

SYNTHESIS OF POLYESTER NANOFILTRATION MEMBRANE FOR REMOVAL  
OF NATURAL ORGANIC MATTER

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## ABSTRACT

Natural Organic Matter (NOM) in surface water usually refers to humic and fulvic acids that are decayed by-products of plants such as those found in surface waters shallow well aquifers. Performance on humic acid removal from natural organic matter using Nanofiltration (NF) has been investigated in this study. Since most of the organic matter in groundwater has smaller between 200-500 D of the pore sizes, the use of the NF process was proposed. Thin-film composite nanofiltration membranes were prepared via interfacial polymerization of triethanolamine and trimesoyl chloride with different concentration and reaction time. The separation process was conducted by separating solution that contains humic acid to get the best result based on varying the concentration and reaction time for interfacial polymerization. In term of concentration of monomers has shown that 4% TEOA has gives more rejection of humic acid. As for reaction time, meanwhile 10 minutes of TMC reaction time shows higher performance in removing humic acid. Fouling is still occur because of the tendency of humic acids appears to be due to their ability to bind to multivalent salts.

## ABSTRAK

Bahan Organik Semula Jadi (NOM) dalam air permukaan biasanya merujuk kepada asid humid dan asid-asid fulvic yang busuk dari produk sampingan tumbuh-tumbuhan seperti yang didapati dalam permukaan akuifer telaga cetek. Prestasi penyingkiran asid humid dari bahan organik semula jadi menggunakan Nanofiltration (NF) membran telah dikaji dalam kajian ini. Kebanyakan daripada bahan organik dalam air bawah tanah mempunyai saiz liang yang begitu kecil antara 200-500 D, maka penggunaan membran NF telah dicadangkan. Filem nipis membran nanofiltration komposit telah disediakan melalui pemolimeran antara muka klorida triethanolamine dan trimesoyl dengan kepekatan dan masa tindak balas yang berbeza. Proses penyingkiran dijalankan dengan menyingkirkan cecair yang mengandungi asid humid untuk mendapatkan keputusan terbaik berdasarkan mempelbagaikan tumpuan dan masa tindak balas untuk pemolimeran antara muka. Dari segi pengaruh monomer-monomer, ianya telah membuktikan bahawa 4% TEOA memberikan lebih penolakan asid humid. Walaubagaimanapun bagi masa tindak balas, 10 minit masa tindak balas TMC menunjukkan prestasi lebih tinggi dalam penyingkiran asid humid. Pembatalan membran masih berlaku disebabkan oleh kecenderungan asid humid muncul disebabkan kemampuan mereka untuk terikat akan garam-garam multivalen.

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**LIST OF SYMBOLS**

$C_p$	concentration of permeate
$C_f$	concentration of feed
h	hour
P	permeability
b	bar
$J$	Permeate flux
L	liter
m	meter
$\Delta P$	Filtration pressure
$R$	rejection
$\Delta t$	filtration time
V	volume

**LIST OF ABBREVIATIONS**

TEOA	Triethanolamine
TMC	trimesoylchloride
PES	Polyethersulfone
PVP	Polyvinylpyrrolidone
NMP	N-metyl-pyrrolidone
UF	Ultrafiltration
RO	Reverse Osmosis
NF	Nanofiltration
IP	Interfacial Polymerization

## CHAPTER 1

### INTRODUCTION

#### 1.1 RESEARCH BACKGROUND

Water is the most important basic need for human to live. 70.9% of the Earth is actually covered by water and mostly found in oceans but 1.7% is in groundwater. Less than 0.3% of all freshwater is in rivers, lakes, and the atmosphere, and an even smaller amount of the Earth's freshwater (0.003%) is contained within biological bodies and manufactured product. Like many substances, water can take numerous forms that are broadly categorized by phase of matter. Nowadays, water is the most popular issues since water becomes more polluted nowadays. One of the supply water is groundwater which comes from rain, snow, sleet, and hail that soak into the ground. The polluted groundwater is a result of human activities such as farming and industry. Leakage from underground storage tanks and chemical and waste storage facilities, leakage from hazardous waste dump sites, sewers, soak pits and also often by accidental discharge of waste also contribute to pollution. Pesticides, chemical and other things can soak through the ground and contaminates the ground water.

Natural Organic Matter (NOM) is the organic material present in surface or ground water. NOM interacts with all of the chemical components of natural waters; these interactions alter the behavior of pollutants in surface and ground waters(Natural Organic Matter, 2011). Natural Organic Matter (NOM) in surface water usually refers to humic and fulvic acids that are decayed by-products of plants such as those found in surface waters

shallow well aquifers. These substances plus tannic acid are the major fraction of Dissolved Organic Matter (DOM) in water. These substances are oxidized very slowly and their solubility in water may vary with pH. The dissolved fraction of NOM may not be fully removed using conventional water treatment practices and have been shown to produce by-products such as trihalomethane during disinfection. The organic compounds in surface waters and ground waters arise from natural and anthropogenic sources. Natural organic matter (NOM) is either formed in place or is formed outside of the water body and then transported into the water body (Wershaw, 2005).

Nanofiltration (NF) is a process with membrane permeability in between RO and UF processes, even still have high rejection of organic matter but it still provides a low retention of monovalent ions (Rautenbach et al. 1989). Interfacial polymerization usually is used for preparation of thin film composite layer. There are many methods in preparing composite membranes for NF but interfacial polymerization is still the main method to produce commercial NF high performance polymers (Li et al. 2008). In this paper presents the results obtained from further studies on ability of NF membrane in removing NOM which is humic acid.

## **1.2 PROBLEM STATEMENT**

The world demand on the access of the clean water is very high. Groundwater can be treated by chemical & physical treatment. One of the successful physical treatments is membrane process. From NOM removal, nanofiltration (NF) is the best choice, however the major problem for the commercial polyamide NF membrane is fouling. In order to investigate fouling problem, new polyester NF will be synthesized and tested.

### **1.3 RESEARCH OBJECTIVE**

The objectives of this study are listed as following:

1. To optimizing the performance of membrane by changing the concentrations of monomers and reaction time.
2. To further understand the polyester NF membrane performance on natural organic matter removal.

### **1.4 RESEARCH SCOPE**

To achieve the objective of this research, scopes have been identified:

1. Preparing polysulfonemicroporous support.
2. Preparing the composite membrane using conventional interfacial polymerization technology by varying monomer concentrations and reaction time.

### **1.5 SIGNIFICANCE OF STUDY**

This study is to prepare the NF membrane is the best choice as the physical treatments for groundwater. NF is suitable for NOM removal if the removal of humic acid is higher by using this filtration method.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 MEMBRANE REVIEW**

##### **2.1.1 Membrane definition**

Membrane can be defined as be some definition aphase that acts as a barrier to prevent mass movement, but allows restricted and / or regulated passage of one or more species. It is proven that membrane processes particularly can separate organics, salts, microorganisms, nutrients, biomass, and even micro pollutants from wastewater (Chang & Kim, 2005). Membrane filtration is defined as a pressure- or vacuum-driven separation process in which particulate matter larger than 1  $\mu\text{m}$  is rejected by an engineered barrier primarily through a size exclusion mechanism and which has a measurable removal efficiency of a target organism. A membrane system is available in variety of separation capabilities have become the technology of choice for these regulations. The capabilities were include from the removal of turbidity, precursors and disinfectant tolerant micro-organisms relating to groundwater and surface water supplies, as well as tapping into new water supplies, such as brackish and seawater (Membrane Filtration Guidance Manual, 2011).

### 2.1.2 Membrane Structure

RO, NF and UF membranes are asymmetric. This differentiates most membranes from common filters, e.g. coffee filters, which are symmetric or, in other words, are identical on both sides of the filter. Membranes have a tight top layer facing the product to be treated. This layer is also called the skin layer. It is thin, typically less than 0.1 micron. The membrane itself is 150 - 250 micron; the bulk of the membrane gives structural support for the skin layer. Asymmetric structure means that the pores are wider from the surface, which prevents the pores from being plugged. This provides good fouling resistance, since foulants have a tendency to either be totally rejected or to pass all the way through a membrane.

The membrane processes can be categorized based on its separation mechanism which mainly consist of size exclusion, solubility and diffusivity and charge (Van Rijn, 2004). Table 2.1 shows the major membrane processes arranged according to the mechanism of separation. Membrane separation processes that occur based on size exclusion involve microfiltration (MF), ultrafiltration (UF) and nanofiltration (NF). Microfiltration (MF) membrane consists of the largest membrane pores (which typical range from 0.1 -10  $\mu\text{m}$ ) as compared to the NF and UF membrane.

**Table 2.1:** Membrane processes arranged according to the mechanism of separation

Separation Mechanism	Major membrane separation process
Size exclusion (filtration)	Nanofiltration, ultrafiltration, microfiltration
Solubility/ diffusivity	Reverse osmosis, gas separation, pervaporation
Charge	Electrodialysis

(Source: Van Rijn, 2004)

These type of membrane commonly used in separation of macromolecular solutes and colloidal material from 2 macromolecular solutes and solvents. The applications of these membranes include concentration of protein/enzyme for pharmaceutical and biomedical industries, food and dairy, pulp and paper and etc (Scott and Hughes, 1996). Table 2.2 shows the other types other membranes filtration with the application.

**Table 2.2:**Membrane filtration applications

		PERMEATE	CONCENTRATE
RO	Dying effluent Water Whey	Clean water Low salinity water Low BOD permeate	BOD, salt, chemicals, waste product Salty water Whey concentrate
NF	Antibiotics Dyeing effluent Water whey	Salty waste product Clean, salty water Softened water Salty waste water	Desalted, concentrated antibiotics BOD/COD, color Water product Desalted whey concentrate
UF	Antibiotics Bio-gas waste Carrageenan Enzymes Milk Oil emulsion Washing effluent Water Whey xantan	Clarified fermentation broth Clarified liquid for discharge Waste product Waste product Lactose solution Oil free water (<10ppm) Clarified water Clarified water Lactose solution Waste product	Waster product Microbes to be recycled Concentrated carrageenan High value product Protein concentrate for cheese production Highly concentrated oil emulsion Dirty water (waste product) Waste product Whey protein concentrate Concentrated xantan

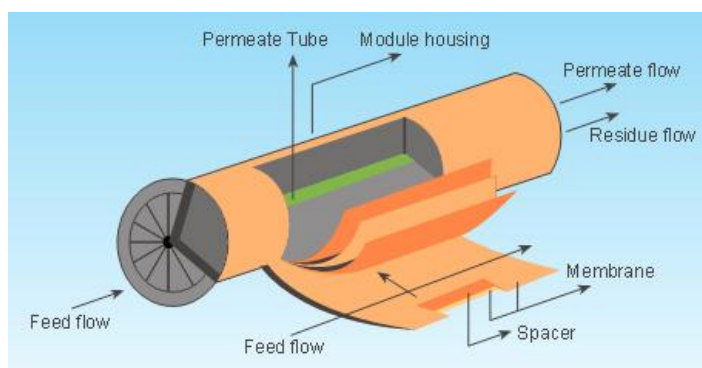
(Source: Wagner, 2001, Membrane Filtration Handbook).



## 2.2 MEMBRANE MODULES

### 2.2.1 Plate and Frame Module

One of the earliest membrane system modules is plate and frames modules. Membrane, feed spacer and product are layered together between two end plates. The feed mixtures is forced across the surface of the membrane (Baker, 2000). A portion passes through membrane, enters the permeate channel and makes its way to a central permeate collection manifold. Figure 2.2 shows the schematic drawing of plate and frame module.



**Figure 2.1:** Schematic drawing of plate and frame module

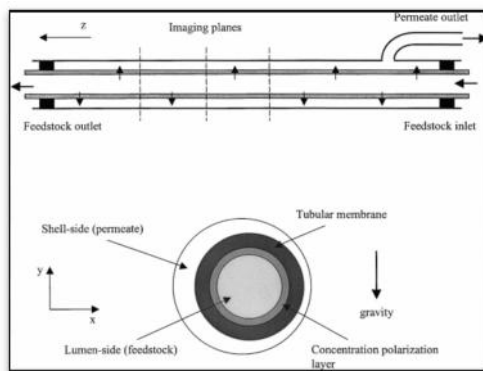
### 2.2.1 Tubular Modules

Tubular membranes were available for laboratory use as early as the 1920's and were first used in industrial applications in the 1960's. Tubular membrane modules have one or more tubes of varying diameter. The tubes themselves are constructed of a microporous substrate material which provides mechanical strength and the membrane is cast on the inside of the tube as a finely porous surface layer. Wastewater is pumped through the membrane tubes and permeate flows through the engineered pores to produce treated water.

Tubular membranes are not self-supporting membranes. They are located on the inside of a tube, made of a special kind of material. This material is the supporting layer for the membrane. Because the location of tubular membranes is inside a tube, the flow in a tubular membrane is usually inside out. The main cause for this is that the attachment of the membrane to the supporting layer is very weak.

Tubular membranes are known for their sturdy construction, long membrane life, and high flux rates. Of all membrane types, they are more robust and can be subjected to high pressures in demanding applications. Tubular membranes are backwashed in some applications. With backwash pressures of up to 15 psi, the large flow in the reverse direction efficiently removes solids from the membrane surface.

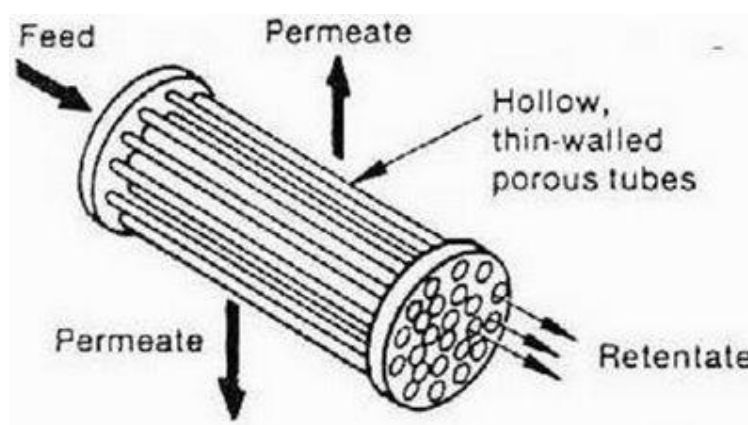
Tubular modules, the membrane is fitted over the inside of a pipe of 10 mm or more inner diameter of the module and can treat high turbid, concentration liquids and water with suspended solids for which hollow fiber modules are not effective. Typical applications of this module are including reclaimed water plants, night soil treatment and enzyme concentration using UF membranes. The modules are also ideal for dye desalination and tomato concentration when combined with RO membranes. The turbulent flow of the retentive stream prevents the cake formation on the inner surface of the tube resulting in high flux and longer shelf life (Prasad, 2010).



**Figure 2.2:** Schematic diagram of tubular module

### 2.2.3 Hollow Fibre Modules

Hollow fiber cellulose acetate membranes with asymmetric structure were prepared using a wet spinning method. In the preparation procedures, spinning parameter (i.e bore liquid flowrate) has been identified to influence the shape and the hollow fiber thickness. Spinneret dimension also influences the hollow fiber thickness produced. The membrane structures are characterised by using Scanning Electron Microscope. The system is pressurized from the shell side. Permeate passes through the fiber wall and exits through the open fiber ends. Figure 2.3 shows the schematic design of hollow fiber module.

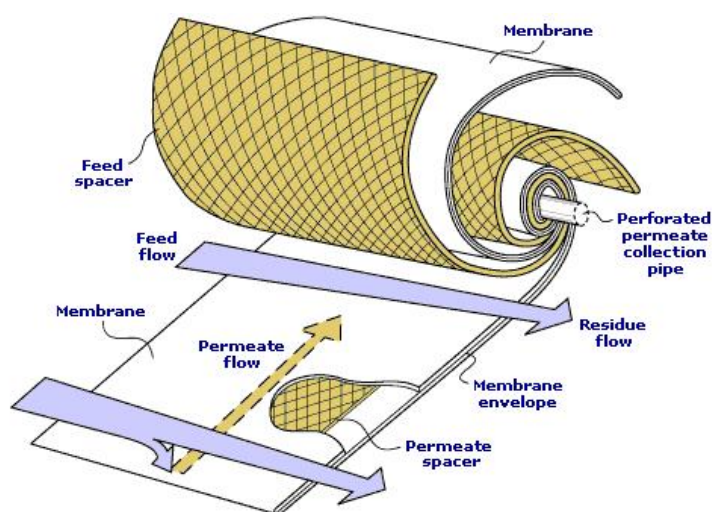


**Figure 2.3:** Schematic design of hollow fiber module

### 2.2.4 Spiral Wound Module

Since 1970s, the demand for spiral wound membrane (SWM) has been rapidly increasing in both local and worldwide market. Current market available SWM possess lifespan between one to three years depends on the applications. The designs of a spiral wound membrane consist of membrane envelopes (leaves) and feed spacers which wound around a perforated central collection tube. These modules were designed in an effort to pack as much membrane surface as possible into a given volume (Senthilmurugan

et al., 2005). Spiral-wound membrane modules are the most commonly used design for commercial membrane installations today. Spiral-wound modules are robust, resistant to fouling, and economical; they are used in 95% of the reverse osmosis desalination industry and 30% of the industrial gas separation market. Figure 2.4 shows the general design features of a spiral-wound membrane module. The module consists of a permeate collection tube with a spiral formation of permeate spacers and feed spacers, which allow the flue gas and separated CO<sub>2</sub> to flow through the device.



**Figure .2.4:** Schematic design of a spiral-wound membrane module

### 2.3 PRESSURE DRIVEN MEMBRANE PROCESS

In pressure-driven membrane processes (reverse osmosis, nanofiltration, ultrafiltration, and microfiltration) a pressure exerted on the solution at one side of the membrane serves as a driving force to separate it into permeate and retentate. Membranes may be polymeric, organo-mineral, ceramic, or metallic, and filtration techniques differ in pore size, from no pores to porous membranes. Depending on the type of technique, small organic molecules, salts, macromolecules, or particles can be retained, and the applied pressure will differ (Bart et al. 2004)

There are a number of filtration systems that use membrane technology but the commonly used approach involves using pressure driven membrane technology. This is based on liquid being forced at high pressure through a filter membrane that has high surface area. The type of membrane used varies but is most broadly dependent on the particle size of the material being filtered.

There four basic pressure driven membrane filtration processes. In descending order of particle size being separated these include microfiltration, ultrafiltration, nanofiltration and reverse osmosis. Table 1.3 shows the different pressure driven membrane processes.

**Table2.3:** Different Pressure Driven Membrane Processes

	Pore size ( $\mu\text{m}$ )	Applied pressure (bar)	Typical application
Microfiltration	10-0.05	0.1-2	Separation of colloids and particles
Ultrafiltration	0.05-0.002	1-10	Separation of macromolecules
Nanofiltration	0.002-0.001	5-20	Separation of low MW solutes
Reverse osmosis	<0.001	10-100	Separation of low MW solutes

(Source:Souren, 2007, Pressure Driven Membrane Processes)

## 2.4 NANOFILTRATION MEMBRANE

Nanofiltration (NF) membrane separation has become important role nowadays in the industrial sector, such as water treatment, pharmaceutical, biochemical industries. In having advantages in low operation pressure, high permeate flux and high retention of multi-valent ion salts gives NF membrane has much advantage in becoming the current trends of membrane separation method(Zhang et.al., 2003).

Narrow openings between the molecules that have formed a crosslinked structure are the reason for the high rejection of ions is that the water can only pass through the membrane. These narrow opening do not go straight through the membrane, the tortuous path need to be through for the water molecules and the accompanied solutes to reach the permeate side from the feed side of membrane. The name nanofiltration was chosen because of the size of the pores is about 1 nanometer (nm) in diameter .The pores of NF membranes have a surface charge which results in that solutes are rejected both because of size and charge of the solvated species. It is possible for ions to shield the charges on the pore wall and still leave enough space in the pore for other ions to pass through when the pore size is larger. In such a case, the monovalent solute permeability through the membrane increase with the solute concentration and the rejection of monovalent ionic species becomes strongly dependent on the solute concentration(Eriksson et.al. 2005).

NF membranes have a composite thin-film structure with an active layer (50-100 nm thick) formed on top of a polysulfone film in turn supported on a non-woven polyester fiber backing. Diversity of physicochemical properties of active layer and aqueous solution constituents interactions with the active layer polymer of NF membranes has affected the water permeability and contaminants rejection characteristic. Generally, thinner active layers of NF membranes would give higher water permeability. Greater water permeability without deterioration in solute rejection could be achieved with NF membranes having thinner active layers because of both water and solute permeation are inversely proportional to the membrane active layer thickness (Suzuki et.al. 2007).

## 2.5 INTERFACIAL POLYMERIZATION

Thin film composite (TFC) was produced via polymerization where interface of two liquids which are insoluble to each other it takes place. TFC membranes with the selective layer on the top surfaces of porous substrates through interfacial polymerization have been considered also as RO membrane. It is suggested by *Rao et. al.*(2003) that TFC membranes should be improved under increased permeability of water and salt rejections, better fouling resistance and chlorine resistance in polyamides membrane to reduce the operating costs and energy consumption. TFC membranes that is regularly used is the polyamide composite membrane with skin barrier layer by the interfacial polymerization technique between two reactive monomers on the top of a resistant polysulfone or polyethersulfone porous support membrane (Ghosh et. al. 2009).

Diversity of applications and worldwide expansion of NF technology are result from the introduction of TFC membrane by interfacial polymerization in 1972 (Zhou et.al. 2005). Water molecules appear to flow preferentially through super-hydrophilic, molecular sieve nanoparticle pores, while solute rejection remains comparable to pure polyamide membranes. *Jeong et.al* (2007) believes that thin film nanocomposite materials represent a breakthrough in the design of reverse osmosis membranes - introducing new degrees of freedom in membrane design, which could lead to the next generation of high performance reverse osmosis membranes. It, is well known that the interfacial polymerization is diffusion-controlled and exists in a self-limiting phenomenon. Thus, the reaction time plays an important role in determining the extent of reaction (Mohammad et. al. 2003)

It is known from the literature that the concentration of the monomer in the organic phase plays an important role in the preparation of TFC membrane by interfacial polymerization; however, the concentration of the monomer in the aqueous phase is not the dominant factor for polymerization (Chen et. al.2002).

A novel polyester composite nanofiltration membrane was prepared Tang et. al. (2002) which studied the performance of TFC membrane was optimized by studying the

preparation parameters, such as the reaction time of polymerization, pH of aqueous phase and the concentration of reactive monomers by interfacial polymerization of TEOA and TMC on the polysulfone supporting membrane. The top skin layer thickness of the composite membrane increases with increasing polymerization time. The top layer will stop growing when the thickness of the thin layer is enough to prevent the monomer diffusing from one phase into the other phase. Thus, the permeability of the membrane is high due to the extent of cross-linking is low for a short reaction time. *Kim et. al* (2000) has stated that after a certain time of reaction, water flux will almost stay constant because of the selective layer is almost fixed. Organic phase is important in the preparation of TFC membrane by interfacial polymerization but the concentration of the monomer in the aqueous phase is not the dominant factor for polymerization (Chen et. al 2002).

## **2.6 TECHNOLOGIES OF HUMIC ACID AND ORGANIC MATTER REMOVAL**

Natural organic matter (NOM) exists in all water systems NOM removal has attracted much attention in water supply technologies such as adsorption, ozonation, chemical coagulation and membrane filtration have widely been studied because of its adverse effect on human health (Jone and Bryan, 1997). Natural organic matter (NOM) in ground waters mainly consists of partially decomposed plant material and most is found as dissolved organic matter such as humic substances (HS), hydrophilic acids, and simple organic compounds. HS make up 50±80% of the dissolved organic matter in natural waters (Thurman, 1985), and ground waters rich in NOM can be termed humic ground waters.

The presence of HS in water causes a yellow to brown colour, may result in the formation of trihalomethanes (THM's) during chlorination (Chadik and Amy, 1983), can result in microbial regrowth in distribution systems, and impedes the removal of iron and manganese from the water (Prakash and McGregor, 1983). While, hydrophilic and/or