RECOVERY OF CAROTENOIDS FROM CRUDE PALM OIL USING ORGANIC SOLVENT NANOFILTRATION

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A thesis submitted in fulfilment of the requirements
for the award of the degree of Master of Engineering
(Bioprocess)

Faculty of Chemical Engineering
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JUNE 2014

To my beloved family and friends

ACKNOWLEDGEMENTS

First and foremost I would like to thank Allah S.W.T for giving me the strength, and courage to conduct this study. The knowledge I have gained throughout this work and all my life is all because of His blessing. Alhamdulillah.

I would like to express my deepest appreciation to my supervisor, Dr. Mohd Nazlee Faisal Ghazali for his guidance, advices, and dedication throughout the research. It was his kind support that helped me complete this study successfully. I am also grateful towards my colleague, Qadly Ameen Pahlawi for his unconditional support and assistance for me towards the success of this research. Also I am grateful to all the staff of Faculty of Chemical Engineering, Universiti Teknologi Malaysia (UTM) for their support that made this research a success.

Last but not least, I would like to thank my beloved family members for their love and moral support throughout every endeavor of mine.

ABSTRACT

The recovery of carotene from crude palm oil prior to refining would add significant value to the country's palm oil industry. Various methods are currently available to yield high concentrations of carotene. These methods however, have many disadvantages when factors such as cost, and energy consumption are weighed into consideration. This work presents the application of organic solvent nanofiltration membranes for the separation of carotene from a crude palm oil (CPO)/solvent system. Filtration tests were conducted using a dead-end filtration set-up, utilizing four different polyimide membranes and four different solvents. PuraMem™ 280 showed the best performance, with a selectivity of 1.25 when hexane was used as the solvent. Runs using DuraMem™ 150, DuraMem™ 300 and DuraMem™ 500 showed low or no selectivity between carotene and triglyceride in all solvents. It was found that rejection of carotene depends strongly on the type of solvents and membrane type and therefore is critical for carotene separation. Lower applied pressure and low feed concentrations improved the membrane selectivity. The effect of solute-solvent coupling was much higher towards lower molecular weight component (carotene) and at lower pressures. Theoretical pore size of the membrane was predicted using sets of equations relating the diffusivity of the solutes and the experimental rejection results. Results showed that the theoretical pore size of membrane PuraMem™ 280 was between 1.38 nm to 1.85 nm. The effect of concentration polarisation was predicted, and result showed that in this system, the effect of concentration polarization was very minimal, where c_w/c_b was only between 1.06 to 1.24. Generally, high feed concentrations increased the feed viscosity, resulting in significant osmotic pressures and reduces the permeate flux.

ABSTRAK

Pemisahan karotena daripada minyak sawit mentah sebelum proses penapisan memberi nilai tambah kepada industri minyak sawit negara. Pada masa ini, terdapat pelbagai kaedah yang berupaya menghasilkan karotena dalam kepekatan tinggi. Walau bagaimanapun, kaedah-kaedah tersebut mempunyai banyak kelemahan apabila faktor seperti kos dan penggunaan tenaga diambilkira. Kajian ini membentangkan aplikasi membran nanopenurasan pelarut organik untuk pemisahan karotena daripada sistem minyak sawit mentah/pelarut organik. Ujian penurasan dijalankan dengan susunan penurasan buntu, menggunakan empat membran poliimida dan empat pelarut berlainan. PuraMem™ 280 menunjukkan prestasi terbaik dengan nilai pemilihan 1.25 apabila digunakan bersama pelarut heksana. Membran DuraMem™ 150, DuraMem™ 300 and DuraMem™ 500 menunjukkan nilai pemilihan yang rendah antara karotena dan trigliserida dalam semua pelarut. Kajian mendapati, kadar penolakan karotena sangat bergantung kepada jenis pelarut dan jenis membran oleh itu sangat kritikal dalam pemisahan karotena. Tekanan yang rendah dan kepekatan suapan yang rendah meningkatkan pemilihan membran tersebut. Kesan gandingan antara bahan larut dan pelarut lebih tinggi terhadap komponen yang mempunyai berat molekul lebih ringan (karotena) dan pada tekanan yang lebih rendah. Saiz liang membran teori boleh diramalkan menggunakan beberapa set persamaan yang mengaitkan kebolehresapan bahan larut dengan penolakan yang didapati dari eksperimen. Keputusan menunjukkan bahawa saiz liang membran teori adalah di antara 1.38 nm hingga 1.85 nm. Kesan kekutuban kepekatan telah diramalkan, dan keputusan menunjukkan kesan tersebut adalah minimum, di mana c_w/c_b adalah antara 1.06 hingga 1.24. Secara umumnya, kepekatan suapan yang tinggi meningkatkan kelikatan, seterusnya meningkatkan tekanan osmotik, dan merendahkan fluks.

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LIST OF ABBREVIATIONS

CPO	-	Crude Palm Oil
MW	-	Molecular weight
MWCO	-	Molecular weight cut-off
TG	-	Triglycerides
MG	-	Monoglycerides
DG	-	Diglycerides
FFA	-	Free fatty acids
POME	-	Palm oil mill effluent
Da	-	Dalton
RSM	-	Response surface methodology
HPLC	-	High performance liquid chromatography
RO	-	Reverse osmosis
RPME	-	Red palm methyl esters
CPME	-	Continuous electrophoresis with porous membranes
PAN	-	Polyacrylonitrile
PI	-	Polyimide
PDMS	-	Polydimethysiloxane

CHAPTER 1

INTRODUCTION

1.1 Research Background

Palm oil is one of the world's richest natural plant sources of carotenoids. The minor components of crude palm oil (CPO) consist of carotenoids, tocopherols, tocotrienols, vitamin E and sterols. It contains high concentration of carotenes of about 0.5-0.7 g/L, 15 times more retinol equivalents (vitamin A) than carrots and 300 times more than tomatoes(Sundram *et al.*, 2003). The primary carotenes that exists in palm oil are α-carotene and β-carotene, which account for 80% of the total carotenes (Ooi *et al.*, 1994). The importance of β-carotene is well documented for human nutrition and serves as a major source of vitamin A, as they can be transformed into vitamin A *in vivo* (Chuang and Brunner, 2006) and serves as a potential alternative means of fighting vitamin A deficiency which is prevalent in many countries (Barison, 1996). Numerous studies have shown that carotenes can prevent certain types of cancer, such as lung, oral, pharyngeal and stomach cancers (Peto *et al.*, 1981). It could also enhance the immune system and help to protect against flu, colds, infections, and toxins. Furthermore, carotenoids are strong dyes, in which at even very low levels of parts per million, they are able to impart the desired properties to foods (Gordon and Bouernfeind, 1982). Due to broad application of natural compounds in cosmetics, pharmaceuticals and food industry, carotenoids have increased in its value and importance. Therefore, their recovery from palm oil or its by-products are very important.

The nation's palm oil industry would benefit significantly from the process of extraction and recovery of carotenes from palm oil. But unfortunately, conventional physical and chemical methods of palm oil refining causes almost all of the carotenes to be either removed or destroyed (Ooi *et al.* 1994). This process also helps to give the light colored oil as favoured by consumers. A few processes have been developed to recover carotenes from palm oil which includes solvent extraction (Tanaka, 1986), molecular distillation, and adsorption (Ong and Boey, 1980). These technologies however require high capital expenditure to set up and have significant operating costs due to vast energy consumption.

Nanofiltration offers a good alternative for the process of separating carotene from crude palm oil due to its lower energy consumption, ambient temperature operation, and retention of thermally sensitive compounds. Recently, organic solvent nanofiltration (OSN) has found numerous applications including homogeneous catalyst recovery, solvent exchange, chiral separation, concentration of natural extracts, and peptide synthesis. There are a few reports on the membrane process of recovering carotenoids from palm oils, in which the oil was first transesterified into methyl esters, then the carotenes are separated from the methyl esters through nanofiltration (Chiu *et al.*, 2009; Darnoko and Cheryan, 2006). The disadvantage of this process is that the edible oil is lost or rendered useless for further consumption. Thus there is a need to find a membrane filtration process to recover the carotenoids from crude palm oil prior to refining without chemical transformation of the triglycerides.

1.2 Problem Statement

During conventional physical and chemical methods of palm oil refining, almost all of the carotenes are removed and destroyed. It is proposed that prior to chemical or physical refining of the crude palm oil, the carotenoids are first recovered by adding an extra step of membrane filtration in the process. There are various methods being applied to recover carotene from crude palm oil, and intensive studies have been done on the extraction and recovery of carotene. These methods include saponification, selective solvent extraction, and transesterification followed by both phase separation and distillation of the ester. The process of transesterification can produce high concentrations of carotene however, has many disadvantages after weighing in factors such as cost, materials, and the large amount of energy required for evaporation. Converting the palm oil into methyl esters also renders the oil useless for further usage and consumption. Organic solvent nanofiltration can address these problems whereby the separation of carotene from crude palm oil is based on different molecular size, lowering energy consumption, minimizing thermal damage and without involving any phase change,. Moreover, the separation maintains the oil quality where the oil is not transformed to methyl esters while increasing yield through gentle ambient temperature separations.

1.3 Research Objectives

The main objective of this study is to explore the potential of OSN process to separate carotene from palm oil. In this present work, the performances of various OSN membranes will be evaluated for the permeation of carotene and retention of triglycerides. The best membrane in terms of flux and selectivity will be further studied on the effects of operating parameters such as pressure and feed concentration towards selectivity of carotene. The specific research objectives are as follows:

1. To identify the best combination of membrane and solvent for the separation of carotene from crude palm oil.
2. To evaluate the effects of varying operating parameters towards the flux and selectivity using the best combination of membrane and solvent.
3. To study the transport phenomenon involved in the OSN membrane separation of carotene and triglyceride using model solutions.

1.4 Scope of Study

In order to achieve the specified objectives, the study involves investigating the performance of OSN membrane in the recovery of carotene from crude palm oil directly without chemical transformation of the triglycerides. Performance was based on the flux (J), rejection (R) and selectivity (α) between carotene and triglycerides (palm oil). The process will use 4 different polyimide membrane materials with different molecular weight cut-off (MWCO) as described by the manufacturer. The membranes are PuraMem™ 280, DuraMem™ 150, DuraMem™ 300, and DuraMem™ 500. The effect of using different types of solvents namely acetone, hexane, ethyl acetate, and isopropanol towards flux and rejection will be studied. The effects of varying operating parameters namely; palm oil feed concentration, and pressure, and towards flux and selectivity will be studied. The rejection characteristics of the selected membrane will then be tested using model solution of carotene in solvent, and triglyceride in solvent. Based on the experimental data, the solute diameters and theoretical pore size of the selected membrane will be predicted using Stokes-Einstein equation. The implications of concentration polarization, osmotic pressure, membrane swelling, and solubility parameters in the membrane system will be investigated.

1.5 Significance of Research

This study will help to improve our understanding on the potential of OSN membranes towards specific applications in the processing of crude palm oil. This research will add significant advantages to the palm oil industry especially in the processing of crude palm oil since the findings will help to add value to the final product.

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