit from the filtration chamber. A cake-retained material builds up on the surface of the membrane, which restricts further flow. Cross flow, filtration causes the retained liquid to be circulated across the membrane surface.

Dead-end filtration



a) Dead-end filtration

Cross-flow filtration



b) Crossflow filtration

Figure 2.1.2 Schematic representation of a) Dead-end and b) cross flow operation

Membrane is a type of separation process utilizes a permeable thin pliable layer acting as boundary, lining, or a partition to separate impurities from streams. The streams can be either gas or liquid. In membrane process, the application of shear-enhanced filtration is technique used to separate the impurities according to their molecular weight and size. Application of pressure within the system causes the membrane act like a sieve. As its function is tantamount to a sieve, membrane construction must engulf thousands of

pores within the surface area. Particle smaller than the size of the pore will pass through likewise, particle larger than pore size will be rejected as concentrate. [5][6]

The performance of membrane systems is determined by transport process. These influence the three independent stages which involve convective and diffusive flows on the feed-side of the membrane, permeation of materials through the membrane and transfer of material into the permeate stream. However, the first two factor need to give more consideration because the resistance associated with the transfer into the permeate stream is insignificant.

A membrane can be considered a permselective barrier between two phases. Figure 2.1.3 is a schematic representation of a semi-permeable membrane, which under the influence of an applied driving force preferentially passes component A. there is thus a convective flow of component A to and through membrane. Component B is also transported towards the membrane by the same convective flow. However, the concentration of component B in permeate is less than that of component B in the feed. Thus, initially component B accumulates on the feed-side of the membrane and its concentration on the face of the membrane increases above the bulk value. There is therefore concentration gradient for diffusive back flow into the bulk on the feed-side. At steady-state which is reached after a few seconds, the following equations represents the relevant fluxes. [2]

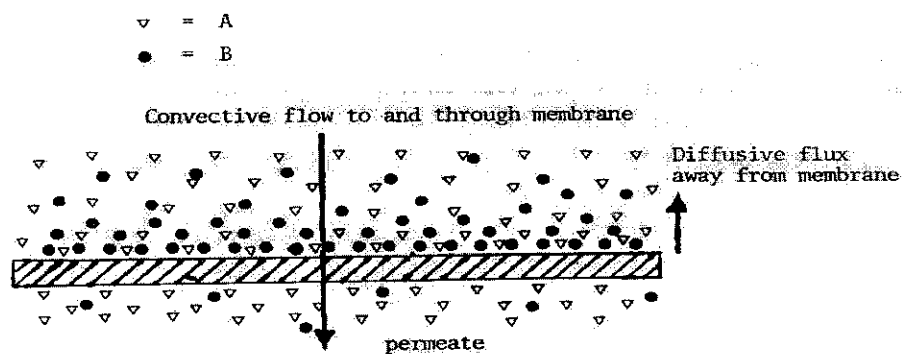


Figure 2.1.3 Convective and Diffusive flow perpendicular to membrane surface

Convective flux of A through Boundary layer to membrane = Convective flux of A through membrane **Equation 2.1.1**

Convective flux of A through Boundary layer to membrane = Convective flux of A through membrane + diffusive flux of B away from membrane **Equation 2.1.2**

The resultant concentration profile is illustrated in Figure 2.1.3 taking the concentration of a general point within the concentration boundary layer to be C, assuming density to be constant and equation 2.1.2 is applied to the element and the equation obtained is:

$$JC = JC_p - D \left(\frac{dC}{dy} \right) \quad \text{Equation 2.1.3}$$

- Where D = diffusion coefficient of the solute (m²/s)
- J = flux (m³/m².s)
- C_p = concentration of permeate (kg mol/m³)
- dC = difference in concentration (kg mol/m³)
- dy = difference in thickness (m)

2.2 Membrane material

MF, UF and RO are applied to separate or remove particles having diameters of from 10^0A to 10 micrometer by using the molecular sieving effect, which reject particles based on the pore radius of the membrane and size of particles. The differences between the process and membrane characteristics are summarized in Table 2.2.1. [8]

Table 2 2.1 Pressure Driven Separation Processes and Membrane Characteristics

Filtration	Material ^a	Structure	Pore size (A°)	Molecular weight cutoff (Da)	Operational pressure (kg/cm ²)
MF	Teflon,PVDF,PP, PE,PC,CA,porous glass	Skinless	10^3 - 10^5	Very high	0.5-1.5
UF	PSF,PES,PI,CA, PAN,PVA,PPS,porous glass	Asymmetric	10-1000	10^3 - 10^6	1.0-3.0
RO	Interfacially polymerized Plyamide,CA	Composite Asymmetric	1-10 (skin layer) 10-1000 (skin layer)	10-100	-

^aPVDF,poly(vinylidene fluoride);PP,polypropylene;pe,polyethylene;PC,polycarbonate; CA,cellulose acetate.PSF,polysulfone,PES,poly(ether polyimide,PAN,polyacrylonitrile,PVA,poly(vinyl alcohol);PPS,poly(phenylene sulfide)

Table 2.2 1Characteristic of RO and UF

	RO	UF
Type	Spiral Wound	Hollow Fiber
Membrane polymer	Composite Polyamide	Polysulfone
Max applied pressure	6.9 MPa (1000 psig)	100 psig
Max operating Temperature	45 ⁰ C (113 F)	75 ⁰ C
pH water range	3.0 – 10.0	3.0 – 10.0
Max feed flow	16 GPM (3.6m ³ /h)	N/A

2.3 Membrane module

There are various shapes and modules of membrane in industries. The basic types of membrane are hollow fiber, tubular, plate and frame, and spiral-wound as shows in Figure 2.3.1 and Figure 2.3.2. The membrane shapes and modules differ according to their application and quality demand of the separations. The spiral-wound membrane is used for RO and is constructed of one or more membrane envelopes wound around a perforated central tube. The permeate passes through the membrane into the envelope and spirals inward to the central tube for collection. Meanwhile, the hollow-fiber module resembles a shell and tube heat exchanger. The pressurized feed enters the shell side at one end and flowing over the fibers channels. Typically, the fibers are sealed at one end and embedded into a tube sheet with epoxy resin at the others end. [9] [21]

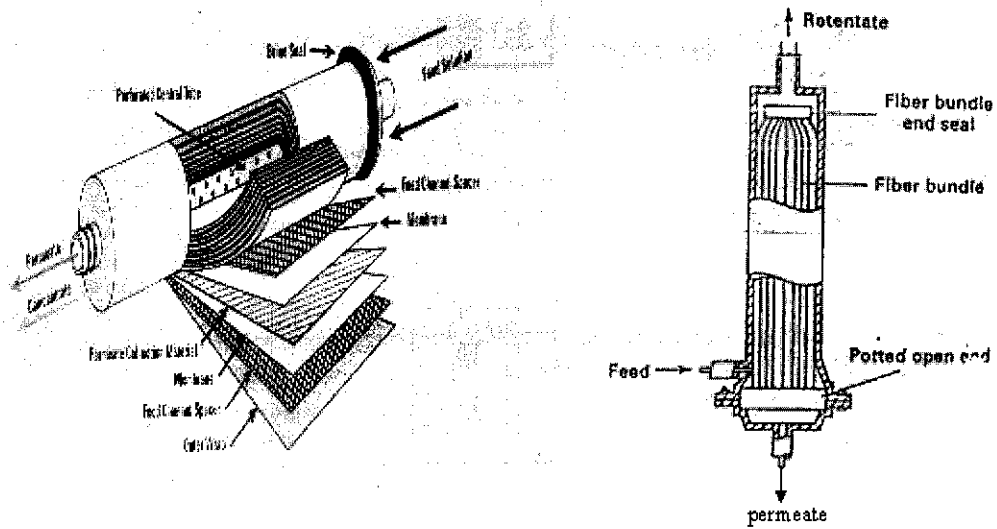
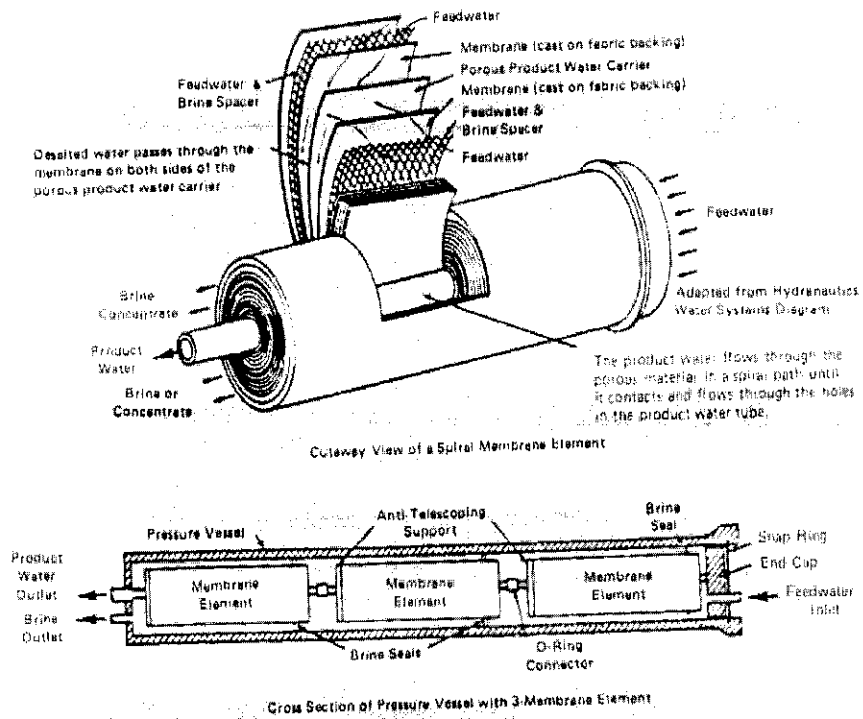


Figure 2.3.1 (a) Spiral-wound membrane element and assembly, (b) Hollow Fiber



Source: O.K. Burns et al. "The USAID Desalination Manual," U.S. Agency for International Development, Washington, DC, prepared by CH2M Hill International Corp., August 1980.

Figure 2.3.2 Schematic diagram of spiral-wound module and pressure vessel assembly

2.4 Ultrafiltration

Ultrafiltration is a membrane process with the ability to separate molecules in solution on the basis of size. An Ultrafiltration membrane acts as a selective barrier. It retains species with molecular weight higher than a few thousand Daltons (microsolutes), while freely passing small molecules (microsolutes and solvents). The separation is achieved by concentrating the large molecules present in the feed on one side of the membrane, while the solvent and microsolutes are depleted as they pass through the membrane. ^{[9][10]}

For example, an Ultrafiltration process will separate a protein (macrosolutes) from an aqueous saline solution. As the water and salts pass through the membrane, the protein is held back. The protein concentration increases and the salts, whose concentration relative to the solvent is unchanged, are depleted relative to the protein. The protein is, therefore, both concentrated and purified by the Ultrafiltration. The process is illustrated in Figure 2.4.1.

Ultrafiltration may be characterized in terms of pore size and porosity, even though there is a little direct evidence for the kinds of pores and the terminology suggests. A frequently used model characterizes the membrane as a flat film with conical pores originating at its surface, as seen in Figure 2.4.2. The surface pores are large enough to permit passage of solvent and microsolutes molecules, but are too small for effective penetration of the larger macrosolutes. The conical shape is desirable, in that any entity that makes it through the opening at the membrane surface can continue unimpeded, there is no danger in pore-plugging. ^{[9][10]}

Ultrafiltration is used in many processes at the present time. An illustrative example of UF is its use for whey processing. Whey production exceeds 4×10^7 tons/year worldwide. It is a byproduct of cheese manufacture. Whey is composed of roughly 0.6 percent true protein, 0.2 percent nonprotein nitrogen, 5 percent lactose, 1 percent salts, some lactic

acid, and the balance water at a pH 3.5 between to 6. It contains trace amount of casein fines and butterfat globules and a large population of bacteria. UF retain protein, large casein and butterfat particles, and the bacteria. UF passes water, lactose, salt and nonprotein nitrogen through the membrane into permeate. When reverse osmosis is used to the whey process, it passes only the water and some of the lactic acid. It is due to the solubility of lactic acid in RO membranes. UF also widely used in separation of oil-water emulsions, concentration of latex particles, processing of blood and plasma, fractionation or separation of proteins, recovery of whey protein in cheese manufacturing, removal of bacteria and other particles to sterilize wine and clarification of fruit juices. (Refer Figure B 4 in appendix B)

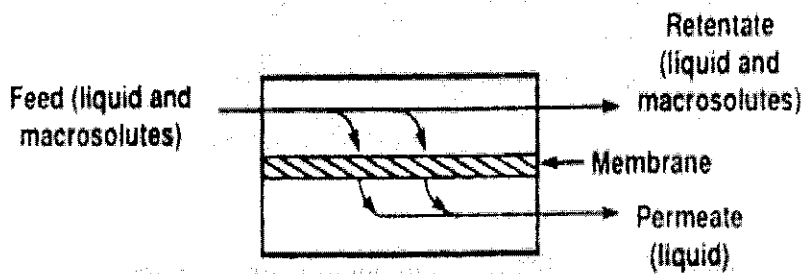


Figure 2.4.1 Schematic diagram of Ultrafiltration process

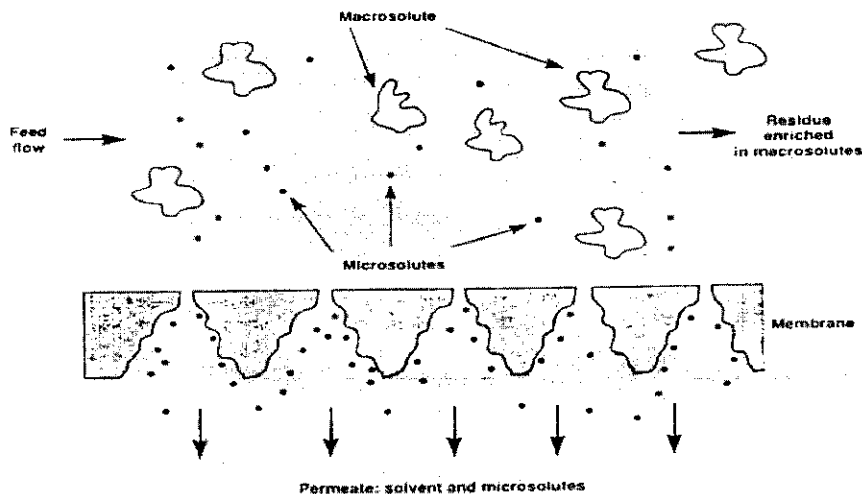


Figure 2.4.2 A model of Ultrafiltration of a solution containing macrosolutes (e.g. proteins) and microsolute (e.g. salts)

2.4.1 Concentration polarization

Since the solute is rejected by the membrane, it accumulates and starts to build up at the surface of the membrane, as pressure drop is increased and/or concentration of the solute is increased, concentration polarization occurs, which is much more severe than in reverse osmosis. This is shown in Figure 2.4.1, where c_1 is the concentration of the solute in the bulk solution, $\text{kg solute}/\text{m}^3$, c_s is the concentration of the solute at the surface of the membrane and c_p is the concentration in the permeate.

As the pressure drop increases, this increases the solvent flux N_w to and through the membrane. This results in a higher convective transport of the solute to the membrane, that is the solvent carries with it more solute. The concentration c_s increases and gives larger back molecular diffusion of solute from the membrane to the bulk solution. At steady state the convective flux equals the diffusion flux.^[7]

$$\frac{N_w c}{\rho} = -D_{AB} \frac{dc}{dx} \quad \text{Equation 2.4 1}$$

Where $\frac{N_w c}{\rho} = \dots \text{kg solute}/\text{s.m}^2$
 $D_{AB} = \dots \text{diffusivity of solute in solvent, m}^2/\text{s}$
 $X = \dots \text{distance, m}$

Further increases in pressure drop increase the value of c_s to a limiting concentration, at which the accumulated solute forms a semisolid gel where $c_s = c_g$ as shown in Figure 2.4.3 still further increases in pressure drop do not change c_g and the membrane is said to be “gel polarized”. Then the equation becomes:

$$\frac{N_w}{\rho} = K_C \ln \left(\frac{c_g}{c_1} \right) \quad \text{Equation 2.4 2}$$

With increases in pressure drop, the gel layer increases thickness, causing the solvent flux to decrease because of the added gel-layer resistance. Finally, the net flux of solute by convective transfer become equal to the back diffusion of solute into the bulk solution because of the polarized concentration gradient as shown in equation 2.4.2. [3]

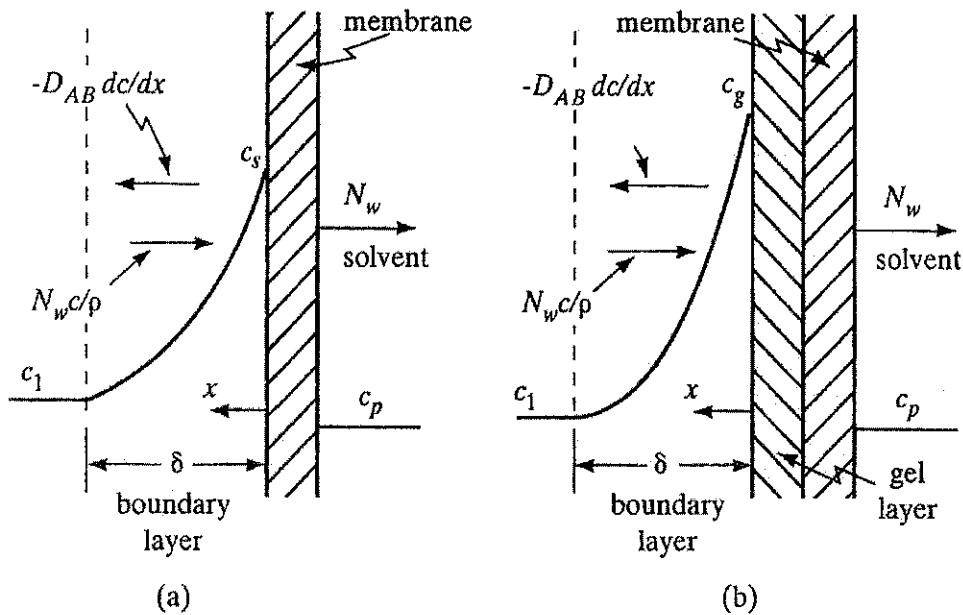


Figure 2.4.3 Concentration polarization in Ultrafiltration: (a) concentration profile before gel-formation, (b) concentration polarization with a gel layer formed at membrane surface

Cutoff characteristics of UF membranes are generally represented by molecular weight cutoff. A membrane will not pass molecules having a molecular weight larger than the molecular weight cutoff. the definition, generally but not universally followed is MWCO is the molar mass of the globular protein, which is 90 percent, retain by the membrane. The section of marker molecule can affect the MWCO measured. Markers for the UF membranes are usually protein, but always polymeric. Polymers of the same molar mass can have very different molecular size, and MWCO is more a measure of size than anything else. [12]

To further complicate, molecular shape can change in the vicinity of a membrane. Linear molecules, such as apolyacrylic acid, with a given molecular mass pass easily through a membrane that retains a globular protein of the same molecular mass. It is necessary to

keep the concentration in the feed and the flux very low to minimize polarization effects. Any polarization of the membrane will alter the measured value and significant accumulation will result in autofiltration. This is a problem for fractionation by Ultrafiltration because microsolute are partially retained by almost all retained macrosolute. As a rule of thumb, higher pressure and more polarization results in more autofiltration.

2.5 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 2.5.1a, a solute dissolved in a solvent in concentrated form is separated from 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 dilute the more concentrated solution. The osmosis continues until equilibrium is established as illustrated in Figure 2.5.1b. 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 because of the presence of the membrane, the osmosis is not useful because the solvent is transferred in wrong direction, resulting in mixing rather than separation. However, applying a pressure to the concentrated solution as shown in Figure 2.5.1c can reverse the direction of transfer of solvent through the membrane. This causes the solvent to permeate through the membrane from concentrated solution to the dilute solution. This separation process, known as *reverse osmosis*, can be used to separate a solvent from a solute-solvent mixture. [3]

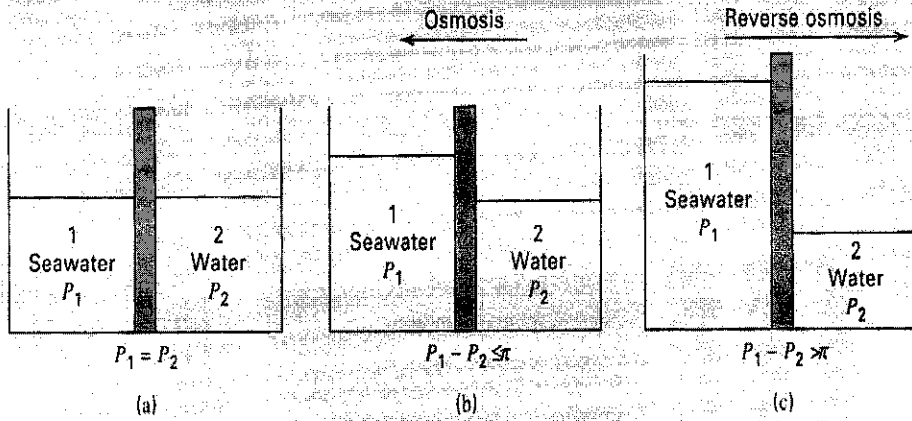


Figure 2.5.1 Osmosis and Reverse Osmosis

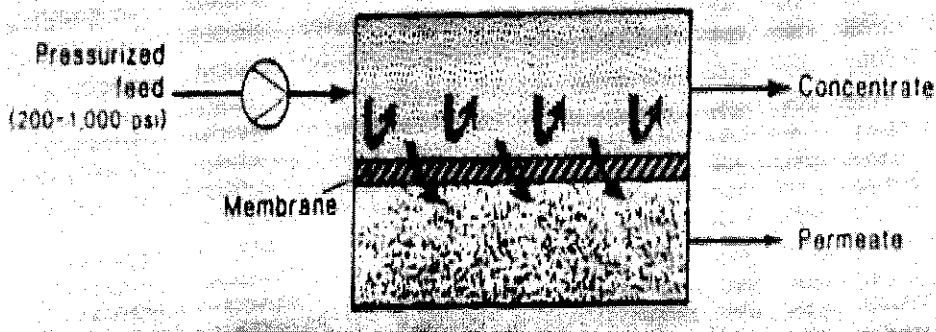


Figure 2.5.2 A schematic diagram of Reverse Osmosis process

The flux through membrane can be written as

$$N_w = \frac{P_w}{L_m} (\Delta P - \Delta \pi) \quad \text{Equation 2.5.1}$$

N_w = solvent (water) flux ($\text{kg}/\text{m}^2 \cdot \text{s}$)

P_w = solvent membrane permeability ($\text{kg solvent}/\text{s} \cdot \text{m} \cdot \text{atm}$)

L_m = membrane thickness (m)

ΔP = $P_1 - P_2$ (hydrostatic pressure difference with P_1 pressure exerted on feed and P_2 on product solution), (atm)

$\Delta \pi$ = $\pi_1 - \pi_2$ (osmotic pressure of feed solution – osmotic pressure of product solution), (atm)

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 bars. As long as the applied pressure is greater than the osmotic pressure of the feed solution, “pure” water will flow from the more concentrated solution to the more dilute through the membrane. The osmotic pressure in equation above can be approximated by Van’t Hoff equation: ^{[3][7]}

$$\Pi = \frac{nRT}{V_m} \qquad \text{Equation 2.5.2}$$

- n = number of kg mol of solute
- V_m = volume of pure solvent (m³)
- R = gas law constant
- T = operating temperature, (K)

Reverse osmosis now is widely applied to desalination and purification of seawater, brackish water, and wastewater. Prior to 1980, multistage flash distillation was the main process for the desalinization of water. By 1990, this situation was dramatically reversed, making RO the dominant process for new construction. Leob made the dramatic shift from a thermally driven process to a more economical pressure-driven process possible through the development and Sourirajan of an asymmetric membrane that allows pressurized water pass through at a high rate, while almost preventing transmembrane flows of dissolved salts, organic compounds, colloids, and microorganisms. Today, more than 1,000 Ro desalting plants are producing more than 750,000,000 gallons per day of potable water worldwide. ^[23]

Other uses of reverse osmosis, usually on a smaller scale than the desalinization of water to produce potable water, include; (1) the treatment of industrial wastewater to remove heavy metal ions, nonbiodegradable substances and other components of commercial value;(2) the treatment of rinse water from electroplating processes to obtain a metal ion

concentrate and a permeate that can be reused as a rinse; (3) the separation of sulfites and bisulfites from effluents in pulp and paper processes; (4) the treatment of wastewater in dyeing processes; (5) the recovery of constituents having food value from wastewaters in food processing plants for example lactose, lactic acid, sugars and starches;(6) the treatment of municipal water to remove inorganic salts, low-molecular weight organic compounds, viruses and bacteria; and (7) the dewatering of certain food products such as coffee, soups, tea, milk, orange juice and tomato juice. In such applications, membranes must have chemical, mechanical, and thermal stability to be competitive with other process. [6][7]

Permeate flux and salt rejection is the key performance parameters of a reverse osmosis process. They are mainly influenced by variable parameters, which are pressure, temperature, recovery, and salt rejection.

2.5.1 Effect of pressure

Feedwater pressure affects both the water flux and salt rejection of RO membranes. Pressure in excess of the osmotic pressure is applied to the concentrated solution and the flow of water is reversed. A portion of the feedwater (concentrated solution) is forced through the membrane to emerge as purified water of dilute solution side. Figure 2.3.1 shows the water flux across the membrane increases as increases in feedwater pressure also results in increased salt rejection but the relationship is less direct than for water flux. As feedwater pressure is increased, some salt passage increasingly overcome as water is pushed through the membrane at a faster rate than salt can be transported. However, there is an upper limit to the amount of salt that can be excluded via increasing feedwater pressure. As shown in figure above at a certain pressure level, salt rejection no longer increases and some salt flow remains coupled with water flowing through the membrane. [3][4]

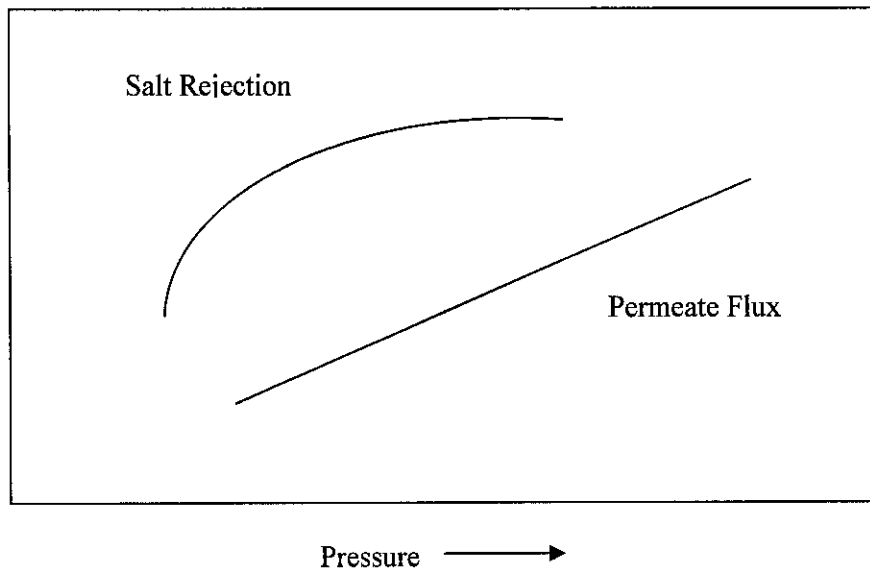


Figure 2.5 3 Effect of Pressure to the Reverse Osmosis

2.5.2 Effect of Temperature

Membrane productivity is very sensitive to changes in feedwater temperature. As water temperature increases, water flux increases almost linearly due primarily to the higher diffusion rate of water through the membrane. Increased feedwater temperature also results in lower salt rejection or higher salt passage. This is due to a higher diffusion rate for salt through the membrane. ^{[4][13]}

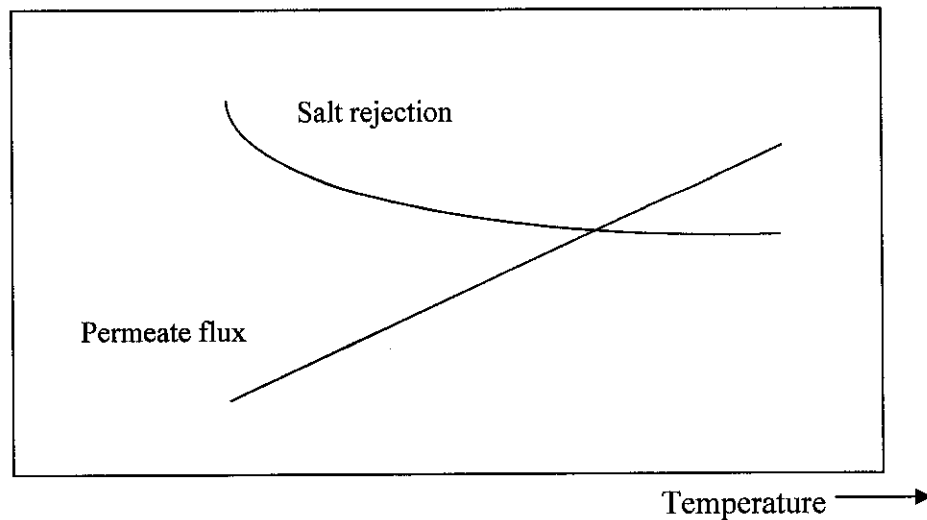


Figure 2.5 4 Effect of Temperature to Reverse Osmosis

2.5.3 Effect of Salt Concentration

Osmotic pressure is a function of the type and concentration of salts or organics contained in feedwater. As salt concentration increases, so does osmotic pressure. The amount of feedwater driving pressure necessary to reverse the natural direction of osmotic flow is, therefore largely determined by the level of salts in the feedwater. Figure 2.3.3 demonstrate that if feed pressure remains constant, higher salt concentration results in lower membrane water flux. The increasing osmotic pressure offsets the feedwater driving pressure. Besides, increases in salt passage through the membrane (decrease in rejection) as the water flux declines. [3][12]

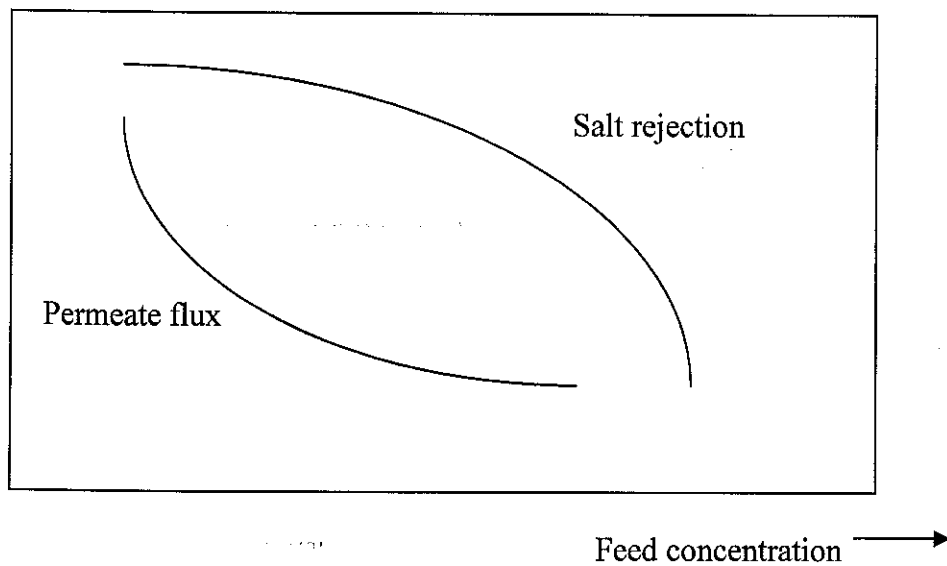


Figure 2.5 5 Effect of Salt Concentration

In this form, the molecule cannot readily permeate the small pores in the membrane. At low pH, the carboxyl groups along the acid polymer are protonated. The resulting neutral molecule is much more flexible and therefore can pass through the membrane. The pH tolerance of various types of membrane can vary widely. pH of most naturally occurring water though a RO membrane is within 7 to 11. Thin film composite membranes are typically stable over a broader pH range than cellulose acetate (CA) and therefore, offer greater operating latitude. [3][12]

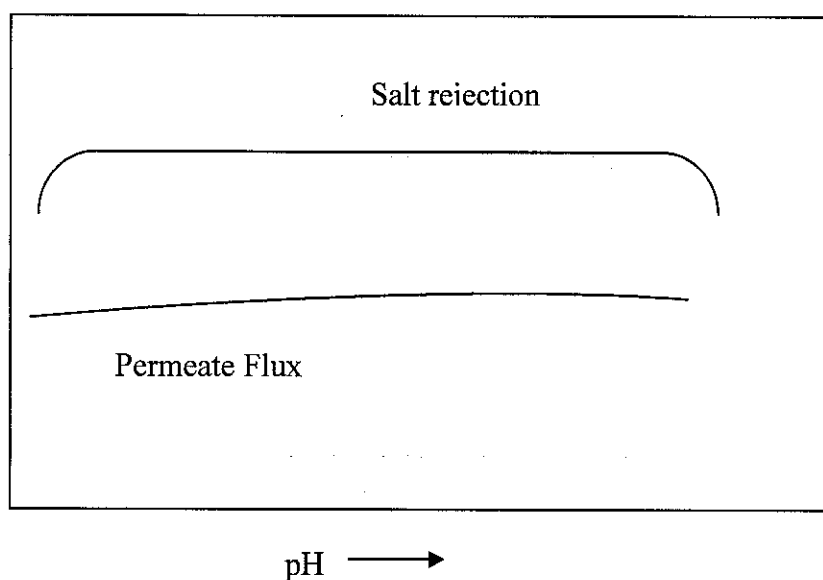


Figure 2.5.7 Effect of feedwater pH on water flux and salt rejection

2.6 Concentration Polarization (RO)

A phenomenon that is particularly important in the design of reverse osmosis units is that of concentration polarization. This occurs if there has ion at the feed-side (concentrated side) of the reverse osmosis membrane. Because the solute cannot permeate through the membrane, the concentration of the solute in the liquid adjacent to the surface of the membrane is greater than that in the bulk of the fluid. This difference causes mass transfer of the solute by diffusion from the membrane surface back to the bulk liquid. The rate of diffusion back into the bulk fluid depends on the mass transfer coefficient for the

boundary layer on feed-side. Concentration polarization is the ratio of the solute concentration at the membrane surface to the solute concentration in the bulk stream. Concentration polarization causes the flux of solvent to decrease since the osmotic pressure increases and the overall driving force ($\Delta P - \Delta \pi$) decreases.^{[3][4]}

In general, the solution-diffusion mechanism for gas permeation is quite slow compared to the diffusion in the gas boundary layers or film adjacent to the membrane, so external mass transfer resistances are negligible. Thus, concentration polarization is commonly neglected for gas permeation. Because diffusion in liquid boundary layers and film can be slow, concentration polarization cannot be neglected in membrane processes that involved liquids, such as dialysis, reverse osmosis and pervaporization. The concentration polarization is more important in reverse osmosis, where the effect can reduce the water flux and increase the salt flux.^[23]

For simplicity, concentration polarization is assumed to occur only on the feed side of the membrane and the membrane flux across the membrane and boundary layer together is written as

$$J_i = k_{ov} (c_{ib} - c_{ip}) \quad , \quad \text{and} \quad \text{Equation 2.6.1}$$

$$\frac{1}{k_{ov}} = \frac{1}{k_m} + \frac{1}{k_{bl}} \quad \text{Equation 2.6.2}$$

Where;

J_i = membrane flux

k_{ov} = overall mass transfer coefficient

k_m = mass transfer coefficient of the membrane

k_{bl} = mass transfer coefficient of the fluid boundary layer

c_{ib} = concentration of component i in bulk feed solution

c_{ip} = concentration of component i in bulk permeate solution

The most easily changed factor that affects CP is the boundary layer thickness. Thus, the most straightforward way of minimizing CP is to reduce the boundary layer thickness by

increasing turbulent mixing at the membrane surface. [The Behavior of Suspensions and Macromolecular Solutions in Cross Flow Microfiltration, 1994] The most direct techniques to promote mixing are to increase fluid flow velocity past the membrane surface. Membrane spacers are also widely used to promote turbulence by disrupting fluid flow in the module channels [Spacer Characterization and Pressure Drop Modeling in Spacer-Filled Channels for Ultrafiltration, 1994].

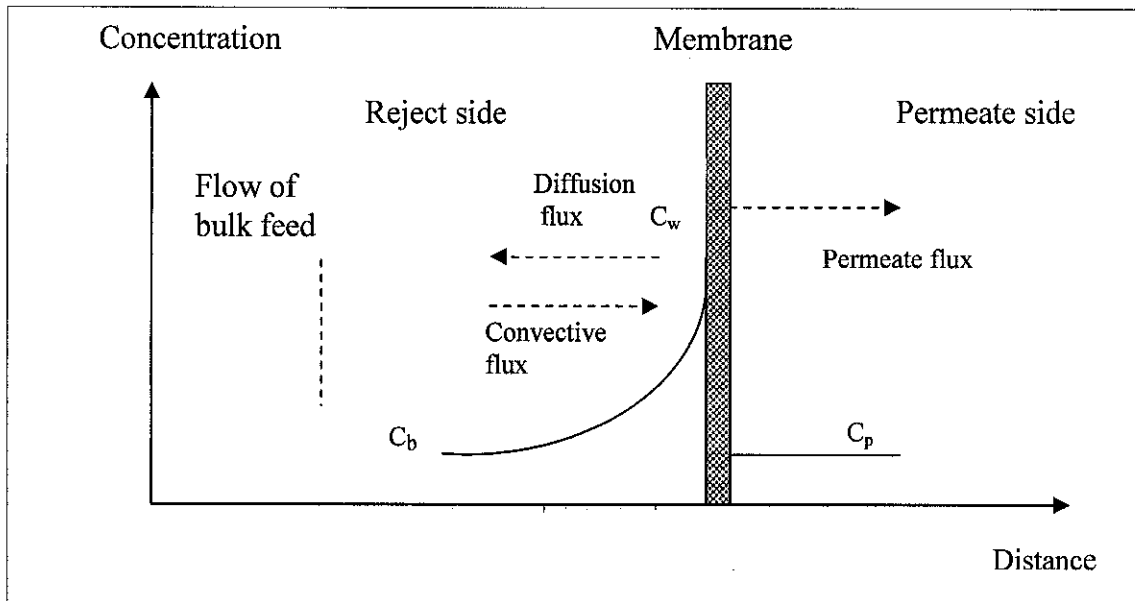


Figure 2.6.1: Concentration and flows around the membrane

2.7 Resistance and retention

The basic approach to the characterization of membranes is using a simple form of *Darcy's equation*

$$J = \frac{\Delta P}{R_m}$$

Equation 2.7.1

Where ΔP is trans-membrane pressure (TMP), which can be calculated as

$$\Delta P = P_{feed} - P_{permeate} \quad \text{Equation 2.7.2}$$

Darcy's equation applies well when only water is flowing in the system and can be used to find the resistance of the membrane to the water flow.^[3]

2.8 Types of amine

2.8.1 Primary Amine (RNH_2)

Monoethanolamine (MEA) and Diglycolamine (DGA) are categorized in primary amines. MEA is effective at removing virtually all H_2S and CO_2 but requires a large quantity of heat to regenerate in order to break the chemical bonds formed. MEA is a viscous hygroscopic liquid with an ammoniacal odor and it miscible with water and many organic solvents. DGA is same with MEA in term of performance but DGA has a lower vapor pressure, which result in less solvent vaporization losses.

... resistance to the water flow.

2.8.2 Secondary Amine (R_2NH)

Secondary amines such as Diethanolamide (DEA) and Disopropylamine (DIPA) are less reactive than primary amines because the hydrocarbon groups are larger than hydrogen group. This extra bulk reduces the ability of incoming reactant molecules to interact with nitrogen. DEA become the dominant commercial absorbents companies such as the Girdler Corporation developed MEA chemistry into a commercial process. DEA is good general purpose solvent. It also moderate organic sulfur removal and moderate solution concentrations due to corrosion concerns.

2.8.3 Tertiary Amine (R_3N)

The third group is tertiary amines, which are Methyl Diethanolamine (MDEA) and Triethylamine (TEA). This group is less corrosive and can be used in higher concentrations but it is not a good choice when the raw gas pressure is low or the specification calls for deep CO_2 removal. Tertiary amines particularly MDEA are well suited for selective absorption on high-pressure gas streams. MDEA has been technically available since 1950, but has only recently become commercially popular. MDEA combines the low generative heat requirement of a physical solvent with the ability to selectively remove H_2S from gas stream containing both H_2S and CO_2 .^{[8][9]}

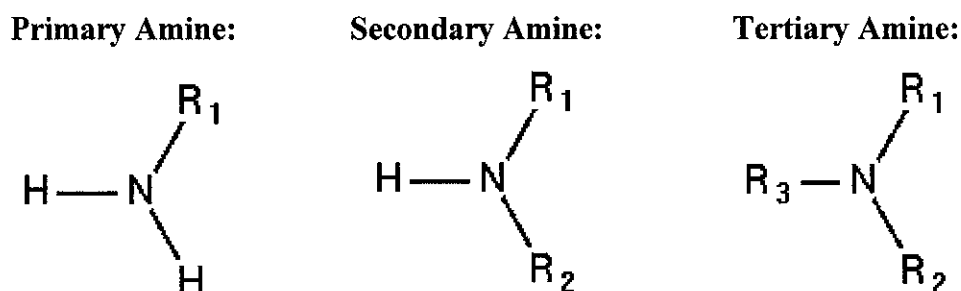
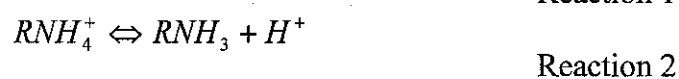
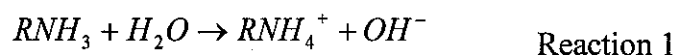


Figure 2.8.1 Structure of amines

R = carbon based groups

2.9 Intermolecular Forces

Amines are polar compounds and both primary and secondary amines form intermolecular hydrogen bonds. An $N-H \cdots N$ hydrogen bond is weaker than an $O-H \cdots O$ hydrogen bond because the difference in electronegativity between nitrogen and hydrogen ($3.0-2.1=0.9$) is less than between oxygen and hydrogen ($3.5-2.1=1.4$). Hydrogen Bonding is a special type of intermolecular attraction that exist between the hydrogen atom in a polar bond and an unshared electron pair on a nearby small electronegativity ion or atom.^[2] The example of chemical reaction between primary amines and water is shown below:



For the first reaction, the primary amines will react with water to form conjugate acid (positive ion) and hydroxide (negative ion). Then, the second reaction takes place in order to dissociate the conjugate acid to form primary amine and positive hydrogen ion. From both reactions, it will produce the third reaction, which is pure water. The chemical reaction is the same for secondary and tertiary amines^[22]

CHAPTER 3

METHODOLOGY AND PROJECT WORK

3.1 Experimental Procedures

3.1.1 System Start Up:

1. The main supply is turned on by turning the main switch and the white light is ensured to illuminate.
2. “General” button is pressed to power on the system and all the digital displays are ensured on.
3. The feed tank is fully filled with solution before each experiment starts.
4. The valves NV1, V1, V2, V4, V5 and V7 are set at open.
5. The “NV2” is regulated to open at 20%.
6. “DV4” is closed.
7. 3/2 way valve V6 is set to either RO or UF path way for different experimental purposes.

3.1.2 System Shut Down:

1. “STOP” button is pressed under “HIGH PRESSURE PUMP” on the control panel to stop high pressure pump running (for RO experiments only).
2. “STOP” button is stopped under “BOOSTER PUMP” to shut down booster Pump (both for RO & UF experiments).

3. The solution can be drained off from membrane and tanks by opening the drainage valves DV1, DV2, DV3 and DV4 when the experiment is completed.

3.1.3 UF backwash

1. The feed tank is filled with sufficient amount of tap water (80L).
2. UF pathway is set.
3. Valve NV2, V4, V5 and V7 are closed.
4. Valve DV4 is set open.
5. The backwash coupling line is then plugged into the system.
6. Booster pump is started and run backwash for about 4 minutes.

For RO and flat sheet, there is no backwash, but flushing with tap water for at least 4 minutes is required.

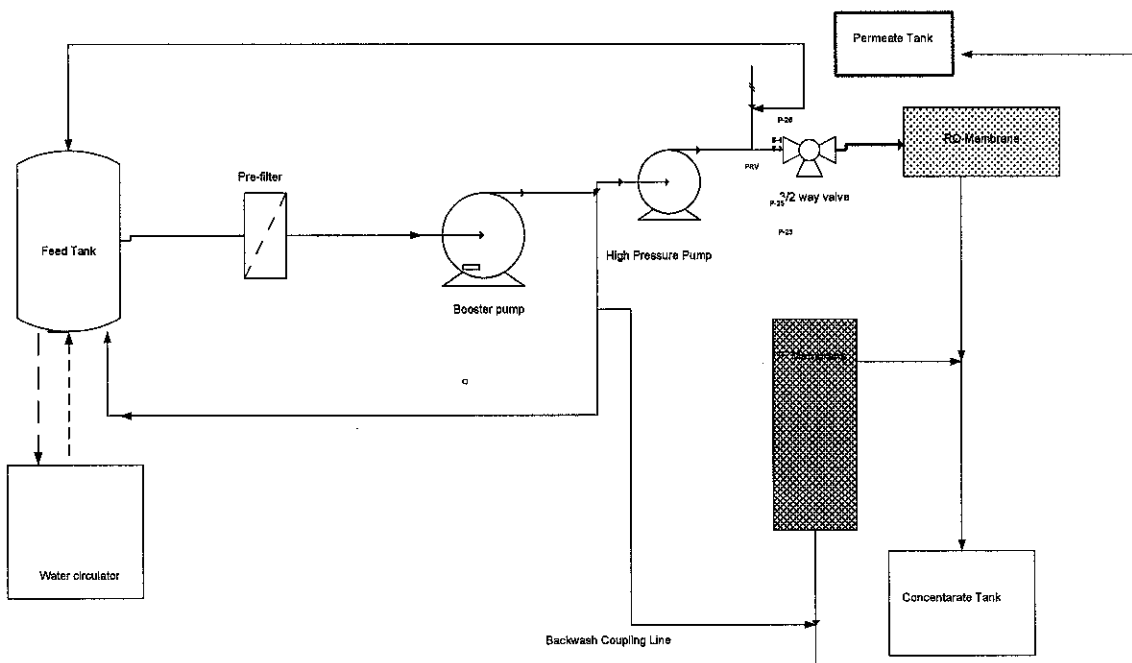
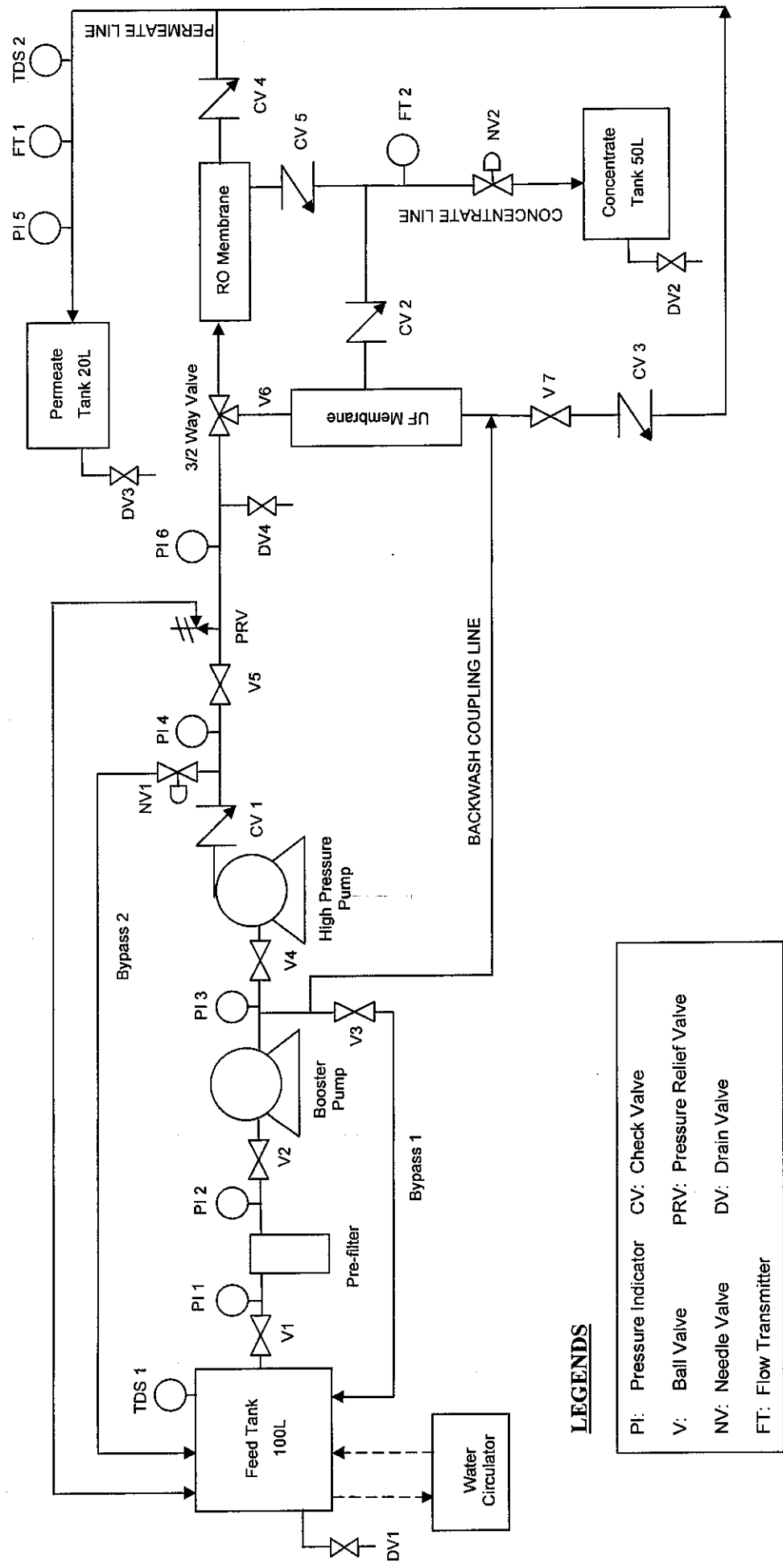


Figure 3.1.1: Reverse Osmosis Pilot System schematic diagram



LEGENDS

PI: Pressure Indicator	CV: Check Valve
V: Ball Valve	PRV: Pressure Relief Valve
NV: Needle Valve	DV: Drain Valve
FT: Flow Transmitter	

Figure 3.2.2 Reverse Osmosis Pilot System

3.1.4 Gas Chromatography with Mass Spectrometer(GCMS)

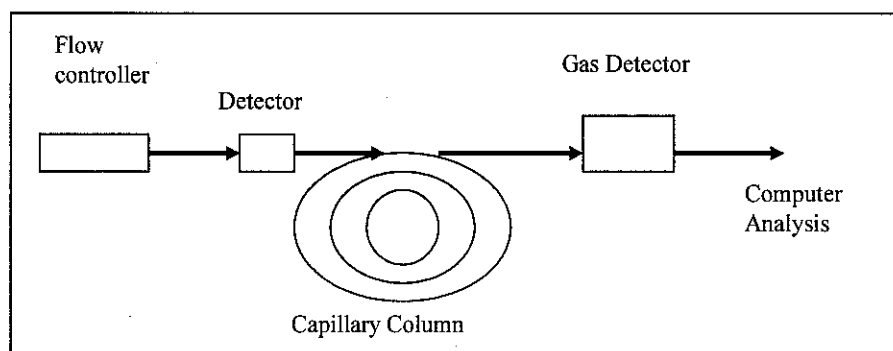


Figure 3.1.3: Gas Chromatography Flow Diagram

To determine the chemical composition in the sample.

1. The GC system is switched on and waited until it is stabilized.
2. A suitable method and duration for the analysis is chosen from the software.
3. 0.5 μ L of sample solution is taken and injected into the capillary column using a syringe.

The peaks obtained are compared and matched with the data in the system's library, to determine the chemical component.

3.2 Equipment and Process Description

Table 3.2 1 Chemicals used during the experiment

NO	CHEMICALS	QUANTITY
1	Monoethanolamine (MEA) (<i>primary amine</i>)	1 L
2	Diethanolamine (DEA) (<i>secondary amine</i>)	1 L
3	Methyl Diethanolamine (MDEA) (<i>tertiary amine</i>)	1 L

Table 3.2 2 Physical properties of chemicals and solution used

PROPERTY CHEMICAL	WATER	MEA	DEA	MDEA
Formula	H ₂ O	OHC ₂ H ₄ NH ₂	(OHC ₂ H ₄) ₂ NH	C ₅ H ₁₃ NO ₂
Molecular weight(g/mol)	18	61.08	105.14	119.16
Boiling point (°C)	100	170.4	269	247
Melting point (°C)	>0	10.5	28	-21
Density (kg/m ³)	1000	1020	1090	1040
Flash point (°C)	-	96.1	130	137
Physical state	Colorless liquid	Colorless liquid	Colorless liquid	Colorless liquid
Solubility in H ₂ O @20°C	-	100%	100%	100%
pH	7	9-11	9-11	9-11

Table 3.2 3 Equipment during the project

No	Equipment	Function
1	Reverse Osmosis Pilot System and Ultrafiltration Membrane	To separate the feed solutions
2	Gas Chromatography with Mass Spectrometer (GCMS)	To analyze chemical compositions in the sample
3	Digital pH meter	To determine sample pH

3.2.1 Reverse Osmosis Pilot System

The system consists of three membrane separation modules: reverse osmosis (RO), ultra filtration (UF) and flat sheet membrane. For RO, two types of pump are used which are booster pump and high pressure pump in order to supply the high pressure required for the liquid separation. The highest pressure that can be reached by this equipment for RO membrane filtration is 1000 psi or 69 bar while for UF is only 100 psi or 6.9 bar, where it does not need the high pressure pump.

In the membrane, the feed solution is filtered and the product or permeate flows to the permeate tank for collection. Several indicators are installed at the permeate line, PI 5, FT 1 and TDS 2 to measure the permeate out pressure, flow rate and concentration. The portion of feed that do not pass through the membrane or called reject, flows to the concentrate line. For safety purposes, a pressure relieve valve (PRV) is also installed on its line. Then the feed flows through the selected membrane module, whether UF or RO, by adjusting the 3/2 way valve.

3.2.1 Gas Chromatography with Mass Spectrometer

A gas chromatographic method has been developed to analyze amine solutions for acid gases, hydrocarbons, water and amine content. Good separations and sharp peaks were

obtained for most components with Tenax-GC and Poropak Q columns when combined with temperature programming. The GC method has numerous advantages over the titration methods. These include approximate 10 minute analysis time, better reproducibility, the capability for on-line sampling and less interference.

The GC method agreed very closely with titration results for CO₂ analysis. Mass Spectrometer is interfaced with gas chromatography to provide structural information and help identify the separated analyses. In the GCMS, the sample injected is heated while flowing in the capillary column and turned into gas. The gases formed are detected at the end of the column by a sensor and transfer the data to the connected computer

3.2.1.1 Analysis method used for GC

Injection volume	: 1.0 μ L
Temperature	: 280 $^{\circ}$ C
Carrier gas	: N ₂ / Air
Pressure	: 99.9 kPa
Total flow	: 18.3 mL/min
Column type	: BPX-5
Column length	: 30.0 m
I. Diameter	: 0.25 mm

CHAPTER 4

RESULT AND DISCUSSION

4.1 Overview of RO and UF Result

The experiment had been done by using two different types of membrane, which are Reverse Osmosis and Ultrafiltration. The main objective of this experiment is to demonstrate the separation amines from water. Three different group of amine had been used in this experiment which are MEA, DEA and MDEA. Different parameters such as concentrations and pressures are manipulated in order to get better separation between amines and water passing through RO and UF membrane.

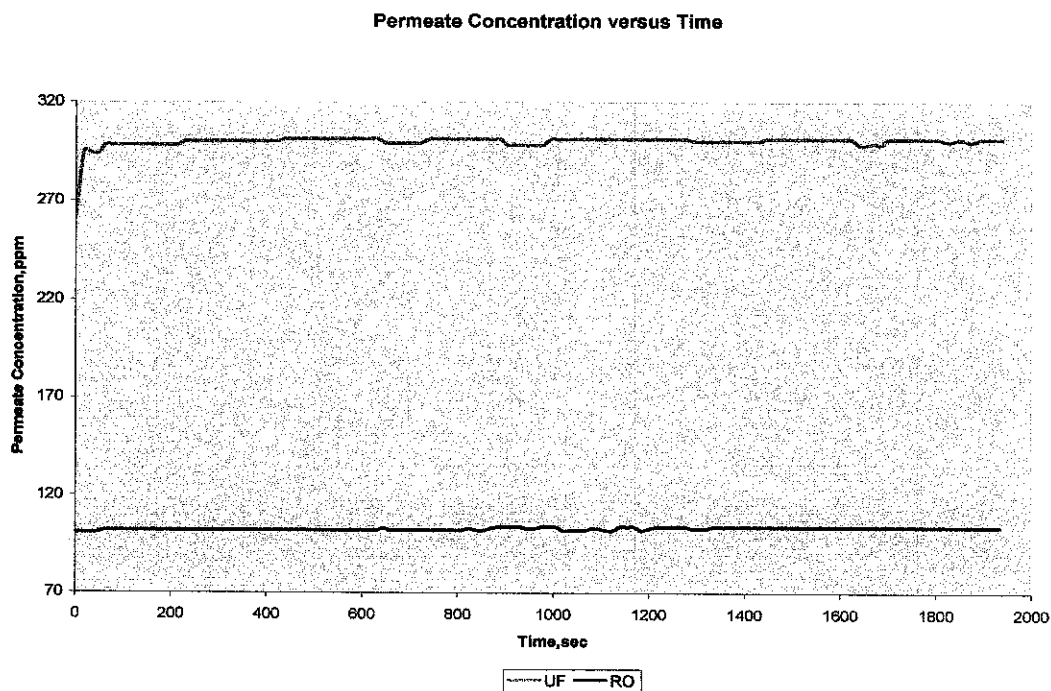


Figure 4.1 Permeate Concentration versus time for different membrane

Figure above shows that the permeate concentration versus time. It means that as the permeate concentration of solute (amine) will increase as the time increase. However,

there is a certain limit where no separation occurred due to the accumulation of the solute at the surface of membrane. From the Figure 4.1 can see that reverse osmosis give lower permeate concentration compared with Ultrafiltration. Based on Figure 2.1.1, the pore size for reverse osmosis is 0.1nm to 1nm that is much smaller than Ultrafiltration, which is 10nm to 100nm. Therefore, the molecular size of solute is smaller than the pore size of the UF membrane that gives very small amine rejection compared with the pore size of RO membrane, which is much smaller than the molecular size of solute, and gives better separation process. (Refer to appendix D)

4.2 Effect of Feed Pressure

4.2.1 Reverse Osmosis

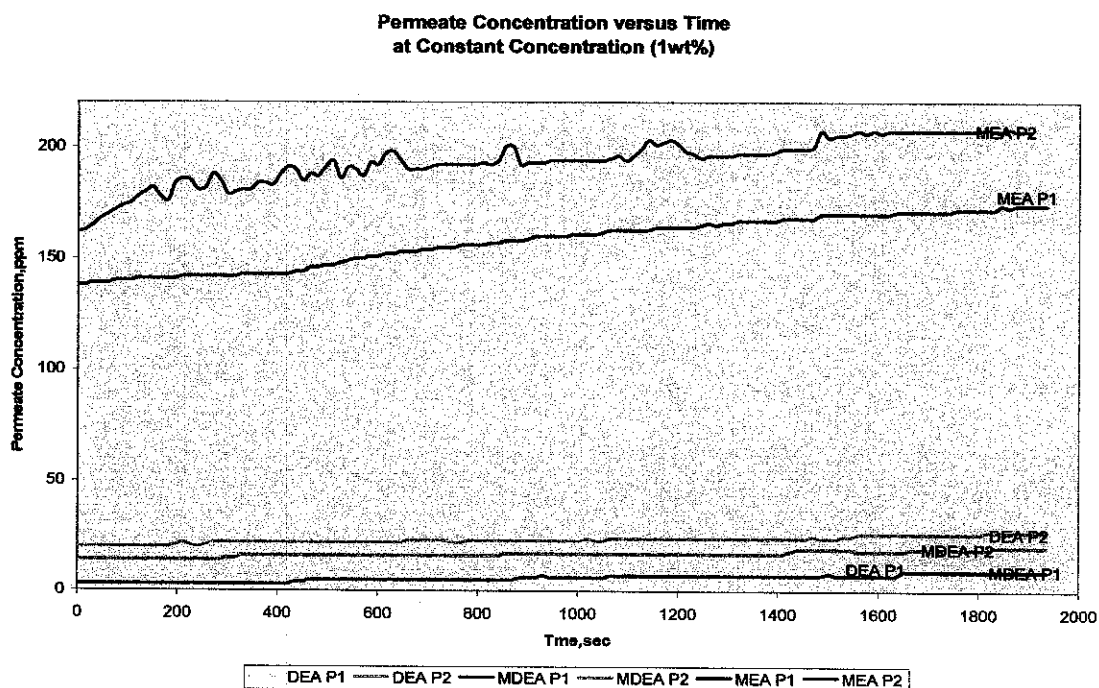


Figure 4.1.1 Permeate Concentration versus Time at 1wt% Amine using RO

Permeate Concentration versus Time
at Constant Concentration (3wt%)

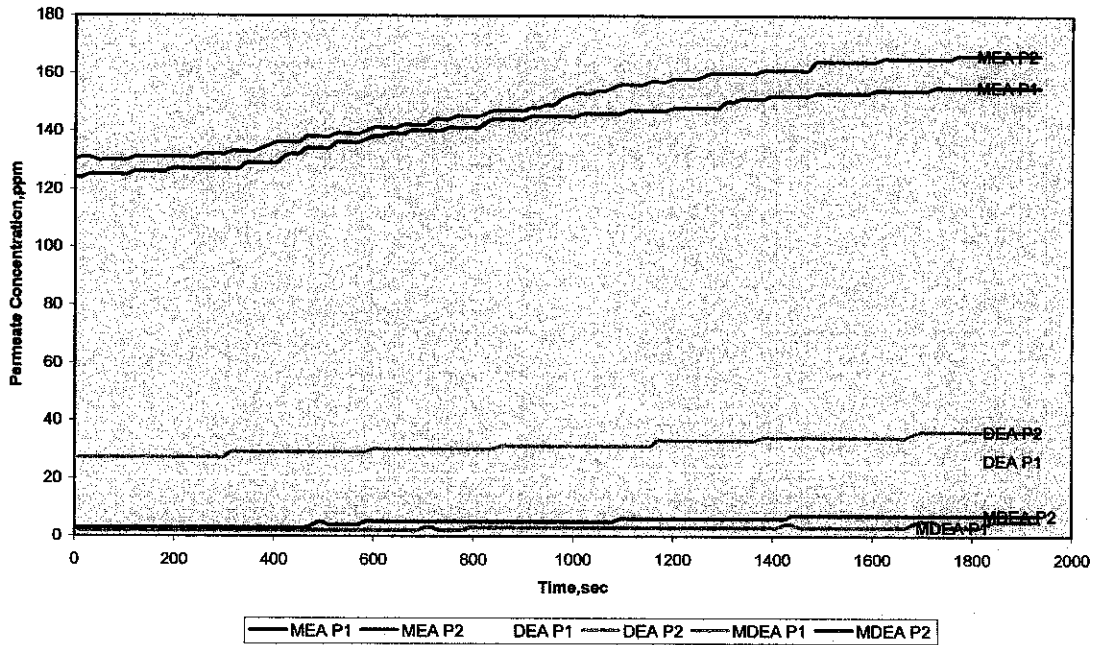


Figure 4.1.2 Permeate Concentration versus Time at 3wt% Amine using RO

Figure 4.1.1 and Figure 4.1.2 show the effect of different feed pressure to the permeate concentration for three different types of amine using RO at constant concentration. The experiment had been done in two different solute concentrations, which are 1 wt% and 3 wt% of amines and two different feed pressure which are 294 psi (20 bar) and 441 psi (30 bar). These pressures are called applied pressure. In this experiment, the applied pressure used is larger than osmotic pressure. This already proved by calculation using the Equation 2.5.2 (Refer Table D.4 in appendix D).

As shown in the figures above, increasing the pressure from 20 bar to 30 bar will reduced the permeate concentration (amine concentration) for all three types of amine passing through the membrane. It means that more purified water pass through the membrane as permeate is increases and the solute (amine) remains approximately constant, giving lower permeate concentration in the product solution compared with the rententate which is has higher solute (amine) concentration. Because RO membranes are imperfect barriers

to dissolved amine in feed, there is always some solute passage through the membrane. As feed pressure is increased, this solute passage increasingly overcome as water is pushed through the membrane at a faster rate than solute can be transported. However, there is an upper limit to the amount of solute that can be excluded via increasing feed pressure.

Both concentration show that, MEA gave higher permeate concentration at higher pressure (30 bar) and followed by DEA and MDEA. It is due to the molecular size of the amines according to their group. Tertiary Amine, which is MDEA, has the best separation process with water. This is because according to the Table 3.2.2, MDEA has the highest molecular weight that indicates the largest molecular size of MDEA compared with MEA and DEA. Therefore, at feed pressure 20 bar the permeate concentration of MDEA for 1wt percentage and 3wt percentage is the lowest compared with the permeate concentration at 30 bar feed pressure as shown in Figure 4.1.1 and Figure 4.1.2.

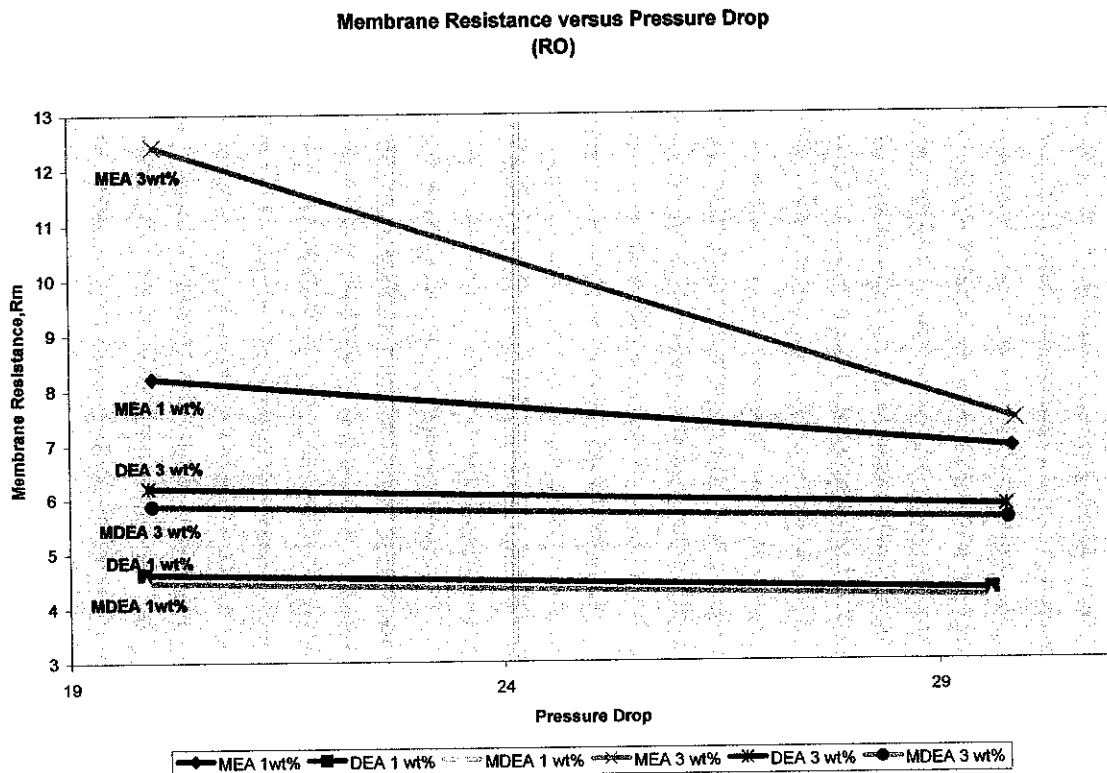


Figure 4.13 Membrane resistance for reverse osmosis

Based on *Darcy's equation* the higher the pressure, more passage can pass through the membrane due less membrane resistance through the membrane which shown in Figure 4.1.3. Meanwhile, Figure 4.1.4 clearly shows that MDEA gives the highest amine rejection compared with the other two types of amine, as the feed pressure increases from 20 bars to 30 bars. This indicates that the best separation is obtained between MDEA and water at higher feed pressure.

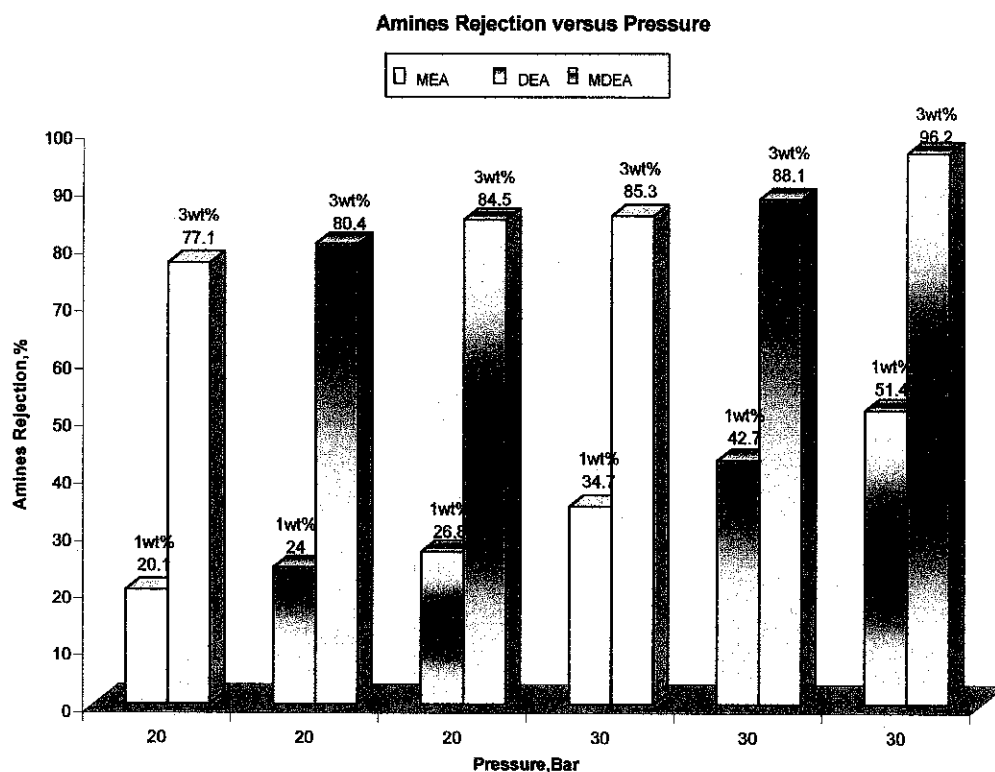


Figure 4.1.4 Amine rejection for different types of amine using RO membrane

4.2.2 Ultrafiltration (UF)

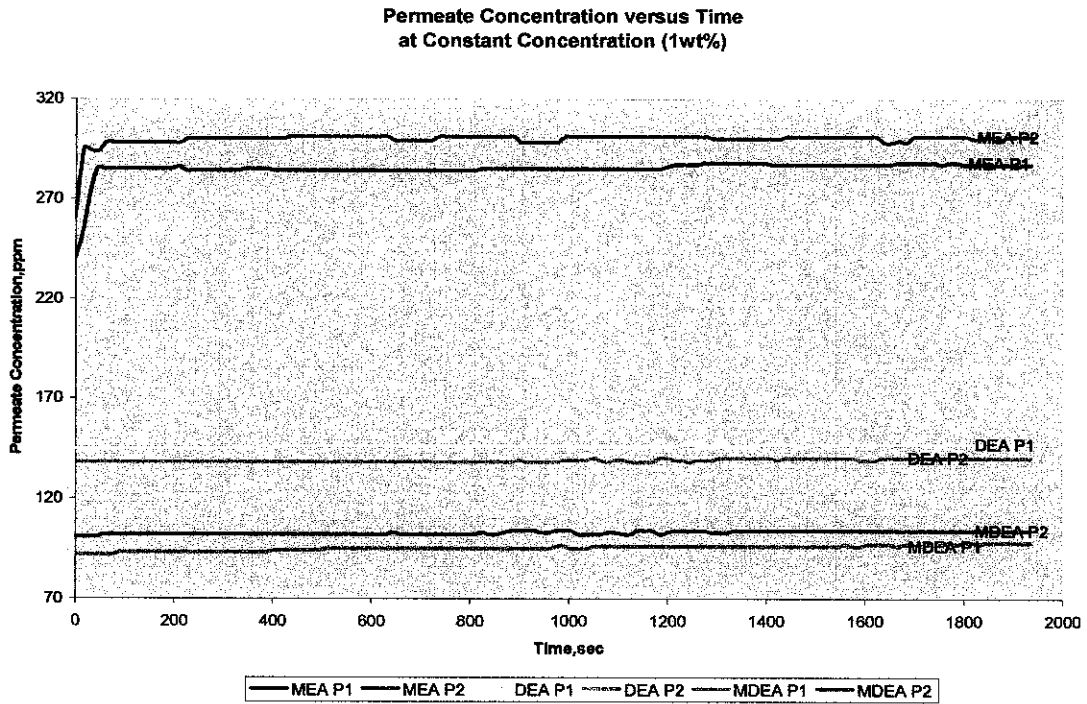


Figure 4.1.5 Permeate Concentration versus Time at 1wt% MEA using UF

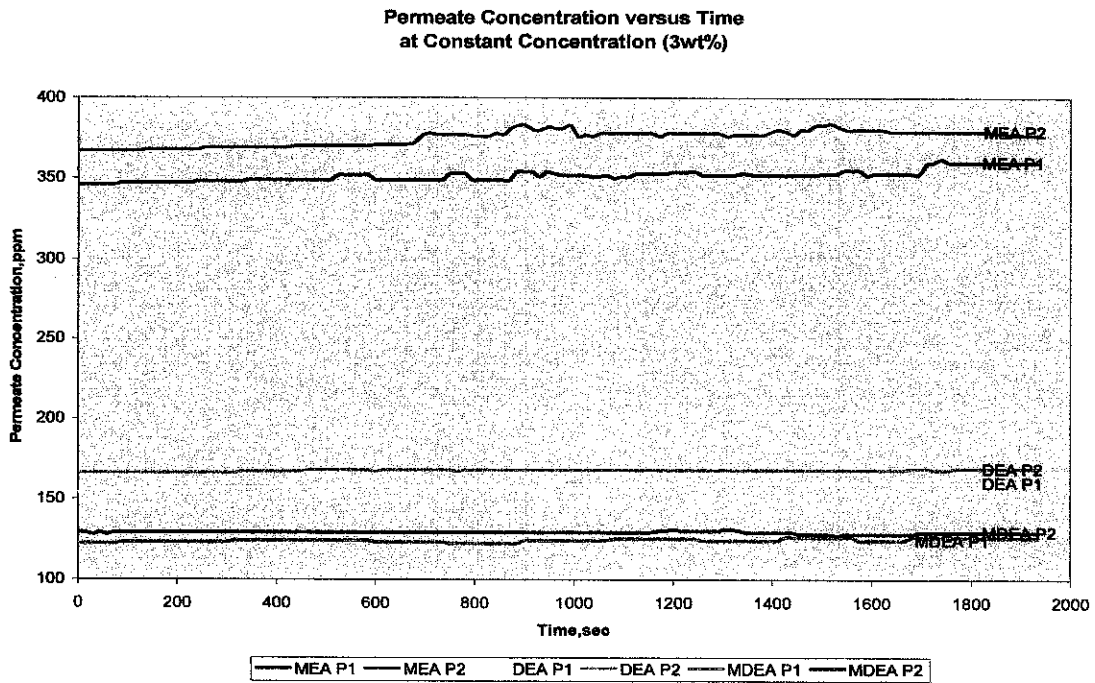


Figure 4.1.6 Permeate Concentration versus Time at 3wt% MEA using UF

Figure 4.1.5 and Figure 4.1.6 show that the effect of different feed pressure that is 51.45 psi (3.5 bars) and 70.56-psi (4.8 bars) using UF membrane. The experiment had be done by varies the pressure while maintaining the temperature and concentration of amine either 1 wt percentage or 3 wt percentage .From both graph, can see that increasing the feed pressure will producing more permeate concentration of three types of amine. Even though, the molecular size of MDEA is the largest compared with the other, the separation between MDEA and water still cannot be obtained. This is because the pore size of the UF membrane (10 nm to 100 nm) is larger than the molecular size of the solute (amine). Therefore, as the separation occurs at higher feed pressure, the solute (amine) can pass through the membrane easily.

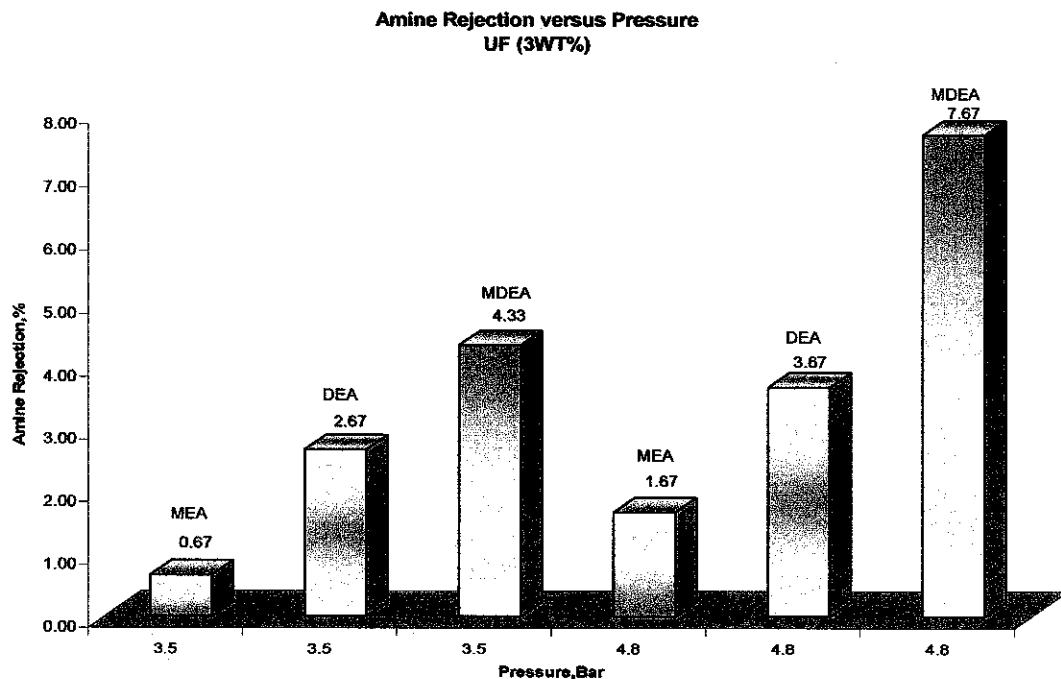


Figure 4.1.7 Amine rejection for different types of amine using UF membrane for 3 wt% feed solute concentration

At 1wt percentage of the feed solute concentration, the permeate concentration produced is almost the same as the feed concentration and it takes shorter time to be stabilized at both pressure applied for three types of amine. However, when the pressure is applied at 3wt percentage feed solute concentration, the permeate concentration is more fluctuating

compared with 1wt percentage feed solute concentration. It is because the concentrated feed solutions is harder to pass through the membrane as pressure is applied compared with the diluted solution (1wt %) but still most of the solute can pass through the UF membrane. MEA shows the highest permeate concentration compared with the other two types of amines. It is due to the smallest molecular sizes that make it easily pass through the UF membrane and there is small percentage of amine rejection at the retentate side for 3 wt percentage feed solute concentration and there is no amine rejection for 1 wt percentage feed solute concentration as shown in Figure 4.1.7. This is because most of the concentration for the feed and permeate is almost the same for 1wt percentage as shown in Figure 4.1.5, therefore there is no amine rejection obtained by passing through the UF membrane.

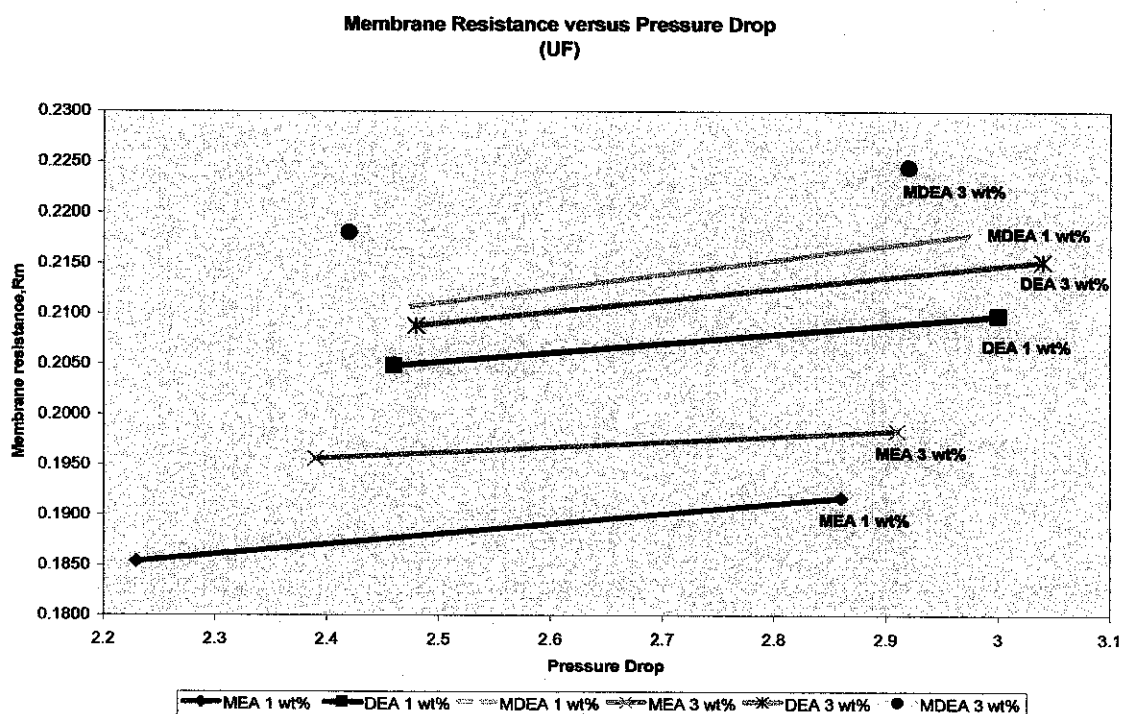


Figure 4.1.8 Membrane resistance for Ultrafiltration

As shown in Figure 4.1.8 MEA also give the lowest membrane resistance as the pressure drop is increased. For example at 1wt percentage solute concentration, the membrane

resistance will increase from 0.1858 to 0.1917 as the feed pressure is increased. This is because the membrane resistance very dependant on the pressure drops across the membrane. This indicates the separation between amines and water is not occurred by using UF membrane system.

Based on the principle of concentration polarization, as the pressured drop increases, this will increases the solvent flux (water) through the membrane. [3][13]. However, this will give higher convective transport of solute to the membrane that is the solvent carries with it more solute. Further increases in pressure drop increase the solute concentration to a limiting concentration, at which the accumulated solute forms a semisolid gel as shown in Figure 2.4.1 by increasing more pressure drop, the gel layer increases in thickness causing the solvent flux to decrease because of added gel-layer resistance.

4.3 Effect of Feed Concentration

4.3.1 Reverse Osmosis

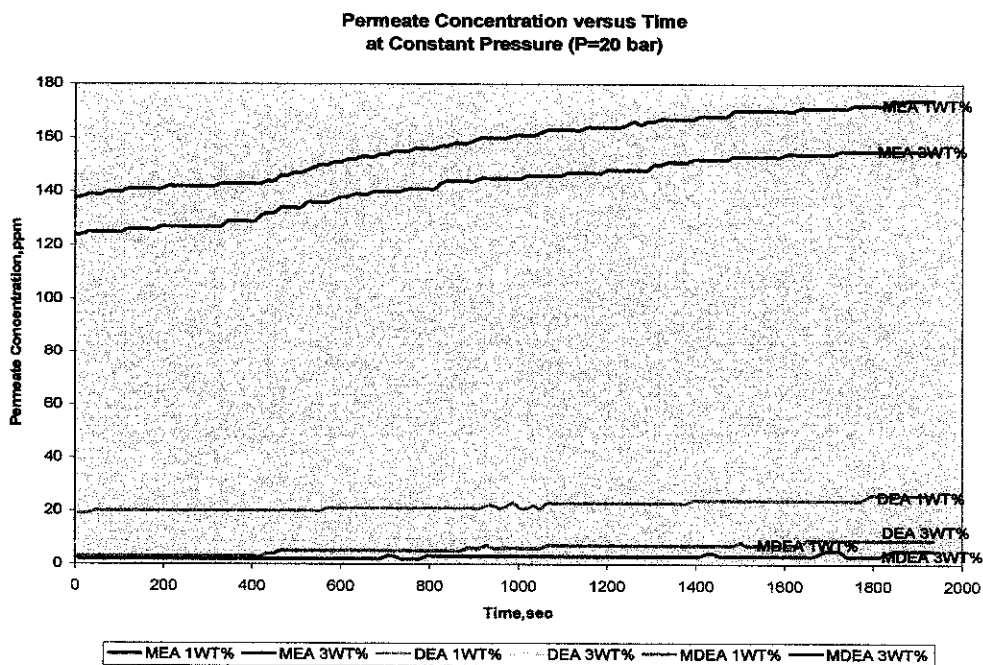


Figure 4.1. 9 Permeate Concentration versus Time at P=20 bar using RO

**Permeate Concentration versus Time
at Constant Pressure (P=30 bar)**

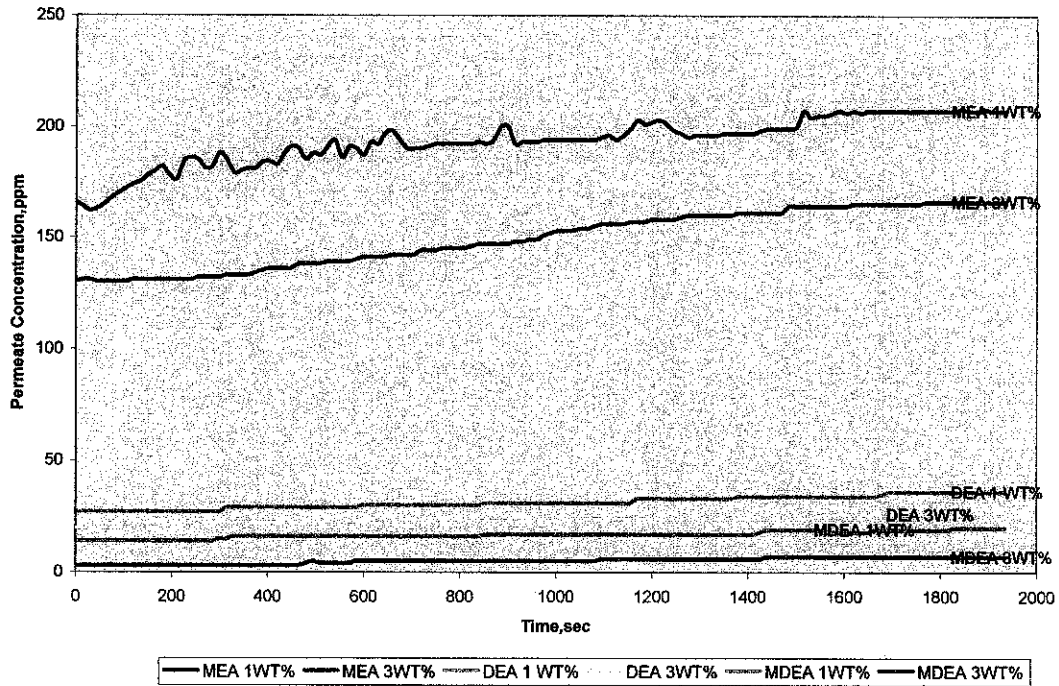


Figure 4.1.10 Permeate Concentration versus Time at P=30 bar using RO

Both of figures show, the separation that had been done at same operating pressure ($p=20$ bar and $P=30$ bar) but in different feed solute concentration in order to study the effect of different feed solute concentration passing through the RO membrane. Figure 4.1.9 and Figure 4.1.10 shows the same trend where 1wt percentage of feed solute concentration will produce higher permeate concentration compared with 3wt percentage of feed solute concentration for both 20 bar and 30 bar applied pressure for three types of amine. This is due to the hydrogen bonding which is stronger in the more concentrated solution compared with the diluted solution which is 1wt percentage. ^[2] Increase in amine concentration means the portion of water is lower, therefore the water flux is lower, and better separation process achieved.

From both figures can see that MDEA at 3wt percentage give the best separation compared with others. It may due to the molecular size of MDEA which is the largest compared with DEA and MEA. Figure 4.1.3 shows clearly, the membrane resistance for

3wtpercentage is higher than 1wt percentage of feed solute concentration. These indicate that the concentrated solution give better separation compared with the diluted solution. As an example, Figure 4.1.6 shows the better separation is obtained for 3wt percentage of MDEA at the same operating pressure. This is due to the membrane resistance of MDEA for 1wt percentage is 4.4674, which is lower than 3wt percentage that is 5.8676.

Another factor that effects the feed solution concentration is the osmotic pressures, which need to be lower the applied pressure to ensure the process of reverse osmosis occurred. The osmotic pressure will increase as the molecular weight and the concentration of solution increases. Since, as more solvent is extracted from the feed solution, the solute concentration becomes higher, and the water flux decreases. Thus, concentration polarization causes the flux of solvent to decrease since the osmotic pressure increases and the overall driving force ($\Delta P - \Delta \pi$) decreases. [3][4]

4.3.2 Ultrafiltration

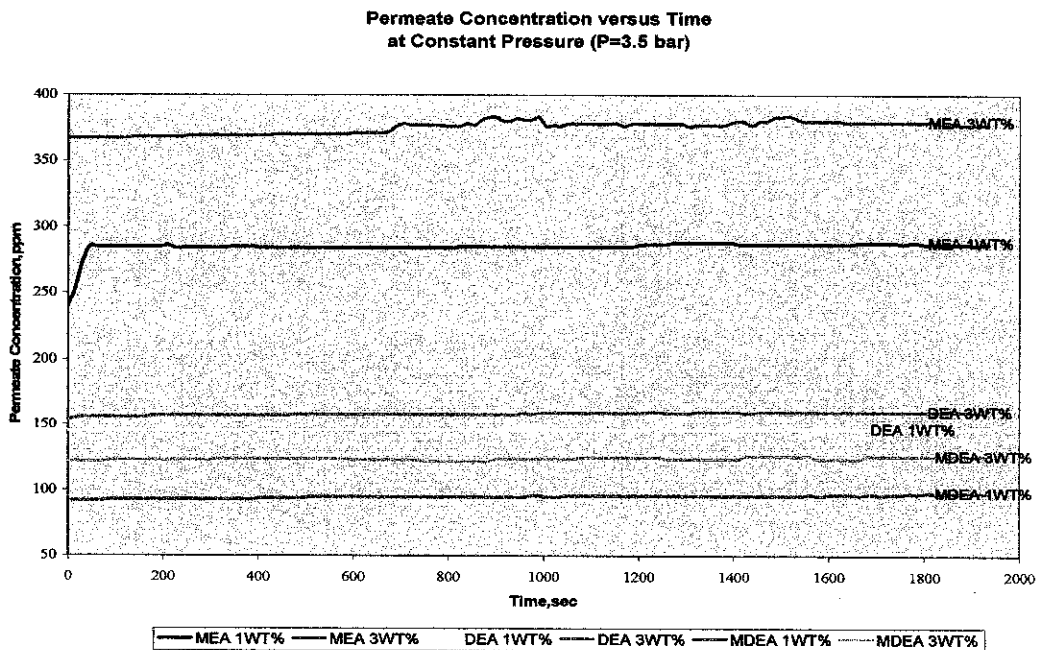


Figure 4.1. 11 Permeate Concentration versus Time at P= 3.5 bar using UF

**Permeate Concentration versus Time
at Constant Pressure (P=4.8 bar)**

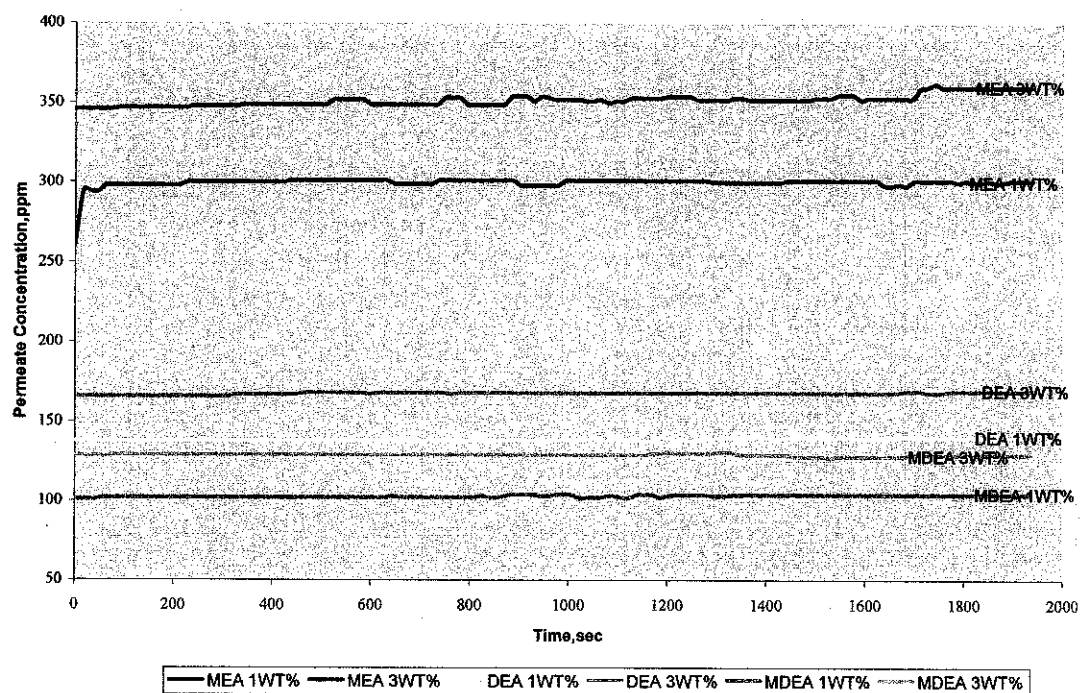


Figure 4.1.12 Permeate Concentration versus Time at P=4.8 bar using UF

Figure 4.1.11 and Figure 4.1.12 have shown the effect of the different in feed solute concentration at the same applied pressure which is 3.5 bar and 4.8 bar passing through the UF membrane system. Both figures gave the same trend which is the feed solute concentration for 3wt percentage will produced higher permeate concentration compared with the feed solute concentration for 1wt percentage. The UF membrane permeate concentration almost the same with the feed concentration or only taking shorter period to be stabilized. It was due to the molecular size, which is smaller than the pores diameter of the membrane causing all the feed solution to pass through the membrane.

The more concentrated feed solution will give higher permeate concentration and will reduced the amine rejection at the retentate or concentrated side. The principle is applied by using UF membrane system rather than using RO membrane system. This is because in the RO membrane system, there is osmotic pressure that prevent the permeate concentration to be higher as the feed solute concentration is increased compared with the

UF membrane system that not apply the principle. Therefore, the higher feed solute concentration will produce more permeate concentration for the UF membrane system. It indicates that, the separation between amine and water is not suitable by using UF membrane. This is because Ultrafiltration is used to separate mixture of different molecular weight proteins. The molecular weight cut off of the membrane is defined as the molecular weight of globular proteins, which are 90% retained by the membrane. A rule of thumb is that the molecular mass must differ by a factor of 10 for a good separation

4.4 Comparison of findings

The experiment had been done for three different types of amine with different operating parameter to investigate the performance of the membrane. The results obtained in this study reveal that the separation of amine from water is feasible by using membrane system, especially RO and MDEA gave the highest amine rejection due to the molecular size of MDEA is larger than the pore size of the RO membrane. Although the results from this project is not equal quantitatively to the theory due to some factors that affecting the performance, the findings still comply with the theory that between the two membrane systems, RO yield the highest salt rejection followed by UF.

Membrane separation holds several advantages over evaporation and other separation processes. A separation process is normally selected based on experience and economic evaluation of the alternatives. Quite often, membrane separation is overlook, although the potential for energy saving is enormous compared with other separation techniques, particularly evaporation. Whether the objective is to recover a valuable solute or to avoid the discharge of polluted water, the energy requirements of evaporation as a separation method are large. To evaporate water in simple single effect evaporator requires an energy input approximately 2,260 kJ/kg. multiple effect evaporation can reduce this by a fraction approaching $1/N$ where N is the number of effects but the required surface area is multiplied by N . vapor recompression can be used to improve the economy of single

evaporator to the equivalent of a 10 to 15 effect system, but the increase in complexity and capital cost is considerable. In contrast to these evaporator methods, a reverse osmosis (RO) system operating at 68 atm with a 60% efficient pump and 50% recovery of permeate, requires only 23 kJ electrical input/kg permeate, which is about 1/100 of the energy required by a simple evaporator and 1/10 that needed by more complex “energy sufficient” evaporation schemes.

In lower pressure Ultrafiltration systems, the energy savings are even greater. Using conservative estimates of capital cost, maintenance and energy requirements, and membrane life, RO unit can replaced an evaporative system in many applications with a payout of one year or less. In some cases, the RO option can make practical the recovery of a valuable but very dilute solute. Even though it may be impractical to concentrate certain solutions fully by membrane separation because of high osmotic pressure, RO can be used to pre-concentrate an evaporator feed, with a substantial energy saving. Aside from energy consideration, RO may have advantages over evaporation in terms of product quality. Because RO is not a thermal process, it can be used to concentrate temperature sensitive material without loss of quality.

Compare to crystallization method where high heat requirement and cost is needed, the membrane process seems more advantageous. Membrane separation is also easier and lower in operating cost compared to the complex distillation method. However, there are some factors in membrane process that should be considered for the commercial amine-water separation. Temperature, process recovery, and pH limitations may become disadvantage factors. Most membrane in the market is provided with pH tolerance between 3 to 10, while the pH of pure amine liquid is about 10 or 11. Higher pH outside the range will only resulting in faster membrane degradation. Furthermore, if the mixture solution is too viscous or has high amine concentration, the driving force for mass transport will be reduced and therefore the filtration rate will also be affected.

Problems associated with the use of RO membrane include potential membrane degradation by chemical action, membrane fouling by particles or precipitation, drops in water flux over time due to mechanical changes (compaction effects) in the membrane and membrane-solute interactions. Membrane polymer-solute interaction consists of sorption of the hydrophobic domain, specific interactions with polymer hydrophilic sites and electrostatic interactions with membrane charged groups. This membrane-solute interaction is a major factor contributing to flux drop.

The focus on the operating problem of membrane fouling reflects the importance of this problem to the reverse osmosis industry. It also reflects the very high performance of current membranes. The best membranes available have salt (NaCl) rejection of greater than 99.5% with corresponding water fluxes of 0.5 m³/m² day. The development of membranes with better salt rejections and/or higher fluxes would enable reverse osmosis operation to operations to operate at lower pressures, but the impact on costs would not be dramatic.

4.5 Error analysis

Changes in operating parameters will have a normal effect on membrane performance. Whilst doing the experiments, the following errors have been identified.

I. Loss of Water Quality

Membrane are rarely perfect and in processes which are concerned with rejecting some material some contamination of permeate will occur or in case the recovering solutes, some material will be lost in permeate. A feature of membrane processes is that the quality of permeate strongly depends on the feed concentration. Changes in operating parameters will result in actual lower quality permeate water, as indicated by an increase in permeate TDS as ppm or conductivity. The main causes of this phenomena is sudden increased in feedwater temperature or decrease in permeate flow, which reduces the water flux and results in less permeate water to dilute the salts that have passed through the membrane. Fouling and damage to the membrane surface, such as exposure to chlorine also allows more salts to pass.

II. Inaccurate readings

The software connected to the equipment is used to record all the parameters during the experiment. However, some of the parameters detected can only be recorded with one decimal place. For example, if the real value is 0.02, the monitor will only display 0.0. Therefore, student has to record the data manually and this promotes to higher human error. The feed analyzer is also suspected malfunction or inaccurate. This is because some of the readings obtained do not comply with the values obtained from other analyzers, which have higher accuracy. Proper and regular maintenance on this equipment should be considered.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

RO is more reliable for the separation of amine from water as compared with the UF membrane. It is because the molecular size of solute is smaller than the pores size of the UF membrane (10nm to 100nm) and shows that almost no separation occurred. However, RO membrane, which has pores size between 1nm to 10 nm, is more suitable to separate amine from water. The membrane resistance in RO is much higher than in UF because the RO membrane has much smaller pore size and compact construction. MDEA give the best separation which is 96.2% compared with MEA and DEA by using RO membrane. This is due to the molecular size of MDEA is the largest among the three types of amine.

The factors affecting membrane performance are feedwater pressure, concentration, temperature, pH, concentration polarization, and the membrane recovery. In RO, as the feed pressure increase, the permeate concentration will reduce. This indicates that more purified water (water flux) is obtained and the separation better separation between amine and water. Meanwhile, if the solute concentration is higher, permeate concentration will be lower which demonstrate that more solute (amine) and the retentate side compared at the permeated side.

However, there is almost no separation take place in the UF membrane. It is due to the molecular size of the solute is smaller than the pore diameter of the membrane. Furthermore, Ultrafiltration membranes are typically rated by molecular weight cutoff, a convenient but fictitious value giving the molecular weight of hypothetical macrosolutes that the membrane will just retain.

Comparing the membrane system with the other technique, the membrane system will give higher potential for energy saving compared with other technique like evaporation, which required larger energy. Aside from energy consideration, RO may have advantages

over evaporation in terms of product quality. Because RO is not a thermal process, it can be used to concentrate temperature sensitive material without loss of quality.

Amine carry over is a commonly discussed in gas plant utilizing amine as a medium to eliminate acid gases from the incoming gas stream. Besides, if improperly discharge had been done, it will pollute the environment. Mixture of water with high amine concentration will resort to high chemical oxygen demand (COD) value, which is a direct measurement of organics contaminants in water. The higher COD value means the higher COD level. In most cases, the use of bio-treated utilizing bacteria as a treatment media is one of the selected solution. However, the extent use of the process is somewhat limited and cannot be used in the case of high COD value. Therefore, the membrane system can be introduced especially RO, in order to separate amine from effluent water prior to discharge. the main objective of introducing the membrane system is to reduce COD value of the effluent to meet the target which is $COD < 100 \text{ mg /L}$.

Few improvements are also recommended for this project for better observation and evaluations.

1. Install feed heater to the equipment

One of the parameter that contributing to get more permeate or product as discussed in the theory. Increases the temperature will increase the production of permeate as well since the temperature is linearly proportional to the temperature. Currently, the RO Pilot system used to conduct the experiments has no temperature regulator or heater that can increase the feedwater temperature. The water regulator is used only to maintain the feedwater temperature at 25°C . Therefore, installing a new heater to the equipment is highly recommended

2. Comparison of different membranes

From the result, shows that the Ultrafiltration is not suitable for the material since most of the permeate concentration is almost the same with the feed concentration. It is because Ultrafiltration usually implies separation of macromolecules such as protein or polyvinyl alcohol from low molecular weight solvents. So, as a recommendation the product or permeate from the reverse osmosis can be run again in to get more purified product while recycling the retentate to feed tank.

3. Install pH Meter

pH is one of the important factors for molecule permeation through the membrane. As discussed previously, at higher pH of the solution, where the molecules are mostly ionized, higher salt rejection will be obtained. So for this purpose, pH meter should be installed at the feed and the permeate side in order to observe the pH of the solutions before and after passing through the membrane. It is because good membrane performance give will reduce the pH after passing through the membrane system.

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APPENDIX A

CO2 Recovery Unit (Benfield Lo-Heat System)

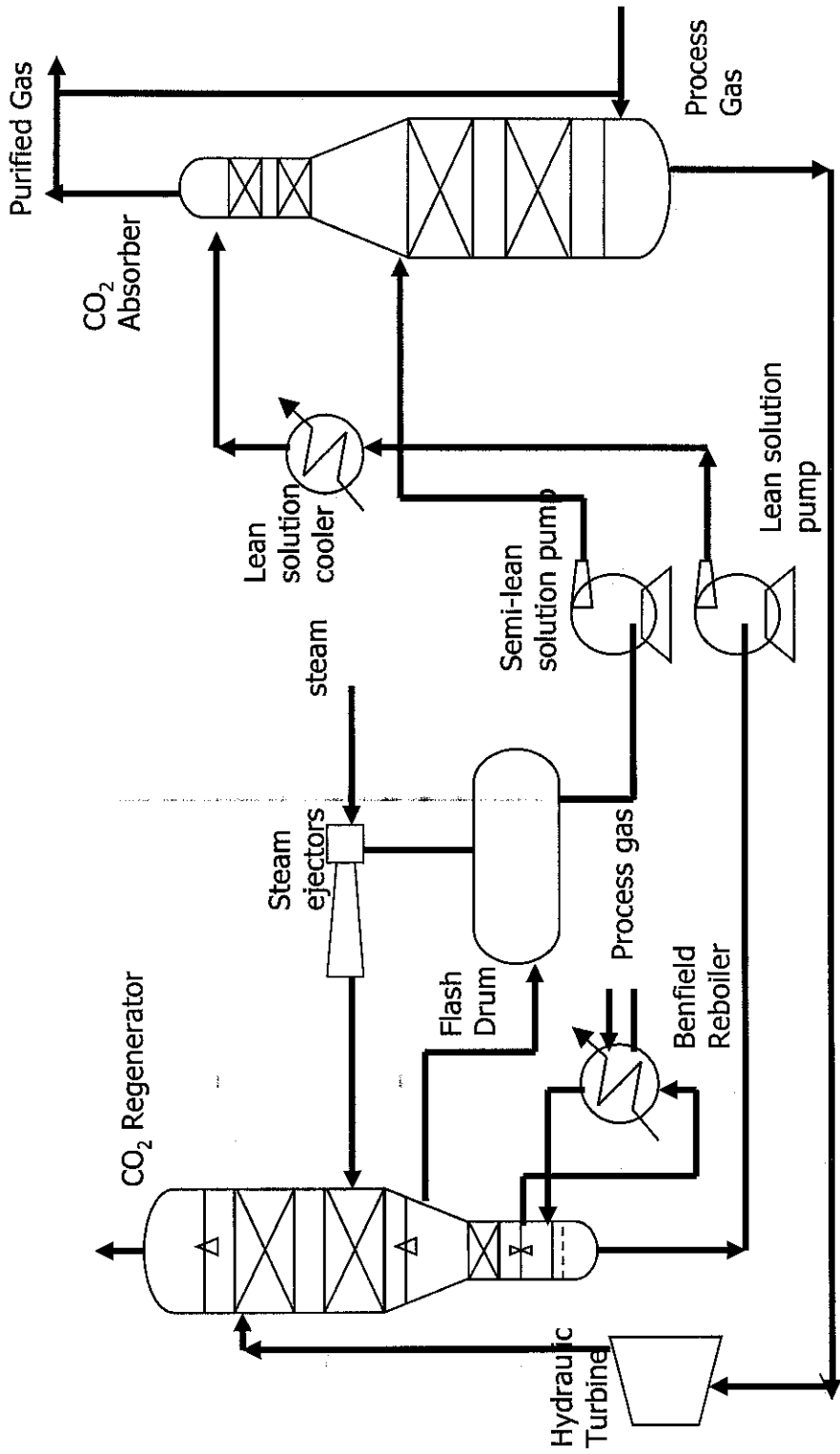


Figure A1 Benfield Unit

APPENDIX B

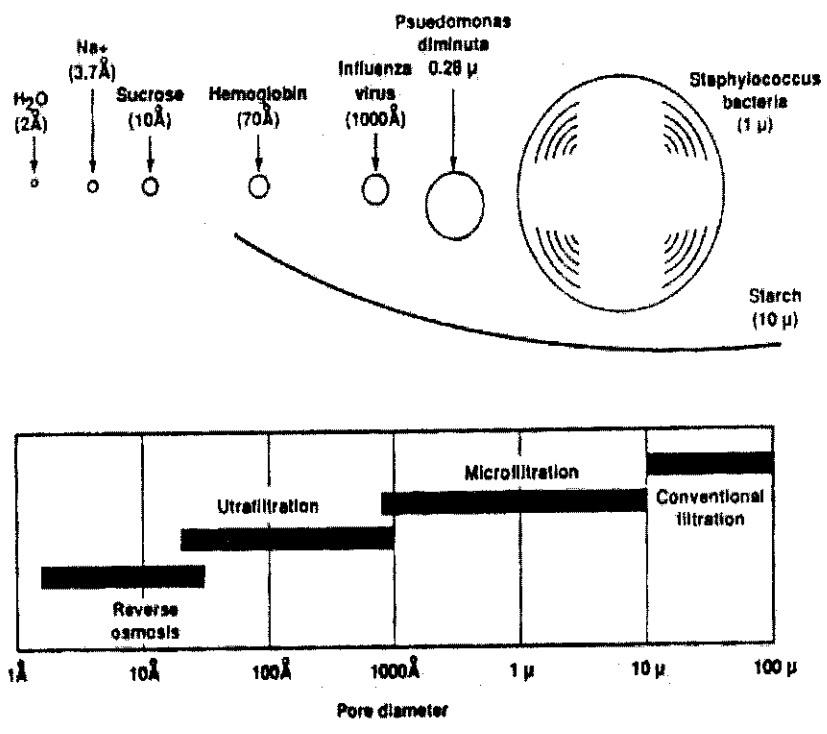


Figure B 1 Reverse Osmosis, Ultrafiltration, Microfiltration and Conventional Filtration are all related process differing principally in the average pore diameter of the membrane filter.

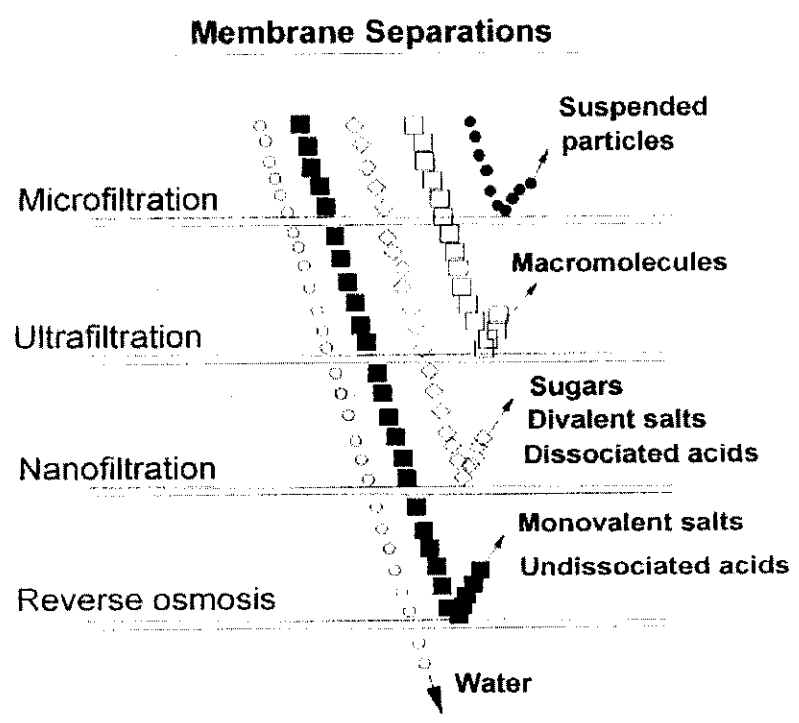


Figure B 2 Pressure-driven membrane processes and their separation characteristics

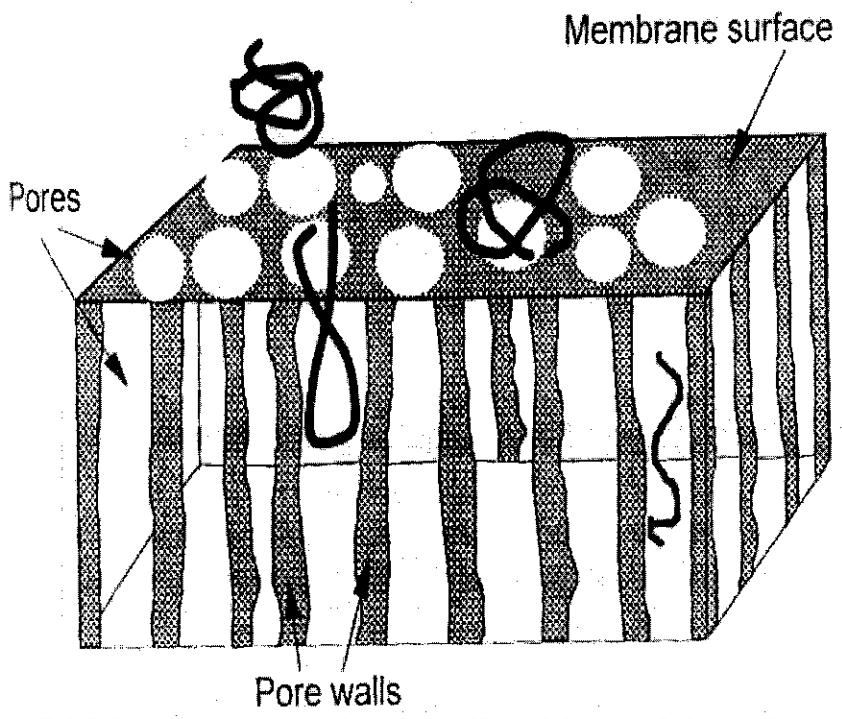


Figure B 3 Schematic representation of the effect of shape and size on the passage of solutes pass through pores.

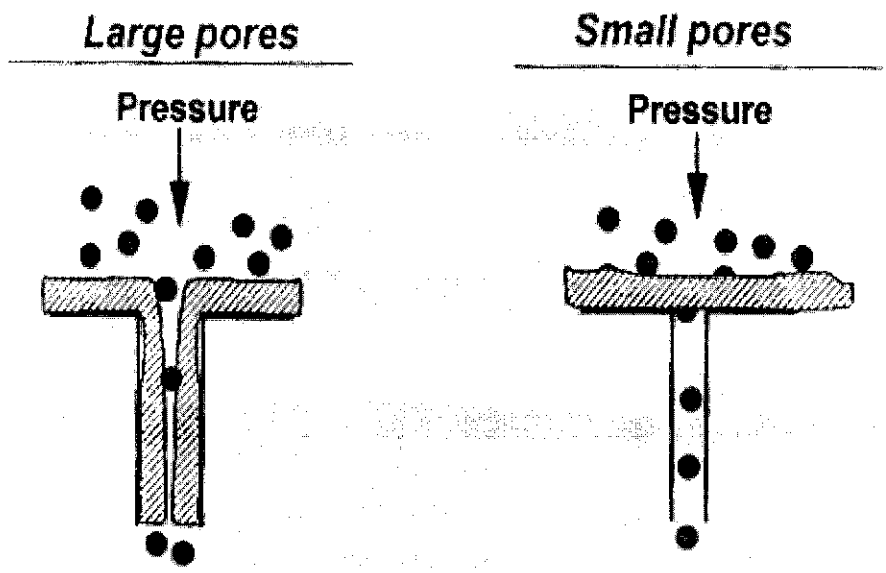


Figure B 4 Permeability of large and small molecules through large and pores membrane

APPENDIX C

Assume that the volume of the tank is 100 liter. The density of the amines as below:

$$\rho_{H_2O} = 1000 \text{ kg/m}^3$$

$$\rho_{MEA} = 1020 \text{ kg/m}^3$$

$$\rho_{DEA} = 1092 \text{ kg/m}^3$$

$$\rho_{MDEA} = 1040 \text{ kg/m}^3$$

Primary Amine (MEA)

$$wt\% = \frac{(V_{MEA} + \rho_{MEA})}{[(V_{MEA} \times \rho_{MEA}) + (V_{H_2O} \times \rho_{H_2O})]}$$

$$wt\% = \frac{(V_{MEA} + 1020)}{[(V_{MEA} \times 1020) + (0.1 - V_{MEA})1000]}$$

$$wt\% = \frac{1020V_{MEA}}{1020V_{MEA} + 100 - 1000V_{MEA}}$$

$$wt\% = \frac{1020V_{MEA}}{20V_{MEA} + 100}$$

If 3 wt% of MEA is used, hence volume of MEA is:

$$3wt\% = \frac{1020V_{MEA}}{20V_{MEA} + 100}$$

$$0.6V_{MEA} + 3 = 1020V_{MEA}$$

$$1019.4V_{MEA} = 3$$

$$V_{MEA} = 0.002943m^3 = 2.943\text{liter}$$

Table C 1 Concentration of Monoethanolamine (MEA)

V _{Tank} (L)	Concentration of Monoethanolamine (MEA)					
	3 wt %		2 wt%		1 wt%	
	V _{MEA} (L)	V _{H2O} (L)	V _{MEA} (L)	V _{H2O} (L)	V _{MEA} (L)	V _{H2O} (L)
80	2.35	77.66	1.57	78.43	0.7844	79.21
60	1.77	58.23	1.18	58.82	0.588	59.41
50	1.47	48.53	0.981	49.02	0.49	49.51
40	1.18	38.82	0.785	39.21	0.39	39.6
35	1.03	33.97	0.69	34.31	0.343	34.66

Secondary Amines (DEA)

$$wt\% = \frac{(V_{DEA} + \rho_{DEA})}{[(V_{DEA} \times \rho_{DEA}) + (V_{H_2O} \times \rho_{H_2O})]}$$

$$wt\% = \frac{(V_{DEA} + 1092)}{[(V_{DEA} \times 1092) + (0.1 - V_{DEA})1000]}$$

$$wt\% = \frac{1092V_{DEA}}{1092V_{DEA} + 100 - 1000V_{DEA}}$$

$$wt\% = \frac{1092V_{DEA}}{92V_{DEA} + 100}$$

If 3 wt% of DEA is used, hence volume of DEA is:

$$3wt\% = \frac{1092V_{MEA}}{92V_{MEA} + 100}$$

$$2.76V_{MEA} + 3 = 1092V_{MEA}$$

$$1089.24V_{MEA} = 3$$

$$V_{MEA} = 0.0027543m^3 = 2.754liter$$

Table C 2 Concentration of Monoethanolamine (DEA)

V _{Tank} (L)	Concentration of Monoethanolamine (DEA)					
	3 wt %		2 wt%		1 wt%	
	V _{DEA} (L)	V _{H2O} (L)	V _{DEA} (L)	V _{H2O} (L)	V _{DEA} (L)	V _{H2O} (L)
80	2.2	77.8	1.47	78.53	0.733	79.27
60	1.65	58.35	1.10	58.9	0.55	59.45
50	1.377	58.27	0.917	49.08	0.458	49.54
40	1.10	38.9	0.7338	39.27	0.367	39.63
35	0.964	34.04	0.642	34.36	0.321	34.68

Tertiary Amines (MDEA)

$$wt\% = \frac{(V_{MDEA} + \rho_{MDEA})}{[(V_{MDEA} \times \rho_{MDEA}) + (V_{H_2O} \times \rho_{H_2O})]}$$

$$wt\% = \frac{(V_{MDEA} + 1040)}{[(V_{MDEA} \times 1040) + (0.1 - V_{MDEA})1000]}$$

$$wt\% = \frac{1040V_{MDEA}}{1040V_{MDEA} + 100 - 1000V_{MDEA}}$$

$$wt\% = \frac{1040V_{MDEA}}{40V_{MDEA} + 100}$$

If 3 wt% of MDEA is used, hence volume of MDEA is:

$$3wt\% = \frac{1040V_{MDEA}}{40V_{MDEA} + 100}$$

$$1.2V_{MDEA} + 3 = 1040V_{MDEA}$$

$$1038.8V_{MDEA} = 3$$

$$V_{MDEA} = 0.002888m^3 = 2.888liter$$

Table C 3 Concentration of Monoethanolamine (MDEA)

V _{Tank} (L)	Concentration of Monoethanolamine (MDEA)					
	3 wt %		2 wt%		1 wt%	
	V _{MDEA} (L)	V _{H2O} (L)	V _{MDEA} (L)	V _{H2O} (L)	V _{MDEA} (L)	V _{H2O} (L)
80	2.31	77.69	1.54	78.46	0.7695	79.23
60	1.723	58.2	1.155	58.85	0.577	59.42
50	1.444	48.56	0.962	49.04	0.481	49.52
40	1.16	38.84	0.7698	39.23	0.385	39.62
35	1.01	33.99	0.6736	34.32	0.337	34.66

Concentration of MDEA (MDEA)

APPENDIX D

Table D 1 Concentration of Amines at 3.5 bar for Ultrafiltration

1 WT%				3 WT%		
Time, Seconds	MEA, ppm	DEA, ppm	MDEA, ppm	MEA, ppm	DEA, ppm	MDEA, ppm
0	240	145	92	367	154	122
15	253	145	92	367	155	122
30	273	145	92	367	156	122
45	285	145	92	367	156	122
60	285	144	92	367	156	122
75	285	145	92	367	156	122
90	285	145	93	367	156	123
105	285	145	93	367	156	123
120	285	145	93	367	156	123
135	285	145	93	368	156	123
150	285	145	93	368	156	123
165	285	145	93	368	156	123
180	285	145	93	368	157	123
195	285	145	93	368	157	123
210	286	145	93	368	157	123
225	284	145	93	368	157	123
240	284	145	93	368	157	123
255	284	145	93	369	157	123
270	284	145	93	369	157	123
285	284	145	93	369	157	123
300	284	145	93	369	157	123
315	284	145	93	369	157	124
330	284	146	93	369	157	124
345	285	145	93	369	157	124
360	285	145	93	369	157	124
375	285	146	93	369	157	124
390	285	146	93	369	157	124
405	284	145	94	369	157	124
420	284	146	94	369	157	124
435	284	146	94	370	157	124
450	284	146	94	370	157	124
465	284	146	94	370	157	124
480	284	146	94	370	158	124

495	284	146	94	370	158	124
510	284	146	95	370	158	124
525	284	146	95	370	158	124
540	284	146	95	370	158	124
555	284	146	95	370	158	124
570	284	146	95	370	158	124
585	284	146	95	370	158	124
600	284	146	95	371	158	123
615	284	146	95	371	158	123
630	284	146	95	371	158	123
645	284	146	95	371	158	123
660	284	146	95	371	158	123
675	284	146	95	372	158	123
690	284	146	95	376	158	123
705	284	146	95	378	158	123
720	284	146	95	377	158	123
735	284	146	95	377	158	123
750	284	146	95	377	158	122
765	284	146	95	377	158	122
780	284	146	95	377	158	122
795	284	146	95	376	158	122
810	284	146	95	376	158	122
825	285	146	95	376	158	122
840	285	146	95	378	158	122
855	285	146	95	377	158	122
870	285	146	95	381	158	122
885	285	146	95	383	158	122
900	285	146	95	383	158	124
915	285	146	95	380	158	124
930	285	146	95	380	158	124
945	285	146	95	382	158	124
960	285	146	95	381	159	124
975	285	146	96	381	158	124
990	285	146	96	383	159	124
1005	285	146	95	376	159	124
1020	285	146	95	377	159	124
1035	285	146	95	376	159	124
1050	285	146	96	378	159	124
1065	285	146	96	378	159	124

1080	285	146	96	378	159	125
1095	285	146	96	378	159	125
1110	285	146	96	378	159	125
1125	285	146	96	378	159	125
1140	285	146	96	378	159	125
1155	285	146	96	378	159	125
1170	285	146	96	376	159	125
1185	285	146	96	378	159	125
1200	286	146	96	378	159	125
1215	287	146	96	378	160	125
1230	287	147	96	378	160	125
1245	287	148	96	378	160	125
1260	287	146	96	378	159	124
1275	288	146	96	378	159	124
1290	288	146	96	378	159	124
1305	288	146	96	376	159	124
1320	288	146	96	377	160	124
1335	288	146	96	377	160	124
1350	288	146	96	377	160	124
1365	288	146	96	377	160	124
1380	288	146	96	377	160	124
1395	288	147	96	379	160	124
1410	287	147	96	380	160	124
1425	287	147	96	379	159	126
1440	287	147	96	377	159	126
1455	287	147	96	380	160	126
1470	287	146	96	380	160	126
1485	287	146	96	383	160	126
1500	287	147	96	383	160	126
1515	287	147	96	384	160	126
1530	287	146	96	382	160	126
1545	287	147	96	380	160	126
1560	287	147	97	380	160	126
1575	287	147	96	380	160	124
1590	287	147	96	380	160	124
1605	287	147	97	380	160	124
1620	287	147	97	380	160	124
1635	287	147	97	379	160	124
1650	287	147	97	379	160	124

1665	288	147	96	379	160	124
1680	288	147	97	379	160	126
1695	288	147	97	379	160	126
1710	288	147	96	379	160	126
1725	288	147	97	379	160	126
1740	288	147	97	379	160	126
1755	287	147	97	379	160	126
1770	288	147	98	379	160	126
1785	288	147	97	379	160	126
1800	287	147	98	379	160	126
1815	287	147	98	379	160	126
1830	287	147	97	379	160	126
1845	287	147	98	378	160	126
1860	287	147	98	378	161	126
1875	287	147	98	377	160	126
1890	287	147	98	377	160	126
1905	286	147	98	377	160	126
1920	287	148	98	377	160	126
1935	287	148	98	377	161	126

Table D 2 Concentration of Amines at 4.8 bar for Ultrafiltration

Time, Seconds	1 WT%			3 WT%		
	MEA, ppm	DEA, ppm	MDEA, ppm	MEA, ppm	DEA, ppm	MDEA, ppm
0	260	138	101	346	166	129
15	295	138	101	346	166	129
30	294	138	101	346	166	128
45	294	138	101	346	166	129
60	298	138	102	346	166	128
75	298	138	102	346	166	129
90	298	138	102	347	166	129
105	298	138	102	347	166	129
120	298	138	102	347	166	129
135	298	138	102	347	166	129
150	298	138	102	347	166	129
165	298	138	102	347	166	129
180	298	138	102	347	166	129
195	298	138	102	347	166	129
210	298	138	102	347	166	129
225	300	138	102	347	166	129
240	300	138	102	348	166	129
255	300	138	102	348	166	129
270	300	138	102	348	166	129
285	300	138	102	348	166	129
300	300	138	102	348	166	129
315	300	138	102	348	166	129
330	300	138	102	348	167	129
345	300	138	102	349	167	129
360	300	138	102	349	167	129
375	300	138	102	349	167	129
390	300	138	102	349	167	129
405	300	138	102	349	167	129
420	300	138	102	349	167	129
435	301	138	102	349	167	129
450	301	138	102	349	167	129
465	301	138	102	349	168	129
480	301	138	102	349	168	129
495	301	138	102	349	168	129

510	301	138	102	349	168	129
525	301	138	102	352	168	129
540	301	138	102	352	168	129
555	301	138	102	352	168	129
570	301	138	102	352	168	129
585	301	138	102	352	168	129
600	301	138	102	349	167	129
615	301	138	102	349	168	129
630	301	138	102	349	168	129
645	299	138	103	349	168	129
660	299	138	102	349	168	129
675	299	138	102	349	168	129
690	299	138	102	349	168	129
705	299	138	102	349	168	129
720	299	138	102	349	168	129
735	301	138	102	349	168	129
750	301	138	102	353	168	129
765	301	138	102	353	167	129
780	301	138	102	353	168	129
795	301	138	102	349	168	129
810	301	138	102	349	168	129
825	301	138	103	349	168	129
840	301	138	102	349	168	129
855	301	138	102	349	168	129
870	301	138	103	349	168	129
885	301	138	104	354	168	129
900	298	139	104	354	168	129
915	298	138	104	354	168	129
930	298	138	104	351	168	129
945	298	138	103	354	168	129
960	298	138	103	353	168	129
975	298	138	104	352	168	129
990	301	139	104	352	168	129
1005	301	139	104	352	168	129
1020	301	139	102	352	168	129
1035	301	139	102	351	168	129
1050	301	140	102	351	168	129
1065	301	139	102	352	168	129
1080	301	138	103	350	168	129

1095	301	139	103	351	168	129
1110	301	139	102	351	168	129
1125	301	138	102	353	168	129
1140	301	138	104	353	168	129
1155	301	138	104	353	168	129
1170	301	138	104	353	168	130
1185	301	140	102	353	168	130
1200	301	140	103	354	168	131
1215	301	139	104	354	168	130
1230	301	139	104	354	168	130
1245	301	138	104	354	168	130
1260	301	139	104	352	168	130
1275	301	139	104	352	168	130
1290	300	139	103	352	168	130
1305	300	140	103	352	168	131
1320	300	140	103	352	168	131
1335	300	140	104	353	168	130
1350	300	140	104	353	168	129
1365	300	140	104	352	168	129
1380	300	140	104	352	168	129
1395	300	140	104	352	168	129
1410	300	140	104	352	168	129
1425	300	139	104	352	168	129
1440	301	140	104	352	168	129
1455	301	140	104	352	168	128
1470	301	140	104	352	168	128
1485	301	140	104	352	168	128
1500	301	140	104	353	168	128
1515	301	140	104	353	168	128
1530	301	140	104	353	168	127
1545	301	140	104	355	168	128
1560	301	140	104	355	168	128
1575	301	140	104	355	168	128
1590	301	139	104	352	168	128
1605	301	139	104	353	168	128
1620	301	139	104	353	168	128
1635	298	140	104	353	168	128
1650	298	140	104	353	168	128
1665	299	140	104	353	168	128

1680	298	140	104	353	169	128
1695	301	140	104	353	169	129
1710	301	140	104	359	169	129
1725	301	140	104	360	168	129
1740	301	140	104	362	168	129
1755	301	140	104	360	168	129
1770	301	140	104	360	169	129
1785	300	140	104	360	169	129
1800	301	140	104	360	169	129
1815	300	140	104	360	169	129
1830	300	140	104	360	169	129
1845	301	140	104	360	169	129
1860	300	140	104	360	169	129
1875	300	140	104	360	169	129
1890	301	140	104	360	169	129
1905	301	140	104	360	169	129
1920	301	140	104	360	169	129
1935	301	140	104	360	169	129

Table D 3 Concentration of Amines at 20 bar for Reverse Osmosis

Time Seconds	1 WT%			3 WT%		
	MEA ppm	DEA ppm	MDEA ppm	MEA ppm	DEA ppm	MDEA ppm
0	138	8	3	124	19	3
15	138	8	3	124	19	2
30	139	8	3	125	19	2
45	139	8	3	125	20	2
60	139	8	3	125	20	2
75	140	8	3	125	20	2
90	140	8	3	125	20	2
105	140	8	3	125	20	2
120	141	8	3	126	20	2
135	141	8	3	126	20	2
150	141	8	3	126	20	2
165	141	8	3	126	20	2
180	141	8	3	126	20	2
195	141	8	3	127	20	2
210	142	8	3	127	20	2
225	142	8	3	127	20	2
240	142	8	3	127	20	2
255	142	8	3	127	20	2
270	142	8	3	127	20	2
285	142	8	3	127	20	2
300	142	8	3	127	20	2
315	142	8	3	127	20	2
330	143	8	3	127	20	2
345	143	8	3	129	20	2
360	143	8	3	129	20	2
375	143	8	3	129	20	2
390	143	8	3	129	20	2
405	143	8	3	129	20	2
420	143	8	3	131	20	2
435	144	8	4	132	20	2
450	144	8	4	132	20	2
465	146	8	5	134	20	2
480	146	8	5	134	20	2

495	147	8	5	134	20	2
510	147	8	5	134	20	2
525	148	8	5	136	20	2
540	149	8	5	136	20	2
555	150	8	5	136	20	2
570	150	8	5	136	21	2
585	151	9	5	137	21	2
600	151	9	5	138	21	2
615	152	9	5	138	21	2
630	152	8	5	139	21	2
645	153	8.5	5	139	21	2
660	153	9	5	139	21	2
675	153	9	5	140	21	2
690	154	9	5	140	21	2
705	154	9	5	140	21	3
720	155	9	5	140	21	3
735	155	9	5	140	21	2
750	155	9	5	141	21	2
765	156	9	5	141	21	2
780	156	9	5	141	21	2
795	156	9	5	141	21	3
810	156	9	5	141	21	3
825	157	9	5	143	21	3
840	157	9	5	144	21	3
855	158	9	5	144	21	3
870	158	9	5	144	21	3
885	158	9	6	144	21	3
900	159	9	6	144	21	3
915	160	9	6	145	21	3
930	160	10	7	145	22	3
945	160	10	6	145	22	3
960	160	9	6	145	21	3
975	160	9	6	145	22	3
990	161	9	6	145	23	3
1005	161	9	6	145	21	3
1020	161	9	6	146	21	3
1035	161	9	6	146	22	3
1050	162	9	6	146	21	3
1065	163	10	7	146	23	3

1080	163	10	7	146	23	3
1095	163	9	7	146	23	3
1110	163	9	7	147	23	3
1125	163	9	7	147	23	3
1140	163	10	7	147	23	3
1155	164	10	7	147	23	3
1170	164	10	7	147	23	3
1185	164	10	7	147	23	3
1200	164	10	7	148	23	3
1215	164	10	7	148	23	3
1230	164	10	7	148	23	3
1245	165	10	7	148	23	3
1260	166	10	7	148	23	3
1275	165	10	7	148	23	3
1290	166	10	7	148	23	3
1305	166	10	7	150	23	3
1320	167	10	7	150	23	3
1335	167	10	7	151	23	3
1350	167	10	7	151	23	3
1365	167	10	7	151	23	3
1380	167	10	7	151	23	3
1395	167	10	7	152	24	3
1410	168	10	7	152	24	3
1425	168	10	7	152	24	4
1440	168	10	7	152	24	4
1455	168	10	7	152	24	3
1470	168	10	7	152	24	3
1485	170	10	7	153	24	3
1500	170	10	8	153	24	3
1515	170	10	7	153	24	3
1530	170	10	7	153	24	3
1545	170	11	7	153	24	3
1560	170	11	7	153	24	3
1575	170	12	7	153	24	3
1590	170	12	7	153	24	3
1605	170	12	7	154	24	3
1620	170	12	7	154	24	3
1635	171	12	7	154	24	3
1650	171	12	9	154	24	3

1665	171	12	9	154	24	3
1680	171	12	9	154	24	4
1695	171	12	9	154	24	5
1710	171	12	9	154	24	5
1725	171	12	9	155	24	5
1740	171	12	9	155	24	3
1755	172	12	9	155	24	3
1770	172	12	9	155	24	3
1785	172	12	9	155	25	3
1796	172	12	9	155	26	3
1815	172	12	9	155	26	3
1830	172	12	9	155	26	4
1845	174	12	9	155	26	5
1860	173	12	9	155	26	5
1875	174	12	9	155	26	5
1890	174	12	9	155	26	5
1905	174	12	9	155	26	5
1920	174	12	9	155	26	5
1935	174	12	9	155	26	5

Table D 4 Concentration of Amines at 30 bar for Reverse Osmosis

Time Seconds	1 WT%			3 WT%		
	MEA ppm	DEA ppm	MDEA ppm	MEA ppm	DEA ppm	MDEA ppm
0	166	20	14	130	27	3
15	164	20	14	131	27	3
30	162	20	14	131	27	3
45	163	20	14	130	27	3
60	165	20	14	130	27	3
75	168	20	14	130	27	3
90	170	20	14	130	27	3
105	172	20	14	130	27	3
120	174	20	14	131	27	3
135	175	20	14	131	27	3
150	178	20	14	131	27	3
165	180	20	14	131	27	3
180	182	20	14	131	27	3
195	178	21	14	131	27	3
210	176	22	14	131	27	3
225	184	21	14	131	27	3
240	186	20	14	131	27	3
255	185	21	14	132	27	3
270	181	22	14	132	27	3
285	182	22	14	132	27	3
300	188	22	15	132	27	3
315	185	22	15	133	29	3
330	179	22	16	133	29	3
345	180	22	16	133	29	3
360	181	22	16	133	29	3
375	181	22	16	134	29	3
390	184	22	16	135	29	3
405	184	22	16	136	29	3
420	183	22	16	136	29	3
435	188	22	16	136	29	3
450	191	22	16	136	29	3
465	190	22	16	138	29	3
480	185	22	16	138	29	4

495	188	22	16	138	29	5
510	187	22	16	138	29	4
525	191	22	16	139	29	4
540	194	22	16	139	29	4
555	186	22	16	139	29	4
570	191	22	16	139	29	4
585	190	22	16	140	29	5
600	187	22	16	141	30	5
615	193	22	16	141	30	5
630	192	22	16	141	30	5
645	197	22	16	141	30	5
660	198	23	16	142	30	5
675	194	23	16	142	30	5
690	190	23	16	142	30	5
705	190	23	16	142	30	5
720	190	23	16	144	30	5
735	191	23	16	144	30	5
750	192	22	16	144	30	5
765	192	22	16	145	30	5
780	192	23	16	145	30	5
795	192	23	16	145	30	5
810	192	23	16	145	30	5
825	192	23	16	146	30	5
840	193	23	16	147	30	5
855	192	23	17	147	31	5
870	194	23	17	147	31	5
885	200	23	17	147	31	5
900	200	23	17	147	31	5
915	192	23	17	148	31	5
930	193	23	17	148	31	5
945	193	23	17	149	31	5
960	193	23	17	149	31	5
975	194	23	17	151	31	5
990	194	23	17	152	31	5
1005	194	23	17	153	31	5
1020	194	24	17	153	31	5
1035	194	23	17	153	31	5
1050	194	23	17	154	31	5
1065	194	24	17	154	31	5

1080	194	24	17	155	31	5
1095	195	24	17	156	31	6
1110	196	24	17	156	31	6
1125	194	24	17	156	31	6
1140	196	24	17	156	31	6
1155	199	24	17	157	31	6
1170	203	24	17	157	33	6
1185	201	24	17	157	33	6
1200	202	24	17	158	33	6
1215	203	24	17	158	33	6
1230	201	24	17	158	33	6
1245	198	24	17	158	33	6
1260	197	24	17	159	33	6
1275	195	24	17	160	33	6
1290	196	24	17	160	33	6
1305	196	24	17	160	33	6
1320	196	24	17	160	33	6
1335	196	24	17	160	33	6
1350	197	24	17	160	33	6
1365	197	24	17	160	33	6
1380	197	24	17	161	34	6
1395	197	24	17	161	34	6
1410	197	24	17	161	34	6
1425	198	24	18	161	34	6
1440	199	24	19	161	34	7
1455	199	24	19	161	34	7
1470	199	25	19	161	34	7
1485	199	24	19	164	34	7
1500	200	24	19	164	34	7
1515	207	24	19	164	34	7
1530	204	25	19	164	34	7
1545	205	25	19	164	34	7
1560	205	26	18	164	34	7
1575	206	26	18	164	34	7
1590	207	26	18	164	34	7
1605	206	26	18	164	34	7
1620	207	26	18	165	34	7
1635	206	26	18	165	34	7
1650	207	26	18	165	34	7

1665	207	26	19	165	34	7
1680	207	26	19	165	35	7
1695	207	26	19	165	36	7
1710	207	26	19	165	36	7
1725	207	26	19	165	36	7
1740	207	26	19	165	36	7
1755	207	26	19	165	36	7
1770	207	26	19	166	36	7
1785	207	26	19	166	36	7
1800	207	26	19	166	36	7
1815	207	27	19	166	36	7
1830	207	27	20	166	36	7
1845	208	27	20	166	36	7
1860	207	27	20	166	36	7
1875	207	27	20	166	36	7
1890	208	27	20	166	36	7
1905	207	27	20	166	36	7
1920	207	27	20	166	36	7
1935	207	27	20	166	36	7

Table D 5 Percentage of amine rejection at different operating condition

Process	Operating at constant	Types of Amine	Permeate wt%	% Rejection
UF 1 wt%	P = 3.5 bar	MEA	1.209	-
		DEA	1.698	-
		MDEA	1.114	-
	P = 4.8 bar	MEA	1.249	-
		DEA	1.877	-
		MDEA	1.095	-
UF 3 wt%	P = 3.5 bar	MEA	2.98	-
		DEA	2.92	-
		MDEA	2.57	-
	P = 4.8 bar	MEA	2.95	-
		DEA	2.89	-
		MDEA	2.77	-
RO 1 wt%	P = 20 bar	MEA	0.799	20.1
		DEA	0.760	24.0
		MDEA	0.732	26.8
	P = 30 bar	MEA	0.653	34.7
		DEA	0.573	42.7
		MDEA	0.486	51.4
RO 3 wt%	P = 20 bar	MEA	0.688	77.1
		DEA	0.589	80.4
		MDEA	0.464	84.5
	P = 30 bar	MEA	0.442	85.3
		DEA	0.352	88.1
		MDEA	0.113	96.2

Table D 6 Membrane resistance for 1wt% concentration of amines

Process	Types of Amine	Pressure (Bar)		Pressure drop, ΔP	Flux, J(L/min)	Membrane Resistance, R_m
		Feed	Permeate			
UF	MEA	3.5	1.27	2.23	12	0.1858
		4.8	1.94	2.86	14.92	0.1917
	DEA	3.5	1.04	2.46	12.01	0.2048
		4.8	1.80	3.00	14.30	0.2098
	MDEA	3.5	1.03	2.47	12.15	0.2033
		4.8	1.82	2.98	14.48	0.2058
RO	MEA	20	0.04	19.96	2.43	8.2140
		30	0.15	29.85	4.32	6.9097
	DEA	20	0.14	19.86	4.29	4.6294
		30	0.39	29.61	6.91	4.2851
	MDEA	20	0.12	19.88	4.45	4.4674
		30	0.4	29.60	7.13	4.1515

Table D 7 Membrane resistance for 3 wt% concentration of amines

Process	Types of Amine	Pressure (Bar)		Pressure drop, ΔP	Flux, J(L/min)	Membrane Resistance, R_m
		Feed	Permeate			
UF	MEA	3.5	1.11	2.39	12.22	0.1956
		4.8	1.89	2.91	14.67	0.1984
	DEA	3.5	1.02	2.48	12.00	0.2067
		4.8	1.76	3.04	14.13	0.2151
	MDEA	3.5	1.08	2.42	12.53	0.1931
		4.8	1.88	2.92	14.85	0.1966
RO	MEA	20	0.01	19.99	1.61	12.4161
		30	0.11	29.89	4.03	7.4169
	DEA	20	0.09	19.91	3.21	6.2025
		30	0.22	29.78	5.12	5.8164
	MDEA	20	0.05	19.95	3.40	5.8676
		30	0.20	29.80	5.34	5.5805

Table D 8 Osmotic pressure for MEA, DEA and MDEA

Concentration		Molecular Weight(kg/kmol)	Density(kg/m ³)	Osmotic Pressure(Bar)
MEA	1 wt%	61.08	1020	8.090
	3 wt%	61.08	1020	18.71
DEA	1 wt%	105.14	1092	4.710
	3 wt%	105.14	1092	14.37
MDEA	1 wt%	119.16	1040	4.150
	3 wt%	119.16	1040	12.75

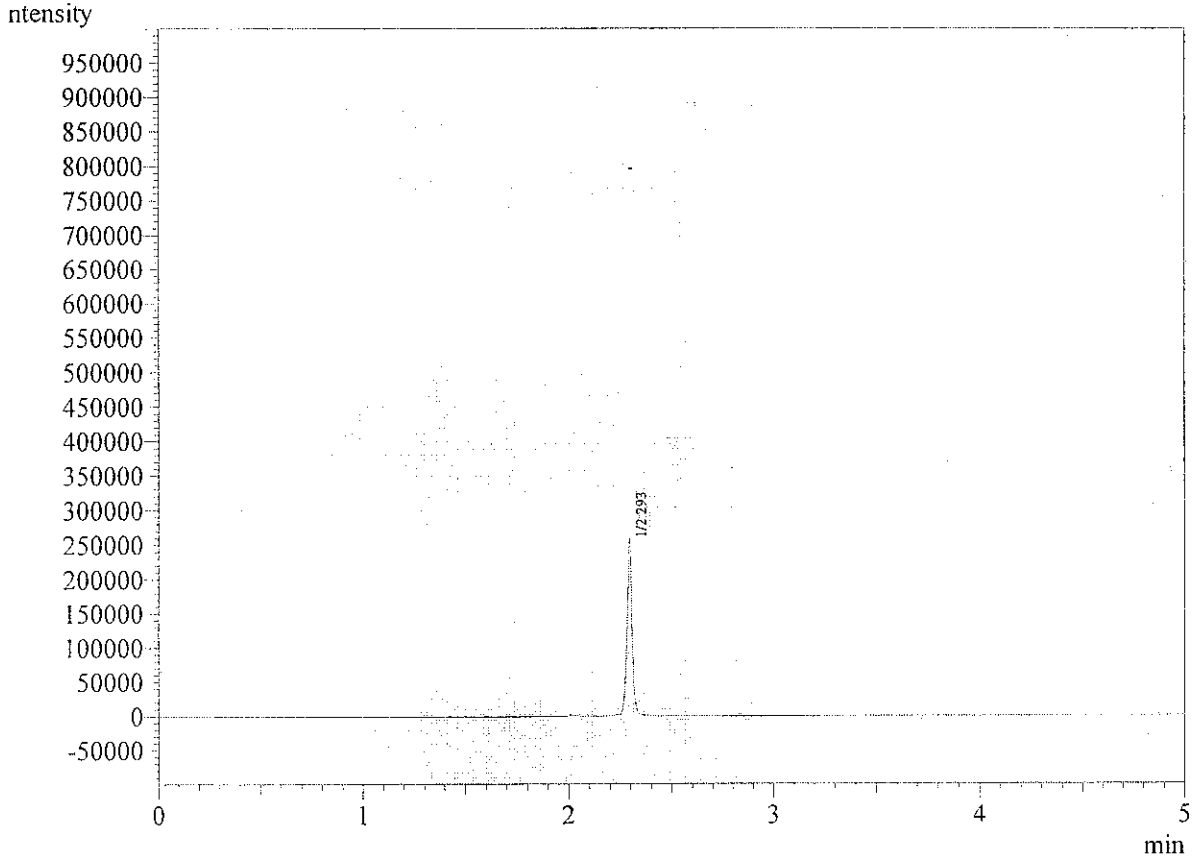
MEA-UF-P = 3.5 BAR

Sample Information

analysis Date & Time : 9/19/2005 11:05:56 PM

Sample Name : uf 1w% s130 p1

Chromatogram - Channel 1 uf 1w% s130 p1 C:\GCsolution\Data\fab\FYP\amiza_004.gcd



Peak Table - Channel 1

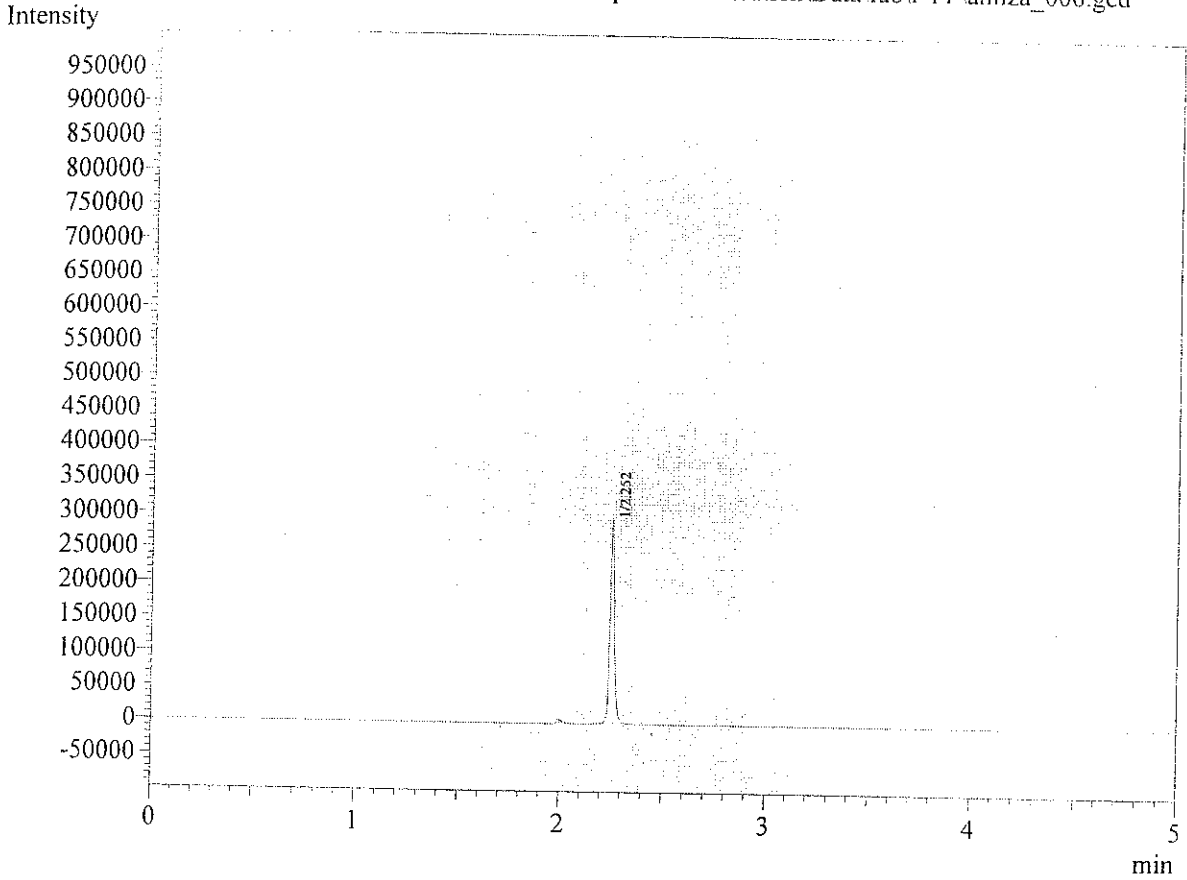
Peak#	Ret.Time	Area	Area%	Height	Height%	A/H	Conc.	Unit	ID#
1	2.293	441858	100.0000	256711	100.0000	1.721	1.209 %		1
Total		441858	100.0000	256711	100.0000				

Sample Information

Analysis Date & Time : 9/19/2005 11:26:11 PM

Sample Name : uf 1w% s40 p3

Chromatogram - Channel 1 uf 1w% s40 p3 C:\GCsolution\Data\fab\FYP\amiza_006.gcd



Peak Table - Channel 1							
Peak#	Ret. Time	Area	Area%	Height	Height%	A/H	Conc. Unit IID#
1	2.252	456463	100.0000	297251	100.0000	1.536	1.249 % 1
Total		456463	100.0000	297251	100.0000		

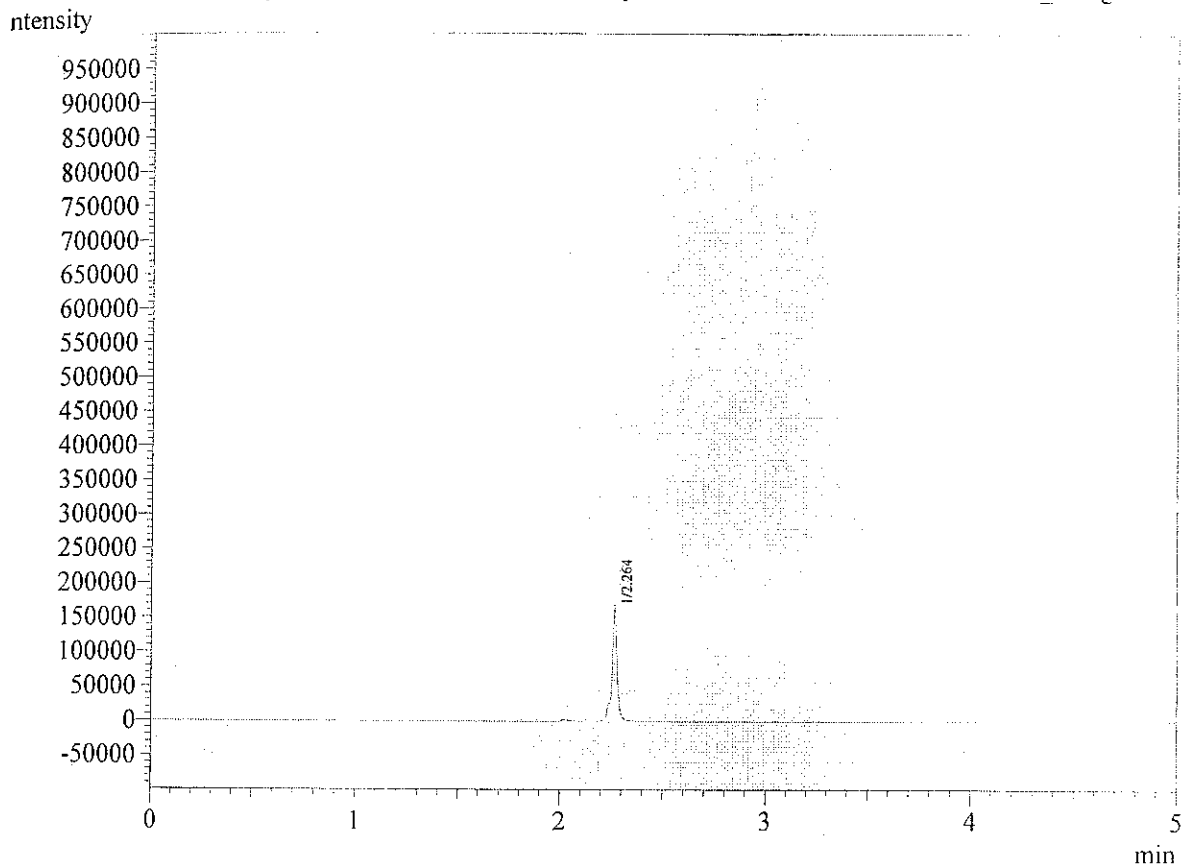
MEA(3 WT%)-RO-P = 20 BAR

Sample Information

Analysis Date & Time : 9/19/2005 11:56:30 PM

Sample Name : RO 1wt% feed p1

Chromatogram - Channel 1 RO 1wt% feed p1 C:\GCsolution\Data\fab\FYP\amiza_009.gcd



Peak Table - Channel 1							
Peak#	Ret. Time	Area	Area%	Height	Height%	A/H	Conc. Unit 1 ID#
1	2.264	292153	100.0000	166754	100.0000	1.752	0.799 % 1
Total		292153	100.0000	166754	100.0000		

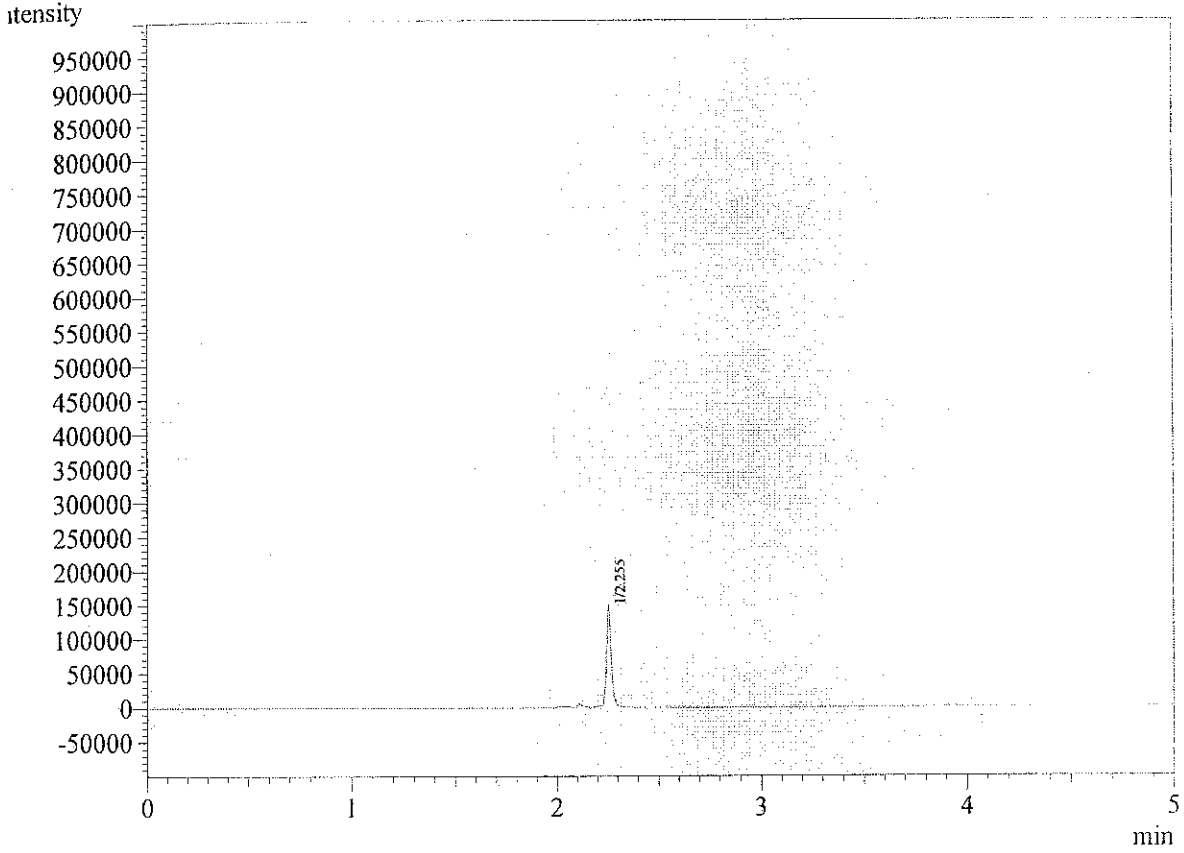
MEA(3 WT%)-RO-P = 30 BAR

Sample Information

Analysis Date & Time : 9/20/2005 12:26:47 AM

Sample Name : RO 1wt% feed p2

Chromatogram - Channel 1 RO 1wt% feed p2 C:\GCsolution\Data\fab\FYP\amiza_012.gcd



Peak Table - Channel 1								
Peak#	Ret.Time	Area	Area%	Height	Height%	A/H	Conc.	Unit 1 ID#
1	2.255	238722	100.0000	147896	100.0000	1.614	0.653 %	1
Total		238722	100.0000	147896	100.0000			

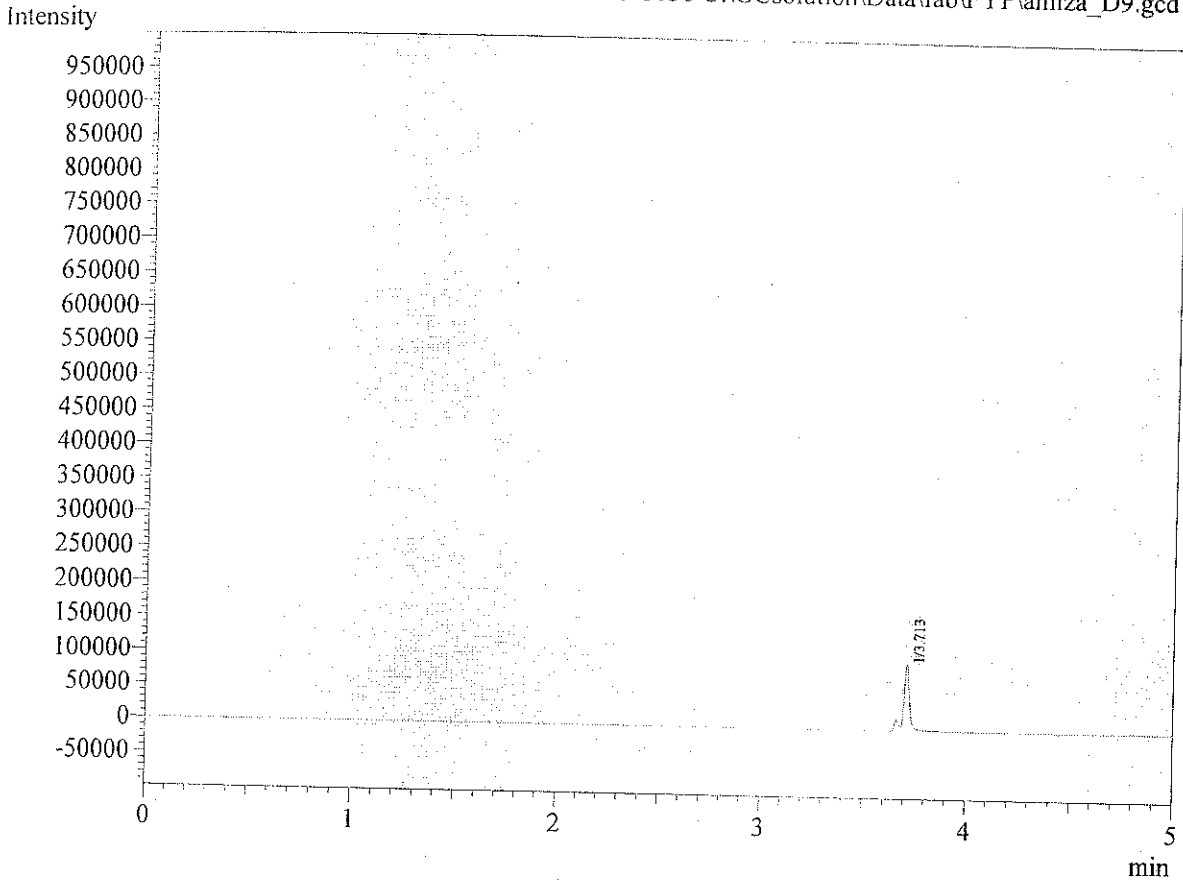
DEA(1 WT%)-RO-P = 20 BAR

Sample Information

Analysis Date & Time : 9/27/2005 2:19:57 AM

Sample Name : DEA 3wt% P1 RO S130

Chromatogram - Channel 1 DEA 3wt% P1 RO S130 C:\GCsolution\Data\fab\FYP\amiza_D9.gcd



Peak Table - Channel 1									
Peak#	Ret.Time	Area	Area%	Height	Height%	A/H	Conc.	Unit	ID#
1	3.713	179163	100.0000	97993	100.0000	1.828	0.760	%	1
Total		179163	100.0000	97993	100.0000				

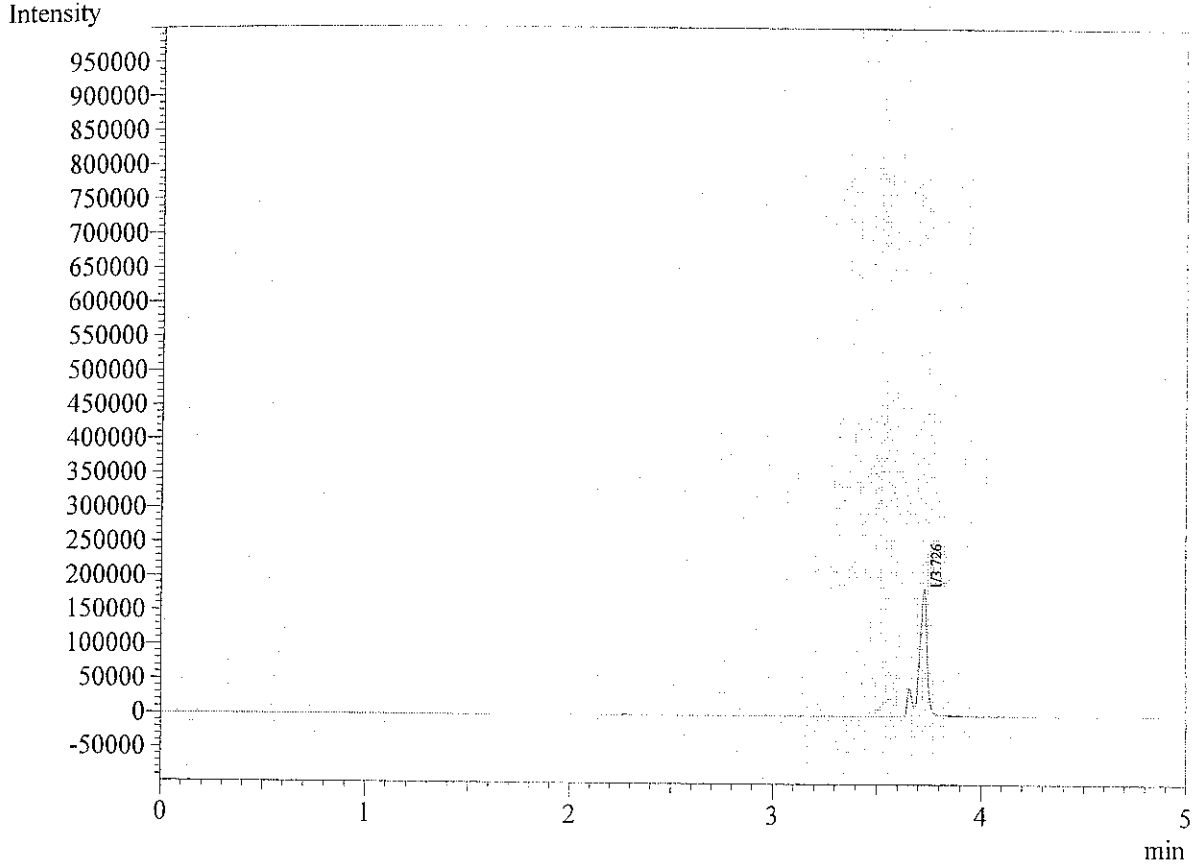
DEA(1 WT%)-UF-P = 4.8 BAR

Sample Information

Analysis Date & Time : 9/27/2005 1:39:20 AM

Sample Name : DEA 1WT% p2 UF S60

Chromatogram - Channel 1 DEA 1WT% p2 UF S60 C:\GCsolution\Data\fab\FYP\amiza_D5.gcd



Peak Table - Channel 1

Peak#	Ret.Time	Area	Area%	Height	Height%	A/H	Conc.	Unit	ID#
1	3.726	442561	100.0000	185446	100.0000	2.386	1.877 %		1
Total		442561	100.0000	185446	100.0000				

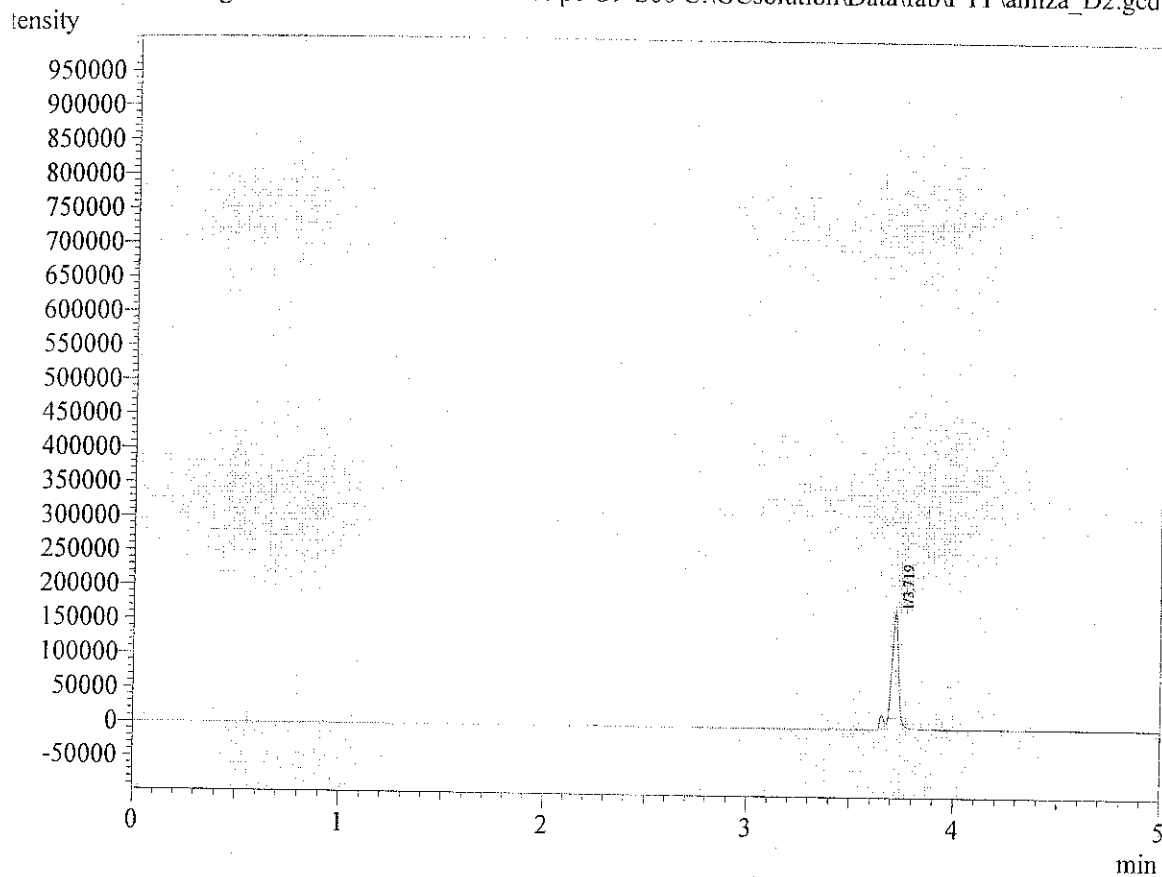
DEA(1 WT%)-UF-P = 3.5 BAR

Sample Information

Analysis Date & Time : 9/27/2005 1:08:55 AM

Sample Name : DEA 1WT% p1 UF S60

Chromatogram - Channel 1 DEA 1WT% p1 UF S60 C:\GCsolution\Data\fab\FYP\amiza_D2.gcd



Peak Table - Channel 1

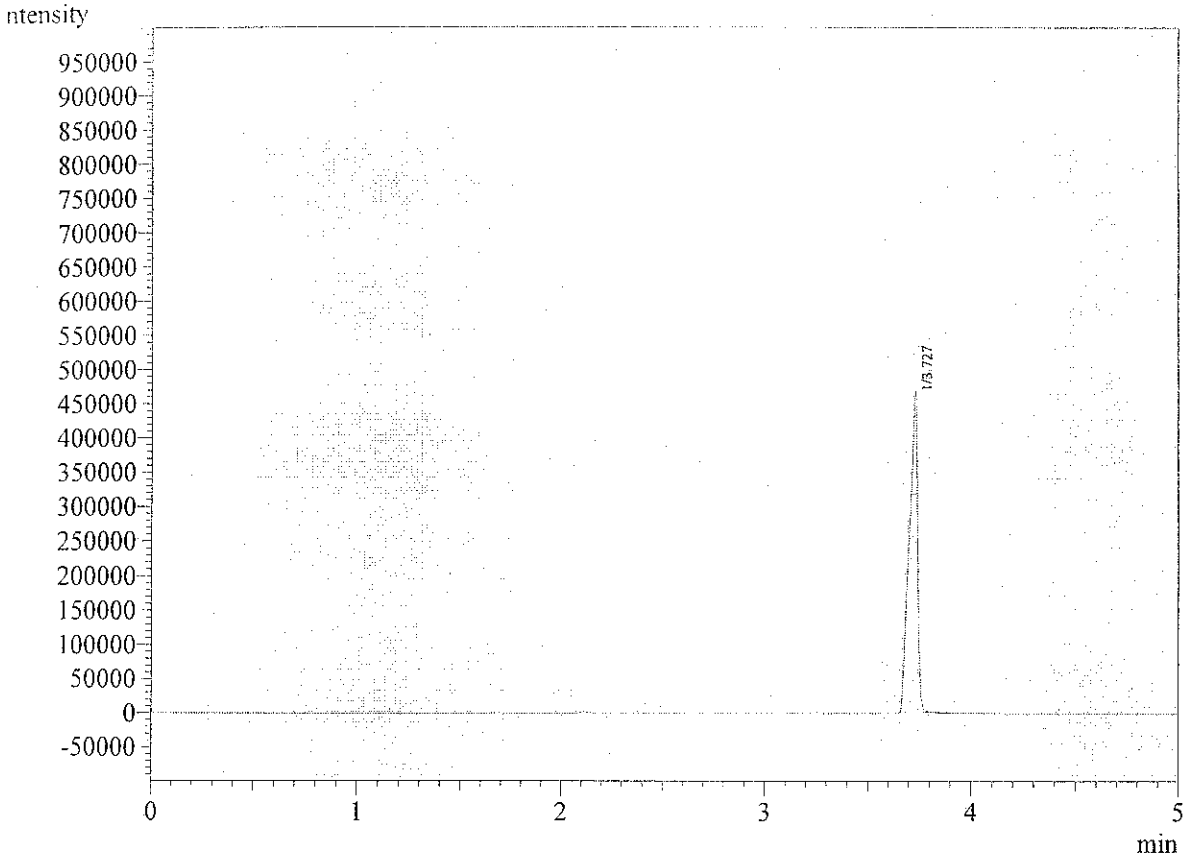
Peak#	Ret.Time	Area	Area%	Height	Height%	A/H	Conc.	Unit	ID#
1	3.719	400388	100.0000	171374	100.0000	2.336	1.698 %		1
Total		400388	100.0000	171374	100.0000				

Sample Information

Analysis Date & Time : 10/5/2005 4:58:37 AM

Sample Name : MDEA 1WT% P1 UF S60

Chromatogram - Channel 1 MDEA 1WT% P1 UF S60 C:\GCsolution\Data\fab\FYP\amiza_MD14.gcd



Peak Table - Channel 1								
Peak#	Ret.Time	Area	Area%	Height	Height%	A/H	Conc.	Unit ID#
1	3.727	1317356	100.0000	466549	100.0000	2.824	1.114 %	1
Total		1317356	100.0000	466549	100.0000			

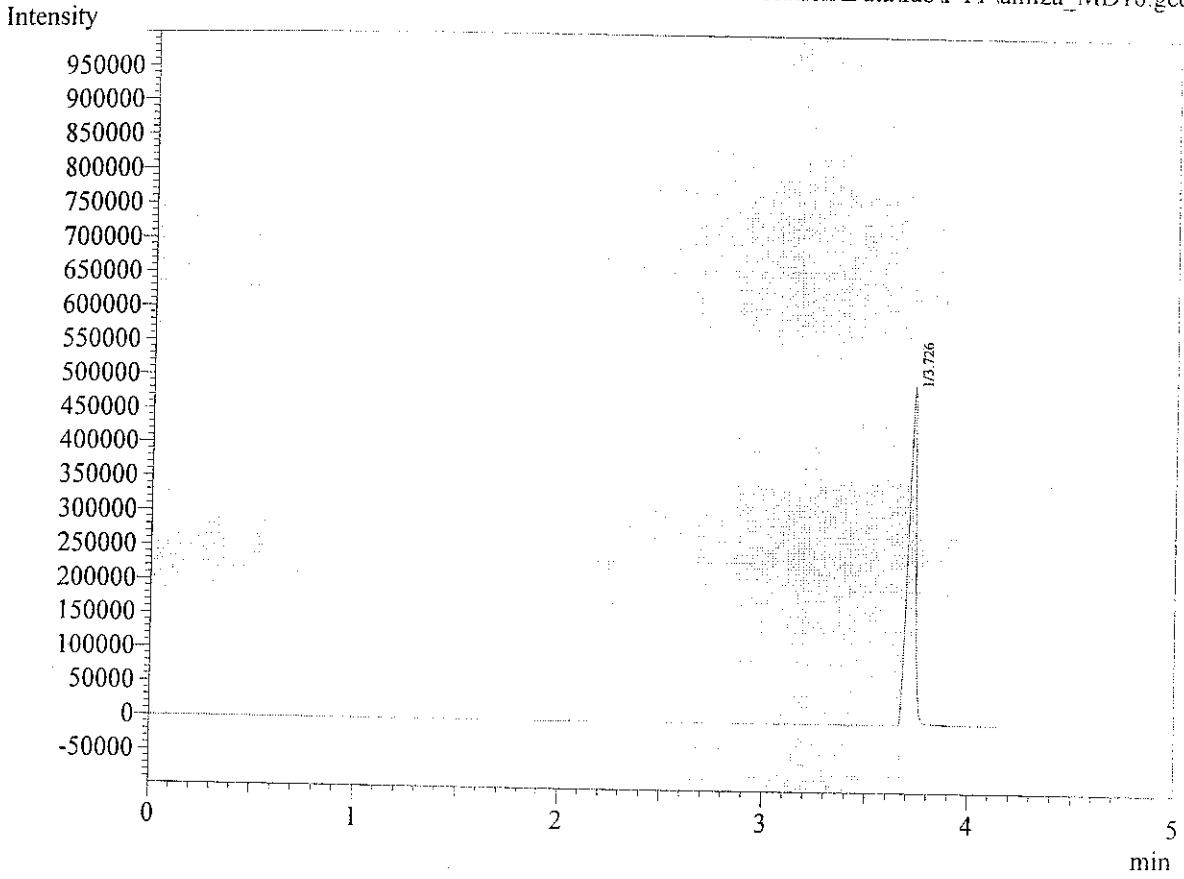
MDEA(1 WT%)-UF-P = 4.8 BAR

Sample Information

Analysis Date & Time : 10/5/2005 5:18:59 AM

Sample Name : MDEA 1WT% P2 UF F

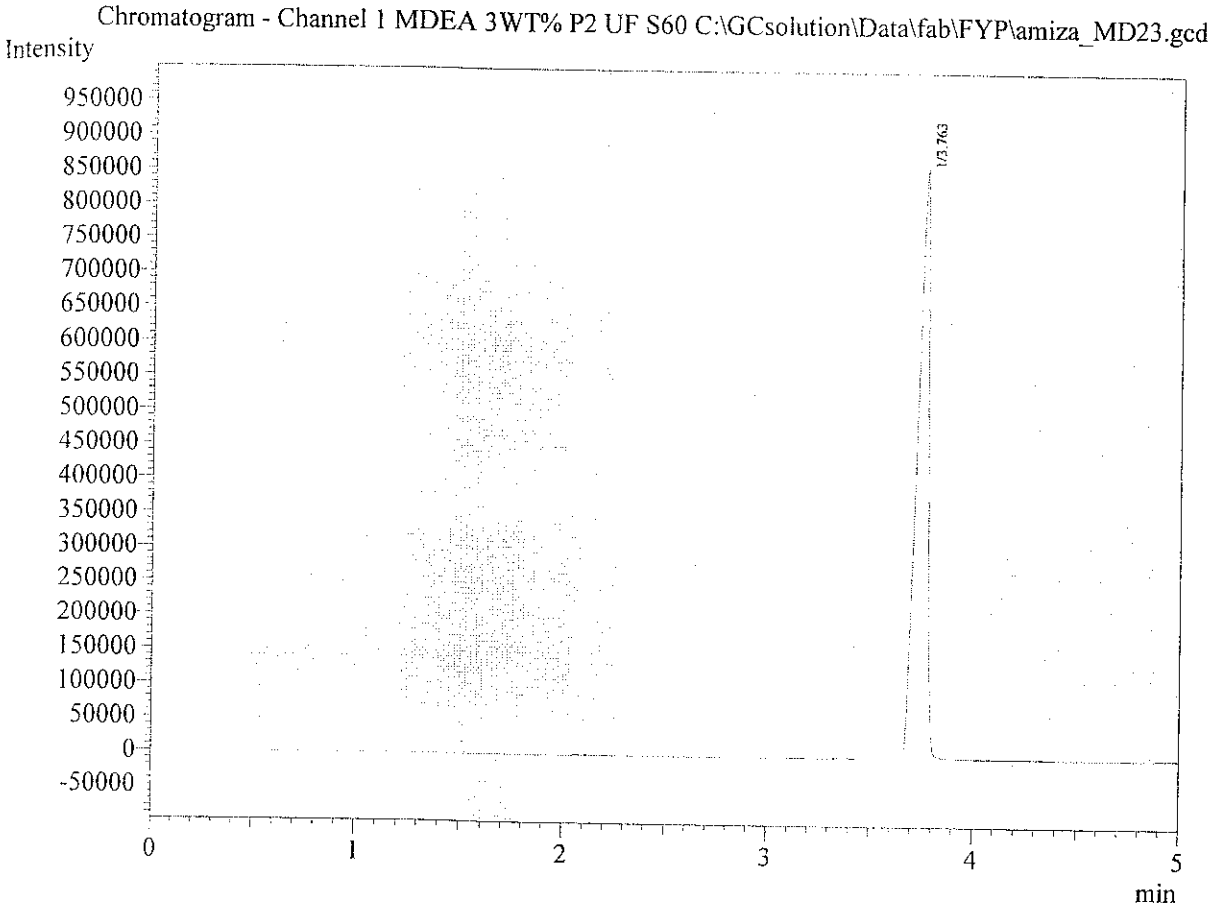
Chromatogram - Channel 1 MDEA 1WT% P2 UF F C:\GCsolution\Data\fab\FYP\amiza_MD16.gcd



Peak Table - Channel 1							
Peak#	Ret.Time	Area	Area%	Height	Height%	A/H	Conc. Unit1 ID#
1	3.726	1294507	100.0000	492474	100.0000	2.629	1.095 % 1
Total		1294507	100.0000	492474	100.0000		

MDEA(3 WT%)-UF-P = 4.8 BAR

Sample Information
Analysis Date & Time : 10/5/2005 6:30:10 AM
Sample Name : MDEA 3WT% P2 UF S60



Peak Table - Channel 1							
Peak#	Ret.Time	Area	Area%	Height	Height%	A/H	Conc. Unit ID#
1	3.763	3274764	100.0000	861441	100.0000	3.801	2.770 % 1
Total		3274764	100.0000	861441	100.0000		

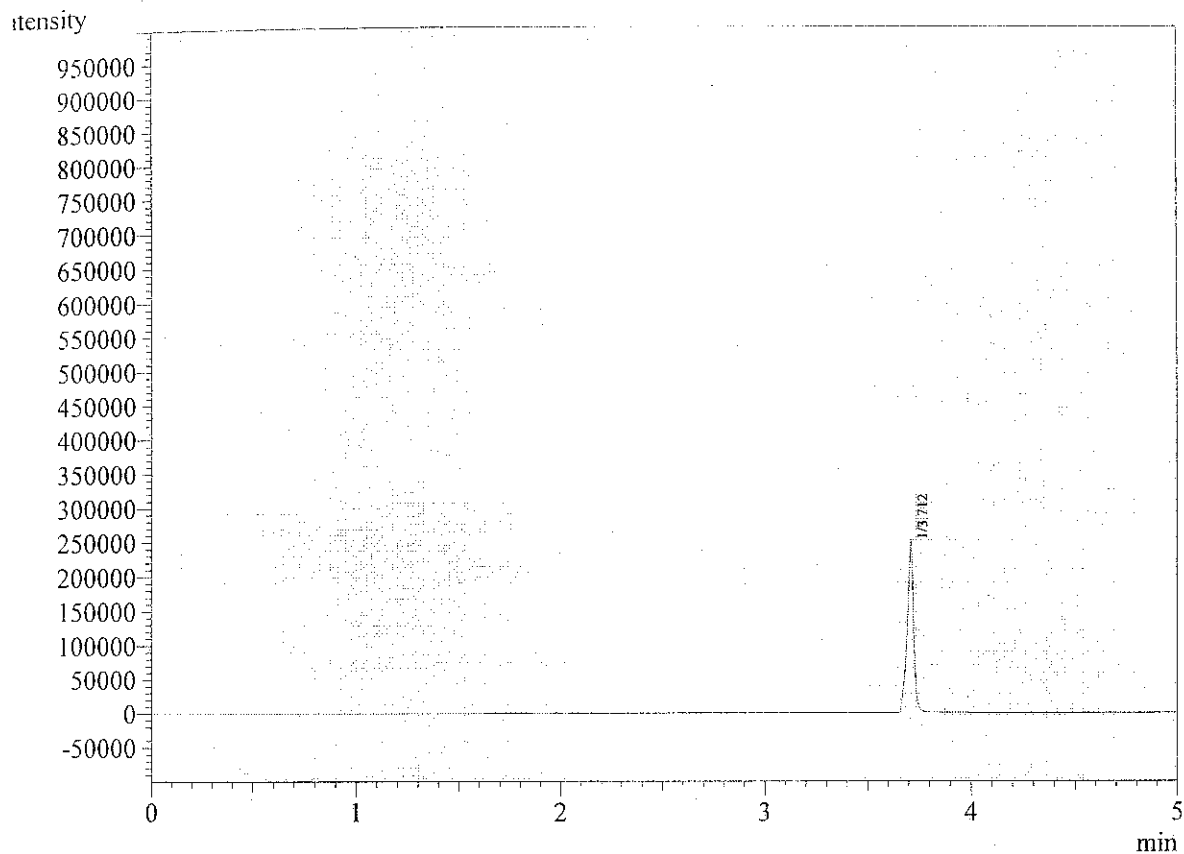
MDEA(3 WT%)-RO-P = 20 BAR

Sample Information

Analysis Date & Time : 10/5/2005 3:57:28 AM

Sample Name : MDEA 3WT% P1 RO S60

Chromatogram - Channel 1 MDEA 3WT% P1 RO S60 C:\GCsolution\Data\fab\FYP\amiza_MD8.gcd



Peak Table - Channel 1

Peak#	Ret.Time	Area	Area%	Height	Height%	A/H	Conc.	Unit	ID#
1	3.712	548287	100.0000	249320	100.0000	2.199	0.464 %		1
Total		548287	100.0000	249320	100.0000				