

**'THE EFFECT OF CLEANING ULTRAFILTRATION MEMBRANE USING
DIFFERENT CONCENTRATION OF NaOH'**

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I declared that this thesis entitled ‘the effect of cleaning Ultrafiltration membrane using different concentration of NaOH’ is the result of my own research except as cited in the references. The thesis has not been accepted for any degree is not concurrently submitted candidature of any degree.

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Date : 20 November 2006

Special dedicated to my beloved mother and father

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ABSTRACT

The developments of membrane technology are currently increased in parallel with the wide requirement. This happen because many application of this membrane technology. One of the applications from membrane technology is treatment of waste water as this experiment withheld. The study of mercury separation in deionized (DI) water using Ultrafiltration (UF) membrane to make sure whether UF membrane can separate mercury in the solution or not. This study using (UF) without the basis of PVDF. Besides, this study was held for pressure from 1.5-10.5 bar. If the optimum pressure of 5 bar, permeate flux value is as low as $1.82\text{Lm}^2/\text{min}$ and retentate flux value is as high as $18.02\text{Lm}^2/\text{min}$ achieved. Concentration value at retentate is high showing that the presences of mercury inhibit in the sample compare to original (DI) water. Concentration value achieved at retentate is about $2.0\mu\text{g/L}$ and permeate value is $1.0\mu\text{g/L}$. After running separation process, observed that the product flow will be constant starting at the first 20 minutes to 40 minutes. This happen because pores on the Ultrafiltration membrane surface clogged with mercury. In order to clean pores, 5 concentration parameter NaOH for 0.1M, 0.2M, 0.3M, 0.4M, 0.5M recommended for cleaning process. From overall results, 0.5M NaOH is suitable for cleaning process of (UF) membrane according to graph plotted as same as standard graph that we get from first experiment.

ABSTRAK

Perkembangan penggunaan membran pada masa ini meningkat selari dengan permintaan yang semakin meluas. Keadaan ini berlaku kerana pelbagai aplikasi yang boleh dilakukan melalui penggunaan membran ini. Salah satu aplikasi yang boleh dilakukan daripada penggunaan membran ini adalah di dalam rawatan air yang tercemar seperti yang telah dijalankan di dalam eksperimen ini. Kajian terhadap pemisahan merkuri didalam air yang tidak bercas menggunakan Ultrafiltration(UF) membran dilakukan bagi memastikan samaada (UF) membran itu dapat memisahkan unsur merkuri didalam air yang tidak bercas tersebut. Kajian ini dijalankan menggunakan (UF) membran berasaskan bahan PVDF. Selain itu juga kajian ini dijalankan pada kadar tekanan 1.5-10bar. Pada tekanan optimum iaitu 5 bar, nilai kadar permeate adalah rendah iaitu $1.82\text{Lm}^2/\text{min}$ dan nilai kadar retentate yang tinggi iaitu $18.02\text{Lm}^2/\text{min}$ telah dicapai. Nilai kepekatan pada retentate adalah tinggi menunjukkan kehadiran unsur merkuri yang masih wujud didalam sample berbanding kepekatan merkuri didalam permeate iaitu menghampiri kepekatan piawai merkuri didalam air yang tidak bercas. Nilai kepekatan yang dicapai pada retentate adalah lebih kurang $2.0\mu\text{g/L}$ dan nilai hasil adalah $1.0\mu\text{g/L}$. Setelah menjalankan menjalani proses pemisahan ini, didapati kadar pengaliran permeate akan menjadi sekata bermula pada minit ke-20 hingga minit ke-40. Ini berlaku kerana liang-liang pada permukaan (UF) membran tersebut telah tersekat oleh unsur merkuri. Untuk membersihkan liang-liang tersebut, 5 kepekatan larutan NaOH iaitu 0.1M, 0.2M, 0.3M, 0.4M, dan 0.5M telah disyorkan untuk menjalankan proses pembersihan tersebut. Hasil keseluruhan proses tersebut didapati kepekatan larutan 0.5M NaOH adalah paling sesuai dalam menjalankan proses pembersihan (UF) membran berdasarkan graf yang didapati adalah hampir sama dengan yang graf piawai yang dijalankan pada permulaan eksperimen.

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LIST OF SYMBOLS

UF	- Ultrafiltration
RO	- Reverse Osmosis
MF	- Microfiltration
$\mu\text{g/L}$	- Unit of concentration

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CHAPTER 1

INTRODUCTION

1.1 Membrane Technology

The word membrane comes from Latin word, “membrane” that means a skin. Today, the word “membrane” has been extended to describe a thin flexible sheet or film, acting as a selective layer between two phases because of its semi permeable properties. Its function is as a separation agent that very selective based on the difference of diffusivity coefficient, electric current or solubility. Actually membrane has become an integral part of our daily lives. All cells composing living things, including ours are surrounded with membrane. Biological membranes (membrane cells) are very selective that transfer only particular species.[1]

Membranes showed an important place in chemical technology especially in biotechnology and have been used extensively in many of application area such as pharmaceutical, purification, and hemodialysis. Membrane separation processes has become one of the emerging technology which undergo a rapid growth during the past few decades. It has drawn the world attention especially in the separation technology field, one of the chemical engineers specialties with is distinguish performance compared to the conventional separation technology.[5]

Membrane separation application allows only one component of a mixture to permeate through the membrane independently while the others component still remain.

Membrane process can be classified according to the driving force that used in the process. This usually exert in reverse osmosis, microfiltration, ultrafiltration, or gas separation. Other relevant driving force is concentration gradient driven process that been applied in pervaporation and dialysis. Meanwhile, and for electrolysis and electro dialysis is exerted with electrical potential gradient. Membrane process is widely used and until now its application is still going. We can see application spread over various industries such as in clean water process, textile industry, palm oil industry, cane sugar industry, and many more. As the example in the clean water process water produced is free from suspended solid and bacteria and a number of dissolved ions especially heavy metal are reduced. Membrane technology is applied in mineral water industry. Ground water or river water can be used as the raw material.[5]

The spectrum of filtration separations runs from the millimeter size scale (beach sand and activated carbon particles) using coarse filters, to the angstrom scale (metal ions and gas molecules) using reverse osmosis or gas separation membranes. In between there is micro filtration (bacteria and emulsions), ultrafiltration (proteins, viruses, and colloids), and Nanofiltration (sugars, herbicides, small organic molecules). In the analytical environment it is likely that membrane and filtration systems involving this entire spectrum will be used to provide high purity water, gases, reagents, and even special functions within instruments. In addition to these applications, membranes and filters will often also be used in sample preparation and perhaps initial characterization. [5]

The Ultra filtration is a process of separating extremely small suspended particles and dissolved macromolecules from fluid using asymmetric membranes of surface pore size in the range of 50 to 1 nm. The primary basis of separation is molecular size although secondary factors such as molecular shape and charge can play a significant role. UF membranes are often operated in a tangential flow where the feed streams weeps tangentially across the upstream surface of membrane as filtration occurs, thereby maximizing flux rates and membrane life. Ultrafiltration is generally defined as effecting

separation in the 0.002 to 0.2 micron range. This is perhaps more usefully described as the 500 to 300,000 molecular weight cut-off (MWCO) range, requiring pore sizes of from 15 to 1000 angstroms. [3]

1.2 Problem Statement

Nowadays, membrane technologies are currently increased in parallel with the wide requirement. This happen because many application of this membrane technology such as in wastewater treatment. There are a lot of membranes available to be applied to the process such as microfiltration (MF), ultrafiltration (UF), and reverse osmosis (RO) but in this research UF process was applied. Pores of the membrane surface will be clogged by the particles of the mercury after running the UF process. Membrane that was been clogged must be clean to be back the real efficiency.

1.3 Objective

The main objective of this research is to determine the optimum concentration of NaOH in cleaning Ultrafiltration(UF) membrane.

1.4 Scope

Scopes is important as a guide to achieve the objective of the research. This is due to the objective to be narrow with certain aspect that should be considered to implement the research. The scope of this research are:

1. Analyze the effect of the pressure on the Ultrafiltration(UF) membrane permeate flow rate.
2. Measure the volume of permeate and rejection after treating and cleaning process in one duration time.
3. Analysis the mercury concentration in permeate and rejection using Hach cold vapor mercury.

CHAPTER 2

LITERATURE REVIEW

2.1 Historical Background of Membrane Development

Systematic studies of membrane phenomena can be traced to the eighteenth century philosopher scientists. The Abbe Nolet, for example, coined the word osmosis to describe permeation of water through a diaphragm in 1748. Through the nineteenth and early twentieth centuries, membranes had no industrial or commercial uses. However, membrane were used as laboratory tools to develop physical/chemical theories in 1887 to develop his limit law, explaining the behavior of ideal dilute solutions. [1]

In 1906 Bechhold devised a technique to prepare nitrocellulose membranes of graded pore size, which he determined by a bubble-test method. Later workers, particularly Elford, Zsigmondy and Bachman, and Ferrys, improved on Bechhold's technique. By the early 1930s microporous coilodion membranes were commercially available. During the next 20 years, this early microfiltration membrane technology was expanded to other polymers, particularly cellulose acetate, and membranes found their first significant applications in the filtration of drinking water samples at the end of World War II. [1]

Membrane science and technology is an expanding field and has become a prominent part of many activities within the process industries. It is relatively easy to identify the success stories of membranes such as desalination and microfiltration and

refer to others as developing areas. This, however, does not do justice to the wide field of separation in which membrane are used. No other 'single' process offers the same potential and versatility as that of membranes. [7]

Improvements and advances in membrane technology over the last two decades have seen applications expand in many industrial sectors; chemical, petrochemical, mineral and metallurgical, food, biotechnology, pharmaceutical, electronics, paper and pulp water etc. Membrane separations are in competition with physical methods of separation such a selective adsorption, absorption, solvent extraction, distillation, crystallization, and cryogenic gas separation. [7]

Membranes have gained an important place in chemical technology and are used in a broad range of applications. The key property that is exploited is the ability of membrane to control the permeation rate of a chemical species through the membrane. In controlled drug delivery, the goal is to moderate the permeation rate of a drug from a reservoir to the body. In separation applications, the goal is to allow one component of a mixture to permeate the membrane freely, while hindering permeation of other components. [1]

2.2 Basic Concept of Membrane

Membranes have gained an important place in chemical technology and are used in a broad range of applications. The key property that exploited is the ability of a membrane to control the permeation rate of a chemical species through the membrane. As showed in Figure 2.1, membrane acts as a semipermeable barrier and separation occurs by the membrane controlling the rate of movement of various molecules between two liquid phases, two gas phases, or a liquid and a gas phases. [14]

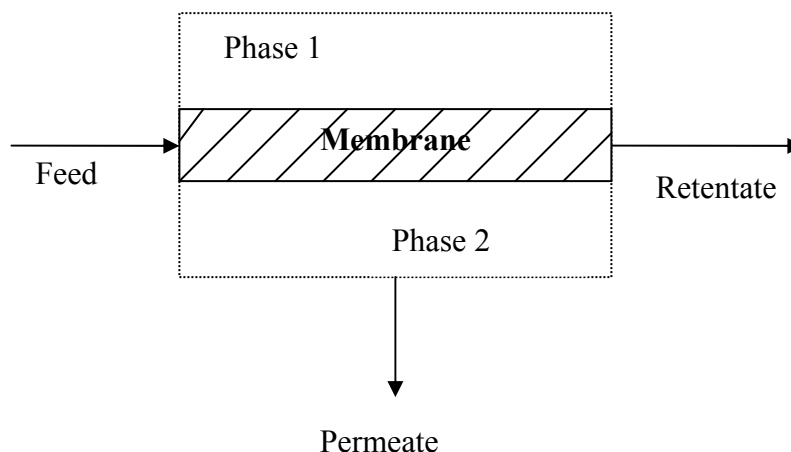


Figure 2.1: Membrane separation process

Membrane also known as selective barrier between two phases that have thin barrier that permits selective mass transport and the phase that acts as a barrier to prevent mass movement, but allows restricted and / or regulated passage of one or more species. [14]

2.3 Types of Membrane Process

There are some of levels for filtration membrane. Each of them has different character such as mechanisms of separation, physical morphology and chemical nature. Generally, there are 3 main types of filtration membrane. There are microfiltration membrane, ultrafiltration membrane, and reverse osmosis filtration membrane. Table 2.1 show the technically relevant membrane separation processes, their operating principles, and their application. From the Table 2.1 below, we know that microfiltration have the pore radius 0.05-5 μ m and hydrostatic pressure 0.5-4 bar mean while for the Ultrafiltration, the pore radius 2-10 nm and the pressure 1-10 bar. [14]

Table 2.1: Technically relevant membrane separation processes

Separation Process	Membrane Type Used	Applied Driving Force	Mode of Separation	Applications
Microfiltration	symmetric porous structure, pore radius 0.05-5 μm	hydrostatic pressure 0.5-4 bar	filtration (size exclusion)	water purification, sterilization
Ultrafiltration	asymmetric porous structure, pore radius 2-10 nm	hydrostatic pressure 1-10 bar	filtration (size exclusion)	Separation & fractionation of molecular mixtures
Diafiltration	asymmetric porous structure, pore radius 2-10 nm	hydrostatic pressure 1-10 bar	filtration & dialysation (size exclusion)	purification of molecular mixtures artificial kidney
Reverse osmosis	asymmetric skin-type solution-diffusion structure	hydrostatic pressure 10-100 bar	solution-diffusion mechanism	sea & brackish water desalination
Dialysis	Symmetric porous or gel-type structure	concentration gradient	diffusion	artificial kidney
Electrodialysis	symmetric ion-exchange membrane	electrical potential	migration Donnan-exclusion	water desalination
Donnan Dialysis	symmetric ion-exchange membrane	concentration gradient of individual ions	diffusion Donnan exclusion	water softening
Electrodialytic Water Dissociation	bipolar membrane	electrical potential	migration, Donnan-exclusion	acid & base production from salts
Gas Separation	homogeneous symmetric structure	vapor pressure gradient	solution-diffusion	oxygen/nitrogen separation
Pervaporation	homogeneous symmetric structure	vapor pressure gradient	solution-diffusion	separation of azeotropic mixtures
Vapor Permeation	homogeneous symmetric structure	vapor pressure gradient	solution-diffusion	recovering of organic vapors from air
Membrane Distillation	symmetric porous hydrophobic structure,	vapor pressure gradient	diffusion	liquid/solid separation
Membrane Contactores	symmetric porous structure, or liquid membrane	chemical potential gradient	diffusion solution	Solvent extraction

2.3.1 Microfiltration Membrane

Microfiltration (MF) as figure 2.2 is oldest of the three pressure driven membrane technologies, which also include reverse osmosis and ultrafiltration. Microfiltration is used primarily for separate particles, viruses, bacteria and microbial removal and it can operate under ultra low pressure. Bacteria, paint pigments, and macromolecules with molecular weights greater than about 300,000. Microfiltration uses a conventional flow path. The input flow is perpendicular to the membrane surface, and all of the solvent to be processed passes through the membrane. [8]

Solutes permeate the membrane by dissolving in the membrane material and diffusing down a concentration gradient. The separation occurs because of the difference in solubility and mobility of the solutes in the membrane. MF uses “loose” membranes, that is, membranes that have relatively large pores. The size of microfiltration of microfiltration pores or cut off weight is 0.05-5 μm (Baker, 2004). The operating pressure requirements of microfiltration systems are low (0.5-4 Bar). [8]

Applications examples include:

- Water purification.
- Sterilization.
- Cell recovery from broths.
- Removal of bacteria from milk and other food products.
- Removal fat and oil.
- Fractionation of proteins.
- Clarification of juice

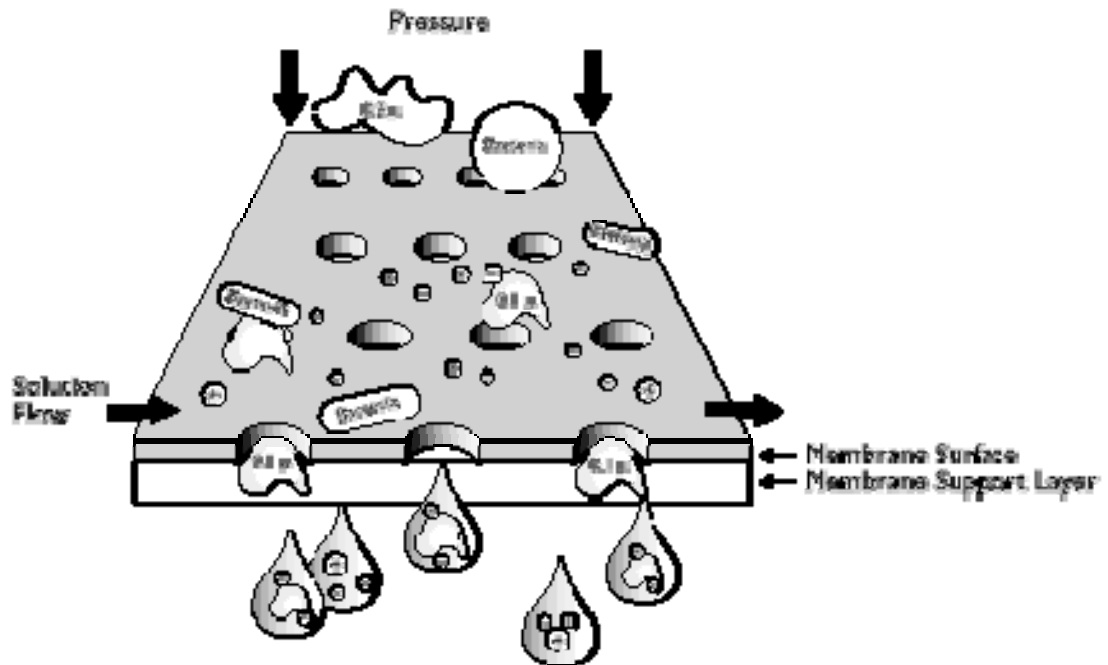


Figure 2.2: Microfiltration Processes

2.3.2 Ultrafiltration Membrane

The basic concept of ultrafiltration (UF) processes as figure 2.3 is the same as in microfiltration processes. The different of these two types separations is the size of the membrane pores. For ultrafiltration, the size of membrane pores is 5-50 nm and the pressure requirements are moderate at 1-10 bar. This mean only the particle that small than the membrane pores can pass through the membrane [14]. Meredith Feins and Kamallesh K. Sirkar had done the research about ultrafiltration technique based on a multimembrane stack. Ultrafiltration, is carried out to separate two proteins relatively close in molecular weight [8]. Ultrafiltration process is used to separate and/or concentrate macro-molecules, protein and enzymes. Typical applications include

recovery of proteins, removal of ash from gelatine and fruit juice concentration and clarification. The process normally rated by their nominal weight cut off, which is defining as the molecular weight of a solute that has a rejection coefficient of 90% [15].

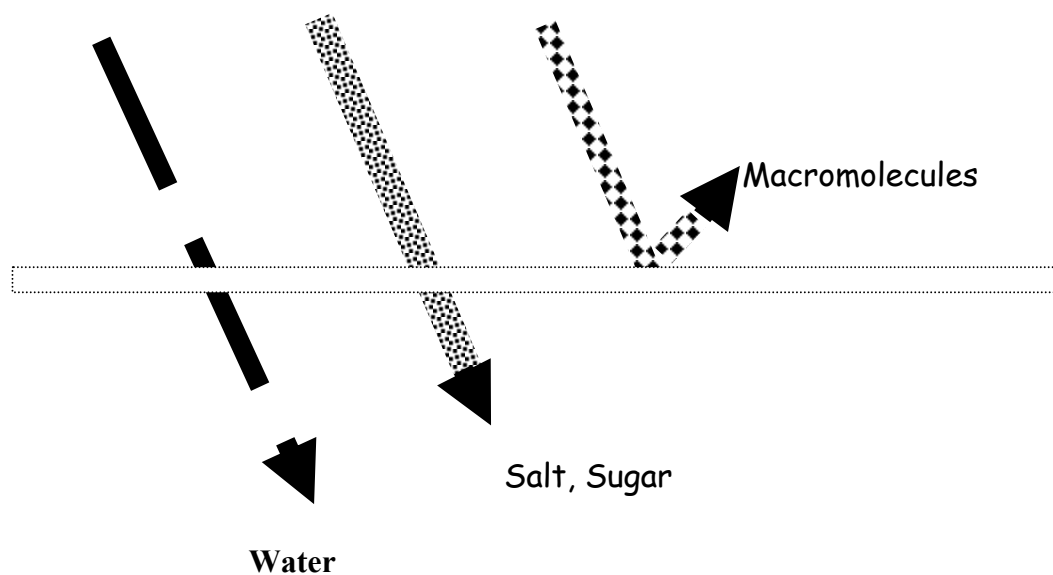


Figure 2.3: Ultrafiltration Membrane Process

2.3.3 Reverse Osmosis Filtration Membrane

Reverse Osmosis (RO) uses membrane with the smallest pores 0.0001- 0.001 microns and has the highest-pressure requirement 10-100 bar. This process is used to remove water separate small molecules such as sugars from dissolved salts, the process will occur when the water move from low concentration to high concentration by using pressure. Typical applications include concentrating dairy or food products and recovery/polishing of water from permeate or evaporator condensate in purification, the principal is to remove undesired components in a feed mixture from the desired species. For example is the purification of acid gases such as sulfur dioxide must be removed

from power plant combustion gas effluents before discharge into atmosphere [14]. Figure 2.4 show the process of reverse Osmosis Membrane.

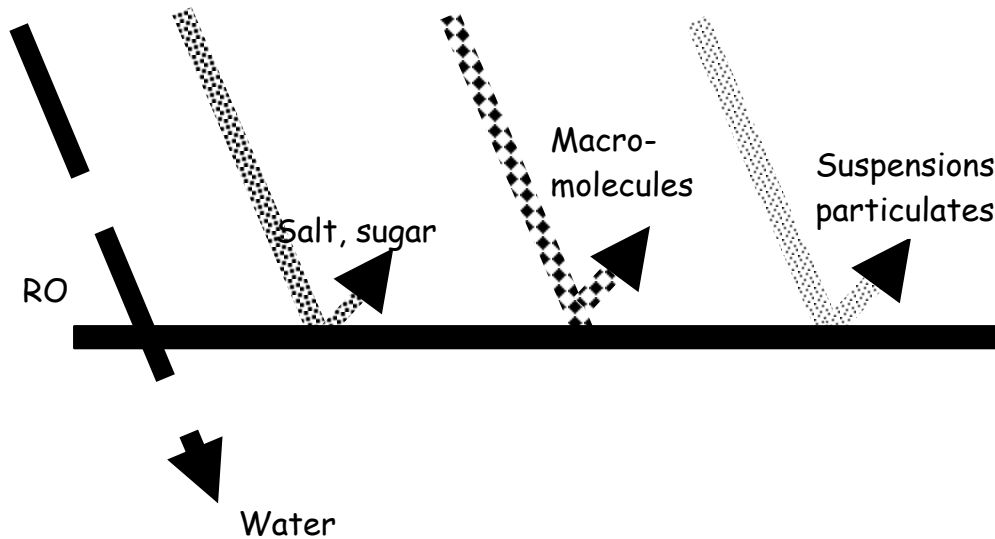


Figure 2.4: Reverse Osmosis Filtration membrane Process

2.4 Advantages and Disadvantages of Ultrafiltration Membrane Technology

2.4.1 Advantages

The technology of membrane separation has been developing to be the top of process separation. It is because there are a lot of advantages by using membrane for ultrafiltration [31]. The advantages of membrane are: