POLYSULFONE BASED POLY(METHYL METHACRYLATE) DUAL LAYER HOLLOW FIBER MEMBRANE INCORPORATED WITH ACTIVATED CARBON FOR UREMIC TOXINS REMOVAL

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DEDICATION

This thesis is dedicated to my father in heaven, Zainol Abidin Bin Anas, my mother, Azizah Binti Senawi and the love of my life, Noresah Binti Said. It is also dedicated to my siblings, Ida Zaliza, Ina Maswati, Muhamad Hafiz, Muhammad Mukhlis, Muhammad Zahid, Muhammad Anis, Muhammad Siddiq, Muhammad Khidzir, Siti Zhulaiqha and Nur Athirah, close family and friends who gave me inspiration, encouragement, and endless support throughout my study. May this thesis be an inspiration and guidance in the future.

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ABSTRACT

Daily and nocturnal hemodialysis practices require a portable dialysis machine. To foster the development of portable dialysis machine, an innovative technology to regenerate dialysate is needed. Hence, the objective of this study is to develop a highly selective polysulfone/poly(methyl methacrylate) (PSf/PMMA) dual layer hollow fiber (DLHF) membrane incorporated with activated carbon (AC) for collective removal of uremic toxins. In the first phase of the study, the urea adsorption capacity of PMMA was enhanced by a surface modification process using 5 %v/v aqueous (3-aminopropyl)triethoxysilane solution. The silane coating on the surface of PMMA particle was observed using transmission electron microscopy and the identification of silicon and nitrogen elements by the energy-dispersive X-ray spectroscopy has confirmed the successful modification of PMMA. A comprehensive adsorption study of urea was then conducted on the PMMA, whereby the isotherm, kinetic and thermodynamic of the adsorption process were determined. Modified PMMA showed urea adsorption capacity of 57 mg/g, which was higher than the unmodified PMMA (23 mg/g) due to the increased number of active adsorption sites. The urea adsorption onto PMMA surface was found as a non-spontaneous physical process that follows Freundlich isotherm model and Lagergren's pseudo-second-order kinetic model. In the second phase of the study, DLHF membranes consisting of PSf inner layer and PSf/PMMA outer layer were fabricated via a single-step co-extrusion technique using a triple orifice spinneret. The effect of PSf/PMMA composition (PSf:PMMA; 18:2, 15:5, 12:8, 10:10 and 8:12) on physical compatibility, molecular sieving properties and urea removal performance of the DLHF membranes were investigated. In conditions where the composition of PMMA is lesser than PSf, there was no sign of delamination between the two membrane layers. Results showed that the DLHF membrane exhibited urea adsorption capacity from 5.5 to 27.6 mg/g. In ultrafiltration adsorption experiment, the membrane with PSf/PMMA composition of 12:8 demonstrated significant urea removal of 39.2% and showed desired sieving properties towards large solute (lysozyme). In the third phase of the study, AC particles were incorporated in the inner layer of the DLHF membrane, where the effect of AC loading (0, 3, 5, 7 and 9 wt%) on the co-adsorptive urea and creatinine removal performance was investigated. The DLHF membrane with the highest AC loading (9 wt%) displayed the highest maximum adsorption capacity of creatinine of 86.2 mg/g. Besides, the membrane demonstrated the highest flux of 16.4 $Lm^{-2}h^{-1}$ and rejection of 35.3% and 73.3% for urea and creatinine, respectively. In the final phase of the study, the long-term stability of the optimized PSf/PMMA/AC DLHF membrane in continuous operation was evaluated. The membrane was tested for 3 cycles of 6-hour operation, whereby in each cycle, the membrane experienced different extents of reduction in flux and solute rejection. The membrane showed promising reusability with a high overall solute rejection recovery rate of 86% and 73% in the second and third cycles, respectively. The PSf/PMMA/AC DLHF membrane was successfully fabricated and showed collective removal of uremic toxins via the combined process of adsorption and filtration, hence becoming a potential candidate for dialysate regeneration.

ABSTRAK

Amalan hemodialisis harian dan malam memerlukan sebuah mesin dialisis mudah alih. Bagi mendorong pembangunan mesin dialisis mudah alih, suatu teknologi inovatif untuk menjana semula dialisat diperlukan. Oleh itu, objektif kajian ini adalah untuk membangunkan suatu membran gentian berongga dwilapisan (DLHF) polisulfon/poli(metil metakrilat) (PSf/PMMA) yang dicampurkan dengan karbon aktif (AC) yang sangat selektif untuk penyingkiran toksin uremik secara kolektif. Pada fasa pertama kajian, kapasiti penjerapan urea PMMA ditingkatkan dengan proses pengubahsuaian permukaan menggunakan larutan akueus 5 %v/v (3-aminopropil) trietoksilana. Lapisan silana pada permukaan partikel PMMA diperhati menggunakan mikroskop elektron transmisi dan pengenalpastian unsur silikon dan nitrogen oleh spektroskopi sinar-X penyebaran tenaga telah mengesahkan pengubahsuaian PMMA yang berjaya. Kajian penjerapan urea komprehensif kemudian telah dilakukan pada PMMA, di mana isoterm, kinetik dan termodinamik proses penjerapan ditentukan. PMMA yang diubahsuai menunjukkan kapasiti penjerapan urea sebanyak 57 mg/g, yang mana lebih tinggi daripada PMMA yang tidak diubah suai (23 mg/g) disebabkan oleh peningkatan bilangan kawasan jerapan aktif. Penjerapan urea ke atas permukaan PMMA didapati sebagai proses fizikal tidak spontan yang mematuhi model isoterm Freundlich dan model kinetik pseudo-urutan kedua Lagergren. Pada fasa kedua kajian, membran DLHF yang terdiri daripada lapisan dalam PSf dan lapisan luar PSf/PMMA dihasilkan melalui teknik penyemperitan bersama satu langkah menggunakan pemintal tiga orifis. Kesan komposisi PSf/PMMA (PSf:PMMA; 18:2, 15:5, 12:8, 10:10 dan 8:12) terhadap keserasian fizikal, sifat penyaringan molekul dan prestasi penyingkiran urea membran DLHF telah dikaji. Dalam keadaan di mana komposisi PMMA lebih rendah daripada PSf, tidak terdapat tanda-tanda penempaan di antara dua lapisan membran. Hasil kajian menunjukkan bahawa membran DLHF tersebut menunjukkan kapasiti penjerapan urea dari 5.5 hingga 27.6 mg/g. Dalam eksperimen penjerapan ultraturasan, membran dengan komposisi PSf/PMMA 12:8 menunjukkan penyingkiran urea yang signifikan sebanyak 39.2% dan menunjukkan sifat penyaringan yang diinginkan terhadap zat terlarut besar (lisozim). Pada fasa ketiga kajian, partikel AC dimasukkan ke dalam lapisan dalam membran DLHF, di mana kesan kandungan AC (0, 3, 5, 7 dan 9 wt%) terhadap prestasi penyingkiran urea dan kretinin secara penjerapan bersama dikaji. Membran DLHF dengan kandungan AC tertinggi (9 wt%) menunjukkan kapasiti penjerapan maksimum kreatinin tertinggi sebanyak 86.2 mg/g. Selain itu, membran tersebut menunjukkan fluks tertinggi sebanyak 16.4 $Lm^{-2}h^{-1}$ dan penyingkiran sebanyak 35.3% dan 73.3%, iaitu masing-masing untuk urea dan kreatinin. Pada fasa akhir kajian, kestabilan jangka panjang membran PSf/PMMA/AC DLHF yang dioptimumkan dalam operasi berterusan telah dinilai. Membran ini diuji selama 3 kitaran operasi 6 jam, di mana dalam setiap kitaran, membran tersebut mengalami pengurangan yang berbeza terhadap fluks dan penyingkiran zat terlarut. Membran tersebut menunjukkan kebolehgunaan semula yang memberangsangkan dengan kadar pemulihan penyingkiran zat terlarut keseluruhan yang tinggi sebanyak 86% dan 73%, masing-masing pada kitaran kedua dan ketiga. Membran PSf/PMMA/AC DLHF berjaya dihasilkan dan menunjukkan penyingkiran toksin uremik secara kolektif melalui gabungan proses penjerapan dan penapisan, justeru menjadi calon yang berpotensi untuk penjanaan semula dialisat.

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LIST OF ABBREVIATIONS

AC	-	Activated Carbon
AFM	-	Atomic Force Microscopy
APTES	-	(3-Aminopropyl)Triethoxysilane
ATIII	-	Antithrombin III
ATR	-	Attenuated Total Reflectance
BET	-	Brunauer-Emmet-Teller
BJH	-	Barrett-Joyner-Halenda
CA	-	Cellulose Acetate
DER	-	Dope Extrusion Rate
DLHF	-	Dual Layer Hollow Fiber
EDX	-	Energy-Dispersive X-Ray Spectroscopy
ESRF	-	End-Stage Renal Failure
FO	-	Forward Osmosis
FTIR	-	Fourier Transform Infrared Spectroscopy
HAP	-	Hydroxyapatite
IUPAC	-	International Union of Pure and Applied Chemistry
PSf	-	Polysulfone
MF	-	Microfiltration
MMM	-	Mixed Matrix Membrane
m-PMMA	-	Modified Poly(Methyl Methacrylate)
NF	-	Nanofiltration
NIPS	-	Non-Solvent Induced Phase Separation
NMP	-	N-Methyl-2-Pyrrolidone
PAI	-	Polyamide-Imide
PBS	-	Phosphate Buffer Saline
PES	-	Polyethersulfone
PMMA	-	Poly(Methyl Methacrylate)
PVDC	-	Polyvinylidene Chloride
PVDF	-	Polyvinylidenefluoride
PVP	-	Polyvinylpyrrolidone

PWF	-	Pure Water Flux
RO	-	Reverse Osmosis
SC	-	Sieving Coefficient
SEM	-	Scanning Electron Microscopy
SLHF	-	Single Layer Hollow Fiber
TEM	-	Transmission Electron Microscopy
TIPS	-	Thermal Induced Phase Separation
UF	-	Utrafiltration

LIST OF SYMBOLS

A	-	Effective surface area
b	-	Langmuir constant
C_e	-	Equilibrium concentration
C_f	-	Concentration of feed
C_o	-	Initial concentration
C_p	-	Concentration of permeate
J	-	Flux
K_a	-	Adsorption distribution coefficient
K_F	-	Freundlich constant
k_1	-	Lagergren's pseudo-first-order model rate constant
k_2	-	Lagergren's pseudo-second-order model rate constant
l	-	Thickness
m	-	Mass
n	-	Number of trials
P	-	Pressure
q_e	-	Equilibrium adsorption capacity
Q_{max}	-	Maximum adsorption capacity
q_t	-	Adsorption capacity at designated time
r	-	Radius
R	-	Rejection
R^2	-	Correlation coefficient
R_a	-	Average surface roughness
R_i	-	Average solute rejection in the first cycle
R_n	-	Average solute rejection in successive number of cycles
R_R	-	Rejection recovery
t	-	Time
T	-	Temperature
V	-	Volume
w_1	-	Mass of wet membrane
w_2	-	Mass of dry membrane

$\Delta G°$	-	Gibbs free energy
$\Delta H°$	-	Enthalpy
$\Delta S°$	-	Entropy
ε	-	Porosity
η	-	Viscosity
λ	-	Wavelength
ρ_w	-	Density of water

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CHAPTER 1

INTRODUCTION

1.1 Research Background

In the 21st century, the number of chronic kidney disease patients has increased terrifically where these patients suffer from the incapability of filtering and removing body waste. According to Malaysia's National Renal Registry, it has been reported that the total amount of people undergoes hemodialysis had risen from 6,689 to 21,159 people in 2009 (Cheng, 2011). The latter report in May 2013 indicated the increase of hemodialysis patients to 26,159 people (Cheng, 2013). The latest statistics issued by National Kidney Foundation revealed that 30,000 Malaysians needed hemodialysis in 2014 (Cruez, 2014), and the number further increased by 8,157 patients in March 2016 (Mustapha and Bavanandan, 2018). This shows the growth of about 4,000 newly registered patients each year and that number was expected to continue to rise in years to come (Hammim, 2017). The patients undergoing hemodialysis are usually termed as the end-stage renal failure (ESRF) patients. ESRF is the last stage of chronic kidney disease, whereby the long-termed kidney failure has caused drastic reduction of glomerular filtration rate to below 15 mL/min (Kalra *et al*., 2006). Besides, the kidney function of these patients is said to fall to 15% of its normal capacity. As the result, the wastes such as the end-products of metabolism reactions occurred in body, together with the electrolytes may accumulate in the blood to cause complications (Liu *et al.*, 2020).

Hemodialysis is the most widely applied extracorporeal treatment to filter and purify blood. It is considered as a highly successful treatment that provides the second chance to live. The treatment includes the removal of waste products, mostly referred as uremic toxins (e.g. urea and creatinine) from blood and the control of water and electrolyte content (e.g. sodium chloride, NaCl) in blood. These processes occur within the heart of hemodialysis, namely dialyzer. Inside a dialyzer, the blood flows into

thousands of semipermeable membrane fibers surrounded by an electrolyte solution called dialysate. The continuous flow of dialysate outside the membrane fibers creates concentration difference for the separation processes to happen. A conventional hemodialysis treatment requires patients to have thrice sessions a week, with an average of four hours for each session. However, the 'unphysiology' of the intermittent hemodialysis treatment has become a concern, as it leads to major complications such as cardiovascular events, impaired cognitive function, anemia, and bone disease (Kim and Ronco, 2011; Kjellstrand *et al*., 1978; Neumann *et al*., 2017). This has resulted in the increase of mortality rate among ESRF patients (Kim and Ronco, 2011). Besides, the hours of conventional hemodialysis may limit patients' freedom and restrict them from dietary choices. In the past decade, there has been a growing interest among researchers and nephrologists regarding slow and frequent form of hemodialysis. Researchers and nephrologists agree that this extended-hours hemodialysis is associated with outstanding clinical outcomes, for example better control of blood pressure and quicker recovery rate after the treatment (Rivara *et al*., 2016).

With the new piece of information on the alternative form of hemodialysis, the research to improve hemodialysis treatment has revolutionized as it is now coming from many aspects of the system, from developing a better dialyzer using high performance dialysis membrane to adjusting the entire system for different modes of hemodialysis. To implement slow and more frequent hemodialysis away from hospital or dialysis center, a portable dialysis machine is practically needed where the patients may have a direct access towards the machine. The idea to develop portable hemodialysis system was brought up in early 1970s to improve the treatment and patients' circumstances (Davenport, 2015). It is the best way to mimic the natural kidney functions by constantly cleansing the blood from harmful toxins. Though several portable dialysis models have been developed (Fissell *et al*., 2013; Gura *et al*., 2009), there is no implementation in clinical practice due to some technical issues particularly the limitation in dialysate regeneration (Kim and Ronco, 2011).

One of the main components of a portable dialysis model is dialysate regeneration system which functions to ensure continuous influx of dialysate in the dialyzer (Cheah, Sim and Yeoh, 2016; Kim and Ronco, 2011). Dialysate is very

important to ensure the smooth operation of the treatment as it controls the movement of solutes across the hemodialysis membrane via concentration gradient. The complexity of dialysate production and the large consumption of water are among the reasons why dialysate regeneration system is needed. Hemodialysis demands a large volume of water to operate in which it consumes in average 120 L of water per session (Gura *et al.*, 2016). Water is used to dilute the dialysate which is normally stored as a concentrated solution. The dilution is typically performed at water:dialysate ratio of 30:1 (Kameda *et al.*, 2019). During the treatment, the uremic toxins contained in blood constantly diffuse into the dialysate. The dialysate becomes saturated and this situation would affect the diffusion rate of the solutes. In order to reduce water consumption and at the same time controlling the concentration of diffused solutes in the dialysate, the produced dialysate should be condensed and regenerated within a small and closed system (Cheah, Sim and Yeoh, 2016).

There have been a very limited number of works conducted to address this matter (Petrella, Orlandini and Bigi, 1975; Talaat, 2009). These works intended to recover the dialysate as much as possible by removing the diffused uremic toxins and to recirculate the dialysate back into the dialyzer. Adsorption is the simplest yet the most effective and practical method to remove small water-soluble molecules, for a lightweight setup of the integrated system. Previously, activated carbon (AC) has been employed as the adsorbent to remove uremic toxins from used dialysate along with the ion-exchangers such as hydrated zirconium-oxide, hydrated zirconium-phosphate and activated aluminum silicate (Agar, 2010; Petrella, Orlandini and Bigi, 1975; Wester *et al*., 2014). AC has been chosen because of its versatility to adsorb a wide range of uremic toxins (Tijink *et al*., 2013). In current practices, selected materials are packed into a cylindrical sorbent cartridge to adsorb the uremic toxins and to restore ions in the dialysate. Even now, the search for the most viable technology for dialysate regeneration never ceases as researchers occasionally look for other alternatives.

Today, one of the fastest growing and the most advanced technologies in separation field is membrane technology. The first ever membrane separation process studied for dialysate regeneration was forward osmosis (FO) (Talaat, 2009; Shaffer *et al.*, 2015) . The FO process was able to reclaim a good portion of water from the used

dialysate. This kind of development has opened the possibility for a combined technological solution by adapting adsorption into membrane separation process to attain the best version of dialysate regeneration system. In this study, attempts were made to fabricate a polysulfone (PSf)-based dual layer hollow fiber (DLHF) membrane incorporated with two adsorptive materials, namely poly(methyl methacrylate) (PMMA) and AC for uremic toxin removal from aqueous solution. The synergism between PMMA and AC in the PSf matrix in demonstrating enhanced membrane adsorption properties and separation performance was investigated.

1.2 Problem Statement

Current prototype models of portable dialysis machine face technical difficulties in developing a reliable dialysate regeneration system. The sorbent cartridges used in these models have failed to meet the technical requirements for the system due to the poor removal of uremic toxins from dialysate. As the result, hemodialysis treatment suffers poor diffusive clearance of uremic toxins because the concentration gradient is not restored inside the dialyzer. The popularly used adsorbents to capture a wide range of uremic toxins include commercial zeolites and AC as they possess unique pore structures and wide pore size distribution (Jaramillo, Álvarez and Gómez-Serrano, 2010; Cheah *et al.*, 2017). However, urea which is the most abundant uremic toxin in blood is hardly removed using the commercial adsorbents especially AC (Cheah, Sim and Yeoh, 2016). The previously proposed enzymatic reaction to aid the adsorption process using urease to break down urea into ammonia and carbon dioxide may complicate the system as the release of the ammonia can favor the precipitation of calcium carbonate in the dialysate (Gura *et al*., 2009; Wester *et al*., 2014).

Among the materials reported to have high urea adsorption capacity are oxidized starch (Shimizu and Fujishige, 1983) and amine-functionalized mesoporous silica (Cheah, Sim and Yeoh, 2016). Each of these materials adopted different strategies to enhance urea adsorption capacity. Oxidation of the starch was intended to introduce aldehyde group that can form a specific interaction with amine group of urea

via nucleophilic substitution reaction (Shimizu and Fujishige, 1983). This approach requires higher energy for the adsorption to take place as it involves chemisorption that is naturally irreversible. Moreover, oxidized starch was later found to be unstable as it dissolves in water and disappears. On the contrary, mesoporous silica was functionalized with an amino-functional silane to improve the urea adsorption capacity by offering extra adsorption sites for urea molecules. Despite the good attempts to produce a promising urea adsorbent, another unmet challenge arises which is the limitation of adsorbents to specifically adsorb the targeted solute among the vast number of solutes. Competitive adsorption is known to occur between middle molecules (>500 g/mol) and small water-soluble molecules (<500 g/mol) or within the same class of solutes. The competitive adsorption among solutes especially amino-compounds (i.e. urea and creatinine) may disrupt the efficiency of the adsorbent as they compete for active adsorption sites.

To address the problems, a one-step adsorption-filtration process using membrane technology is an attractive alternative to selectively remove the targeted molecules. This can be done using adsorptive membrane that allows two mechanisms of separation namely molecular sieving and adsorption to take place simultaneously during the process to cater different target groups. The selection of materials to form the adsorptive membrane is based on the sieving and adsorption properties required. Typical ceramic and polymeric membranes both have disadvantages in their current state to be used as adsorptive membrane. Ceramic membranes, although can be prepared using adsorptive materials such as zeolites (Adam *et al.*, 2019), tend to have large pore size and require a number of post-fabrication treatments. On the other hand, almost all polymeric membranes have poor adsorption capacity which is the only downside of using these membranes. Nevertheless, PSf and polyethersulfone (PES) membranes have been reliably utilized as low pressure-driven membranes to reject a wide range of solutes (Said *et al.*, 2019a, b; Kumari, Modi and Bellare, 2020). In this situation, polymeric membrane is the better pick due to its easily tailored pore size to reject middle molecules and its proven capability as a host for adsorbents to adsorb small water-soluble molecules.

Adsorptive polymers serve as a good alternative for adsorptive membrane fabrication. PMMA membranes are known for their pronounced adsorption property for middle molecules like cytokines and β_2-microglobulin (Moachon *et al.*, 2002; Masakane *et al.*, 2017; Uchiumi *et al.*, 2018). It was evident that PMMA membrane adsorbed 28.6 μg β_2-microglobulin and 0.72 μg lysozyme (Moachon *et al.*, 2002). The results also revealed that neat PMMA membrane was not well suited for ultrafiltration (UF) process as it rejected only 2.6% β_2-microglobulin and 14% lysozyme via size exclusion. Similar outcome was reported when PMMA was added as a secondary polymer to polyvinylidenefluoride (PVDF) single layer membrane (Ai *et al.*, 2012; Tan *et al.*, 2014). This trade-off effect is the common limitation encountered to develop membranes for adsorption-filtration process. To compensate the effect, methods such as surface coating and interfacial polymerization were used to uphold the intrinsic properties of the polymers (Choi *et al.*, 2019). For hollow fiber configuration, a more practical way to pair up the polymers without losing their unique properties is by preparing a DLHF membrane. This membrane setup allows the denser layer to filter middle molecules within UF range and the more porous layer containing adsorptive polymer to adsorb the small water-soluble molecules.

Understanding the limitation of each adsorbent, combination of two or more adsorptive components in a system is an interesting approach to be explored in order to achieve efficient removal of a wide range of uremic toxins. Therefore, in this study, DLHF membranes which composed of PSf/AC inner layer and PSf/ PMMA outer layer were prepared via co-extrusion technique for co-adsorptive removal of small water-soluble molecules (urea and creatinine) from aqueous solution. Prior to membrane fabrication, a surface modification was performed on PMMA to improve the adsorption capacity of PMMA. PSf with polyvinylpyrrolidone (PVP) as a pore former was used as a base material in both membrane layers to ensure good compatibility between the layers. In the outer layer, PMMA was added to induce adsorption capabilities to the membrane, particularly to adsorb urea. Meanwhile. the inner layer of the membrane served as the first barrier to reject middle molecules (lysozyme) by size exclusion. AC as the universal adsorbent was then incorporated in the inner layer of the membrane to adsorb creatinine, hence minimizing competitive adsorption. This was the first attempt of producing membrane that can effectively reject uremic toxins

via the combined effects of molecular sieving and adsorption using two adsorptive components.

1.3 Objectives of the Study

The main objective of this study is to fabricate a highly selective PSf/PMMA DLHF membrane embedded with AC via a single-step co-extrusion technique for collective removal of uremic toxins. Based on the research background and problem statement, the specific objectives of this study are listed below:

1. To perform surface modification and characterization on PMMA and evaluate the urea adsorption properties of PMMA.

2. To study the effects of PSf/PMMA composition on the physical compatibility, molecular sieving properties and adsorption performance of the DLHF membranes.

3. To investigate the effects of AC addition on the co-adsorptive urea and creatinine removal performance of the resultant membrane in terms of flux and rejection.

4. To evaluate the long-term stability of the optimized membrane in continuous operation and the reusability of the membrane.

1.4 Scopes of the Study

To fulfil the objectives, the following scopes of work are outlined:

1. Modifying the surface of PMMA through amino-silanization using 5 %v/v aqueous (3-Aminopropyl)triethoxysilane (APTES) solution at fixed reaction conditions.

2. Characterizing the PMMA before and after modification by transmission electron microscopy (TEM), energy-dispersive X-ray spectroscopy (EDX), pore and surface analysis and Fourier transform infrared spectroscopy (FTIR).

3. Conducting the adsorption isotherm, kinetic and thermodynamic studies of urea on the PMMA by varying the initial urea concentration (0, 500, 1000, 1500, 2000, and 2500 mg/L), contact time (0-24 hours) and absolute temperature (281.15, 298.15, 310.15 and 323.15 K).

4. Preparing an inner dope solution containing 18 wt% PSf and 3 wt% PVP and several outer dope solutions by varying the PSf/PMMA composition (PSf:PMMA; 18:2, 15:5, 12:8, 10:10 and 8:12) using N-methyl-2-pyrrolidone (NMP) as solvent.

5. Fabricating a PSf single layer hollow fiber (SLHF) membrane and the PSf/PMMA DLHF membranes of different PSf/PMMA compositions in the outer layer via one-step co-extrusion technique using a triple orifice spinneret at fixed spinning parameters.

6. Examining the morphology of PSf SLHF membrane and PSf/PMMA DLHF membranes by scanning electron microscopy (SEM) and atomic force microscopy (AFM), surface hydrophilicity using contact angle goniometer, overall porosity, water uptake, average pore size, and confirming the chemical functionality by FTIR.

7. Evaluating the separation features of the PSf SLHF membrane and PSf/PMMA DLHF membranes in terms of flux and sieving coefficient for various solutes (NaCl, urea, vitamin B_{12}, and lysozyme) at the transmembrane pressure of 1.0 bar.

8. Evaluating the adsorption capacity and the UF adsorption performance of PSf/PMMA DLHF membranes for urea removal.

9. Preparing several inner dope solutions by varying the AC loading (0, 3, 5, 7 and 9 wt%) and an outer dope solution using the optimized PSf/PMMA composition.

10. Fabricating a PSf/AC SLHF membrane and the PSf/PMMA/AC DLHF membranes of different AC loadings in the inner layer via one-step co-extrusion technique using a triple orifice spinneret at fixed spinning parameters.

11. Characterizing the PSf/AC SLHF membrane and the PSf/PMMA/AC DLHF membranes in terms of morphology, chemical functionality, surface hydrophilicity, porosity, water uptake and pure water flux (PWF).

12. Investigating the creatinine adsorption properties of PSf/AC SLHF membrane, PSf/PMMA DLHF membrane and PSf/PMMA/AC DLHF membranes by studying the effect of initial concentration (500, 1000, 1500 and 2000 mg/L) and contact time (0-24 hours) on the adsorption capacity of the membranes and determining the adsorption isotherm and kinetic involved in the process.

13. Studying the competitive adsorption between urea and creatinine for PSf/AC SLHF membrane, PSf/PMMA DLHF membrane and PSf/PMMA/AC DLHF membrane in a binary batch adsorption system at various solute concentrations (500, 1000, 1500 and 2000 mg/L).

14. Performing a UF adsorption experiment on PSf/AC SLHF membrane, PSf/PMMA DLHF membrane and PSf/PMMA/AC DLHF membrane to evaluate the co-adsorptive removal performance of the membranes in terms of flux and rejection of urea and creatinine.

15. Conducting a long-term UF adsorption experiment consisting 3 cycles with a total duration of 18 hours on the PSf/PMMA/AC DLHF membrane with optimized PSf/PMMA composition and AC loading to study the stability and reusability of the membrane based on flux, solute rejection and the solute rejection recovery.

16. Studying the leaching phenomenon of adsorbed urea and creatinine from membrane during the UF adsorption experiment through water permeation.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Abidin, M. N. Z.**, Goh, P. S., Said, N., Ismail, A. F., Othman, M. H. D., Hasbullah, H., Abdullah, M. S., Ng, B. C., Kadir, S. H. S. A., Kamal, F., Mansur, S. (2020). Co-adsorptive removal of creatinine and urea by a three-component dual layer hollow fiber membrane. *ACS Applied Materials & Interfaces*, *12*, 33276-33287. https://doi.org/10.1021/acsami.0c08947**. (Q1, IF: 8.758)**

2. **Abidin, M. N. Z.**, Goh, P. S., Said, N., Ismail, A. F., Othman, M. H. D., Hasbullah, H., Abdullah, M. S., Ng, B. C., Kadir, S. H. S. A., Kamal, F., Mansur, S. (2019). Polysulfone/amino-silanized poly(methyl methacrylate) dual layer hollow fiber membrane for uremic toxin separation. *Separation and Purification Technology*, *236*, 116216. https://doi.org/10.1016/j.seppur.2019.116216. **(Q1, IF: 5.774)**

3. **Abidin, M. N. Z.**, Goh, P. S., Ismail, A. F., Said, N., Othman, M. H. D., Hasbullah, H., Abdullah, M. S., Ng, B. C., Kadir, S. H. S. A., Kamal, F. (2018). Highly adsorptive oxidized starch nanoparticles for efficient urea removal. *Carbohydrate Polymers*, *201*, 257-263. https://doi.org/10.1016/j.carbpol.2018.08.069. **(Q1, IF: 7.182)**

4. **Abidin, M. N. Z.**, Goh, P. S., Ismail, A. F., Othman, M. H. D., Hasbullah, H., Said, N., Kadir, S. H. S. A., Kamal, F., Abdullah, M. S., Ng, B. C. (2017). Development of biocompatible and safe polyethersulfone hemodialysis membrane incorporated with functionalized multi-walled carbon nanotubes. *Materials Science and Engineering C*, *77*, 572-582. https://doi.org/10.1016/j.msec.2017.03.273. **(Q1, IF: 5.880)**

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1. **Abidin, M. N. Z.**, Goh, P. S., Ismail, A. F., Said, N., Othman, M. H. D., Hasbullah, H., Abdullah, M. S., Ng, B. C., Kadir, S. H. S. A., Kamal, F. (2019). Polysulfone/iron oxide nanoparticles ultrafiltration membrane for adsorptive removal of phosphate from aqueous solution. *Journal of Membrane Science & Research*, *5*, 20-24. https://doi.org/10.22079/JMSR.2018.87665.1195. **(Indexed by SCOPUS)**

2. **Abidin, M. N. Z.**, Goh, P. S., Ismail, A. F., Othman, M. H. D., Hasbullah, H., Said, N., Kadir, S. H. S. A., Kamal, F., Abdullah, M. S., Ng, B. C. (2017). The effect of PCA-g-MWCNTs loading on the performance of PES/MWCNTs hemodialysis membrane. *Chemical Engineering Transactions*, *56*, 1609-1614. https://doi.org/10.3303/CET1756269. **(Indexed by SCOPUS)**

Book Chapter

1. Ismail, A. F., **Abidin, M. N. Z.**, Mansur, S., Zailani, M. Z., Said, N., Raharjo, Y., Rosid, S. M., Othman, M. H. D., Goh, P. S., Hasbullah, H. (2018). Hemodialysis membrane for blood purification in *Handbooks in Separation Science: Membrane Separation Principles and Applications from Material Selection to Mechanisms and Industrial Uses*, 283-309, ISBN: 978-0-12-812815-2. **(ELSEVIER)**