FORWARD OSMOSIS THIN FILM COMPOSITE MEMBRANE INCORPORATED WITH METAL ORGANIC FRAMEWORK FOR ARSENIC (V) REMOVAL

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DEDICATION

This thesis is dedicated to my family, who taught me that the best kind of knowledge to have is that which is learned for its own sake. They also taught me that even the largest task can be accomplished if it is done one step at a time.

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ABSTRACT

The importance of the substrate layer in composite membranes lies not only in providing mechanical strength to the active layer, but also in serving as a foundation for the formation of polyamide. Therefore, the objectives of this study were to investigate the physicochemical properties of water stable metal organic framework University of Oslo-66 (UiO-66) nanoparticle and thin film composite (TFC) mixed matrix membrane (TFC-MMM). The membranes were fabricated by a phase inversion process that consists of UiO-66 nanoparticles embedded in a polysulfone matrix ranging from 0, 0.05, 0.1, 0.3 and 0.5 wt%. Then, an interfacial polymerization process has taken place to form polyamide on the outer membrane surface. These nanoparticles and membranes were characterized with field emission scanning electron microscopy, x-ray diffraction, contact angle, overall porosity, atomic absorption spectroscopy, attenuated total reflectance Fourier transform infrared, atomic force microscopy, pore size distribution and zeta potential. Based on the characterizations, the membranes have the potential to be used for arsenic (V) rejection in water flux tests. The forward osmosis process was utilized to determine water flux and solute reverse flux. Pure water and 1 M NaCl solution were used as feed and draw solution, respectively. The water flux was increased up to 20 LMH at TFC-0.3 and it went down to 17 LMH at TFC-0.5 while the solute reverse flux kept elevated but at a controlled rate. Then, 100 ppm arsenic (V) was used as feed for As rejection performance. It was demonstrated that the physicochemical properties of MMM affect the interfacial polymerization of polyamide, leading to greater arsenic (V) rejection which up to 96%. Then, the pH of the feed solution was adjusted to 5, 6, 7, 8 and 9. The membrane performs optimally at a pH of 9 due to electrostatic repulsion between $HAsO_4^{2-}$ and polyamide.

ABSTRAK

Kepentingan lapisan substrat dalam membran komposit bukan sahaja untuk memberi sokongan kekuatan mekanikal kepada lapisan aktif tetapi juga berfungsi untuk menjadi asas kepada pembentukan poliamida. Tujuan penyelidikan ini dijalankan adalah untuk menyiasat sifat-sifat fizikokimia nanopartikel kerangka logam organik stabil dalam air Universiti Oslo-66 (UiO-66) dan komposit filem nipis (TFC) membran matriks campuran (TFC-MMM). Membran tersebut dihasilkan melalui proses fasa berbalik yang mengandungi nanopartikel UiO-66 dimasukkan ke dalam matriks polisulfon antara 0, 0.05, 0.1, 0.3 dan 0.5%. Kemudian, proses pempolimeran antaramuka telah berlaku untuk membentuk poliamida di atas permukaan luar membran. Nanopartikel dan membran tersebut dicirikan dengan mikroskopi imbasan pancaran medan elektron, pembelauan sinar-x, sudut sentuh, keliangan keseluruhan, spektroskopi penyerapan atom, pantulan menyeluruh dilemahkan infra merah jelmaan Fourier, mikroskopi daya, taburan saiz liang dan potensi zeta. Berdasarkan pencirian tersebut, membran ini berpotensi untuk menyingkirkan arsenik (V) semasa ujian fluks air. Proses osmosis ke hadapan digunakan untuk menentukan fluks air dan fluks berbalik bahan larut. Air tulen dan larutan NaCl berkepekatan 1M masing-masing diletakkan sebagai larutan suapan dan larutan penarik. Fluks air telah meningkat sehingga 20 LMJ untuk TFC-0.3 dan menurun kepada 17 LMJ untuk TFC-0.5 sementara fluks larutan berbalik terus meningkat tetapi pada kadar yang terkawal. Kemudian, 100 ppm arsenik (V) digantikan sebagai larutan suapan untuk ujian prestasi penyingkiran arsenik. Kajian menunjukkan ciri-ciri fizikokimia MMM mempengaruhi pempolimeran antaramuka poliamida yang menunjukkan peningkatan kadar penyingkiran arsenik (V) sehingga 96%. Seterusnya, pH larutan suapan diubah kepada 5, 6, 7, 8 dan 9. Membran tersebut bertindak pada tahap yang optimum pada pH 9 disebabkan oleh penolakan elektrostatik antara $HAsO_4^{2-}$ dengan poliamida.

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LIST OF ABBREVIATIONS

WHO	-	World Health Organization
UF	-	Ultrafiltration
NF	-	Nanofiltration
RO	-	Reverse osmosis
MOF	-	Metal organic framework
MMM	-	Mixed matrix membrane
TFC	-	Thin film composite
UiO-66	-	University of Oslo
AL-FS	-	Active layer facing feed solution
FESEM	-	Field emission scanning electron microscopy
XRD	-	X-ray diffraction
FTIR	-	Fourier transform infrared
AFM	-	Atomic force microscopy
AAS	-	Atomic absorption spectroscopy
FO	-	Forward osmosis
MF	-	Microfiltration
PRO	-	Pressure retarded osmosis
ICP	-	Internal concentration polarization
AL-DS	-	Active layer facing draw solution
CTA	-	Cellulose triacetate
PSf	-	Polysulfone
PVDF	-	Polyvinylidene fluoride
PA	-	Polyamide
PEI	-	Polyetherimide
MPD	-	m-phenylenediamine
TMC	-	Trimesoyl chloride
IP	-	Interfacial polymerization
NMP	-	N-methyl-2-pyrrolidone
PES	-	Polyethersulfone

CNT	-	Carbon nanotube
GO	-	Graphene oxide
MIL	-	Materials of Institute Lavoisier
ZIF-8	-	Zeolite imidazolate framework
TFN	-	Thin film nanocomposite
BDC	-	Benzedicarboxylate
HKUST-1	-	Hong Kong University of Science and Technology
PAN	-	Polyacrylonitrile
HDPE	-	High density polyethylene
DMF	-	Dimethylformamide
PI	-	Phase inversion
PVP	-	Polyvinylpyrrolidone

LIST OF SYMBOLS

Jw	-	Water flux
Js	-	Reverse solute flux
A	-	Effective area
C_p	-	Permeate concentration
Δt	-	Time interval
Rpm	-	Revolution per minute
Ppm	-	Parts per million
Π	-	Van’t Hoff equation
Re	-	Reynold Number
C_b	-	Bulk concentration
V_d	-	Volume of draw solution
V_p	-	Volume of permeate water
M	-	Molarity
h	-	Hour
LMH	-	Litre per cubic meter per hour
nm	-	nanometer

CHAPTER 1

INTRODUCTION

1.1 Research Background

Surface water is a major backbone of drinking water supply because the cost of operating per volume of water is low, considering the pollutant containing in the water is relatively low. However, this resource is finite, meaning that the source is depleting with continuous use. Although the resource is not going to run out in short time, the main issue would be the raise of wastewater generation being left untreated because the treatment plant may not be able to accommodate in large quantities. Wastewater comprises of organic and inorganic pollutant is generated from daily consumption such as for household cleaning, drinking and manufacturing of goods has led to thousands of different pollutant contain in the water.. The major drawbacks in the interest of treating wastewater is higher cost faced in the treatment plant compare to groundwater or surface water supply. A research in pursuit to find alternative approach or materials are in needed that is more cost effective yet be able to maintain high quality of treated water.

Arsenic, being classified as heavy metal, is commonly found in wastewater as a result of anthropological activities. It is much used in agricultural, glass, wood preservatives, herbicides and electronics industries (Ishiguro, 1992). Due to its toxicity and carcinogenicity, water containing arsenic is not safe to be directly consumed (Singh *et al.*, 2015). Some diseases related with arsenic consumption are including skin cancer, vascular disease, hypertension and anhydremia (Duker *et al.*, 2005). According to World Health Organization (WHO), it is recommended that the permissible As content in water to be not more than 0.01 mg/L for both raw and drinking water purpose which is also in-line as prescribed by Ministry of Health Malaysia (Choong *et al.*, 2007). In water bodies, As may exist in various concentration, pH and forms such as arsenite (As^{3+}) and arsenate (As^{5+}). Recently, there was rise in contamination level of arsenic due to vast growth of industrial activities as much 21 times fold compared to the past 60 years (Chen *et al.*, 2015). The situation urge the need of efficient treatment method to control arsenic contamination.

To remove As and to comply with the abovementioned standard, in need to undergo a primary treatment by means of coagulation-flocculation. At this stage, a major number of foreign particles including As were eliminated but yet to be safe for end-user consumption. Secondary treatment took place to remove leftover soluble organic matter that escapes from primary treatment. Then, As was completely removed at tertiary stage which can be done by membrane separation process. Membrane is a semi-permeable, selective barrier that allow certain molecules to pass through while retaining solutes from permeating. Membrane can be in range of microfiltration, ultrafiltration, nanofiltration or reverse osmosis where each of them are differentiated by the pore size. The selection of membrane range is based on molecular size of the solute. For instance, UF and NF are applied for heavy metal and dye removal while RO is more suitable for desalination (Ammar *et al.*, 2015). Membrane separation is superior than other treatment process because it does not produce by-product requiring further treatment. Furthermore, membrane is able to reduce solute concentration to comply WHO standard.

Porous materials had gained interest among researchers because it is very much useful for application in mixture separation and chemical storage. Metal organic framework (MOF) is a unique class of porous materials built with metal cluster and organic linker to form a crystal structure of one, two or three dimensions. The term "MOF" may be self-explanatory among scientific community that describe metal clusters are surrounded by organic ligand to form a framework or coordination network. Due to its highly crystalline structure, MOF possess higher surface area and porosity with more uniform pore dimension compare to other porous materials like activated carbon, zeolite, silica and carbon nanotube. Another unique feature of MOF is its pore size tunability that can be tailored for specific application. MOF was initially applied for gas storage which later the scope was expanded for gas separation, drug delivery, sensing, catalysis and recently in water treatment. The tunability characteristic is attributed to the degree of freedom to synthesize the MOF through altering precursors ratio, synthesizing temperature or guest removal. For wastewater treatment field, MOF has potential for adsorbing solutes such as organic materials and heavy metal for its high adsorption properties or may be integrated as filler for membrane separation.

Polymeric membrane used in wastewater treatment are relatively low compare to inorganic molecular sieve materials because polymer membrane suffers from trade-off between selectivity and permeability. On the other hand, there is difficulty in processing inorganic membrane not to mention the cost is higher which hinder it from production in large scale. Given that both of these materials have their own pros and cons, combining them together had led into discovery of mixed-matrix membrane (MMM) concept, that improved the membrane performance. MMM is often combined with thin film composite (TFC) because its denser layer is more effective to reject solute particularly for heavy metals and salt.

1.2 Problem Statement

One of the most common issues in polymeric membrane is the trade-off between permeability and selectivity. It is not beneficial to acquire such low permeability as it will not utilize the capability to produce clean water at high amount. On the other hand, attempting to tune the membrane so it can yield high permeability may compromise the quality of treated water making it unsafe for human consumption.

To overcome this problem, MMM is one of the promising approach as it provides preferential path for water while undesired molecule is retained. It has been found that MMM has improved the performance compare to traditional polymeric membrane but the extent of finding the best dispersed phase loading remained a challenge. The critical issue in MMM is the compatibility between polymer and the filler. Without appropriate chemical bonding between them, agglomeration is likely to occur. Agglomeration is an occurrence where the filler is distributed unevenly throughout the polymer matrix and clumped in certain area. Agglomeration may also occur when excessive filler is loaded into the polymer and tend to sediment to the bottom layer of substrate. Studies on overcoming agglomeration had much drawing attention but most of them are involving additional steps. A simpler and straightforward approach through blending between filler and polymer is critically needed so as to develop easier controlled procedure.

Extensive research had been carried out for development of high performance membranes. In specific, the use of thin film composite membrane which originally used in reverse osmosis had yield high water flux. However, water transport passing through the membrane is reduced in forward osmosis due to absence of hydraulic pressure.

Given that MOF is built with inorganic metal clusters and organic linkers is what makes it has better compatibility with polymer matrix. Through covalently H_2 bonding, MOF can be dispersed more uniformly and mitigate unwanted voids formation. However, it was found that most MOF are unstable in water and other chemicals making them unsuitable to be applied for water treatment field. A deterioration in performance could be seen when unstable MOF were directly exposed towards water indicating the crystalline structure has collapsed.

UiO-66 as a type of MOF, had been recognized for its water stability and may withstand in wide range of pH for prolong exposure. However, there is lack of understanding on how the incorporation of UiO-66 in MMM helps to improve overall membrane performance because of limited numbers of research regarding to it.

In forward osmosis for heavy metal rejection, the thin film composite (TFC) active layer is responsible in performing the separation based on size exclusion and electrostatic repulsion (Zhang *et al.*, 2017). The dependency on charge repulsion may cause performance deterioration when not in optimal pH condition thus requiring pH adjustment, at the cost of adding complexity to the treatment operation.

1.3 Objectives of Study

Based on the abovementioned problem statements, the main goal of this research is to develop thin-film composite polyamide on top of PSf-UiO-66 MMM consists of synthesizing UiO-66 to treat aqueous arsenic under forward osmosis process. The specific objectives of this research are as follows:

1) To synthesize and characterize the UiO-66 nanoparticles.

2) To fabricate and characterize TFC-MMM

3) To identify the TFC-MMM performance on pure water permeability and As (V) rejection via forward osmosis process.

1.4 Scopes of Study

To achieve the aforementioned objectives, the following scopes are outlined:

(a) UiO-66 consists of $ZrCl_4$ and 1,4-benzenedicarboxylic acid was synthesized via solvothermal method at 120^oC for 24 h.

(b) The synthesized UiO-66 nanoparticle was characterized for particle shape (FESEM), average particle size (ImageJ), particle crystallinity (XRD) and presence of functional group (FTIR).

(c) A series of PSf substrates were prepared by hand casting, non-solvent phase inversion method with different loading of UiO-66 nanoparticle (0, 0.05, 0.1, 0.3 and 0.5 wt%).

(d) PSf substrate was characterized for top and bottom surface morphology (FESEM), functional group peak at different UiO-66 loading (FTIR), membrane hydrophilicity (contact angle), porosity (wet-dry method) and average pore size (ImageJ).

(e) On top of the substrate, a thin film composite (TFC) polyamide active layer made up of m-phenilenediamine (MPD) and trimesoyl chloride (TMC) was fabricated by interfacial polymerization.

(f) Fabricated TFC was analysed for top and cross-section morphology (FESEM) and membrane surface roughness (AFM).

(g) Pure water flux was evaluated in FO system by using pure water as feed and 1 M NaCl as draw solution at 300 rpm

(h) For rejection testing, the membrane was tested by non-pressurized FO system with active layer facing feed solution (AL-FS) with feed and draw solution were 100 ppm As (V) solution at fixed pH 6.5 and 1 M NaCl, respectively.

(i) The final concentration of As (V) at draw solution was measured by atomic absorption spectroscopy (AAS).

(j) The best performed membrane was selected for further As (V) but with variation of feed solution at pH 5, 6, 7, 8 and 9.

1.5 Significance of Study

Forward osmosis is usually applied for desalination process as alternative to intensive energy consumption from reverse osmosis. Aside from that, FO is also suitable for heavy metals removal given that their hydrated ion diameter is larger than salts. Therefore, this research provided insight on how wastewater containing heavy metal can be treated using non-pressurized system.

In most wastewater treatment plant, the influent wastewater is typically existing in various pH due to presence of different chemical constituents and concentration. The membrane used in the system not only should be able to handle such harsh chemical strength, but also maintaining the performance at its peak. The fabricated UiO-66 membrane has potential to advance towards next-generation membrane for its high performance in treating water at wider pH range between 3 to 9 (Wang *et al.*, 2015; He *et al.*, 2017; Liu *et al.*, 2019).

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LIST OF PUBLICATIONS

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