

**DEVELOPMENT OF QUATERNARY SYSTEM
PES POLYMERIC HYBRID MEMBRANES WITH
BLENDING METHOD: STUDY ON THE EFFECT
OF POWDERED ACTIVATED CARBON AND
NANOSILICA**

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MEMBRANES WITH BLENDING METHOD: STUDY ON THE EFFECT OF
POWDERED ACTIVATED CARBON AND NANOSILICA**

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**A project report submitted in partial fulfilment of the
requirements for the award of the degree of
Bachelor of Civil Engineering (Environmental) with Honours**

**Faculty of Engineering and Green Technology
Universiti Tunku Abdul Rahman**

May 2023

DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

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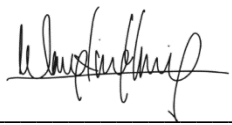
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Specially dedicated to
my beloved grandmother, grandfather, mother, father, brother and sister

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ABSTRACT

Membrane technology has found extensive use in wastewater treatment due to its outstanding ability in pollutant separation. In this study, surface modification is carried out by undergoes blending method to enhance the performance of the membrane. A study was conducted by blending polyethersulfone (PES) as polymer, N-methyl pyrrolidone (NMP) as solvent, nanosilica and powdered activated carbon (PAC) as additives to produce Quaternary System PES polymeric hybrid membranes. The Fourier transform infrared spectroscopy (FTIR) test will be carried out to analyse the functional group of nanosilica and PAC. Several tests are conducted to analyse the performance of the membrane that has been produced. The cross section of the produced membrane will be analyzed using Scanning Electron Microscopy (SEM). Pure water flux and solute rejection flux and membrane fouling performance of the produced membrane will be evaluated. Turbidity, color and chemical oxygen demand (COD) removal efficiency of domestic wastewater will also be tested with the quaternary system PES polymeric hybrid membranes. Among different weight percentage (wt.%) of additives blended in the membranes, the result shows that 2.5 wt.% nanosilica and 2.5 wt.% PAC membrane has the highest pure water flux which range from 133.863 L/m²h - 372.598 L/m²h while 0 wt.% nanosilica and 0 wt.% PAC membrane has the lowest pure water flux which range from 1.574 L/m²h - 4.738 L/m²h. 2.5 wt.% nanosilica and 2.5 wt.% PAC membrane also shows the highest volume of permeate with the shortest fouling time compared to other membranes. However, the highest treatment efficiencies were obtained by 1.5 wt.% nanosilica and 3.5 wt.% PAC

membrane for turbidity, color and COD removal which are 100%, 97.53% and 94.31% respectively.

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LIST OF SYMBOLS / ABBREVIATIONS

AC	Activated carbon
BSA	Bovine serum albumin
CO ₂	Carbon dioxide
COD	Chemical Oxygen Demand
DES	Deep eutectic solvent
FTIR	Fourier transform infrared spectroscopy
GO	Graphene oxide
HA	Humic acid
HNT	Halloysite nanotubes
ITF	Iron-tannin-framework
MC	Methylcellulose
MCM-41	Mobil Composition of Matter No. 41
MF	Microfiltration
MMM	Mixed-matrix membrane
NaCl	Sodium chloride
NF	Nanofiltration
NIPS	Non-solvent induced phase separation
NMP	N-Methyl-2-pyrrolidone
FAU	Formazin Attenuation Unit
OH	Hydroxyl
PA-6	Polyamide
PAC	Powdered Activated Carbon
PEI	Polyethylenimine
PEG	Polyethylene glycol
PES	Polyethersulfone
PSf	Polysulfone

PVC	Polyvinyl chloride
PVDF	Polyvinylidene fluoride
PVP	Polyvinylpyrrolidone
RO	Reverse osmosis
SEM	Scanning Electron Microscopy
SiO ₂	Silicon dioxide
TiO ₂	Titanium oxide
UF	Ultrafiltration
WT.%	Weight percentage

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CHAPTER 1

INTRODUCTION

1.1 Background

The production of domestic wastewater is rising quickly along with urbanisation. As the human population continues to grow, significant amounts of wastewater are generated daily from domestic, industrial, and agricultural sources. However, the demands of an expanding population and their water usage are not met by the renewal of freshwater supplies. Due of this, there is intense competition and unfairly distributed of the limited freshwater resources among many sectors (Obotey Ezugbe & Rathilal, 2020). Currently, a fifth of the world's population has water stress. Over 2.8 billion people across 48 nations are predicted to have water shortage issues by 2025. Nearly 700 million people in 43 different nations are now having trouble getting enough water (Kodali, 2017). When domestic wastewater treatment facilities are not available, several issues arise. The main issues that follow are the eutrophication of water bodies, pollution with organic chemicals, and the development of illnesses (Admirasari et al., 2022). Traditional wastewater treatment methods, such as activated sludge, are effective but can be energy-intensive and produce a significant amount of solid waste (Xu et al., 2020). This has led to an increasing interest in exploring alternative, more sustainable wastewater treatment methods, including the use of membrane technology.

Membrane technology has experienced significant growth as a technique to treat wastewater during this time. Membrane technology offers some potentials for wastewater treatment due to low energy requirements, low or no chemical usage, significant equipment size reduction, inexpensive capital costs and environmentally

friendly (Obotey Ezugbe & Rathilal, 2020). However, the main problem that occur in membrane technology is membrane fouling. Membrane fouling has the potential to significantly reduce productivity, permeate quality, shortened membrane lifespan and lead to high maintenance cost (Goh et al., 2018). Blending method is widely used in improving the membrane performance and hydrophilicity. Additives are added into PES membrane in blending method. Due to its hydrophilic nature, it can enhance hydrophilicity and minimize the fouling effects (Otitoju, Ahmad and Ooi, 2018). In this study, Quaternary system PES polymeric hybrid membranes is produced. In this study, the term "quaternary" refers to the use of four different materials that combine together to produce a membrane. Hybrid membrane is a type of membrane which can get advantages from both polymeric and inorganic membranes by combining polymer with inorganic/hybrid materials. Inorganic particles act as adsorbents and dispersed into a polymeric matrix (Cheng et al., 2017). In this study, Quaternary system PES polymeric hybrid membrane is produced by blending nanosilica and PAC as additives, PES as polymer and NMP as solvent to control the membrane fouling problem and enhance the membrane performance in a more sustainable and effective method.

The nanosilica and PAC can be extracted from rice husk and wood biomass. Rice husk is a waste product that is generated during the processing of rice. Rice husk is the source of nanosilica as rice husks have a high silica content (Moosa and Saddam, 2017). Wood biomass is also a solid waste if it is produced as a by-product or residue from industrial or agricultural processes, and is not utilized or sold for any other purpose. PAC can be extracted from wood biomass. Wood biomass mostly consists of lignocellulosic compounds (carbonaceous by-product) and the compound can be used as a precursor for manufacturing of activated carbon. Activated carbon can be synthesis to form PAC (Kharrazi et al., 2020). Blending nanosilica and PAC into a membrane can lead to significant improvements in membrane performance. Aside from its major advantages, the blending of nanosilica and PAC in membrane also has the potential to reduce waste by utilizing these materials which can be extracted from waste sources. This can lead to a more sustainable and environmentally friendly approach to membrane fabrication.

1.2 Problem Statements

The greatest obstacle to optimize the membrane performance is membrane fouling. Decreasing in membrane performance, reducing of membrane lifespan, high energy consumption, inclining of transmembrane pressure and other problems are the effects caused by membrane fouling (Liao et al., 2018). By blending nanosilica and PAC as additives in hybrid membrane, membranes with enhanced properties and improved performance can be produced. The synthesis of nanosilica and PAC from rice husk and wood biomass can be used as additives to blend into the PES hybrid membrane to solve with the membrane fouling problem in this study. As the membrane fouling in Quaternary system PES polymeric hybrid membrane in this study is controlled, the membrane performance will increase, hence enhancing the domestic wastewater treatment by investigating the parameters such as COD, turbidity and colour before and after the treatment. Blending nanosilica and PAC as additives in membrane not only improves membrane performance but also offer benefits of materials extracted from waste sources such as rice husk and wood biomass. This approach can lead to a reduction in waste production.

The improper disposal or management of waste such as rice husk and wood biomass have become a severe problem. Around 750 million tonnes of rice husks are grown each year, producing 160 million tonnes of rice husk that are eventually thrown on the environment as waste, filling up significant areas of landfills and contributing to pollution issues (Mosaberpanah & Umar, 2020). Burning rice husk can release pollutants such as particulate matter, carbon monoxide, and sulfur dioxide, which can have negative impacts on air quality and human health. Besides, most biomass wastes such as wood biomass, especially in poor nations, are not used or handled. They are either burnt publicly resulting in air pollution. Besides, when biomass waste is left to decay in the fields, it can cause soil degradation by depleting the soil of nutrients and organic matter, making it less fertile and reducing crop yields (Chen et al., 2021). These wastes can be synthesised to form additives that can be used in membrane manufacturing to enhance membrane performance. Due to its high amount of silica content, rice husk can be synthesis to become nanosilica (Mosaberpanah & Umar, 2020) while wood biomass can be used to produce powdered activated carbon (PAC) due to the presence of carbon content (Kharrazi et al., 2020).

1.3 Aims and Objectives

The following are the objectives of this thesis:

- i) To develop quaternary system PES polymeric hybrid membranes with nanosilica and PAC as additive using blending method.
- ii) To analyse the functional group of the nanosilica and powdered activated carbon (PAC) using Fourier transform infrared spectroscopy (FTIR).
- iii) To evaluate the performance of the quaternary system PES polymeric hybrid membranes in treating the domestic wastewater.

1.4 Scope of work

The functional group of the nanosilica and powdered activated carbon (PAC) will be analysed using Fourier transform infrared spectroscopy (FTIR). Quaternary system PES polymeric hybrid membranes will be fabricated by incorporating the prepared nanosilica and PAC using blending method. PES will act as polymer and NMP will act as solvent. Different weight compositions of nanosilica and PAC will be used when producing membrane dope solution. The cross-section surface morphology of quaternary system PES polymeric hybrid membranes will be analysed using Scanning Electron Microscopy (SEM). The domestic wastewater sample which will be used in this study will be collected from INDAH WATER SDN BHD RC KAMPAR. In order to determine the performance of quaternary system PES polymeric hybrid membranes, pure water flux, rejection flux and membrane fouling performance will be tested using dead-end module. Several parameters, including COD, turbidity, and colour removal efficiency, will be used to determine the membrane performance.

CHAPTER 2

LITERATURE REVIEW

2.1 Domestic wastewater

Domestic wastewater is defined by the World Health Organization (WHO) as water used for all customary domestic functions, such as consumption (drinking and cooking), hygiene (covering the fundamental requirements for personal and home cleanliness), and amenity use (such as car washing and grass watering) (Howard et al., 2020). The minimal amount of water needed for drinking and hygiene is 20 l/c/d, while the maximum amount, which includes bathing, meal preparation, washing, and personal hygiene, is at least 100 l/c/d (Wulan et al., 2022). Domestic wastewater from these sources will either be treated in septic tanks or linked to a centralised or communal sewerage system (Wulan et al., 2022).

2.1.1 Classification and Characteristics of domestic wastewater

As the human population continues to grow, there will be a corresponding increase in the production of domestic wastewater. This leads to various issues such as the eutrophication of water bodies, organic chemical pollution, and the spread of illnesses. Admirasari et al. stated that wastewater is recognised to be a potential source of water, nutrients and energy (Admirasari et al., 2022). Domestic wastewater is commonly categorized into black water and grey water. (Wulan et al., 2022). Black water is the wastewater that comes out of toilets and has high levels of nitrogen, phosphorus, and

organics. Grey water is any wastewater from sources other than the toilet, such as the sink, shower, and laundry (Wulan et al., 2022). This type of wastewater is considered to have a high volume but low strength, and presents significant potential for utilization and reutilization. (Oteng-Peprah, Acheampong and DeVries, 2018). COD value has a high concentration in domestic wastewater. It shows that there are high concentrations of nutrients and organic matter inside the domestic wastewater due to the organic compounds source such as residual food, oil and grease (Wulan et al., 2022).

2.1.2 Effects of untreated domestic wastewater

The major causes of water pollution include the discharge of untreated domestic wastewater, water tank leaks, marine dumping, industrial effluent waste, atmospheric deposition and radioactive waste. Haseena et al. stated that the water contamination is primarily caused by untreated domestic wastewater, which accounts for approximately 75% to 80% of the total. (Haseena et al., 2017). Hazardous substances present in untreated domestic sewage such as solid waste, plastic litter, and bacterial pollutants, contribute to water pollution. Some of the issues brought on by untreated domestic wastewater include eutrophication, rising treatment costs, a decline in the water's value as a recreational resource, dangers to human and animal health, oxygen loss, and unfavourable changes to the aquatic ecology (Haseena et al., 2017).

Untreated domestic wastewater is categorised as having low nitrogen strength. However, the rate of eutrophication may increase due to the exceeding ammonia concentration (Wijaya & Soedjono, 2018). Algal growth increases quickly as a result of eutrophication, and oxygen depletion, biodiversity reduction, fish kills, odour, and increased toxicity will occur. There will be increased competition among aquatic living organisms for dissolved oxygen (Wijaya & Soedjono, 2018). Besides, water sources contaminated with fecal matter can cause various waterborne infectious illnesses through the fecal-oral route of infection. The exposure to untreated domestic wastewater poses a health risk that can lead to respiratory illnesses, cancer, neurological disorders, diarrhoea and cardiovascular problems. Low water quality of

untreated domestic wastewater also endangers human and aquatic life, destroy crop production and contaminates the food (Haseena et al., 2017).

2.1.3 Domestic wastewater treatment

Domestic wastewater is collected from various homes and discharged in sewage and deliver to the wastewater treatment plants for treatment. Most wastewater treatment plants were constructed to prepare wastewater for reuse or release into streams or other receiving waterways (Kodali, 2017). Pretreatment, primary, secondary, and tertiary treatment are the four main processes in complete sewage treatment (Ullah et al., 2020).

Membrane filtration, adsorption, disinfection and advanced oxidation processes are the examples of tertiary treatment. Tertiary treatment is utilized to eliminate nutrients and other micropollutants that are present in the secondary effluent. Tertiary treatment can be also used to remove non-biodegradables, metals, soluble minerals, inorganic dissolved solids, colour, taste, odour and pathogens in wastewater (Poerio, Piacentini and Mazzei, 2019). In this study, membrane filtration (PES membrane) and adsorbents (additives) are discussed.

2.2 Membrane technology

By using a membrane as a barrier, two phases are separated from one another by being selectively impermeable to certain components (Asif & Zhang, 2021). When compared to conventional water treatment, membrane processes provide reliable technologies that may be used to provide drinkable water from both regular groundwater and sources with a compromised quality. membrane technology has gained acceptance as an efficient separation procedure for wastewater treatment due to its dependability and effectiveness of pollutant rejection, as well as the flexibility given by different membrane materials and pore sizes, (Esfahani et al., 2019).

2.2.1 Pressure Driven Techniques

Pressure-driven separation techniques are the most commonly used membrane processes in various stages of wastewater treatment, ranging from pre-treatment to post-treatment. Applications for pressure-driven membranes include desalination, wastewater reclamation, and water treatment (Asif & Zhang, 2021). To accomplish separation, the four main types of the pressure driven processes such as ultrafiltration, nanofiltration, nanofiltration and reverse osmosis rely on hydraulic pressure. The Table 2.1 below shows the difference of each type of pressure-driven membrane.

Table 2.1: The difference of each type of pressure-driven membrane.

Process	MF	UF	NF	RO	References
Pore size (µm)	0.05 - 10	0.002 – 0.05	0.001 – 0.002	<0.001	(Fatima, Du and Kommalapati et al., 2021)
Operating pressure (bar)	0.1 - 2	1 - 10	5 - 20	10 - 100	(Fatima, Du and Kommalapati et al., 2021)
Membrane type	Porous, asymmetric or symmetric	Micro porous, asymmetric	Tight porous, asymmetric, thin film composite	Semi porous, asymmetric, thin film composite	(Obotey Ezugbe & Rathilal, 2020)
Separation mechanism	Sieve	Sieve	Sieve, solution/diffusion, exclusion	Sieve, solution/diffusion, exclusion	(Li et al., 2021)
Material retained	Bacteria, suspended particles	Salt, lactose, glucose, micropollutants,	Macromolecules, colloids	Dissolved salts	(Gul, Hruza and Yalcinkaya, 2021)
Material passed	Water, dissolved solutes	Water, monovalent salts	Water, dissolved salts	Water	(Gul, Hruza and Yalcinkaya, 2021)

Membrane technology offers various advantages. For example, membrane technology utilizes zero chemical constituents, the permeate was safe for discharge and potential reuse (Fatima et al., 2021). Besides, membrane-based separation techniques offer high-quality treated effluent and are compact and robust. Membrane technologies also offer several additional advantages such as easily integrated with other processes which enabling hybrid processing, ease of scaling up, continuous separation, separation at mild conditions, low energy consumption, and lack of need for additives (Hakami et al., 2020). However, one of the primary challenges faced by membrane technology is membrane fouling.

2.3 Membrane fouling

Membrane fouling is the accumulation of different elements in the pores or surface of membrane that affects the amount and quality of the permeate. Besides causing flow reduction and reduce the life of the membranes reduce membrane lifetime, productivity, and permeate quality, membrane fouling also needs extremely expensive chemical cleaning in order to be resolved (Zulkefli et al., 2022). It is a main problem will occur in membrane technologies.

2.3.1 Categories of membrane fouling

Membrane fouling can be classified into three categories such as internal fouling, external fouling and concentration polarisation fouling (Du et al., 2020). Internal fouling, which is also known as pore blockage, occurs when solutes and colloidal particles are deposited and adsorbed on the interior of the membrane pores, resulting in fouling. External fouling is the term used to describe the deposition of particles, macromolecules and colloids on the surface of the membrane. On the surface of membrane, external fouling creates a layer of fouling. There are two types of fouling

layers: cake layers and gel layers (Du et al., 2020). The figure 2.1 below shows the pore blockage, gel layer and cake layer in the membrane fouling.

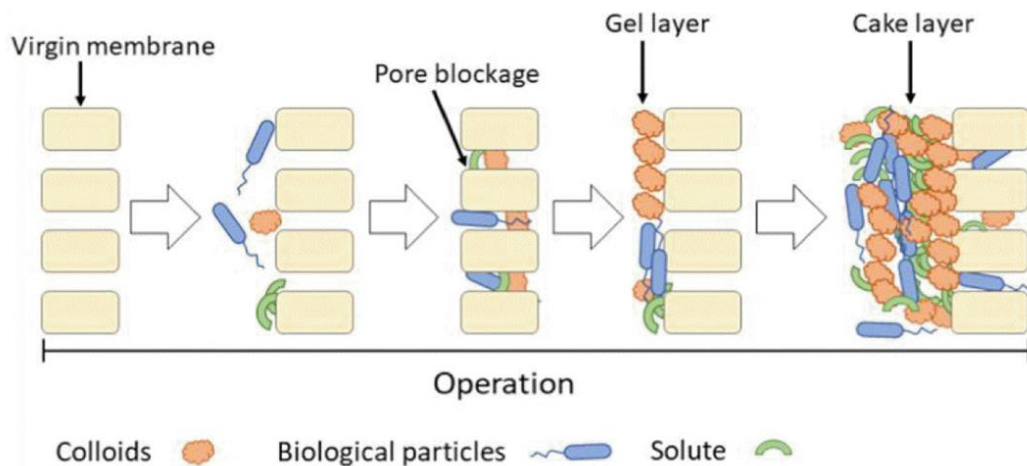


Figure 2.1: Different categories of membrane fouling (Li et al., 2022)

In a gel layer blockage, a gel layer which composed of deposited colloids, solute or biological particles develops on the fouling layer's surface adjacent to the membrane caused by the difference in pressure between the feed and permeate sides of the membrane. When a membrane is clogged, a cake layer is created when solids with higher particle sizes (exceeding the membrane pore size) start to accumulate on the surface of membrane during filtration process (Li et al., 2022). Increased solute concentration close to the membrane surface lead to the formation of a boundary layer close to the membrane walls, which causes concentration polarisation. Surface fouling is caused by concentration polarisation and modification techniques often target both due to their interdependency simultaneously (Golgoli et al., 2021).

2.3.2 Classification of membrane fouling in term of degree of foulant removal

Membrane fouling can be classified into two types based on the degree of foulant removal such as reversible fouling and irreversible fouling. The portion of foulants that may be removed physically, such as backwashing or operating membranes

intermittently while performing cross-flow filtration, is referred to as reversible fouling. Non-reversible fouling is defined as fouling that cannot be eliminated by physical cleaning but instead requires chemical cleaning. The loose deposition of contaminants on the membrane surface causes reversible fouling while the obstruction of membrane pores and the strong adhesion of contaminants to the membrane surface causes irreversible fouling. (Du et al., 2020).

2.3.3 Classification of membrane fouling in term of source of foulants

In terms of the source of foulants, fouling can be categorized as biofouling, organic fouling, inorganic scaling or colloidal fouling as shown in Figure 2.2.

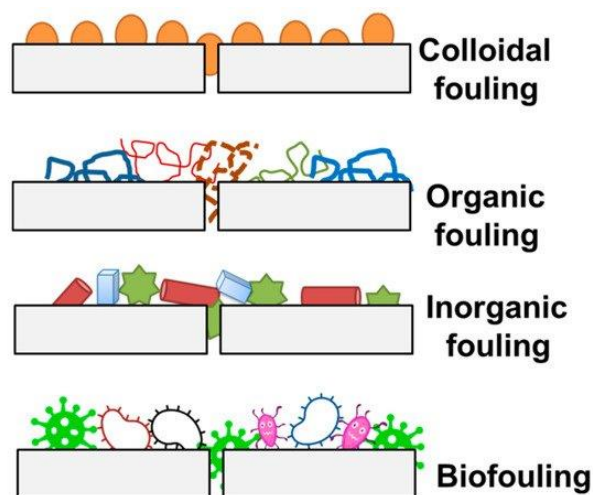


Figure 2.2: Different types of membrane fouling in term of source of foulants (Gul, Hruza and Yalcinkaya, 2021).

Biofouling refers to the adhesion and proliferation of microorganism on membrane surfaces. It can also be defined that the level of biofilm formation has reached to an unacceptable level. The organic fouling occurs as a result of the presence of organic matters. Humic compounds, polysaccharides, proteins, lipids, nucleic acids, amino acids, organic acids, and cellular components are a few examples of these organic substances. Inorganic scaling or inorganic fouling refers to the accumulation

of inorganic materials on membrane surfaces or inside membrane pores. Scaling occurs when inorganic scalants that have a limited solubility or a significant amount concentration of ions in the water exceed the equilibrium solubility product, leading to supersaturation. These substances deposition on the surface or pores of the membrane will cause scaling. Colloidal fouling is membrane fouling caused by colloids or particles accumulating on the host materials. There are two categories of common colloidal foulants such as inorganic foulants and organic macromolecules. Aluminium silicate minerals, silica, iron oxides/hydroxides are the main inorganic pollutants in nature's water, whereas polysaccharides, proteins, and other naturally organic matters make up the majority of the water's organic macromolecules (Jiang, Li and Ladewig, 2017).

2.3.4 Previous study about the membrane fouling

Due to the membrane rejection flux declination of previous studies range from 20% to 40%, when the membrane rejection flux declination in this study more than or equal to 30% will set as membrane fouling. When the membrane reaches the membrane fouling point, the rejection flux test will be stopped. The table 2.2 below shows the previous study of the membrane flux reduction.

Table 2.2: Previous study about the membrane flux reduction.

Membrane type	Time of permeation (min)	Pollutants	Membrane flux reduction	Reference
HNT-SiO ₂ -PEI blended PVC	50	BSA solution	Range from 20% to 40%	(Vatanpour et al., 2022)
MCM-41 membrane	50	Methyl green dye	Range from 25% to 34%	(Alardhi et al., 2020)
Nano-hybrid PES/nanosilica/PEG	480	Produced water	30.6%	(Kusworo, Aryanti and Utomo, 2018)

PVDF-AC	60	HA+BSA	Range from 26% to 29%	(Liu et al., 2018)
PVDF-TiO ₂ -AC	60	HA+BSA	Range from 32%-34%	(Liu et al., 2018)

2.4 Solutions to overcome membrane fouling

Several fouling control strategies, including membrane monitoring and cleaning, pre-treatment and membrane modification are used to control membrane fouling. The table 2.3 shows some of the membrane fouling control mitigations and its examples.

Table 2.3: Membrane fouling control mitigations and its examples.

Mitigation	Examples	References
Membrane monitoring and cleaning	Mechanical cleaning	(Kim, Li and Ghaffour, 2020)
	Chemical cleaning	
Pre-treatment	Coagulation	(Jiang, Li and Ladewig, 2017).
	Flocculation	
	Sedimentation	
	Media filtration for removal of larger or coagulated particles	
Membrane modification	Physical blending	(Du et al., 2020).
	Chemical copolymerization	

In the physical blending method, the membrane material matrix is mixed with the modifying additive in a specific ratio physically, without any reaction between the additive and the bulk of the membrane material. The benefits of both obtaining better

cast film materials and balancing the pros and cons of the bulk membrane material and additives can be achieved through physical blending modifications (Du et al., 2020).

Two of the major disadvantages of blending method are the homogeneity of the final product and the miscibility of the blend's component parts (Halder et al., 2017). For advantages, blending method is cost-effective and easily to be applied for PES membrane preparation. To enhance the performance and hydrophilicity of PES membranes in moderate situations, the blending method provides a versatile and convenient process. The addition of extra functional groups and an increase in the water flux as a result of the increased effective membrane surface area are two additional benefits of blending method with hydrophilic additives (Otitoju, Ahmad and Ooi, 2018). In this study, blending method is used to blend PES with additives to form a hybrid membrane in order to enhance the membrane performance.

2.5 Polyethersulfone (PES) hybrid membrane

Hybrid membrane is a membrane that integrate polymer with inorganic or hybrid materials. Hybrid membranes can feature a variety of interactions between the polymer matrix and fillers, including hydrogen bonds, van der Waals forces, covalent bonds, and ionic bonds (Cheng et al., 2017). The use of a hybrid membrane offers several benefits such as high recovery rates, capacities to treat challenging feeds and minimized environmental impact (Ahmed, Hashaikeh and Hilal, 2020). In this study, polyethersulfone (PES) is used as polymer to create the Quaternary System PES polymeric hybrid membrane. Figure 2.3 illustrates the PES, a well-known polymeric material for the manufacture of ultrafiltration (UF), microfiltration (MF), and gas separation membranes.

PES is used in this study as polymer due to its high hydrolytic stability, high heat resistance, and capacity to maintain mechanical properties in hot and humid conditions. PES has a glass transition temperature (T_g) of 230°C, which is suitable for pH toleration and resistance to chlorine. The hydrophilicity of PES high and can

improve the penetration and antifouling capabilities of UF membranes (Shockravi et al., 2017).

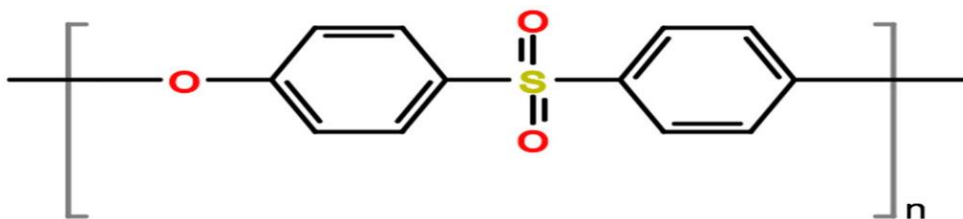


Figure 2.3: The chemical structure of PES (Alenazi et al., 2017).

However, the inherent hydrophobic properties of PES that allow hydrophobic interactions between the membrane and solute in the feed solution, resulting in non-specific protein adsorptions on both the membrane surface and within the membrane pores. This can result in significant membrane fouling and a reduction in permeation flux. This is a significant problem in membrane filtration technology that leads to an increase in energy consumption, a reduction in membrane lifespan, and unpredictability in separation performance. To get around these restrictions, it has been known to modify PES membranes using the blending method (Shockravi et al., 2017). The Table 2.4 below shows some of the previous study by blending the PES hybrid membrane with different types of additives.

Table 2.4: Previous study about the example of blended PES hybrid membrane with different additive.

Membrane type	Advantages of additives	Membrane improvement	References
PES-GOPVP membranes Additive: Graphene oxide (GO) and polyvinylpyrrolidone (PVP)	High chemical stability, nontoxicity, great solubility in polar solvents	Water contact angle reduce from 72° (pure PES) to 42° (1.0 wt.% GOPVP) and pure water flux increase from 32L/m ² h (pure PES) to 230L/m ² h (1.0 wt.% GOPVP)	(Junaidi et al., 2019)
ITF/PES ultrafiltration membranes Additive: Iron-tannin-framework (ITF) complex is formed by combining iron ion (FeIII) with tannic acid (TA).	Inorganic and organic building blocks, abundant phenolic hydroxyl groups that improve hydrophilic.	Water contact angle reduce from 75° (pure PES) to 34° (0.9 wt.% ITF) and pure water flux increase from 197.8L/m ² h (pure PES) to 246.4L/m ² h (0.9 wt.% ITF)	(Fang et al., 2017)

<p>PES/chitosan membranes Additive: chitosan</p>	<p>The hydroxyl group (OH) in chitosan cause high degree of hydrophobicity, good biocompatibility, non-toxicity, low cost, renewable resource, absorbents for variety of dyes, macromolecules and heavy metals</p>	<p>Water contact angle reduce from 84.2° (pure PES) to 71.1° (1.0 wt.% chitosan) and pure water flux increase from about 4 L/m²h (pure PES) to 11.2 L/m²h (0.9 wt.% ITF)</p>	<p>(Fathanah et al., 2020)</p>
<p>PA-6/PES blend membranes Additive: Polyamide (PA-6) is produced by combining biphenyl-2,2-dicarboxylic acid with a diamine (DA) monomer</p>	<p>The polyamide backbone in the PA-6 contains highly polarized groups in the form of -C=S-, -C=N-, and -C-NH- linkages increase hydrophilicity, permeability and antifouling properties</p>	<p>Water contact angle reduce from 75.6° (pure PES) to 65.2° (5.0 wt.% PA-6) and pure water flux increase from about 7.5 L/m²h (pure PES) to 80.4 L/m²h (2.0 wt.% PA-6)</p>	<p>(Shockravi et al., 2017)</p>
<p>PES/PVP/DES membrane Additive: Polyvinyl pyrrolidone (PVP) and ethaline deep eutectic solvent (ethaline DES)</p>	<p>Increase in membrane porosity and more macrovoids within the membrane matrix, increase the flux efficiency, increase hydrophilicity and enhance viscosity.</p>	<p>Water contact angle reduce from 55.6° (pure PES) to 42.3° (1.0 wt.% PVP/4.0 wt.% DES), maximum pure water flux at 241.3 L/m²h (1.0 wt.% PVP/2.0 wt.% DES) and viscosity increase from 51.3% (pure PES) to 58.2% (1.0 wt.% PVP/4.0 wt.% DES)</p>	<p>(Vatanpour et al., 2020)</p>

PES/GO membrane Additive: Graphene Oxide (GO)	Due to its large surface area and functional groups (OH, COOH and GO), it can absorb suspended and water-soluble compounds. It also increases in the amount of flux passing through the membrane	More hydrophilic membrane as water contact angle reduce from 79.3° (pure PES) to 61.8° (5.0 wt.% GO), both heavy metal removal such as Zn removal and dye rejection such as methylene blue achieve about 90% (3.0 wt.% GO)	(Marjani et al., 2020)
PES/PVP/PEG membrane Additive: Polyethylene glycol (PEG) and polyvinylpyrrolidone (PVP)	PVP exhibits better compatibility with PES due to its amorphous nature, closer glassy transition temperature (T _g), and solubility parameters while PEG has a noticeable impact on membrane permeability.	More hydrophilic membrane as water contact angle reduce from 72.8° (pure PES) to 64.8° (0.4 wt.% PVP 0.2 wt.% PEG), maximum pure water flux at 25.2L/m ² h (0.3 wt.% PVP/PEG) and maximum urea solution permeability at 19.8 L/m ² h (0.3 wt.% PVP/PEG)	(Hasheminasab, Barzin and Dehghan, 2020)

2.6 Additives

Phase inversion's fundamental progression can be changed by using alternative membrane-forming polymers, solvents as well as non-solvent or polymeric additives. The polymer and any potential non-solvent or polymeric additions must be dissolved in the proper solvent before creating a membrane casting solution (Kahrs & Schwellenbach, 2020). The casting solution may be improved by adding the suitable additives which act as pore producers and surface modifiers, to increase the permeability of the membranes while also enhancing membrane structure and morphology. Many sorts of additives in the dope solution are being used to produce NF membranes with superior performance. In most cases, the additions can be categorized into low molecular weight organic chemicals, surfactants, amphiphilic copolymers, inorganic salts, water soluble polymers, and inorganic nanoparticles (Vatanpour, Dehqan and Harifi-Mood, 2020). In this study, the additives used are nanosilica and PAC.

2.6.1 Nanosilica

One of the agricultural waste items, rice husk, is coated over a rice seed grain and being used to produce nanosilica or silica (Moosa and Saddam, 2017). Similar to other common organic fibres, rice husk has a chemical makeup that includes 40-50% cellulose, 25–30% lignin, 15-20% ash, and 8–15% moisture. (Jyoti et al., 2021). Compared to other agricultural plants, rice husks have a high silica content and may absorb a variety of silicates and minerals from the soil (Moosa and Saddam, 2017). If rice husk is burned, the majority of the components slowly evaporate until silicates are produced. The purity and surface area of the produced rice husk ash after burning may be reduced by a number of metallic contaminants, including Fe, Na, K, and Ca. By leaching rice husk in hydrochloric or sulfuric acid and then regulating combustion, high quality silica (above 99%) can be obtained by removing these metallic impurities. Sol –Gel method, Precipitation Method and In situ emulsion polymerization are the methods to synthesis nanosilica (Moosa and Saddam, 2017).

In this study, nanosilica is used as one of the additives to produce Quaternary System PES polymeric hybrid membrane. It is to reduce the rice husk waste produced and also improve the Quaternary System PES polymeric hybrid membrane performance. The Table 2.5 below shows the advantages and disadvantages of nanosilica while the Table 2.6 below shows some of the previous study about the nanosilica as an additive.

Table 2.5: Advantages and disadvantages of nanosilica.

Advantages	Disadvantages	References
<ul style="list-style-type: none"> • Low cost • Low toxicity to aqueous system • Good monodispersity • High water affinity, cause high permeability • Ability to generate OH-bond • Moderate production process • Ease of modification • Prevent the fouling of polymeric membrane due to hydrophilicity or antibacterial characteristics • Dispersed at low concentrations ranging from 0.10% to 0.30% both outside and inside the PES matrix. 	<ul style="list-style-type: none"> • High doping nanosilica concentrations pose the risk of membrane pore clogging. • Aggregation occur in high concentration interval ranging from 0.40 to 0.50%. 	<ul style="list-style-type: none"> • (Lin et al., 2016) • (Mulyati et al., 2020)

Table 2.6: Previous studies about the nanosilica as an additive.

Additive	Sources	Pollutants	Types of membrane	Water contact angle	Rejection efficiency	Permeability coefficient	References
	Rice husk or sugarcane powder	Humic acid	PES membrane	82° (pure PES) → 52-60° (5 wt.% rice husk silica and 5 wt.% sugarcane bagasse silica)	78% (pure PES) → 62-67% (5 wt.% rice husk silica and 5 wt.% sugarcane bagasse silica)	10.3 L/m ² .h.bar (pure PES) → 23.7-29.5 L/m ² .h.bar (5 wt.% rice husk silica and 5 wt.% sugarcane bagasse silica)	(Mulyati et al., 2020)
Nanosilica	Rice husk	Humic acid	PES membrane	-	75% (pure PES) → 62% (5 wt.% rice husk silica)	Double of the flux (5 wt.% rice husk silica)	(Mulyati et al., 2018)
	Synthesis		PVDF membrane	-	-	The pure water permeability increases by 4 times (3 wt.% silica)	(Fernandes et al., 2018)

Additive	Sources	Pollutants	Types of membrane	Water contact angle	Rejection efficiency	Permeability coefficient	References
	Synthesis	NaCl	PES membrane	75° (pure PES) → 65° (1.5 wt.% nanosilica)	Increase about 10% (1.5 wt.% nanosilica)	Double of the flux (1.5 wt.% nanosilica)	(Kusworo and Utomo, 2017)
Nanosilica	Synthesis	COD (emulsified oils and glycols)	PES membrane	-	Increase about 10% (1.0 wt.% nanosilica)	Highest flux of 28.5 L/m ² .h.bar (0.5 wt.% nanosilica)	(Kusworo et al., 2017)
	Rice husk	COD (Domestic wastewater)	PES membrane	-	Increase about 5% (5.0 wt.% nanosilica)	The pure water permeability increase by 6 times (5 wt.% nanosilica)	(Chan, 2021)

2.6.2 Powdered Activated Carbon (PAC)

Activated Carbon (AC) is a crude form of graphite and due to the presence of graphite structure, AC has large surface area which can adsorb the compounds in large range. Activated carbon can be produced from biomass wastes such as wood waste (Fazal-ur-Rehman, 2018). By using biomass waste to produce activated carbon, it is a smart pollution management method. First, it can fix the carbon in biomass, which stops the creation of CO₂ or CH₄. Besides, it can make produce activated carbon with industrial applications and low environmental impact that can be incorporated into the soil and the natural carbon cycle. Wood biomass mostly consists of lignocellulosic compounds (Danish & Ahmad, 2018). The most accessible and reasonably priced carbonaceous by-products that might be employed as a precursor for the manufacture of activated carbon are lignocellulosic wastes (Kharrazi et al., 2020). Hemicellulose, cellulose and lignin are the examples of lignocellulosic components. The C-O-C and C-C bonds in hemicellulose, cellulose or lignin provide a high amount of carbon. Wood waste as a carbon-rich material need to undergo activation process which initiated with the carbonization of the high carbon content wood wastes. Carbonization is used to remove volatile substances from raw materials at low temperatures. The process of activation occurs in the presence of supporting gases at temperatures ranging from 700°C to 1100°C. This prevents CO₂ production and produce activated carbon with holes of various sizes (Danish & Ahmad, 2018).

PAC is one of the main forms of AC. This is crushed carbon with a size that is primarily under 0.18 mm (Fazal-ur-Rehman, 2018). In this study, as to improve the membrane performance and control the waste such as wood biomass, powdered activated carbon is used as one of the additives to produce Quaternary System PES polymeric hybrid membrane. The Table 2.7 below shows the advantages and disadvantages of PAC while the Table 2.8 below shows some of the previous study about the PAC as an additive.

Table 2.7: Advantages and disadvantages of PAC.

Advantages	Disadvantages	Reference
<ul style="list-style-type: none"> • Fouling reduction. • Adsorption of the foulants and subsequent biodegradation. • Raising critical flux. • Improve scouring of particles on the surface of membrane. • Enhance the strength of microbial flocs. • Offers a solid support for bacterial development. • Large available specific surface area, enhancing adsorption kinetics. • Reduce the concentration of polymer and form large pores. 	<ul style="list-style-type: none"> • Cause additional sludge or solid handling problems. • One time usage, replenish process is required. • Drawbacks occur if there are excessive PAC used in treatment system. • The scouring is feeble due to its small size, especially for adherent gelatinous materials. 	<ul style="list-style-type: none"> • (Kårelid, Larsson and Björleinius, 2017) • (Ng, Bashir and Ng, 2018) • (Lei et al., 2019) • (Aghili et al., 2017)

Table 2.8: Previous studies about the PAC as an additive.

Additive	Additional additive	Pollutants	Types of membrane	Water contact angle	COD Removal efficiency	Colour Removal efficiency	Permeability coefficient	References
	-	POME	PES	-	10.11% (pure PES) → 81.65% (11wt.% PAC)	35.43% (pure PES) → 67.21% (11wt.% PAC)	89.3 L/m ² .h (pure PES) → 231.39 L/m ² .h.bar (11wt.% PAC)	(Ng, Bashir and Ng, 2018)
Powder activated carbon (synthesis)	methylcellulose (MC)	-	polysulfone (PSf)	66° (pure PES) → 48.5° (2.5wt.%PA C / 3.0wt.%MC)	-	-	4.71 L/m ² .h.bar (pure PES) → 39.85 L/m ² .h.bar (2.5wt.%PAC / 0.5wt.%MC))	(Nadour, Boukraa and Benaboura, 2019)
	-	Stabilized landfill leachate	polyvinylidene fluoride (PVDF)	-	Maximum reading of 36.63% (14.9wt.% PVDF / 1.0wt.%PAC)	Maximum reading of 49.50% (14.9wt.% PVDF / 1.0wt.%PAC)	61.10 L/m ² .h (14.9wt.% PVDF / 1.0wt.%PAC at 0.64bar)	(Abuabdou et al., 2021)

-	Domestic wastewater	dynamic membrane bioreactor (DMBR)	-	88.9% (0g/L PAC) → 90.3% (13g/L PAC)	86.4% (0g/L PAC) → 92.9% (13g/L PAC)	Stable flux with 15 and 5 L/m ² (3g/L PAC) (higher flux than 0 and 1 g/L PAC)	(Hu et al., 2017)
	Domestic wastewater	PES	-	61.74% (0wt.% PAC) → 73.04% (5wt.% PAC)	92.34% (0wt.% PAC) → 95.06% (5wt.% PAC)	40.56 L/m ² .h (pure PES) → 140.13 L/m ² .h (0.5wt.% PAC)	(Kee, 2022)
-	cheese whey wastewater (CWW)	powdered activated carbon mixed-matrix membrane (PAC-MMM)	Almost constant	39% (neat membrane) → 44% (0.5wt.% PAC-MMM)	-	120 L/m ² .h (neat membrane) → 198 L/m ² .h (0.5wt.% PAC-MMM)	(Aghili et al., 2017)

CHAPTER 3

METHODOLOGY

3.1 Overview of methodology

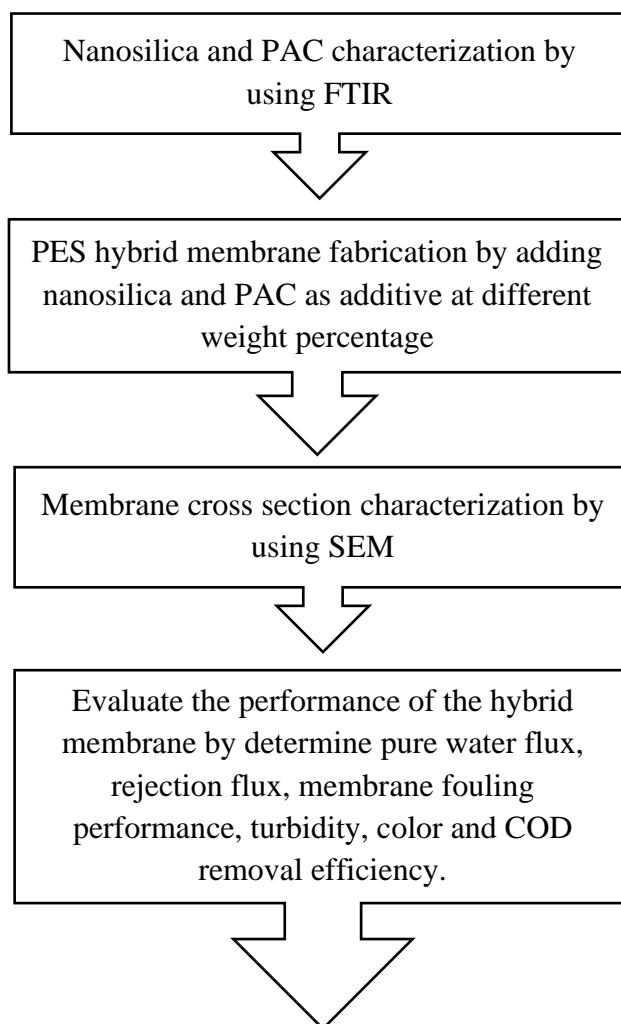


Figure 3.1: The flow chart of the experiment methodology.

3.2 Domestic wastewater samples collection

The domestic wastewater is collected from INDAH WATER SDN BHD RC KAMPAR that is located near Taman Mahsuri. The domestic wastewater samples are stored inside a refrigerator in the EV lab (E003) which located in Block E under temperature of 4°C. This is to ensure that the domestic wastewater samples do not undergoes the chemical or biological reactions and remains at its original condition.

3.3 Nanosilica and PAC Characterization

3.3.1 Functional Group Analysis

Fourier transform infrared spectroscopy (FTIR) is used to determine the chemical functional group that present in nanosilica and powder activated carbon. The test is carried out by using SEM machine which is located at Faculty of Science, UTAR (Block D).

3.4 Preparation of quaternary system PES polymeric hybrid membranes

3.4.1 Dope preparation

With the use of N-Methyl-2-pyrrolidone (NMP) as a solvent; nanosilica and PAC as additives, the dry and wet phase inversion method is applied to create a quaternary system PES polymeric hybrid membrane. The mass of nanosilica and powder activated carbon are altered in order to determine how it affects the performances of membranes in domestic wastewater treatment. The calculation below shows the steps to calculate the mass of polymer, nanosilica and PAC needed with different ratio.

Example of calculation of 15g of PES polymer with 5 wt. % of nanosilica and 0wt. % of PAC

Mass of nanosilica required

$$= \text{Mass of PES polymer} \times \text{nanosilica weight percentage}$$

$$= 15\text{g} \times 5\%$$

$$= 0.75\text{g mass of nanosilica needed}$$

Mass of PES polymer required

$$= \text{Mass of PES polymer} - \text{Mass of nanosilica required}$$

$$= 15\text{g} - 0.75\text{g}$$

$$= 14.25\text{g}$$

The Table 3.1 below shows the mass ratio of polymer PES, solvent NMP, nanosilica and PAC.

Table 3.1: Mass ratio of polymer PES, solvent NMP, nanosilica and PAC (Mahdavi and Moradi-Garakani, 2017) (Ali et al., 2021).

Membrane	Polymer PES (g)	Solvent NMP (g)	Mass of nanosilica (g)	Mass of PAC (g)
0 wt.% nanosilica and 0 wt.% PAC	15.000	85.000	0.000	0.000
5 wt.% nanosilica and 0 wt.% PAC	14.250	85.000	0.750	0.000
0 wt.% nanosilica and 5 wt. % PAC	14.250	85.000	0.000	0.750
2.5 wt.% nanosilica and 2.5 wt.% PAC	14.250	85.000	0.375	0.375
1.5 wt.% nanosilica and 3.5 wt.% PAC	14.250	85.000	0.225	0.525
3.5 wt.% nanosilica and 1.5 wt.% PAC	14.250	85.000	0.525	0.225

The PES polymer, nanosilica and PAC were dried overnight at 90°C in vacuum oven before dope preparation. 85 g of NMP solvent was heated on a heating mantle and stirred by using magnetic stirrer until a temperature reach between 55°C and 65°C

as shown in Figure 3.2. PES Polymer was added with certain weight by using a spatula. The PES polymer was ensured fully dissolved in the solvent. After the heat was turned off, the stirring operation was continued. The dope solution was cool down to room temperature. The cooled dope solution was poured into the reagent bottle as shown in Figure 3.3. The different weight percentage of nanosilica and PAC were added into different reagent bottles to mix with the dope solution. The reagent bottles were put into the sonicator bath for well mixed under the constant time of 8 hours as shown in Figure 3.4. The well mixed dope solution was left overnight to remove air bubble.



Figure 3.2: The set up of dope preparation apparatus.

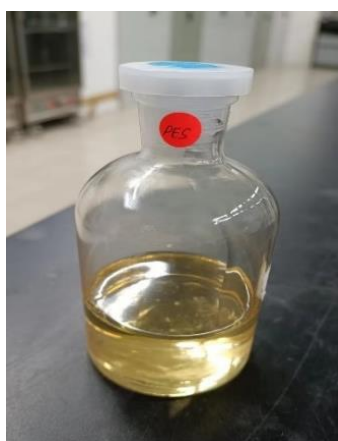


Figure 3.3: The dope solution in reagent bottle.



Figure 3.4: Reagents bottles in the sonicator bath.

3.4.2 Membrane fabrication

The hybrid membrane was casted by using membrane auto casting machine which located in Block J - Engineering workshop as shown in Figure 3.5. A casting knife with 10 micrometer thick was used to cast the prepared dope solution on a glass plate. Phase inversion process was carried out by dipping the casted glass plate into a non-solvent water bath which contain distilled water. Approximately 5 to 10 minutes was needed for the membrane to separate from the glass plate when submerging in the water bath. The membrane was washed and immersed in the distilled water for 24 hours (shown in Appendix B) and then was immersed in methanol solution for 8 hours undergoes post treatment. The methanol solution which immersed with membranes should be covered (shown in Appendix C) as methanol solution will evaporate under room temperature. The membrane was then dried at room temperature as shown in Figure 3.6.



Figure 3.5: Membrane auto casting machine.



Figure 3.6: The membranes were dried at room temperature.

3.5 Hybrid Membrane Cross Section Surface Morphological Characteristic Analysis

The Scanning Electron Microscopy (SEM) is used to investigate the cross-section surface morphology of the quaternary system PES polymeric hybrid membranes. The machine is located at Faculty of Science, UTAR (Block D).

3.6 Hybrid Membrane Performance Analysis

3.6.1 Pure Water Flux

Dead-end membrane test rig (shown in Appendix A) is used to determine the pure water flux. The equipment is located at Environmental Engineering Workshop, UTAR (Block J). The membrane was cut into symmetrical form with diameter of 50 mm. The circular membrane with an active surface layer was placed in the direction of feed inlet. The dead-end membrane test rig was set up and the distilled water was used as feed solution to conduct the permeability test. The pressure of **0.5 bar, 1.0 bar and 1.5 bar** was applied onto the membrane for the test. The permeate volume was set at **10ml** and the time for the permeate to completely passing through the membrane was recorded. The pure water flux, J was calculated with the equation (Junaidi et al., 2019)

$$J = \frac{V}{A\Delta t} \text{-----(3.1)}$$

J = Pure water flux (L/m²h)

V = Volume of permeate (L)

A = Effective area of membrane (m²)

Δt = Sampling time (h)

3.6.2 Application in Domestic wastewater

Rejection flux and membrane fouling performance is tested. Rejection flux test had the same procedures with pure water flux. However, domestic wastewater sample from INDAH WATER SDN BHD RC KAMPAR was used as feed solution to replace distilled water in the rejection flux test. The pressure of 1.5 bar was applied onto the membrane used for the test. The permeate volume was set at 5ml and the time for the permeate to completely passing through the membrane was recorded. The rejection flux, J was calculated with the equation (3.1). The rejection flux will be first measured at 5 minutes to get the initial flux so that the membrane will be completely wetted. For the subsequent flux will be collected at every 30 min and compared with the first rejection flux to calculate the rejection flux reduction with the equation (3.2).

$$R = \left(1 - \frac{J_2}{J_1}\right) \times 100\% \text{-----(3.2)}$$

R= Membrane rejection flux reduction (%)

J₁ = First rejection flux (L/m²h)

J₂ = Final rejection flux (L/m²h)

(Shockravi et al., 2017)

The experiment will be continued until the membrane fouling occur. Based on the previous study in Table 2.2, the membrane fouling will have the membrane rejection flux reduction from 20% to 40%. In this study, 30% is set as the membrane fouling. When the membrane rejection flux reduction is equal or more than 30%, the

Dead-end membrane test rig will be stopped. The permeate will then undergo the turbidity, color and COD test.

3.6.3 Turbidity Rejection Efficiency

DR/890 Colorimeter (shown in Appendix D) programme 95 is used to measure the turbidity before and after the domestic wastewater samples from INDAH WATER SDN BHD RC KAMPAR passing through the hybrid membrane. The turbidity meter needs to be calibrated before conducting the test. The samples were then added to the sample cell until the marked line is reached. The sample cells were cleaned with a clean cloth before the measurement to prevent inaccurate findings. Formazin Attenuation Unit (FAU) are used to measure the turbidity of the sample. The rejection coefficient was calculated by using the equation (3.3).

$$R = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \text{-----(3.3)}$$

R = Rejection coefficient (%)

C_p = Solute concentration in permeate (mg)

C_f = Solute concentration in feed (mg)

(Fathanah et al., 2020)

3.6.4 Color Removal

The color of the domestic wastewater samples from INDAH WATER SDN BHD RC KAMPAR before treated by using quaternary system PES polymeric hybrid membranes are measured using DR/890 Colorimeter. The programme 19 in the equipment is set to measure the color. The 10 ml of samples and distilled water is poured into two empty glass vials respectively. Distilled water is served as blank sample and set as zero. The obtained color value is in Platinum-Cobalt scale, PtCo.

The measurement is repeated to get more accurate average reading. The color removal coefficient was calculated by using the equation (3.3).

3.6.5 COD Removal

The COD digester is preheated until 150°C. 2 ml of distilled water are pipetted into a high range vial at a 45° angle to create a blank solution. The bottle is gently tilted many times for mixing after adding. For several samples of treated wastewater, these two stages are repeated. The well-mixed vials were then put into the COD digester for two hours digestion process at constant 150°C. The vial was moved to a cooling rack after two hours for cooling. The vials' external surfaces were cleaned with tissue paper before measurement. The COD of the domestic wastewater samples from INDAH WATER SDN BHD RC KAMPAR are tested by using DR/890 calorimeter. The programme 17 in the equipment is set to measure the COD. The vial containing distilled water was tested first to act as the zero base solution, and the COD readings of the remaining sample vials were then determined. For greater precision of the results, the COD measurement for each vial was performed numerous times. The COD removal coefficient was calculated by using the equation (3.3).

CHAPTER 4

RESULT AND DISCUSSION

4.1 Characterization of nanosilica and PAC

4.1.1 Fourier transform infrared spectroscopy (FTIR) of nanosilica

The range of FTIR spectrophotometer measurements of nanosilica was carried out from 4000-400 cm^{-1} . The FTIR spectrum of the nanosilica is shown in Figure 4.1.

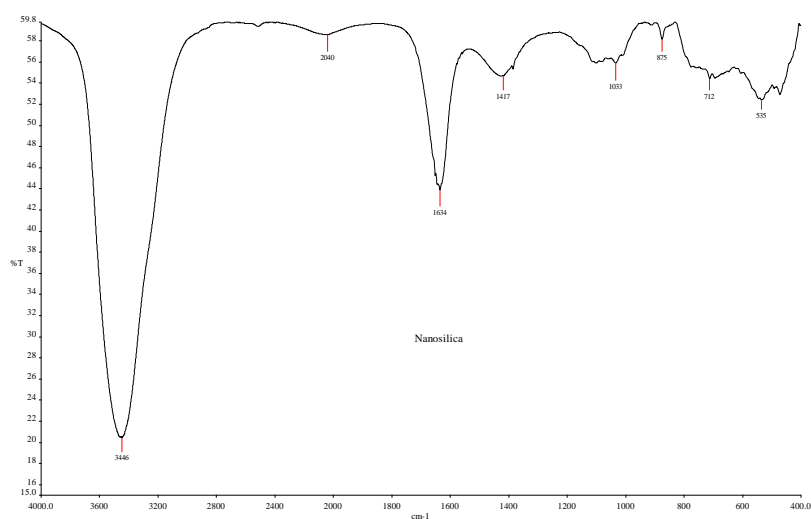


Figure 4.1: FTIR spectrum of nanosilica.

The significant result of FTIR spectrum for nanosilica in Figure 4.1 showed significant peaks at 3446cm^{-1} , 1634cm^{-1} , 1033cm^{-1} , 875cm^{-1} and 535cm^{-1} . The FTIR spectrum of the nanosilica in Figure 4.1 indicated that the peak at 1033, 875 and 535cm^{-1} were as a result of the asymmetric, symmetric and bending modes of O-Si-O,

respectively (Al-Abboodi et al., 2020) (Pham et al., 2018). The presence of hydroxyl and carboxyl groups in the sample is shown by the absorption peaks at 3446 cm^{-1} and 1634 cm^{-1} respectively. These findings support the presence of Si-O-Si stretch (Jyoti et al., 2021).

4.1.2 Fourier transform infrared spectroscopy (FTIR) of PAC

The range of FTIR spectrophotometer measurements of PAC was carried out from $4000\text{-}400\text{ cm}^{-1}$. The FTIR spectrum of the PAC is shown in Figure 4.2.

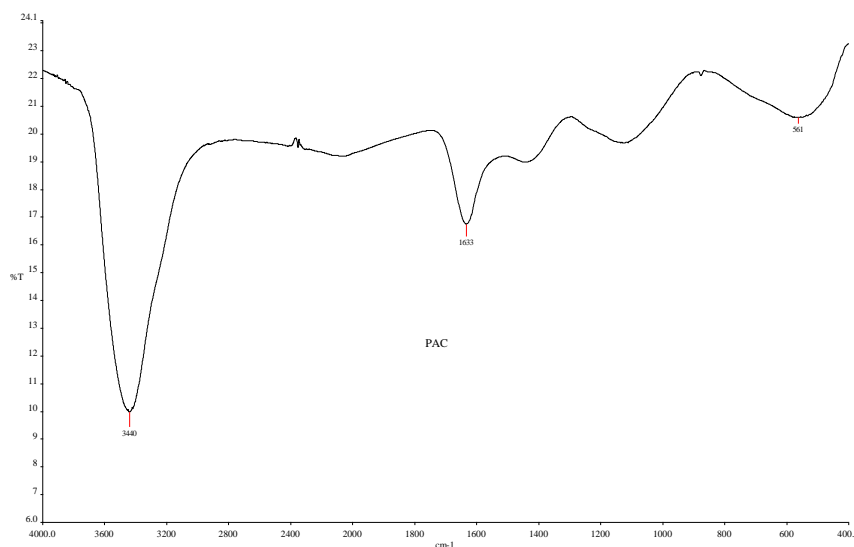


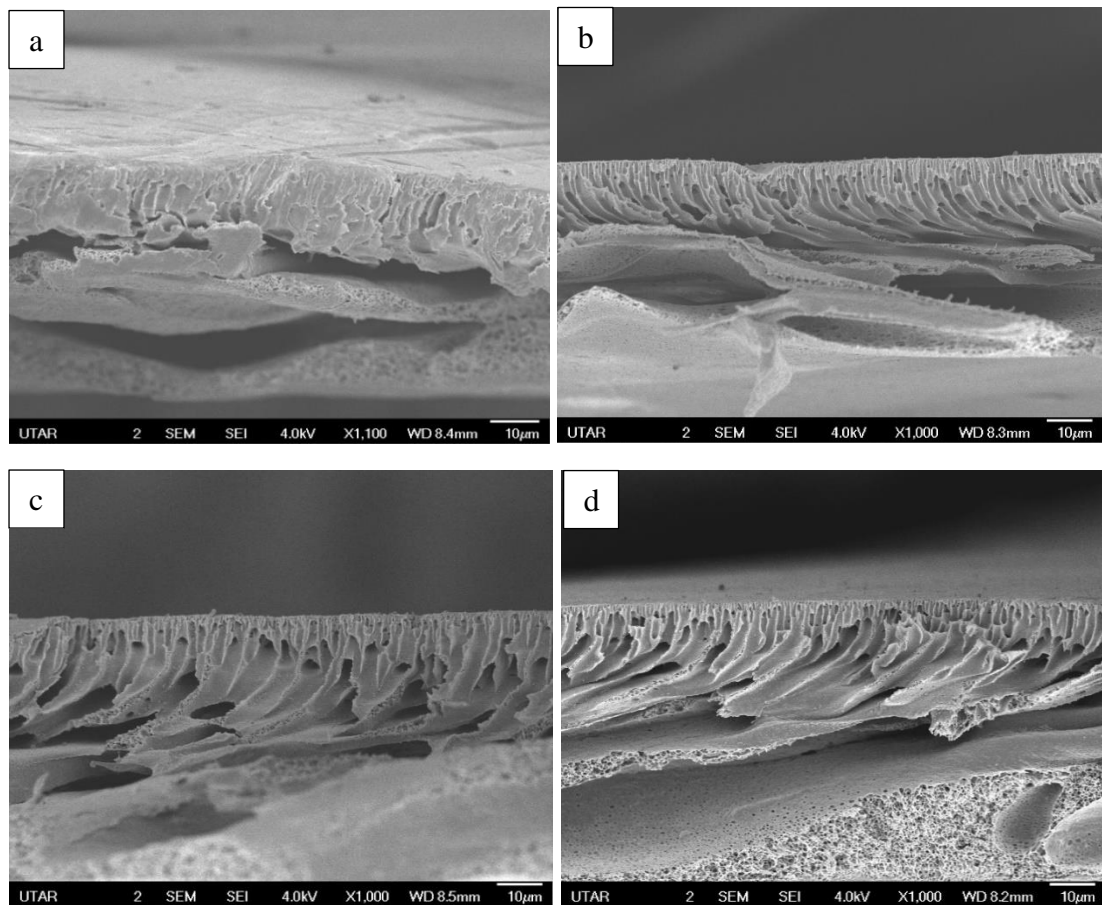
Figure 4.2: FTIR spectrum of PAC.

The FTIR spectrum for PAC in Figure 4.2 show peaks at 3440cm^{-1} , 1633cm^{-1} and 561cm^{-1} . These spectra were similar showing absorption bands around 3400cm^{-1} (3440 in the Figure 4.2) and 1600cm^{-1} (1633cm^{-1} in the figure) that was caused by O-H bond vibrations, indicating the presence of phenolic groups (Danish et al., 2018). The band at 1633cm^{-1} was related to the stretching of the C=C bond and C=O in carbonyl group (Li et al., 2021) (Niazi, Lashanizadegan, and Sharififard, 2018). The band at 561 cm^{-1} is attributed to the Si-O-Si vibration (Kan et al., 2017).

4.2 Characterization of hybrid membrane

4.2.1 SEM image of membrane cross section

SEM test of different types of membrane cross section was conducted. With the phase separation method of non-solvent induced phase separation (NIPS), the membranes formed with water as the nonsolvent exhibited an asymmetric structure with a flat skin layer (Pagliero et al., 2020). Based on the study of Mohsenpour et al. (2022), asymmetric membrane has a dense skin layer with small pores and a more open sub-layer with bigger or finger-like pores and macrovoid. The top layer solidifies spontaneously when the casting solution is submerged in the nonsolvent (water) bath. Large pores are produced as a result of the non-solvent inward diffusion into the solution (Mohsenpour et al., 2022). As a result, the membrane contains a dense skin layer as well as a porous sublayer as shown in Figure 4.3 (a)-(f).



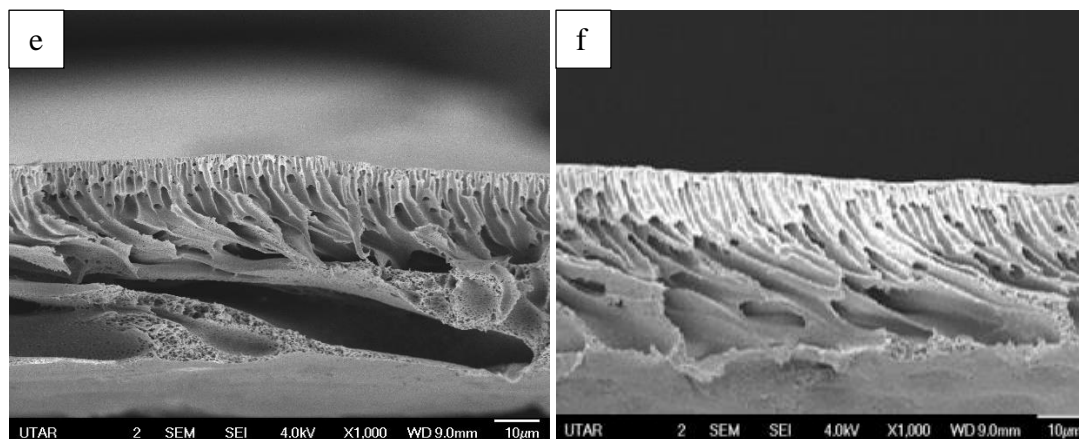


Figure 4.3 (a)-(f): Cross section of (a): 0 wt.% nanosilica and 0 wt.% PAC membrane; (b): 5 wt.% nanosilica and 0 wt.% PAC membrane; (c): 0 wt.% nanosilica 5 wt.% PAC membrane; (d): 3.5 wt.% nanosilica 1.5 wt.% PAC membrane; (e): 1.5 wt.% nanosilica 3.5 wt.% PAC membrane; (f): 2.5 wt.% nanosilica 2.5 wt.% PAC membrane.

As showed in the Figure 4.3 (a)-(f), the membrane with 0 wt.% nanosilica 0 wt.% PAC membrane has the smallest pore size. Besides, the membrane is not well connected to the underlying sublayer that will result in reduced permeability of the membrane. By adding nanosilica and PAC which act as pore formers, the porosity of the membrane will increase as nonsolvent elements likes additives are often leached out of the casting solution. The porosity and pure water flux of the membranes will increase when the concentration of the additive was raised to a level where the thermodynamic parameter surpassed the kinetic parameter (Mohsenpour et al., 2022). It can be observed that all the membranes blended with nanosilica and PAC have showed the increasing in pore size and connecting in the dense top layer and sublayer. The formation of macrovoids and the connection of top layer and sublayer are increasing and more uniformly from 5 wt.% nanosilica 0 wt.% PAC membrane, 0 wt.% nanosilica 5 wt.% PAC membrane, 3.5 wt.% nanosilica 1.5 wt.% PAC membrane, 1.5 wt.% nanosilica 3.5 wt.% PAC membrane and the membrane with largest pore size and best uniform connectivity is 2.5 wt.% nanosilica 2.5 wt.% PAC membrane. The 2.5 wt.% nanosilica 2.5 wt.% PAC membrane might be the optimum dose usage concentration for the membrane to produce biggest and best uniform pore size.

4.3 Dead-end filtration performance of membrane

Different wt.% of nanosilica and PAC of hybrid membrane were blended to evaluate the effectiveness of nanosilica and PAC additives. Membrane flux measurements were conducted using dead end filtration system to measure flow rate of the permeate. Pure water flux, solute rejection flux, membrane fouling performance and the performance in removing pollutant (turbidity, color, COD) are main performance indication of these membranes.

4.3.1 Pure water flux

Each of hybrid membranes had same membrane thickness and same membrane surface area. Distilled water was served as feed solution in permeability test. Different pressures of 0.5 bar, 1.0 bar and 1.5 bar were used in testing the membrane pure water flux. The data that required to record during the test is time taken for collecting 10ml of permeate. The collected time taken is used to calculate pure water flux based on the equation (3.1). Table 4.1 below shows the pure water flux produced by different wt.% of membrane while Figure 4.4 below illustrates the relationship between pure water flux of different wt.% membranes and pressure.

Table 4.1: Pure water flux produced by different wt.% of membrane.

Pure water flux produced by different wt.% of membrane (L/m ² h)						
Pressure (bar)	0 wt.% nanosilica and 0 wt.% PAC	5 wt.% nanosilica and 0 wt.% PAC	0 wt.% nanosilica and 5 wt.% PAC	1.5 wt.% nanosilica and 3.5 wt.% PAC	3.5 wt.% nanosilica and 1.5 wt.% PAC	2.5 wt.% nanosilica and 2.5 wt.% PAC
0.5	1.574	1.798	13.262	51.179	62.539	133.863
1	2.637	3.616	25.709	100.999	126.478	253.928
1.5	4.738	6.098	37.124	161.224	202.811	372.598

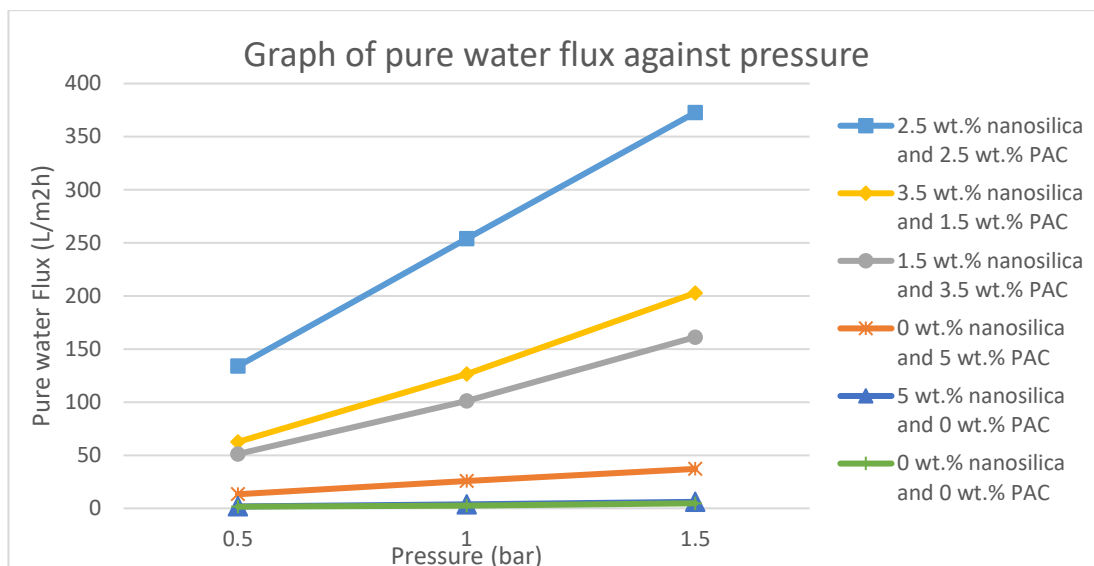


Figure 4.4: Relationship between pure water flux of different wt.% membranes and pressure.

Since the filtration system in this study was carried out based on pressure-driven, hence the increasing in pressure will affect the pure water flux of membranes. According to Figure 4.4, the pure water flux of all wt.% membranes is directly proportional to the applied pressure. The higher the applied pressure, the higher the pure water flux. This proves that the test is conducted in accurate and consistent way as the stability of the membrane in generating flux is ensured. This means that the membrane can be used to carry out the following test.

2.5 wt.% nanosilica 2.5 wt.% PAC membrane has the highest pure water flux which range from 133.863-372.598 L/m²h as it might be the optimum dose usage concentration to form the largest pore size for the membrane as observed based on Figure 4.3 (a)-(f) in this study. The 3.5 wt.% nanosilica 1.5 wt.% PAC membrane has the second highest flux with 62.539-202.811 L/m²h, followed by 1.5 wt.% nanosilica 3.5 wt.% PAC membrane (51.179-161.224 L/m²h), 0 wt.% nanosilica 5 wt.% PAC membrane (13.262-25.709 L/m²h) and 5 wt.% nanosilica 0 wt.% PAC membrane (1.798-6.098 L/m²h). 0 wt.% nanosilica 0 wt.% PAC membrane has the lowest flux which is from 1.574 L/m²h to 4.738 L/m²h. As being observed in Figure 4.3 (a)-(f), the sequence of pure water flux of the membranes corresponds with the sequence of the connectivity and sizes of pores.

The result shows that the membrane permeability is increasing by using nanosilica and PAC as additives. The membrane blended with both nanosilica and PAC significantly improve the membrane permeability if compared to membrane blended with one type of additive due to the increasing permeability and pore size of the produced membrane. This is due to the O-H functional group of both nanosilica and PAC which can be shown in FTIR analysis in Figure 4.1 and Figure 4.2. The O-H functional group will attract water molecules, making the membrane more hydrophilic (Ayyaru & Ahn, 2017).

Both 5 wt.% nanosilica 0 wt.% PAC membrane and 0 wt.% nanosilica 5 wt.% PAC membrane do not have good performance as a higher dose usage of nanosilica and PAC can result in a more compact and dense membrane structure, which can restrict the passage of molecules or ions through the membrane. It can be proved that based on the studies at Table 2.6, the weight percent of nanosilica used for the studies range from 0.5-5 wt.%. It might be said that 5 wt.% nanosilica used in this study have highly over dosage. From the Table 2.8, the weight percent of PAC used for the studies range from 0.5-11 wt.% and the permeability of 5 wt.% PAC membrane is higher than 5 wt.% nanosilica membrane. It might due to the 5 wt.% PAC membrane has lower over dosage rate if compared to the 5 wt.% nanosilica membrane. Based on the study of Lin, et al. (2016), concentration of nanosilica or PAC increase, viscosity of the polymer casting solution will increase, delaying the diffusion of the non-solvent (water) through the polymer matrix. The rising viscosity of the casting solution acts as a physical impediment to mass transfer between non-solvent and solvent, slowing and inhibiting macrovoid formation in the membrane sublayer. The aggregation of nanosilica or PAC will occur causing the pore blockage when the concentration of nanosilica or PAC is too high. It is because nanosilica and PAC has a high affinity for water molecules (Mulyati et al., 2020) (Lei et al., 2019). The competitive mass transfer between nanosilica or PAC with solvent restricts solvent diffusion through the polymer solution, resulting in the creation of a sponge-like structure in the modified membranes with high nanofiller concentrations and cause reduction in the pore size (Lin et al., 2016).

The pure water flux of 3.5 wt.% nanosilica 1.5 wt.% PAC membrane is the second highest while the 1.5 wt.% nanosilica 3.5 wt.% PAC membrane is the third

highest and these are different with the pore size sequence in Figure 4.3 (a)-(f). It might be due to the 3.5 wt.% of both nanosilica and PAC additives in membrane is also considered over dosage. If based on the result of 5 wt.% nanosilica 0 wt.% PAC membrane and 0 wt.% nanosilica 5 wt.% PAC membrane, 1.5 wt.% nanosilica 3.5 wt.% PAC membrane (contain higher amount of PAC) is supposed to have higher pure water flux than 3.5 wt.% nanosilica 1.5 wt.% PAC membrane (contain lower amount of PAC). Besides, based on the Figure 4.3 (a)-(f), 1.5 wt.% nanosilica 3.5 wt.% PAC membrane has larger macrovoid and more vertically-aligned pores if compared to 3.5 wt.% nanosilica 1.5 wt.% PAC membrane. Vertically-aligned pores allow for a more direct path for fluid flow, which can reduce resistance to flow and improve permeability. However, the actual result is different as 3.5 wt.% of nanosilica or PAC in membrane will cause agglomerate additive. This is because the agglomerates may not disperse evenly throughout the dope solution during membrane preparation, leading to non-uniform distribution of the additive (Al-Timimi et al., 2022). The non-uniform distribution of the additive may result in regions of the membrane with higher concentrations of the agglomerated additive, as well as regions with lower concentrations. These variations in concentration might impact the pure water flux.

4.3.2 Application in domestic wastewater

In this test, domestic wastewater was used as the solution to pass through the hybrid membrane with pressure 1.5 bar as 1.5 bar shows the best performance in flux production. The volume will be collected and recorded in fixed time which is minutes. Rejection flux was also calculated based on equation (3.1) which same as the equation used to calculate pure water flux but in this experiment the flux will be recorded and stop at where the membrane fouling occurs with fouling rate more than or equal to 30%. The first membrane flux will be recorded at the 5 min after the filtration process started. For the subsequent flux will be collected at every 30 min until the membrane fouling occurred. Table 4.2 and Figure 4.5 below shows the rejection flux of different additive of hybrid membrane and relationship between time needed for the hybrid membrane to foul.

Table 4.2: Fouling analysis for the produced hybrid membranes (Time versus flux).

Time	Flux produced by the membranes fabricated in the study					
	2.5 wt.% nanosilica and 2.5 wt.% PAC	1.5 wt.% nanosilica and 3.5 wt.% PAC	3.5 wt.% nanosilica and 1.5 wt.% PAC	0 wt.% nanosilica and 5 wt.% PAC	5 wt.% nanosilica and 0 wt.% PAC	0 wt.% nanosilica and 0 wt.% PAC
5 min	67.244	37.124	35.268	30.163	9.924	2.445
35 min	44.829	34.602	33.103	31.619	11.986	2.343
1 hour 5 min	35.099	29.110	28.926	26.656	14.261	2.343
1 hour 35 min	30.565	25.330	25.542	24.323	15.975	2.241
2 hours 5 min	-	-	22.753	22.042	15.131	2.241
2 hours 35 min	-	-	-	-	15.684	2.241
3 hours 5 min	-	-	-	-	14.983	2.241
3 hours 35 min	-	-	-	-	14.625	2.241
4 hours 5 min	-	-	-	-	14.509	2.241
4 hours 35 min	-	-	-	-	13.999	2.241
5 hours 5 min	-	-	-	-	13.485	2.140
5 hours 35 min	-	-	-	-	13.099	2.140
6 hours 5 min	-	-	-	-	12.915	2.140
6 hours 35 min	-	-	-	-	12.648	-
Total Volume (ml)	136.9	106.1	120.1	114.9	162.3	26.8
Fouling (%)	54.546	31.769	35.485	31.751	20.826	12.474

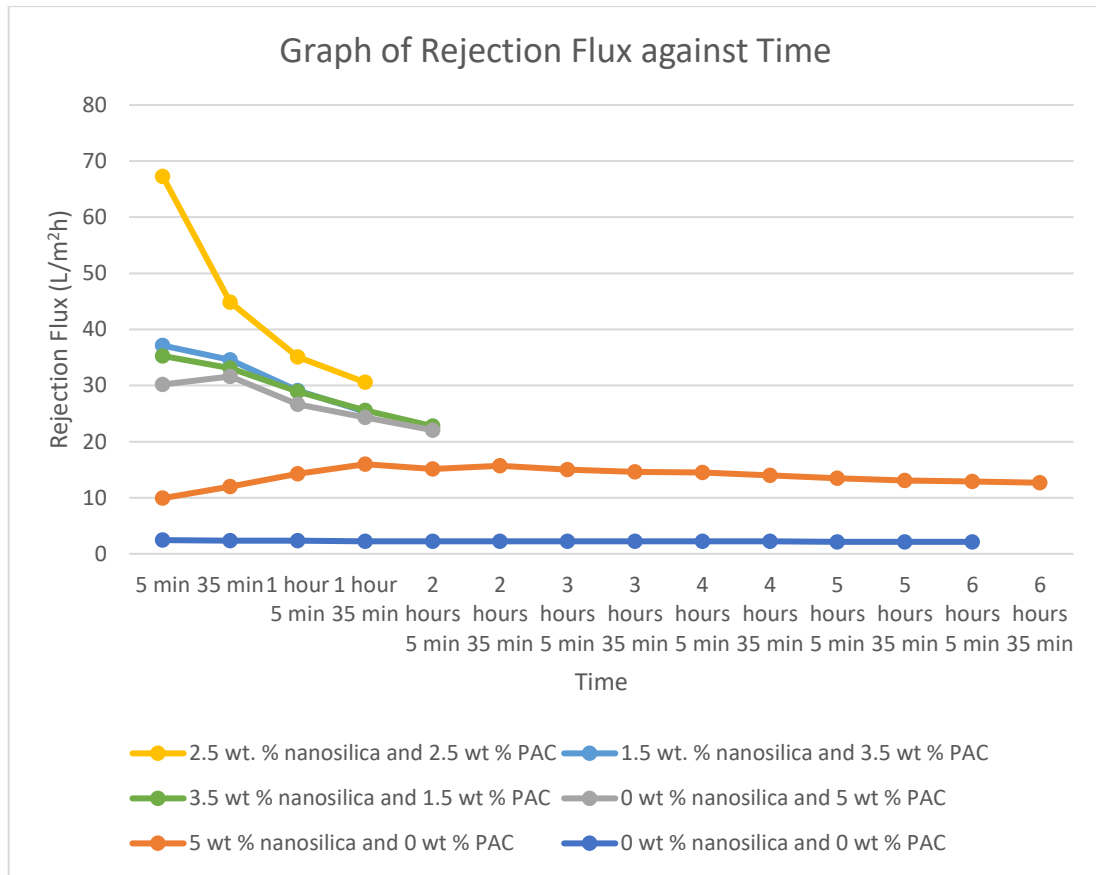


Figure 4.5: Relationship between rejection flux and time needed to foul.

Rejection flux and membrane fouling performance were observed until where the flux drop more than or equal to 30% compare to the initial flux rate at pressure of 1.5 bar. As 5 wt.% nanosilica 0 wt.% PAC membrane and 0 wt.% nanosilica 0 wt.% PAC membrane did not cause any membrane fouling, the testing was continued until the laboratory's closing time. For 2.5 wt.% nanosilica 2.5 wt.% PAC membrane, although the membrane had reached membrane fouling, the test is still continued until 1 hour 35 minutes to check whether the membrane rejection flux will be stable after a period of time.

2.5 wt.% nanosilica 2.5 wt.% PAC membrane had the highest initial rejection flux which is 67.244 L/m²h, followed by 1.5 wt.% nanosilica 3.5 wt.% PAC membrane (37.124 L/m²h), 3.5 wt.% nanosilica 1.5 wt.% PAC membrane (35.268 L/m²h), 0 wt.% nanosilica 5 wt.% PAC membrane (30.163 L/m²h), 5 wt.% nanosilica and 0 wt.% PAC membrane (9.924 L/m²h) and the lowest initial rejection flux membrane is 0 wt.% nanosilica 0 wt.% PAC membrane (2.445 L/m²h). The sequence of the initial

membrane rejection flux is same with the membrane pure water flux. This prove that the membrane is under a stable and uniform condition. Only the rejection flux of the 1.5 wt.% nanosilica 3.5 wt.% PAC membrane and 3.5 wt.% nanosilica 1.5 wt.% PAC membrane is different with pure water flux and do not have much difference. These prove that the agglomerates may not disperse evenly throughout the dope solution during membrane preparation, leading to non-uniform distribution of the additive which has stated before.

2.5 wt. % nanosilica 2.5 wt. % PAC membrane foul first at 35 minutes with total volume of 136.9 ml, followed by 1.5 wt. % nanosilica 3.5 wt. % PAC membrane fouled at 1 hour 35 minutes with 106.1 ml. 3.5 wt. % nanosilica 1.5 wt. % PAC membrane and 0 wt. % nanosilica 5 wt. % PAC membrane both fouled at 2 hours 5 minutes with total volume of 120.1 ml and 114.9 ml respectively. Although 5 wt.% nanosilica 0 wt.% PAC membrane and 0 wt.% nanosilica 0 wt.% PAC membrane did not foul for 6 hours 35 minutes and 6 hours respectively, the rejection flux of the membrane is low and only collect 162.3 ml for 6 hours 35 minutes and 26.8 ml for 6 hours. These findings indicate that these two membranes are not recommended for treating domestic wastewater due to their low rejection flux. Additionally, the membrane blended with nanosilica and PAC has the potential to foul, but exhibits a higher rejection flux due to the enhancing of membrane permeability and pore size (Ayyaru & Ahn, 2017).

The fouling of a membrane occurs when substances accumulate on the surface or pores, resulting in a decline in membrane performance, such as a reduction in membrane flux over time (Zulkefli et al., 2022). The hybrid membrane's macrovoids, which are larger in size, allow for increased water adsorption and higher flow rates of substances through the pores, leading to a faster accumulation of substances at the pore and subsequent membrane fouling (Zhao et al., 2022). Based on the SEM images of the membrane cross-section in Figures 4.3(a)-(f) and the pure water flux shown in Figure 4.4, it can be observed that the 2.5 wt. % nanosilica and 2.5 wt. % PAC membrane has the largest pore size, highest pure water flux which will result in the shortest time to reach membrane fouling. Furthermore, nanosilica and PAC exhibits greater efficiency in removing organic compounds, resulting in a higher percentage of membrane fouling for hybrid membranes containing nanosilica and PAC as an additive.

Although the 2.5 wt.% nanosilica 2.5 wt.% PAC membrane is easier to foul if compared to other membranes, it still offering a better choice due to its high permeability for domestic wastewater to pass through with 136.9 ml for only 1 hour 35 minutes. Besides, from the Figure 4.3 (a)-(f), although the membrane foul at the first 35 minutes, the membrane rejection flux reduction rate is decrease until 1 hour 35 minutes. It means than the membrane might be more stable after a period of time and with a higher rejection flux than other membranes.

4.4 Pollutant removal efficiency

The treated domestic wastewater after the rejection flux and membrane fouling test will undergo turbidity, color and COD test. The result will compare with the untreated domestic wastewater in order to calculate the pollutants removal efficiency. The removal efficiency of turbidity, color, and COD of different types of membrane were shown in Table 4.3 and Figure 4.6.

Table 4.3: Pollutants removal efficiencies of different types of membrane at 1.5 bar.

Types of membranes	Turbidity (%)	Color (%)	COD(%)
0 wt.% nanosilica 0 wt.% PAC	97.01	92.31	79.44
5 wt.% nanosilica 0 wt.% PAC	97.01	95.33	90.61
2.5 wt.% nanosilica 2.5 wt.% PAC	97.01	91.48	92.45
0 wt.% nanosilica 5 wt.% PAC	98.51	93.68	92.89
3.5 wt.% nanosilica 1.5 wt.% PAC	97.01	94.23	93.18
1.5 wt.% nanosilica 3.5 wt.% PAC	100	97.53	94.31

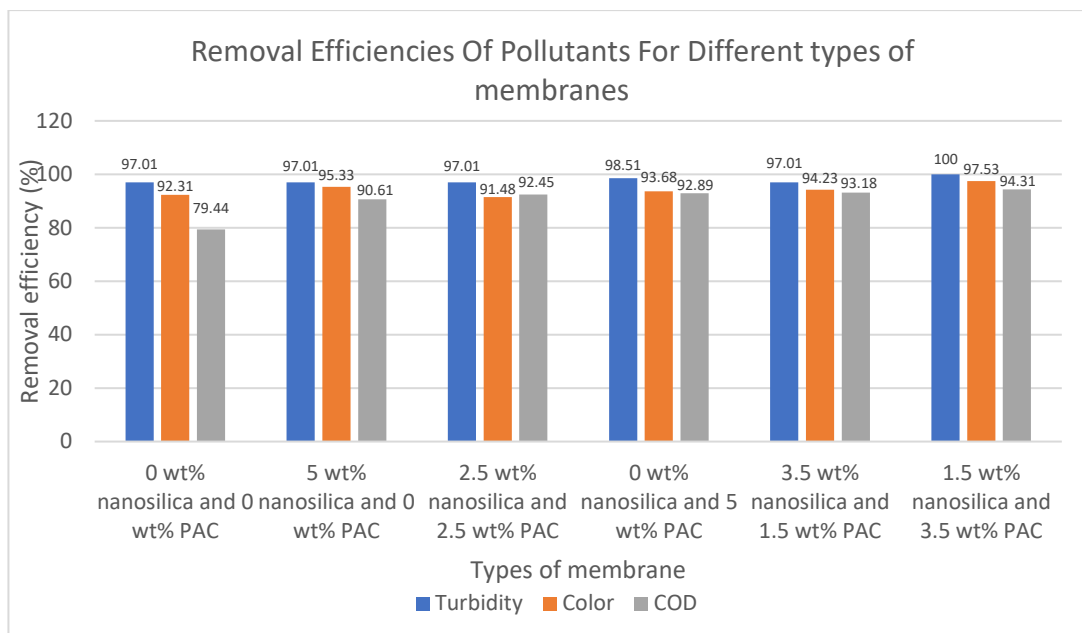


Figure 4.6: Pollutants removal efficiencies of different wt.% membranes at 1.5 bar.

Figure 4.6 demonstrated that all the produced hybrid membranes had similar turbidity removal efficiency which range from 97.01% to 100%. These findings demonstrate that PES membrane can effectively reduce turbidity in domestic wastewater without the need for additional additives. For color removal, 2.5 wt.% nanosilica 2.5 wt.% PAC membrane has the lowest removal efficiency which are 91.48%. Other membranes had the color removal efficiency range from 92.51% to 97.53%.

2.5 wt.% nanosilica 2.5 wt.% PAC membrane did not show good COD and color removal efficiency if compared to other membranes combined with both nanosilica and PAC. As based on the study of Chen et al. (2018), increasing the pore size or enhancing membrane permeability can improve the membrane's flux or throughput, which is beneficial for filtration efficiency. However, it may also lead to a reduced ability to remove pollutants from the wastewater, as larger pores can allow some pollutants to pass through more easily (Chen et al., 2018).

From the Table 4.3 and Figure 4.6, the membrane with 1.5 wt.% nanosilica 3.5 wt.% PAC membrane shows the highest COD removal efficiency which is 94.31%,

followed by 3.5 wt.% nanosilica 1.5 wt.% PAC membrane (93.18%), 0 wt.% nanosilica 5 wt.% PAC membrane (92.89%), 2.5 wt.% nanosilica 2.5 wt.% PAC membrane (92.45%), 5 wt.% nanosilica and 0 wt.% PAC membrane (90.61%) and 0 wt.% nanosilica 0 wt.% PAC membrane (79.44%). The addition of additives (nanosilica and PAC) resulted in COD removal efficiencies ranging from 90.61% to 94.31%. These results range are not significant, suggesting that the impact of the weight percentage of the nanosilica and PAC on COD removal efficiency was negligible, and that these additives exhibited consistent COD removal performance.

It also shows that PAC has a higher removal efficiency than nanosilica. It can be proved based on Table 2.6 and Table 2.8, membrane with PAC as additives can have the maximum COD and color removal efficiency increased by around 60% and 30% respectively while membrane with nanosilica as additive only have the maximum COD removal efficiency increase by 10%. This is why 1.5 wt.% nanosilica 3.5 wt.% PAC membrane has the highest removal efficiency. However, 0 wt.% nanosilica 5 wt.% PAC membrane with the highest PAC content do not show best removal efficiency might due to the 5 wt.% of PAC is over dosage and will cause agglomerate of PAC which will cause a decrease in the effective surface area of the membrane (Ismail et al., 2020). This might result in lower pollutant adsorption and removal capacity.

The presence of nanoparticles (nanosilica and PAC), which function as a molecular sieve with outstanding separation capabilities, improves the rejection of organic compounds (Kusworo et al., 2017). Hence, COD and color of domestic wastewater was reduced and increase the removal rate. The organic matters and color in domestic wastewater were adsorbed on the membrane surface with the aid of outstanding adsorption ability of nanosilica and PAC (Kusworo et al., 2017) (Hu et al., 2017). Besides, the high surface area and a strong affinity for organic and inorganic compounds of nanosilica and PAC aid in the removal of color-causing compounds and COD from domestic wastewater (Mulyati et al., 2020) (Aghili et al., 2017). The functional group such as O-Si-O bond, hydroxyl, carboxyl group of nanosilica and C=C bond, C=O bond and O-H bond in PAC which had shown in previous FTIR test aid in modifying membrane hydrophilicity and surface charge (Al-Abboodi et al., 2020) (Niazi, Lashanizadegan, and Sharififard, 2018). This can improve the anti-fouling properties of the membrane, reducing the accumulation of organic and inorganic

fouling agents on the membrane surface, and thus enhancing the COD and color removal efficiency.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The flat sheet asymmetric membranes with different nanosilica and PAC concentration were successfully produced through phase inversion method. The functional group such as O-Si-O bond, hydroxyl, carboxyl group of nanosilica and C=C bond, C=O bond and O-H bond in PAC were shown in FTIR test. The cross-sectional surface morphology of the produced membranes was characterized by conducting a SEM analysis. The performance of the membranes was carried out by analyzing various parameters including permeability, solute rejection flux, membrane fouling performance, turbidity, color, and COD removal rate. Based on the SEM result, 2.5 wt.% nanosilica and 2.5 wt.% PAC membrane has the biggest and best uniform pore sizes. 2.5 wt.% nanosilica and 2.5 wt.% PAC membrane also shows the highest pure water flux. Upon completion of the study, it was determined that 2.5 wt.% nanosilica and 2.5 wt.% PAC membrane resulted in the highest rejection flux due to its biggest pore size and highest pure water flux. Due to the highest rejection flux, the membrane had the shortest time to reach membrane fouling. While blending the additives such as nanosilica and PAC into the membrane could lead to a shorter time to membrane fouling, it could also result in improved flux and increased rejection effectiveness. Furthermore, the use of both nanosilica and PAC in combination had the potential to further improve the flux. Besides, the increment of flux occurred when higher pressure applied during dead-end filtration. 1.5wt.% nanosilica and 3.5 wt.% PAC membrane was found to have the highest pollutants removal efficiency due to its high PAC content.

The high weight percentage of additive in 5 wt.% nanosilica 0 wt.% PAC membrane and 0 wt.% nanosilica 5 wt.% PAC membrane had resulted in inadequate pure water flux and pollutant removal efficiency due to the agglomeration of the additives.

5.2 Recommendations

Study on membrane filtration has significantly more topics to be explored and improved. Hence, there are some improvements should be considered and included in the further research. I would like to include some recommendation which stated as below:

- i. Compare the performance of membrane that employing different additives.
- ii. Investigate the optimum concentration of nanosilica and PAC that needed to achieve the highest effectiveness through increasing number of concentration variables.
- iii. Prolong the fouling performance test of 2.5 wt.% nanosilica 2.5 wt.% PAC membrane in order to determine whether there is a decrease or increase in the rejection flux.
- iv. Determine the membrane removal efficiencies by using other wastewater samples.

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APPENDICES

Appendix A: Dead-end membrane test rig.



Appendix B: Membranes immersed in distilled water for 24 hours.



Appendix C: Methanol solution that membranes immersed was covered to prevent evaporation at room temperature.



Appendix D: DR/890 calorimeter.

