DEPOSITION OF SYNTHESIZED ZIRCONIUM-BASED METAL ORGANIC FRAMEWORK ON CERAMIC HOLLOW FIBRE FOR FORWARD OSMOSIS DESALINATION

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ABSTRACT

This study was aimed to develop zirconium (Zr)-based metal organic framework (MOF) ceramic membrane that can be used for forward osmosis (FO) desalination. As the first step, ceramic substrate surface was first modified with zirconium dioxide (ZrO_2) using sol-gel Pechini's method to provide active seeds that can favour MOF nucleation. Using this modified substrate, a series of solvothermal synthesis conditions were tested to build the FO applicable membrane. In the unmodulated procedure, only sample synthesized at 220°C with 0.3 M and 16 h gave positive water flux. Field emission scanning electron microscopy (FESEM) and themogravimetric analysis (TGA) results have shown that the non-uniform coverage and defect frameworks of MIL-140B (Materials of Institute Lavoisier –MIL) on the substrate layer was the cause to this problem. Hence, to tune the defect, modulated synthesis was introduced. Increment in modulator amount by increasing precursor concentration from 0.58 M to 2.32 M had successfully lowered the percentage of defect framework from 26.03% to 16.87%. Despite this framework enhancement, FO test result of this sample still displayed worse performance than the previous synthesis due to its high tendency of agglomeration. Loosely joint particles that formed during agglomeration at high temperature synthesis were easily brushed off during FO test. Therefore, lower synthesis temperature of 120°C and longer synthesis time of 24 h was employed in the next procedure to allow slow nucleation process that can form better connected crystals. Instead of MIL-140B, UiO-66-NDC (University of Oslo – UiO) framework was found at $2\theta = 10.36°$ and the crystal shape appeared in octahedron. Even with this reformatted crystal shape, the FO performance result still could not be in positive value. Therefore, the UiO-66-NDC membrane active layer was polymerised with fluorinated polymer as the last resort. Integration between this polymer and UiO-66-NDC had successfully treated the membrane defects by building new bonds inside the framework as proven by FESEM, atomic force microscopy, x-ray diffraction, Fourier-transform infrared spectroscopy and TGA results. With better connected crystals, smoother deposition layer and perfect frameworks, FO performance of all UiO-66-NDC samples finally gave positive water flux results and the highest value was 16.189 $L/m^2.h$. Its lowest reverse solute flux achieved was 0.003 $L/m^2.h$ with sodium chloride rejection of up to 80 % which is definitely better than the previous study. Therefore, polymer-synthesized UiO-66-NDC on ceramic hollow fibre can definitely serve as an excellent FO membrane option that can be used in the desalination process.

ABSTRAK

Kajian ini bertujuan untuk membangunkan membran seramik berkerangka metal-organik (MOF) berasaskan logam zirconium (Zr) yang boleh digunakan untuk osmosis proses penyahgaraman ke hadapan (FO). Langkah pertama, permukaan gentian berongga seramik telah diubah suai dengan zirkonium dioksida menggunakan kaedah *sol-gel* Pechini untuk menghasilkan benih aktif yang membantu proses nukleasi MOF. Dengan menggunakan substrat yang telah dimodifikasi ini, pelbagai keadaan sintesis *solvothermal* telah diuji untuk menghasilkan membran yang mampu berfungsi untuk FO. Melalui prosedur tanpa modulasi, hanya sampel yang disintesis pada suhu 220°C dengan 0.3 M dan 16 jam sahaja dapat memberikan nilai positif fluks air. Keputusan mikroskop imbasan elektron pancaran medan (FESEM) dan analisis thermogravimetri (TGA) menunjukkan bahawa perlindungan yang tidak seragam dan kecacatan kerangka MIL-140B (bahan dari Institut Lavoisier - MIL) di atas lapisan substrat merupakan penyebab kepada permasalahan tersebut. Oleh itu, untuk membaiki kecacatan ini, sintesis bermodulasi diperkenalkan. Penambahan jumlah modulator melalui kenaikan kepekatan bahan dari 0.58 M kepada 2.32 M berjaya menurunkan kecacatan kerangka dari 26.03% kepada 16.87%. Walaupun penambahbaikan telah dibuat ke atas kecacatan kerangka, keputusan ujian FO masih memaparkan prestasi yang negatif malahan lebih teruk daripada sampel sebelum ini disebabkan oleh kecenderungan terhadap penggumpalan. Partikel longgar yang terbentuk pada sintesis suhu tinggi lebih mudah terhapus semasa ujian FO. Maka, suhu sintesis yang lebih rendah dan masa sintesis yang lebih lama iaitu 120 °C dan 24 jam diperkenalkan dalam prosedur seterusnya bagi membolehkan proses nukleasi perlahan berlaku seterusnya membentuk gabungan kristal yang lebih baik. Selain MIL-140B, pada $2\theta = 10.36°$, formasi kerangka UiO-66-NDC (Universiti Oslo - UiO) dapat diperoleh dan kristal terpapar dalam bentuk bongkah bersegi lapan. Walaupun bentuk kristal telah berubah, keputusan ujian FO masih belum dapat diperoleh dalam nilai positif. Oleh itu, lapisan aktif membran UiO-66-NDC dipolimerkan dengan polimer florin sebagai langkah terakhir. Integrasi antara polimer ini dengan UiO-66-NDC berjaya merawat kecacatan kerangka dengan membina ikatan-ikatan baru yang dibuktikan melalui keputusan FESEM, mikroskop daya atom, pembelaun sinar-X, spektroskopi infra merah transformasi Fourier dan TGA. Melalui gabungan kristal yang lebih baik, permukaan lapisan bersepadu yang lebih licin dan kerangka yang sempurna, keputusan ujian FO bagi semua sampel UiO-66-NDC akhirnya memberikan nilai fluks air yang positif dan nilai tertinggi yang diperoleh adalah 16.189 L/m^2.h. Nilai terendah bahan larut fluks balikan adalah 0.003 L/m^2.h dengan prestasi penolakan garam natrium klorida sehingga 80% berbanding dengan membran sebelumnya. Selaras dengan keputusan ini, polimer-disintesis UiO-66-NDC di atas permukaan serat berongga seramik adalah antara pilihan membran FO yang terbaik untuk digunakan dalam proses penyahgaraman..

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LIST OF ABBREVIATIONS

Al_2O_3	-	Aluminium oxide
Ar	-	Argon
CO_2	-	Carbon dioxide
Cu-BTC	-	Copper benzene-1,3,5-tricarboxylate
H_2NDC	-	2,6-naphthalenedicarboxylic acid
H_2O	-	Water
H_2	-	Hydrogen
Zr^{4+}	-	Zirconium ion
$ZrCl_4$	-	Zirconium tetrachloride
ED	-	Electrodialysis
MFD	-	Multistage flash distillation
YSZ	-	Yttria-stabilized zirconia
MOF	-	Metal organic framework
MIL	-	Materials of Institute Lavoisier
SEM	-	Scanning electron microscopy
XRD	-	X-ray diffraction
TGA	-	Thermogravimetric analysis
BET	-	Brunauer-emmett-teller
MF	-	Microfiltration

UF	-	Ultrafiltration
NF	-	Nanofiltration
RO	-	Reverse osmosis
FO	-	Forward osmosis
PSF	-	Polyethersulfone
PTFE	-	Polytetrafluoroethylene
TFC	-	Thin-film composite
LBL	-	Layer-by-layer
SBU	-	Secondary building unit
ZIF	-	Zeolitic imidazolate framework
UiO	-	University of Oslo
DUT	-	Dresden University of Technology
BUT	-	Beijing University of Technology
HCl	-	Hydrochloric acid
NaOH	-	Sodium hydroxide
Cl	-	Chlorine
Pd	-	Palladium
DMF	-	N,N-dimethylformamide
DEF	-	N,N-diethylformamide
NMP	-	N-methylpyrrolidine
TEA	-	Triethylamine
NaCl	-	Sodium chloride

LIST OF SYMBOLS

°	-	Angle degree
%	-	Percentage
ΔM_{FS}	-	Different mass feed solution
ρ	-	Density
A	-	Area
Å	-	Angstrom
°C	-	Degree celcius
C_p	-	Concentration permeate salt
C_f	-	Concentration of feed solution
D_o	-	Outer diameter
DS_i	-	Initial draw solution
FS_f	-	Final feed solution
J_w	-	Pure water flux
K	-	Kelvin
kV	-	Kilovolt
Le	-	Effective length
mg	-	Milligram
mℓ	-	Microlitre
nm	-	Nanometre
µL	-	Microlitre
µm	-	Micrometre
m	-	Meter
wt%	-	Weight percent
t	-	Time
θ	-	Angle

CHAPTER 1

INTRODUCTION

1.1 Research Background

Although this world is mostly covered by water in its portion, fresh water supply is still shortening everywhere. Worsening water pollution, risen of industrial activities and rapid growing of human population are some causes of it. Back then, desalination was one of the ways to convert the cheapest and largest water source (seawater) into fresh water. This seawater desalination is still believed nowadays to offer a steady supply of high-quality water without damaging the natural freshwater ecosystems [1]. Many desalination technologies had been developed since then such as electrodialysis (ED) and multistage flash distillation (MFD). However, those technologies still could not meet the demand and quality acquired for a truly-called fresh water, besides having high operational cost as part of its disadvantages. Hence, another option called membrane-based technology is introduced to encounter previous ineffectiveness. Membrane had exposed its reliability and efficiency in water treatment process throughout the years. The fact that it works without additional chemicals with low energy utilization and by physical process only make it easy to conduct the process and at the same time, producing high quality recycled water [2].

One of the available membrane processes is forward osmosis (FO). It brings numerous benefits such as high water recovery, low membrane fouling and low energy consumption when compared to the others. Its system works based on natural-build osmotic pressure created by two different solutions used. FO membrane which is placed in between the solutions induced the separation process when it allows only water molecules to pass through and retaining the unwanted solutes. Common FO membranes which can be found nowadays were polymeric or composite polymeric membranes. They undoubtedly had shown exceptional water flux performance in FO process but at the same time, they continue to face problems in sustaining their rejection properties. Also, these type of membrane especially polymeric has significant disadvantages of abrasion, mineral scaling and short life span [1–3]. Therefore, to alleviate the problems, MOF ceramic membrane has been introduced.

Ceramic membrane is a class of inorganic membrane which consists of materials such as carbon, silica, oxides (alumina, titania, zirconia) and metals. In terms of better properties compared to polymeric, ceramic membrane possess superior thermal and mechanical stability, high resistance towards solvents and well-defined stable pore structure [4], [5]. However, despite of this benefits, ceramic membrane has certain shortcomings that need to be solved such as formation of cracks in its synthesis process and inter-crystalline defects which later on could affect the membranes' performances [3], [6]. A solution to this problem was exposed by incorporating nanoporous material such as metal-organic framework (MOF) on the ceramic membrane active layer. MOF has an outstanding features which consists of metal ions and organic linkers in its framework. Effectiveness of this material can be found in broad applications such as water separation, gas storage, and catalytic reaction previously [7], [8]. Incorporation of such interesting material on ceramic membrane during dope preparation or coating process was believed to give an enhance properties to the pristine membrane [9]. Furthermore, crack issue in the ceramic membrane also expected to be solved since MOF membrane synthesis does not require thermal burning of organic templates such in the synthesis of zeolite MFI membrane [3].

Among the available MOF materials, zirconium-based MOF, especially MIL series (MIL: Materials from Institute Lavoisier) and UiO series (UiO: University of Oslo) had shown their high stability in water and acidic condition [10]. Their strong ionic bonding between Zr^{4+} and carboxylate oxygen atoms in these MOF was what cause it to possess strong chemical and mechanical stability [11]. Moreover, their unique feature which none other MOF could offer was their pore. It can be tuned readily only by changing its organic linker to fit for selected separation application [12], [13]. This tuneable framework characteristic enables efficient combination of Zr-based MOF with other materials that will result to synergistic effect between them [7].

Hence, this kind of materials were definitely suitable for FO process due to their amazing characteristics. There are various methods available on producing MOF membranes including solvothermal/hydrothermal method, interfacial (contradiffusion) method and liquid phase epitaxy method [14]. Compared to others, solvothermal synthesis emerged as a facile and competitive method besides showing the most applicability in synthesizing existing MOF membranes [3], [6], [15], [16].

1.2 Problem Statement

Despite the solvothermal capability, this method of in situ and seeded growth has some difficulty in controlling the heterogeneous nucleation sites on the membrane substrates. Without any resolution, it would further results to non-uniform deposition, big inter-crystal gaps, and cracks on the membrane surface. Structure defects like these are undesirable as they would affect the membrane performance since gaps existed would allow solutes to pass through instead of water molecules. According to Fang et al., recent fabricated FO membrane with double skin (RO-like and NF-like) layers also could not prevent the reverse solute flux from happened during FO process although it did improved the water flux performance. Besides that, some studies indeed increased its water flux performance to a better level but at the same time, the reverse solute flux still increased rapidly. Other than that, fabrication of MOF membrane without any delamination issue was still remained as a challenge since it is difficult to obtain MOF crystals that can form strong coordination bonds with the mother substrates. Therefore, it remains a challenge to fabricate a competitive FO membrane that possess incredible structure which can perform high FO water flux with high rejection and low or none occurrence of reverse solute flux. Formation of a continuous MOF ceramic membrane might be a great option to build another potential FO membrane with high performance since excellent properties from both materials will be combined together. This research will discover the possibility of fabricating such membrane by varying the solvothermal synthesis conditions and polymerised the active layer with fluorinated polymer.

1.3 Research Objectives

To achieve the aim of this research, the following objectives were fulfilled respectively:

a) To study the effects of varying synthesis conditions on physicochemical properties and FO performance of MIL-140B and UiO-66-NDC hollow fibre produced through solvothermal method.

b) To investigate the effects of adding polymer on physicochemical properties of UiO-66-NDC hollow fibre.

c) To evaluate performance of the polymer-integrated UiO-66-NDC ceramic hollow fibre in term of water flux, reverse solute flux and salt rejection.

1.4 Research Scopes

As stated below are the steps needed to accomplish the aforementioned objectives:

Scope objective 1:

a) Preparation of Al_2O_3/YSZ substrate using phase inversion and sintering technique.

b) Modified the Al_2O_3/YSZ substrate surface with ZrO_2 layer using sol-gel Pechini's method.

c) Synthesize MIL-X and MIL-D at same solvothermal condition of 0.3 M and 16 h at 220 ºC.

d) Varying synthesis time (6 h and 16 h) and synthesis concentration of (0.15 M and 0.30 M) for MIL (A to D) at temperature of 220 ºC.

e) Regulating modulator equivalent condition (0, 2.8 and 10) and synthesis concentration of (0.58 M, 1.16 M, 1.74 M and 2.32 M) for MIL (2.8-A, 10-A, 10-B, 10-C and 10-D) at temperature of 220 ºC.

f) Lowering synthesis temperature to 120 ºC for sample UiO (2.8-A2, 10-A2, 10-B2, 10-C2 and 10-D2).

g) Regulating modulator equivalent (0, 2.8 and 10), synthesis concentration of (0.58 M, 1.16 M, 1.74 M and 2.32 M) for UiO (2.8-A2, 10-A2, 10-B2, 10-C2 and 10-D2) at temperature of 120 ºC.

h) Characterize all samples using Field Emission Scanning Electron Microscopic (FESEM), X-ray Diffraction (XRD), Thermogravimetric Analysis (TGA) and Brunauer-Emmett-Teller (BET) tests.

i) Evaluate the performance of all samples in term of water flux and reverse solute flux using forward osmosis test: feed solution (deionized water) and draw solution (100,000 ppm NaCl).

Scope objective 2:

a) Deposit sample UiO (2.8-A2, 10-A2, 10-B2, 10-C2 and 10-D2) hollow fibre active layer with fluorinated polymer (DEFENSA OP-4003) using dip-coating method and activated it with UV curing process.

b) Characterized the polymer-integrated UiO-66-NDC hollow fibre using Field Emission Scanning Electron Microscopic (FESEM), Atomic Force Microscopy (AFM), contact angle, X-ray Diffraction (XRD), Thermogravimetric Analysis (TGA), Fourier-Transform Infrared Spectroscopy (FTIR) and Brunauer-Emmett-Teller (BET) tests.

c) Evaluate the performance of polymer-integrated UiO-66-NDC hollow fibre in terms of water flux and reverse solute flux using forward osmosis test (active layer facing feed solution, AL-FS): feed solution (deionized water) and draw solution (100,000 ppm NaCl).

Scope objective 3:

a) Evaluate the monovalent salts rejection performance of the best polymer-integrated UiO-66-NDC hollow fibre produced in this study using active layer facing draw solution (AL-DS) configuration system: feed solution (deionized water) and draw solution (40,000 ppm KCl and NaCl).

1.5 Significance of Study

This research was the first to provide an insight on depositing MIL-140B and UiO-66-NDC on ceramic substrate for FO desalination process. Besides that, new reaction ability of Zr-based MOF especially UiO-66-NDC towards fluoride ion (F^-) has been discovered which helps gaining deeper knowledge on the materials properties. Also, throughout this research, the negative performance of FO membrane has been made possible to give positive value when synthesis temperature of 120°C and membrane active layer is polymerized with polymer resin using UV-curing method. Therefore, it is noteworthy to acknowledge that this study is important based on the stated significances. The final product of this research definitely fulfill the research aim by fabricating FO ceramic membrane that has high water flux, low reverse solute flux and at the same time has higher rejection that the existing FO membrane in literature.

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