POLYIMIDE BASED MIXED MATRIX NANOFILTRATION MEMBRANE FOR REFINING PALM OIL

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy

Malaysia-Japan International Institute of Technology
Universiti Teknologi Malaysia

APRIL 2022

DEDICATION

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

ACKNOWLEDGEMENT

In preparing this thesis, I was in contact with many people, researchers, academicians, and practitioners. They have contributed towards my understanding and thoughts. In particular, I wish to express my sincere appreciation to my main thesis supervisor, Dr. Mohd Nazlee Faisal Md Ghazali, for encouragement, guidance, critics and friendship. I am also very thankful to my co-supervisor Dr. Tan Lian See and Dr. Mariam Firdhaus Mad Nordin for their guidance, advices and motivation. Without their continued support and interest, this thesis would not have been the same as presented here.

I am also indebted to Universiti Teknologi Malaysia (UTM) for funding my Ph.D study. Laboratory technicians in UTM also deserve a special thanks for their assistance during various unforeseen events in the laboratory.

My fellow postgraduate student should also be recognised for their support. My sincere appreciation also extends to all my colleagues and others who have provided assistance at various occasions. Their views and tips are useful indeed. Unfortunately, it is not possible to list all of them in this limited space. I am grateful to all my family member.

ABSTRACT

The purification of vegetable oil is an important process to obtain purified vegetable oil for various applications. However, conventional processes in vegetable oil purification, such as deacidification, require a huge amount of energy which is not only costly, but also contributes to a high rate of carbon footprints. To improve the current state of purification, the capability of membrane technology in purifying vegetable oil was investigated. The main objective of this study is to investigate the potential of polyimide-based mixed matrix membrane (MMM) in refining palm oil by nanofiltration. To achieve the objective, preliminary investigations on the performance of commercially available membranes were made. Subsequently, polyimide-based MMM with different additives loadings were fabricated and characterized. Additionally, membrane transport models were used to describe and predict the membrane separation process and a suitable multistage configuration was also proposed. The structural and physical characteristics of the fabricated membranes were studied. The separation performances of the fabricated membranes were investigated by using a dead-end stirred cell and ethyl acetate as the diluting solvent for palm oil. From the membrane characterization, it was found that MMM with 0.5wt% of β-cyclodextrin functionalized multi-walled carbon nanotubes (βCD-fMWCNT) achieves the highest separation of palmitic acid from the feed diluted palm oil at 3.84 LMH/bar. The rejection of palmitic acid was found to be 60% and tocopherol, carotene and triglyceride at 94.43%, 98.74%, and 95.18% respectively. The membrane separation process was found to be best described by using the Solution-Diffusion model. Additionally, a theoretical study by using different multistage configurations found that the separation process can be further improved. The proposed multistage configuration was able to yield triglyceride with 99.28% purity with only 9.8% of oil loss in the purified permeate stream with a 41% of solvent recovery rate. Therefore, from this study, it is proven that membrane separation technology is a promising purification alternative and mixed matrix polyimide membrane has the potential in improving the conventional palm oil purification process.

ABSTRAK

Penulenan minyak sayuran adalah proses yang penting untuk mendapatkan minyak sayuran yang tulen untuk pelbagai aplikasi. Walau bagaimanapun, proses deasidifikasi minyak konvensional, memerlukan jumlah tenaga yang tinggi yang bukan sahaja mahal, malahan menyumbang kepada jejak karbon dalam kadar yang tinggi. Untuk menambahbaik proses penulenan pada masa kini, keupayaan teknologi membran dalam pemprosesan minyak sayuran telah dikaji. Objektif utama penyelidikan ini adalah untuk menyiasat potensi membran matriks campuran (MMM) berasaskan poliimid dalam penulenan minyak sawit. Untuk mencapai objektif utama, siasatan awal dengan menggunakan membran komersial telah dijalankan. Seterusnya, penghasilan and pencirian MMM berasaskan poliimid turut dilakukan. Pemerihalan proses pengasingan menggunakan membran telah dijalankan melalui model pengangkutan membran (MPM). Malahan, konfigurasi untuk nanofiltrasi secara bertingkat yang bersesuaian juga telah dicadangkan dalam kajian ini. Ciri-ciri struktur dan fizikal membran yang dibuat telah dikaji. Prestasi proses pengasingan melalui MMM telah dijalankan dengan menggunakan sel pengaduk hujung mati dan etil asetat sebagai bahan pelarut minyak sawit. Dari pencirian membran, adalah didapati bahawa MMM dengan 0.5% gabungan nanotiub karbon dinding-berganda berfungsikan beta siklo dekstrin (βCD-fMWCNT) mencapai kadar pengasingan asid palmitik yang tertinggi dari minyak sawit dalam kadar 3.84LMH/bar. Kajian ini juga mendapati bahawa penyingkiran asid palmitik, tokoferol, karotena, dan trigliserida adalah masing-masing pada kadar 60%, 94.43%, 98.74% dan 95.18%. Tambahan pula, dari segi perbandingan MPM, adalah didapati bahawa model resapan-larutan adalah model yang paling sesuai dalam perihalan proses nanofiltrasi minyak sawit dalam larutan etil asetat. Selain itu, dengan menggunakan nanofiltrasi secara bertingkat, pengumpulan semula bahan pelarut pada kadar 41% dan peningkatan ketulenan minyak sawit tulen (99.28%) dapat dicapai. Dari kajian ini, adalah terbukti bahawa teknologi membran adalah sesuai sebagai teknologi alternatif untuk menggantikan proses penulenan minyak sayuran yang konvensional.

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LIST OF ABBREVIATIONS

ACM	-	Aspen Custom Modeler
AFM	-	Atomic Force Microscopy
ANN	-	Artificial Neural Network
ATR	-	Attenuated Total Reflectance
βCD	-	Beta-Cyclodextrin
CA	-	Cellulose Acetate
CFSD	-	Combined Film Theory/ Solution Diffusion
DG	-	Diglyceride
EOS	-	Equation of State
FESEM	-	Field Emission Scanning Electron Microscopy
FFA	-	Free Fatty Acid
fMWCNT	-	Functionalized Multi-Walled Carbon Nanotubes
FTIR	-	Fourier-Transform Infrared Spectroscopy
GLCF	-	Group Contribution Lattice Fluid
HPLC	-	High-Pressure Liquid Chromatography
MB	-	Methylene Blue
MF	-	Microfiltration
MG	-	Monoglyceride
MWCNT	-	Multi-Walled Carbon Nanotubes
NF	-	Nanofiltration
OSN	-	Organic Solvent Nanofiltration
PA	-	Palmitic Acid
PDMS	-	Polydimethylsiloxane
PEEK	-	Polyether Ether Ketone
PEG	-	Polyethyleneglycol
PES	-	Polyethersulfone
PI	-	Polyimide
PI1	-	Polyimide P84 Structure 1
PI2	-	Polyimide P84 Structure 2
PI3	-	Polyimide P84 Structure 3

PS	-	Polysulfone
PVA	-	Polyvinyl Alcohol
PVDF	-	Polyvinylidene Difluoride
RB	-	Rose Bengal
RO	-	Reverse Osmosis
SFM	-	Selectivity Figure of Merit
Si	-	Silicon
SRNF	-	Solvent Resistant Nanofiltration
SWCNT	-	Single-Walled Carbon Nanotubes
TG	-	Triglyceride
TGA	-	Thermogravimetric Analysis

LIST OF SYMBOLS

δ_d	-	Energy from dispersion forces between molecules [$MPa^{1/2}$]
δ_p	-	Energy from the dipolar intermolecular force between molecules [$MPa^{1/2}$]
δ_h	-	Energy from hydrogen bonds between molecules [$MPa^{1/2}$]
ΔV	-	Permeate Volume [L]
Δt	-	Time Duration [h]
A	-	Surface Area of Membrane [m^2]
$\%R$	-	Percentage of Rejection [%]
Re	-	Reynold Number [-]
C_P	-	Permeate Concentration [mg/L]
C_R	-	Retentate Concentration [mg/L]
R_0	-	Observed Rejection [-]
k	-	Mass Transfer Coefficient [m/s]
J_V	-	Solvent Flux [$Lm^{-2}h^{-1}$]
J_S	-	Solute Flux [$Lm^{-2}h^{-1}$]
$\frac{D_{AM}K}{\delta}$	-	Solute Transport Parameter [m^2/s]
σ	-	Reflection Coefficient [-]
P_M	-	Permeability Coefficient [m/s]
D_i	-	Diffusion Coefficient [cm^2/s]
l	-	Thickness of Membrane [µm]
p_o	-	Pressure at the Retentate/Feed Side [bar]
p_l	-	Pressure at the Permeate Side [bar]
v_i	-	Molar Volume of Species *i* [cm^3/mol]
c_o	-	Concentration at the Retentate Side [mg/L]
c_l	-	Concentration at the Permeate Side [mg/L]
d_i	-	Effective Diameter of Solute *i* [m]
k_B	-	Boltzmann's Constant [-]

μ_s	-	Dynamic Viscosity of Solvent [cP]
ε	-	Membrane Porosity [nm]
Q	-	The volume of Pure Water Permeated Per Time [L]
η	-	The viscosity of Pure Water [cP]
Φ_i	-	Molecular Volume Fraction of Species *i* [-]
θ_i'	-	Molecular Surface Area Fraction of Species *i* [-]
r_i	-	Van der Waals Volume of Species *i* [-]
q_i	-	Van der Waals Surface Area of Species *i* [-]
x_i	-	Mole Fraction of Species *i* [-]
v^i_k	-	Number of Group *k* in Species *i* [-]
Γ_k	-	Residual Activity Coefficient in Group *k* in a mixture [-]
Γ^i_k	-	Residual Activity Coefficient in Group *k* in a Pure Solution of Species *i* [-]
Ψ_{nm}	-	Interaction Parameter of Structural Group *m* and *n* [-]
Φ_i	-	Volume Fraction of Solvent Penetrant *i* [-]
χ	-	Membrane Solvent Penetrant Interaction Parameter [-]
ρ_m	-	The density of polymer *m* [g/cm^3]
Pe	-	Peclet number [-]

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CHAPTER 1

INTRODUCTION

1.1 Background of Research

Vegetable oils such as oil palm, soybean, rapeseed, and sunflower oil have been studied widely for their production processes since the early years of this century due to their wide availability and uses. The processed oil from plants can be used in fields such as in food products, supplement and nutrition products, beauty products, and most recently biofuels (Pal and Pratap, 2017; Panchal *et al.*, 2017). Vegetable oil processing involves the removal of undesirable components such as phospholipids, free fatty acids (FFA), sterols, trace metals, and oxidation products from edible oil which affects the taste and texture of the vegetable oil. The main stages in conventional vegetable oil processing are solvent extraction, evaporation, degumming, deacidification, bleaching, dewaxing, and deodorization (Gupta, 2008). Although these stages have been used widely in the industry, it possesses some major drawbacks which can be improved by current technology (de Morais Coutinho *et al.*, 2009; Vaisali *et al.*, 2015). Some of the highlighted drawbacks are high energy usage, oil losses, and contaminated effluents produced during the processes (de Morais Coutinho *et al.*, 2009; Vaisali *et al.*, 2015). As Malaysia and Indonesia primarily produce palm oil with approximately 84% of the world's production, the technological enhancement in the palm oil production industry would potentially bring about a significant cost-saving and improved energy-efficiency in obtaining refined oil which is aligned with the current efforts of reducing carbon footprints.

Membrane technology is one of the promising alternatives for the industrial processing of vegetable oil (as can be seen in Figure 1.1) and it has been studied extensively by researchers on a lab-scale to industrial scale (Vaisali *et al.*, 2015). In the field of vegetable oil processing, this technology is capable of removing undesirable products and retrieve valuable components from crude vegetable oil

(Marchetti *et al.*, 2014; Priske *et al.*, 2016). Besides that, the solvent used during the filtration process can also be recovered. Moreover, the membrane technology is relatively energy efficient as compared to conventional approaches such as distillation. Various studies on membrane-aided vegetable oil purification have been published by researchers in the recent years (Azmi *et al.*, 2015; Firman *et al.*, 2017a; Shi *et al.*, 2019; Werth *et al.*, 2017a). From their studies, it was found that vegetable oil needs to be diluted to allow a better permeation flux and separation. As organic solvent can dissolve polymers through solvent diffusion and chain disentanglement, membrane polymer with high solvent-resistance properties is usually used in the study (Gugliuzza, 2015; Lim *et al.*, 2017). Solvent-resistant polymers such as polybenzimidazole (PBI), polyimide (PI), and poly (ether ether) ketone (PEEK) were some of the polymers used for the fabrication of membrane (Galizia and Bye, 2018). The use of solvent-resistant membranes was found to be successful in rejecting triglycerides at high rate of rejection with acceptable solvent permeation fluxes.

However, research on the separation of free fatty acids and nutritional compounds from vegetable oil are still on-going due to the difficulty of separating compounds of similar sizes with acceptable solvent permeability (Vaisali *et al.*, 2015). In the study by Shi *et al.* (2019), they had performed the separation of linoleic acid from glyceryl trilinoleate by using commercial solvent-resistant membranes. It was found the DuraMem 500 was able to reject glyceryl trilinoleate and linoleic acid at around 86% and 35% respectively at acetone permeation of 1.02 LMH/bar. In another study, Werth *et al.* (2017a) have investigated the separation between rapeseed oil and oleic acid by using PuraMem 280 membrane. They have discovered that the membrane was able to reject 97% and 34.7% of rapeseed oil and oleic acid respectively at 0.1135 kg $m^{-2}h^{-1}bar^{-1}$ of ethanol permeation. From the investigation by the researchers, it is proven that polymeric membrane is capable of separating compounds in vegetable oil. Nevertheless, more investigations are needed to improve the permeation flux of the solvent without compensating the membrane selectivity and rejection performances.

In recent decades, nanoparticles have been employed in different fields of industry such as paint, surface coating, and polymer products (Stark *et al.*, 2015). The nano-size particles were found to enhance the properties of the materials and products when a certain composition (of nanoparticles) was added to the base material (Nazari and Riahi, 2011; Stark *et al.*, 2015; Sun *et al.*, 2011). In the field of membrane technology, nanoparticles such as metal oxides, carbon molecular sieves, carbon nanotubes, and zeolites have been studied (Cheng *et al.*, 2018). In the research by Soroko and Livingston (2009), it was found that titanium dioxide was able to improve the structural stability of the membrane. In another study, carbon nanotubes were used as the filler which improves the permeation flux of the fabricated membrane (Farahani *et al.*, 2018). From these studies, the addition of nanoparticles was found to have positive effects on the performance of the fabricated membranes.

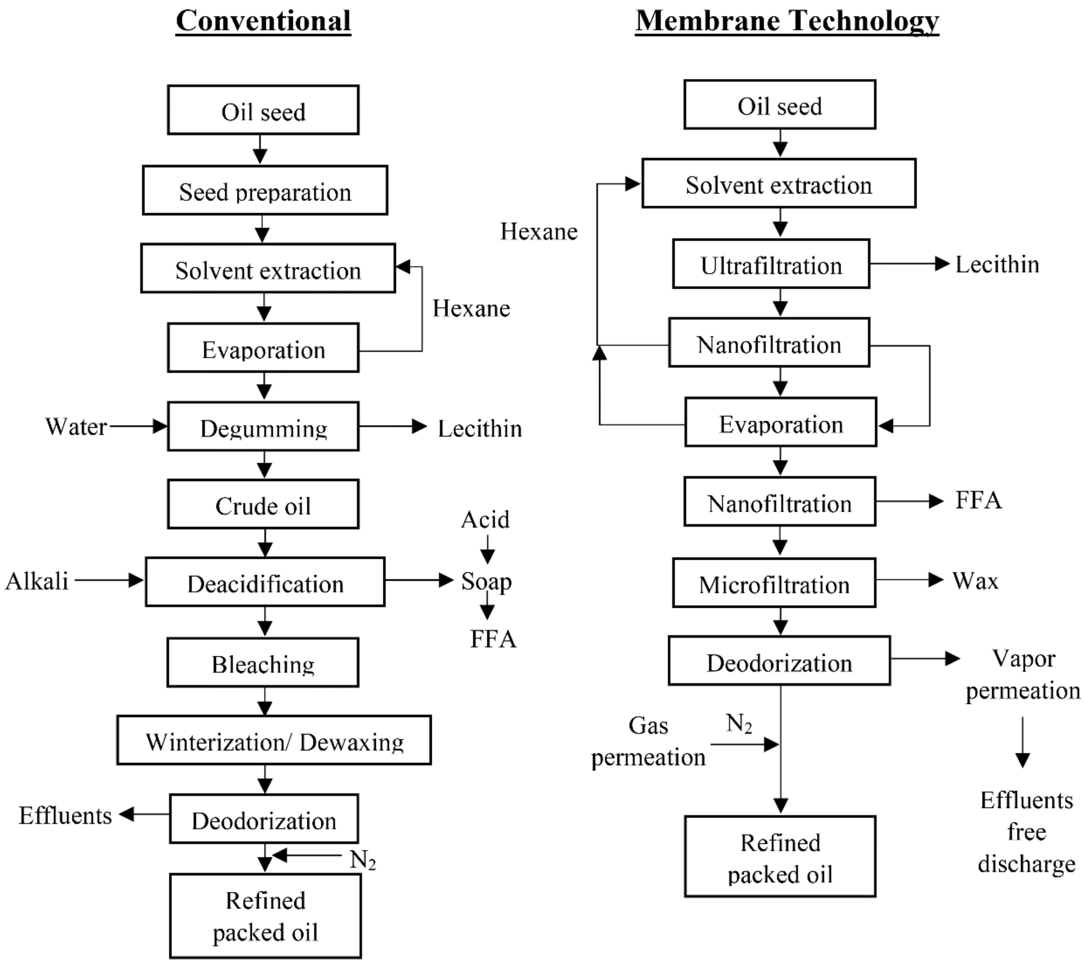

Figure 1.1 Comparison between conventional oil refining method and membrane refining method (Cheryan, 2005).

The prediction of membrane separation performance on the other hand can be performed through the use of membrane transport models as proposed by researchers

(Marchetti and Livingston, 2015). There are various membrane transport models available, such as the widely known solution-diffusion model, pore-flow model, and irreversible thermodynamic models. Through the correlation of experimental data to a certain membrane transport model, the mathematical description of a certain separation process can be obtained (Peshev and Livingston, 2013). The correlation of data to the model will require the estimation of model parameters which can be obtained through non-linear regression of data to the selected model. Besides that, the performance of the separation process can be further improved by employing multistage nanofiltration (Renouard *et al.*, 2018). By using a selected multistage configuration, the separation process could be more effective in separating solutes as well as enabling the recovery of solvents.

1.2 Problem Statement

The refining of vegetable oil is indeed important for the production of different consumable products in our daily life. However, the conventional refining process of vegetable oil particularly stages that involve high energy requirements such as steam distillation during the deacidification stage and winterization during the dewaxing process should be improved (Shi *et al.*, 2019; Werth *et al.*, 2017b). According to the researchers, the current refining process also resulted in the unnecessary loss of oil from the hydrolysis process, production of low-value soap stocks, and highly cost explosion-proof equipment (Vaisali *et al.*, 2015). Therefore, an alternative way to remove the undesirable compounds from the vegetable oil is desired. Membrane technology is a preferable way to separate the compounds as it has low energy requirement, the ability to recover compounds in their natural state, as well as the capability to recover solvents (Marchetti *et al.*, 2014). Although there are studies on the use of membranes to separate oil compounds, their results were either low in selectivity or low in permeation flux which makes them unsuitable for industrial applications (Shi and Chung, 2020; Werth *et al.*, 2017a). In the recent decades, there is an emerging technology known as nanoparticle technology which can improve the overall structure and performance of materials. There are already studies on the use of nanoparticles in the fabrication of membrane, which resulted in mixed matrix membrane (MMM) or thin-film nanocomposite (TFN). However, to our best of knowledge, there is a lack of study on the fabrication of MMM for the separation of vegetable oil (Abdellah *et al.*, 2019; Ali *et al.*, 2021; Shi and Chung, 2020). Therefore, through different formulations of polymer dope solution, the fabrication of suitable MMM for vegetable oil purification can be obtained in this study.

1.3 Research Objectives

Based on the aforementioned issues, the following objectives were constructed:

i) To formulate and fabricate integrally skinned asymmetric (ISA) membranes and mixed matrix membranes (MMM) with different additive loadings.

ii) To perform membrane characterization on fabricated membranes and membrane performance study.

iii) To predict the performance of selected fabricated membrane by using different membrane transport models.

iv) To theoretically evaluate the selected membrane performance using multi-stage nanofiltration configuration.

1.4 Research Scope

This study focuses on the separation of palmitic acid from synthetically prepared palm oil diluted in the organic solvent. Hence, the following scopes of the study were identified and listed as follows:

- Membrane performance studies of commercial solvent-resistant membranes by solute rejections, solvent permeation fluxes, and selectivity of membranes towards different solutes (palmitic acid, tocopherol, carotene, triglyceride) in the solvent-diluted (acetone, ethyl acetate, isopropanol) synthetically prepared palm oil.

- Membrane swelling studies of the commercial solvent-resistant membrane in different solvents (acetone, ethyl acetate, isopropanol).
- Solute-solvent-polymer interaction studies by using molecular modeling.
- Membrane formulation by using polyimide P84 at different solvent/cosolvent ratios.
- Membrane fabrication by using polyimide P84 as the polymer and beta-cyclodextrin (β-CD) and beta-cyclodextrin functionalized multi-walled carbon nanotubes (βCD-fMWCNT) as additives.
- Membrane performance studies of fabricated membranes by using solvent-diluted synthetically prepared palm oil at different additive loadings.
- Membrane characterization of fabricated membranes by using FESEM, AFM, FTIR, TGA, contact angle, pore size, wettability, and dye rejection.
- Membrane performance prediction by using different membrane transport models, where parameter estimation was performed by using MATLAB.
- Evaluation of different multistage nanofiltration configurations for improving membrane selectivity and solvent recovery.

1.5 Significance to knowledge/ Contribution

The technology for fractionating vegetable oil has been stagnant for the past 30-50 years due to the successful separation of the oil through physical and chemical refining methods (Vaisali *et al.*, 2015). However, in recent years, there is an increasing need to improve the conventional method due to its negative impact on the environment and equipment lifespan (Szekely and Zhao, 2022). The use of membrane separation technology was introduced in different industries that involve purification and separation. However, as of current, the vegetable oil refineries still rely on the conventional method due to the low permeation flux, low selectivity, and lack of studies on solvent-resistant membranes in fractionating vegetable oil components (Vaisali *et al.*, 2015). There were several studies on the use of a membrane in vegetable oil purification, but the results were either low in permeation flux or low in the selectivity of desired vegetable oil constituents (Shi and Chung, 2020; Shi *et al.*, 2019). This report is the first to describe the use of polyimide P84 nanofiltration membrane with βCD-fMWCNT additives for the purification of palm oil. The present study found that the use of polyimide P84 membrane constructed through the use of DMF and 1,4-Dioxane as solvent was able to separate palmitic acid from triglyceride at a high selectivity and permeation flux. The addition of βCD-fMWCNT additives at certain compositions also further improves the selectivity of palmitic acid/ triglyceride and permeation flux. Furthermore, it was found that the solution-diffusion model is the best to describe the nanofiltration of ethyl acetate-diluted palm oil. By using the data from the fabricated membrane, it was also found that the selectivity and solvent recovery of the nanofiltration process can be further improved through a proposed multistage configuration. From the findings, it is demonstrated that polyimide P84 mixed matrix membrane is a promising candidate for vegetable oil deacidification applications. This work contributes to the development of membrane technology in the vegetable oil processing field, especially in the deacidification process. This work particularly provides useful data and information for the implementation of mixed matrix membrane in deacidifying vegetable oil. Besides that, through the use of different membrane transport models and multistage, this work can contribute to the future development of artificial intelligence (AI) models, in which the results from the experiments and nonlinear regressions can be used as part of the database.

1.6 Thesis Outline

This thesis consists of 5 chapters. Chapter one explains the background, objectives, scope and problem statement of this research. Chapter two provides literature review related to this research, which also includes the mathematical equations which are useful in describing and comparing the different experimental results. Chapter three outlines the methodology of the research. The methodology describes all the materials, equipment, experimental procedures, as well as process flow or description in obtaining related experimental results. Chapter four of this thesis provides explanation and discussion on the results obtained from the experiments. Finally, chapter five concludes the research and also summarizes the important findings of the study. Chapter five also include recommendations which describes the future directions of this study.

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1. Lim, K. M., & Ghazali, N. F. (2021). Nanofiltration of binary palm oil/solvent mixtures: Experimental and modeling. Materials Today: Proceedings, 39, 1010-1014. (Indexed by WOS)
2. Lim, K. M., & Ghazali, N. F. (2020). Estimation of solute transport parameter and mass transfer coefficient in nanofiltration for solvent-diluted palm oil. In IOP Conference Series: Materials Science and Engineering (Vol. 736, No. 2, p. 022070). IOP Publishing. (Indexed by SCOPUS)

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Book Chapter

1. Ghazali, N. F., & Lim, K. M. (2022). Sustainable Separations using Organic Solvent Nanofiltration. Szekely, G., and Zhao, D., Sustainable Separation Engineering: Materials, Techniques and Process Development. John Wiley and Sons Ltd. Accepted for publication.