MULTIWALL CARBON NANOTUBES/LITHIUM SALTS/POLYETHERSULFONE MEMBRANE FOR MICROALGAE HARVESTING

NUR FARAHAH BINTI MOHD KHAIRUDDIN

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School of Chemical and Energy Engineering
Faculty of Engineering
Universiti Teknologi Malaysia

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ABSTRACT

Membrane filtration for microalgae harvesting has been hampered by bio-fouling. In this research work, feasibility of microalgae harvesting using good anti fouling polyethersulfone (PES) membranes was examined. The main objective of the study is to develop a high-performance membrane with anti-fouling effect for microalgae harvesting. The antifouling PES membranes were fabricated using PES, multiwall carbon nanotubes (MWCNT) and two different additives i) lithium bromide (LiBr) ii) lithium chloride (LiCl) in dimethylacetamide. PES/MWCNT is the control membrane. The membranes were prepared via two methods; non-solvent induced phase separation (NIPS) and thermally induced phase separation (TIPS). The membrane performances were evaluated in terms of membrane flux, molecular weight cut-off and fouling performances. The results show that the morphology of the hybrid PES/MWCNT/LiCl and PES/MWCNT/LiBr membranes were very much influenced by the phase separation method. Lithium salts helped to increase membrane porosity. Flux rates of the membranes were improved dramatically with increasing amount of additives when prepared using TIPS. Both NIPS and TIPS membranes can separate 100% of the microalgae. In terms of fouling propensity, TIPS membrane with LiCl exhibited more than 80% flux recovery while TIPS membrane with LiBr showed 100% flux recovery which exhibits excellent anti-fouling property. The membrane fabricated with 1 wt% MWCNT, 5 wt% LiBr and 18 wt% PES via TIPS process possessed an excellent filtration performance and anti-fouling effect. 5.5 g/l *Nannochloropsis* sp. have been fully retained using the fabricated membrane with average flux 28.9 L/m^2h. Furthermore, the membrane demonstrated excellent anti-fouling effect owing to its higher membrane hydrophilicity (33.76^o). Thus, the fabricated membrane can help to improve sustainability in algae-based production.

ABSTRAK

Penurasan membran untuk penuaian mikroalga telah terhalang oleh biokotoron. Di dalam kajian ini, kebolehan penuaian mikroalga menggunakan antikotoran membran polietersulfon (PES) yang baik telah dikaji. Objektif utama kajian ini ialah untuk menghasilkan sebuah membran berprestasi tinggi dengan kesan antikotoran untuk penuaian mikroalga. Antikotoran membran PES telah dibuat menggunakan PES, karbon nanotiub dinding berbilang (MWCNT) dan dua bahan tambah berlainan i) litium bromida (LiBr) ii) litium klorida (LiCl) dalam dimetilasetamid. PES/MWCNT adalah membran kawalan. Membran telah dihasilkan melalui dua kaedah; fasa permisahan bukan-pelarut teraruh (NIPS) dan fasa permisahan haba teraruh (TIPS). Prestasi membran telah dinilai dari segi fluks membran, potongan berat molekul dan prestasi kotoran. Keputusan menunjukkan bahawa morfologi hibrid membran PES/MWCNT/LiCl dan PES/MWCNT/LiBr sangat dipengaruhi oleh kaedah fasa pemisahan. Garam litium telah membantu untuk meningkatkan keliangan membran. Kadar fluks membran bertambah baik secara dramatik dengan penambahan jumlah bahan tambah apabila menggunakan TIPS. Kedua-dua membran NIPS dan TIPS boleh memisahkan mikroalga 100%. Dari segi kecenderungan untuk kotor, membran TIPS dengan LiCl mempamerkan lebih 80% perolehan fluks manakala membran TIPS dengan LiBr menunjukkan 100% perolehan fluks iaitu mempamerkan kecemerlangan sifat antikotoran. Membran yang diperbuat dengan 1 wt% MWCNT, 5 wt% LiBr dan 18 wt% PES melalui proses TIPS mempunyai prestasi penurasan dan kesan antikotoran yang cemerlang. 5.5 g/l *Nannochloropsis* sp. telah sepenuhnya ditahan menggunakan membran yang dibuat dengan purata fluks 28.9 L/m^2h. Tambahan pula, membran tersebut menunjukkan kesan antikotoran yang cemerlang kerana ketinggian hidrofilik membran (33.76^o). Oleh itu, membran yang dibuat dapat membantu untuk membaiki kelestarian pengeluaran berasaskan alga.

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LIST OF ABBREVIATIONS

Ag	-	Silver nanoparticles
AOM	-	Alga organic matter
Al_2O_3	-	Alumina oxide
AFM	-	Atomic force microscope
BSA	-	Bovine serum albumin
CO_2	-	Carbon dioxide
$CaCO_3$	-	Calcium carbonate
CNT	-	Carbon nanotubes
CA	-	Cellulose acetate
CP	-	Concentration polarization
CF	-	Concentration factor
DMAC	-	Dimethyl acetamide
DMF	-	Dimethyl formamide
DMSO	-	Dimethyl sulfoxide
EDX	-	Energy dispersive X-ray
EOM	-	Extracellular organic matter
EPS	-	Extracellular polymeric substances
FESEM	-	Field emission scanning electron microscopy
FRR	-	Flux recovery ratio
FTIR	-	Fourier transform infrared spectroscopy
FFA	-	Free fatty acid
H_2SO_4	-	Sulfuric acid
HNO_3	-	Nitric acid
LiBr	-	Lithium bromide
LiCl	-	Lithium chloride
LiF	-	Lithium fluoride
MBR	-	Membrane bioreactor
MF	-	Microfiltration
MWCO	-	Molecular weight cut-off
MWCNT	-	Multiwall carbon nanotube

$NaClO$ - Sodium hypochlorite
$NaOH$ - Sodium hydroxide
NF - Nanofiltration
NIPS - Non-solvent induced phase separation
NMP - N-methylpyrrolidone
PAN - Polyacrylonitrile
PE - Polyethylene
PES - Polyethersulfone
PEG - Polyethylene glycol
PSf - Polysulfone
PTE - Polytetrafluorethylene
PVA - Polyvinyl alcohol
PVC - Polyvinyl chloride
PVDF - Polyvinylidene fluoride
PVP - Polyvinylpyrrolidone
PP - polypropylene
RO - Reverse osmosis
SEM - Scanning electron microscopy
SiO_2 - Silicon Dioxide
SMP - Soluble microbial products
TIPS - Thermally induce phase separation
TiO_2 - Titanium oxide
TMP - Transmembrane pressure
TAG - Triglyceride
UF - Ultrafiltration
VIPS - Vapor induced phase separation
VPSEM - Variable pressure scanning electron microscopy
VRF - Volume reduction factor
ZnO - Zinc oxide

LIST OF SYMBOLS

A	-	Area
Ca	-	Calcium
^{o}C	-	Degree celcius
ε	-	Porosity
Fe	-	Ferum
Fr	-	Reversible fouling
F_{ir}	-	Irreversible fouling
F_T	-	Total fouling
g/L	-	Gram per Liter
J_{wo}	-	Initial water flux
J_{w1}	-	Final water flux after cake layer removal
J_{w2}	-	Final water flux before cake layer removal
J_s	-	Microalgae permeation and steady state
J_o	-	Clean membrane initial flux
J_1	-	Flux of membrane before cleaning
J_2	-	Flux of membrane after cleaning
kDa	-	Kilo Dalton
Kg	-	Kilogram
kg/m^2hr	-	Permeation rate
L/m^2h	-	Liter meter square per hour
Mg	-	Magnesium
m/s	-	Meter per second
Na	-	Sodium
O_p	-	Optical density permeate
O_f	-	Optical density feed
R_T	-	Total membrane resistance
R_{cp}	-	Concentration polarization resistance
Rz	-	Height of the surface
Rq	-	Root mean square roughness
Rpv	-	Maximum depth of valleys

t - Time

V - Volume

V_f - Volume of feed

V_c - Volume of concentrate/ retentate

η - Viscosity

ΔP - Different of pressure

CHAPTER 1

INTRODUCTION

1.0 Overview

Membrane filtration has emerged as a promising tool for many separation processes. This is because it is easy in operation, requires only low operating pressure and temperature and does not require any chemical addition. The concept of membrane filtration is molecular sieving through membrane pores which can be divided into nanofiltration, reverse osmosis, microfiltration (MF) and ultrafiltration (UF). A membrane itself is a thin permeable or semipermeable layer that only allows specific molecules to pass through it according to it pore sizes. A desirable membrane filtration process is one with high selectivity and flux and possesses good antifouling properties. The efficiency of membranes is always determined based on their flux, the percentage of rejection, concentration factor and volume reduction factor.

A membrane can be prepared either from polymers, ceramic or metal. Basically, ceramic membranes have better permeability and rejection (Lee and Cho, 2004). Ceramic membranes such as titanium dioxide (TiO_2), zirconium dioxide (ZrO_2), aluminium oxide (Al_2O_3) and silicon dioxide (SiO_2) possess higher fluxes and lower fouling (Hoffs *et al.*, 2011). However, filtration using ceramic membranes is rarely applied in water filtration compared to polymeric membrane. This is because ceramic membranes require high initial installation and production cost compared to polymeric membrane besides they are brittle that they need to be handled carefully.

In order to meet the economic feasibility, the price of ceramic membrane module at least need to be less than 4.25 times of polymeric membrane's price (Park *et al*, 2014). At present, the excellent characteristic of polymer membranes made the polymer as the biggest competitor to the ceramic membrane. Various polymers have been used to make membranes including polyvinylidene fluoride (PVDF), polyethersulfone (PES), and cellulose acetate (CA). The PES membrane main attraction is its properties in chemical stabilities, mechanical strength and membrane-forming properties. Pristine PES membrane is hydrophobic and has problem in permeation and fouling when is used (Susanto and Ulbricht, 2009). Thus additives are always being introduced to the PES polymer to fabricate a membrane that is hydrophilic.

Normally, non-solvent hydrophilic polymer additives such as polyvinyl polyvinylpyrrolidone (PVP) and polyethylene glycol (PEG) are added in PES casting solution to increase hydrophilicity. They also acted as pore-former agents to the PES membrane and help to create spongelike or fingerlike structures in the membrane sub-layer. Polyvinyl alcohol (PVA) is also another common additives added in PES membrane fabrication due to its excellent hydrophilicity. Since PVA is unstable in organic solvent, it was usually grafted and cross-linked on the PES membrane surface (Liu, Kim and Kim, 2008; Guo *et al*., 2008). The additives give the advantage to the PES membrane in terms of high flux rate.

However, the non-solvent additives are soluble in water and there are possibilities for the additives to leach out during immersion in the coagulation bath and resulted in adverse effects on membrane structure and hydrophilicity (Ahmad *et al*., 2013). In order to overcome the mentioned issues, inorganic salts additives such as lithium bromide (LiBr) and lithium chloride (LiCl) were introduced in polymer casting. The significant effect of inorganic additives in polymer membrane is not only on improvement of membrane hydrophilicity but in membrane rejection rate. PES membrane with inorganic salt additive gives greater association with PES moieties and reduction in polymer chain mobility.

These additives make slow polymer precipitation due to weak non-solvent/solvent exchange and lead to a dense membrane with small pore size (Idris, Ahmed and Limin, 2010). The smaller the pore size, the greater the selectivity of the membrane which means the membrane molecular cut-off is improved and high rejection achieved. Nowadays, blending of PES with inorganic nanoparticles has attracted research interests because of the promising membrane results. The emerging technologies in nanoparticles industry have produced variety of nanoparticles such as alumina oxide (Al_2O_3), zinc oxide (ZnO), titanium oxide (TiO_2) and carbon nanotubes (CNT) nanoparticles.

These nanoparticles have been used in PES membrane making as additives to enhance membrane performances and anti-protein fouling. Nanoparticles additives have contributed in the membrane structure change to a fingerlike structure in membrane sub-layer which mainly promotes high permeation rate of the membrane (Sotto *et al*., 2011). Membrane filtration is among one of the many techniques used for microalgae harvesting. It is able to harvest 99-100% (harvesting efficiency) of microalgae as depicted in Table 1.1 and only UF and MF processes are involved in microalgae harvesting.

Table 1.1 Performance of UF/MF membranes for microalgae harvesting

Author	**Filtration Type**	**Velocity (m/s)**	**TMP (bar)**	**Filtration Flux (L/m^2h)**	**Harvesting Efficiency**
Castaing *et al*., 2010	MF	N/A	0.3	29	99%
Castaing *et al*., 2011	MF	N/A	0.3	108	99%
Frappart *et al*., 2011	UF	1	1	>100	100%
Bilad *et al*., 2013	MF	N/A	N/A	>50	100%
Hwang *et al*., 2015	UF	1	2-3	96	100%
Hwang and Wu, 2015	MF	1	2	≈ 105	100%

Note: N/A= information not available

The application of membrane filtration in microalgae harvesting requires the maintenance of a pressure drop across the system to force fluid flow through a membrane. During the process, microalgae will deposit on the membrane and can grow thicker throughout the process which later cause a pressure drop across the membrane and decrease filtration flux (Barros *et al.*, 2015). This phenomenon is known as fouling. One of the advantages when using UF and MF techniques is the low required transmembrane pressure (TMP) and velocity that can reduce fouling propensity. This is because the high velocity and TMP that is attained via pumping through a highly restrictive valve can induce high shear on microalgae.

Shear is responsible for broken cells and release of microalgae products. The sheared algae can cause more drastic fouling than non-sheared microalgae (Ladner, Vardon and Clark, 2010). Membrane filtration for microalgae harvesting is still in its infant stage, unlike centrifugation that has been the most common technique for microalgae harvesting. Centrifugation is used in lab and pilot scale production. Centrifugation applies high rotational and shear forces to separate microalgae but consumes huge amounts of energy if it is being used for vast production. Normally, centrifuge is adjusted to maximize capture efficiency where the energy is consumed. According to Barros *et al.* (2015), high solid capture of 94% consumed 20 kWh of energy and 17% of solid capture consumed only 0.80 kWh but obviously is less efficient.

Meanwhile, belt filter system can be used for up-scale harvesting. A belt-filter system separation is based on gravity drainage followed by compression of filtered material. However, belt filter system is only suitable for high concentration algae culture. A study revealed that a belt filter system can recover microalgae suspension with minimum concentration is 6 g dry wt/L because when 4 g dry wt/L of microalgal suspension was used, the percent of microalgae recovered dropped significantly due to leakage in the filter section (Sandip, Smith and Faddis, 2015). Microalgae are eukaryotic unicellular organisms that can be found in saline or freshwater bodies.

There are numerous microalgae species around the world but only a handful such as *Scenedesmus*, *Chlorella*, *Heamatocuccus* and *Nannochloropis* algae are known for their useful products. *Nannochloropsis* is known as one of the source of biodiesel because of its high percentage of triglyceride yield in relation to overall lipid content (Brennan and Owende, 2010). Previously, animal fats and vegetable oils are used for biodiesel production but they are not practical due to food competitor issues and large area requirement. Microalgae such as *Dunaliella* sp., *Chlorella* sp., and *Scenedesmus* sp., contain various pigments molecules like β-carotene, chlorophyll and carotenoids that have been used as colorants in cosmetic and food (Shah *et al*., 2016) for a long time.

Meanwhile, *Haematococcus pluvialis* contains astaxanthin. Astaxanthin is used in cosmetics products, food supplements and pharmaceutical industries because of its free radical scavenging capacity and powerful antioxidant activity (Khanra *et al*., 2018). Since microalgae holds economic value in various applications, they are sometimes cultivated indoor. Harvesting of cultivated microalgae is necessary so as to obtain their biomass before further processing them into valuable products.

1.2 Problem Statements

Filtration has been found satisfactory at recovering many type of microalgae cell. However its performance has been hampered by rapid bio-fouling (Zhang and Fu, 2018). The main foulants can be classified as algae cell, algae debris and extracellular polymeric substances (EPS). Fouling in microalgae filtration is mainly due to the formation of a cake layer of algae cell on the membrane surface (Marbelia *et al*., 2016). The EPS which is usually in soluble form and loosely bound will tightly bind with the algae cell and become part of the bio-fouling layer (Chang, Lee and Lee, 2019). The presence of EPS is always associated with slimy features due to the algae biofilm.

In nature, the biofilms are the main form of microbial life and they are important to ensure the algae survive in a hostile, nutrient-limited and rough aqueous environment (Upadhyayula and Gadhamshetty, 2010). After fouling of algae membrane cleaning is required in order to provide the membrane with adequate flux and separation. Generally, membrane cleaning can be divided into two types; physical and chemical cleaning. Physical cleaning includes backwash, forward flushing, back-flushing and back-pulsing which imposes shear forces on the membrane surface to loosen and dislodge the foulant. Generally, most membrane systems have backpulse/backwash device to minimize fouling by cleaning the membrane intermittent in between filtration period and rest period.

Especially in cross-flow filtrations where fast fouling can be observed thus periodic washing is a must. However, the adverse effect form frequent cleaning is less working time which lowered the filtration efficiency (Bhave *et al*., 2012;Chen *et al*., 2012). If the backwash frequency is too low, resistance due to fouling can become higher, which can also result in a low flux (Kwon *et al*., 2014). Furthermore, some membrane cannot withstand backwashing especially the flat panel membrane (Baerdemaeker *et al.*, 2013). Chemical cleaning method has been the popular method to remove algae foulant in many studies (Zhang *et al*., 2010; Ríos *et al*., 2012; Monte *et al*., 2018; Gerardo *et al*., 2015; Bilad *et al*., 2012).

Normally, sodium hypochlorite (NaClO) and citric acid in various concentrations were normally pumped into the membrane module. After each cleaning, membranes were flushed with water. However, this method can shorten the life-span of the membrane itself. Thus regular membrane cleaning may not be the first option. Since 2010, a significant focus on application of auxiliary in membrane configuration for combating fouling has been found. The strategy to reduce fouling has focused on increasing the feed flow by installing vibrator, rotating disk, stirrer and blower. All of the added equipment is about configuration improvement and this means that less attention was given on membrane modification.

Additional, a review study by Liao *et al*. (2018) suggests that very few studies have been accomplished to optimize the properties of membrane for microalgae harvesting. Castaing *et al*. (2010) applied hollow fiber submerged filtration system with aeration effect for *Heterocapsa triquetra* harvesting. A blower was set at the bottom of the hollow fiber membrane to generate bubbles and it was found to slow down the fouling occurrence. Critical flux achieved for the harvesting was 29 L/m^{2}h after 180 min of filtration under 0.3 bar TMP. Frappart *et al.* (2011) applied a rotating disk in cross-flow membrane to create dynamic movement during filtration and found that the permeation flux increased by two folds. However broken cells were observed recirculating through the throttling valve.

Bilad *et al*. (2013) performed a flat sheet submerged filtration for harvesting *Phaeodactylum tricornutum* and *Chlorella vulgaris*. The filtration system was equipped with vibrator machine. The vibrations generated from the vibrator machine were from magnetic repulsion. The vibration was only subjected to the area of the membrane. The critical flux achieved was slightly higher than achieved by Castaing *et al*. (2010) which were above 50 L/m^{2}h. Thus, the vibrated system is better than aerated system. Nurra *et al*. (2014) had performed microalgae harvesting using vibrated filtration system in pilot scale. A pilot plant with six photo bioreactors with a total capacity of 53,000 L are developed for cultivation, harvesting, cell disruption and lipid extraction.

The harvesting process is performed using a membrane vibrating set-up from New Logic Research Inc., model VSEP Series LP. Results from membrane filtration achieved microalgae filtration at 28.5 L/m^2/h/bar using a PES with a molecular weight cut-off of 7000 Da. To support microalgae biomass demand, centrifugation was used in parallel with membrane filtration to harvest microalgae. In the centrifugation process a total of 28,100 L was treated in 11 batches. Each batch duration was 3 hours approximately at a recirculating flow rate of 1000 L/h. The total concentrated volume obtained was 20.3 L and the total dry biomass obtained was 2.64 kg.

Kim *et al.* (2014) used cross-flow electro-filtration system as a step to anti-fouling harvesting. A platinum plate has been placed on the opposite side of the electro-membrane with 5 mm distance to cause water electrolysis during filtration and served as the counter anode. The electro-membrane used caused electrical repulsion between the membrane surface and microalgae cell and fouling decreased which was indicated by the high concentration factor achieved. Kim *et al.* (2015) and Hwang and Wu (2015) performed microalgae harvesting using a cross-flow cell equipped with rotating disk. The rotation from the rotating disk can generate sheer stress on membrane surface to mitigate the algae fouling. However, these systems are expensive due to current energy and limiting space in the rotating disk system.

Recently, Ye *et al.* (2018) combined the use of stirrer with forward osmosis (FO) type of filtration. The result achieved was not impressive as Bilad *et al.* (2013) with 23.3 L/m^{2}h. However, the membrane fouling was reversible by simple hydraulic flushing which made the pure water flux remained more than 97% of original pure water flux. Amazing impact of vibration on membrane filtration was also recorded in a recent study by Zhao *et al.* (2018). A uniform shearing vibration was applied by using a simple shaker and it is not same as other vibration machines with the variable shear rate. The purpose was to produce more stable shear action on the membrane. The membrane fouling had a remarkable decline only with little power increment (2 Hz) and at a low frequency of 5 Hz.

Furthermore, according to Kim *et al.* (2019) rotating disks, vibration, and bubbling was not appropriate for hollow fiber membrane. Thus, Kim *et al.* (2019) introduced the use of a turbulent jet. The turbulent jet module has its own design with a perforated cylinder at the center of the module to create turbulent jets. The perforated cylinder consists of 40 holes and the diameter and length is 0.3 mm and 200 mm, respectively. Each hole acts as an inlet port located very close to the membrane. The turbulent jet worked by generating a locally high velocity and shear stress near the membrane. The turbulent jets directly impinging on the membrane surface in the radial directions while removing foulant.

Hence, a study on development of membrane with good properties to combat algae fouling is an opportunity. Drexler and Yeh (2014) mentioned that continuous research in polymer science or interfacial phenomena would help develop membrane that are better able to resist fouling. Previously, Hwang *et al.* (2015) studies the effects of hydrophilic additives Pluronic F-127 on PVDF membrane and found 100% of algae retention with permeation flux of 96 L/m^2h that was larger by approximately 50% than a commercial hydrophilic membrane. Thus, the purpose of this research work is to contribute in the development of a new membrane with a combination of additives which able to reduce fouling issue in membrane filtration for microalgae harvesting.

PES is the chosen base polymer since it has high chemical and thermal stability. It is known for its good membrane forming properties that makes it one of the most popular polymers in producing membrane for water filtration application. Besides that, it is also one of membrane material that is commonly used in protein separation (Celik *et al*., 2011). In this study, the PES will be blended with functionalized multiwall carbon nanotube (MWCNT) and lithium salts. MWCNT is a unique nanoparticle due to its electrical properties that makes it different from other nanoparticle (Bonard *et al*., 2002). The electrical property is mainly due to the extra negative electron charge it consists which can benefit to polymer.

The lithium salts consist of lithium bromide (LiBr) and lithium chloride (LiCl) will be combined together with MWCNT to study their hybrid effects. The hybrid effect of lithium salts and MWCNT is the novel part in this research. The lithium salts alone has been known to increase the performance of membranes pure water permeation rate and rejection rate (Idris *et al*., 2010).

1.3 Objectives of the Study

The main objective of the study is to develop a high-performance membrane with anti-fouling effect for microalgae harvesting by using polyethersulfone (PES) as the base polymer and functionalized multiwall carbon nanotubes (MWCNT) and lithium salts as additives. PES/MWCNT is the control membrane. In order to achieve the main objective, the following objectives need to be addressed;

1) To synthesize membrane with different concentrations of functionalized MWCNT and different lithium salts; LiBr and LiCl in PES polymer using two different phase inversion techniques; thermally induce phase separation (TIPS) and non-solvent induced phase separation (NIPS).

2) To evaluate the hybrid effect of functionalized MWCNT and lithium salts on PES membrane performance in terms of flux, molecular weight cut-off (MWCO) and rejection rate.

3) To use the membrane for harvesting *Nannochloropsis* sp. and determine the extent of bio-fouling of the fabricated membranes.

1.4 Scope of the Study

The scope of the study mainly focuses on the development of anti-fouling PES membrane to be used in microalgae harvesting.

I. Preparation of various PES membranes with two different additive of lithium salts (LiCl and LiBr) and MWCNT varied from 1-5wt% via blending and phase inversion techniques. Pristine membrane PES/MWCNT was also be prepared as the benchmark.

II. Two types of phase inversion were used to form the membranes; non-solvent induced phase separation (NIPS) and thermally induced phase separation (TIPS).

III. The fourier transform infrared spectroscopy (FTIR) and field emission scanning electron microscopy (FESEM) were used to characterize the fabricated membranes.

IV. The surface roughness and hydrophilic property of the membranes were determined using atomic force microscope (AFM) and contact angle measurements respectively.

V. The performance of fabricated membranes was initially evaluated in terms of MWCO, rejection rate, pure water and fluxes because these are the important parameters deciding the separation performance and was compared with the control membrane.

VI. The membranes performances were then evaluated for microalgae harvesting and then anti-fouling properties were then evaluated.

VII. The fouling propensity of the fabricated membrane were determined in term of reversible fouling (F_r) and irreversible fouling (F_{ir}). Only membrane that shows better anti-fouling was evaluated for harvesting efficiency. The microalgae harvesting efficiency was determined using volume reduction factor (VRF) and concentration factor (CF).

VIII. Microalgae genus *Nannochloropsis* that has been receiving much research interest due to its ability to synthesize lipids for biodiesel production has been used as algae model.

1.5 Significance of the Study

The work herein intends to demonstrate the synergistic effect of combining MWCNT with LiBr/LiCl as additives to improve the membrane property and at the same time prevent fouling. This antifouling behavior developed was demonstrated by its ability to harvest microalgae successfully and at the same time be reused again without losing its initial properties. The novel PES membrane formulated with both LiBr and functionalized MWCNT not only producing membrane with excellent anti-fouling behavior but also possess high permeation rate and durability.

The TIPS method used was able to produce membrane with excellent hydrophilic characteristic with zero irreversible fouling ratio that translates to 100% flux recovery. Thus, this study able to demonstrates novel membrane fabrication for better anti-fouling property for microalgae harvesting.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Khairuddin, N.F.M.**, Idris, A. & Hock, L.W. (2019). Harvesting *Nannochloropsis sp*. using PES/MWCNT/LiBr membrane with good antifouling properties. *Separation and Purification Technology*, 212, 1–11. https://doi.org/10.1016/j.seppur.2018.11.013. **(Q1, IF: 3.927)**

Indexed Journal

1. **Khairuddin, N.F.M.**, Idris, A., Irfan, M. & Loong, T. C. (2017). Microalgae harvesting of *Nannochloropsis sp.* using polyethersulphone/lithium chloride/functionalised multiwall carbon nanotube membranes fabricated via temperature induced phase inversion and non-solvent induced phase inversion. *International Journal of Nanoparticles*, *9*, 71-87, https://doi.org/10.1504/IJNP.2017.086133. **(Indexed by SCOPUS)**

2. **Khairuddin, N.F.M,** Idris, A., Ahmed, I., & Mohd Yusof, N. (2014). Influence of multiwall carbon nanotube on polyethersulfone/ polyvinyl alcohol blend membranes. *Jurnal Teknologi*, *67*, 31-35, https://doi.org/10.11113/jt.v67.2730 **(Indexed by SCOPUS)**