UNIVERSITI PUTRA MALAYSIA

ENRICHMENT OF BIOACTIVE MINOR COMPONENTS FROM CPO AND PPMFO BY SEQUENTIAL ADSORPTION-DESORPTION TECHNIQUE

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FK 2018 116

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By

PHOON KAH YEE

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

April 2018

Dedicated to my family

For your endless love, support and encouragement

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

ENRICHMENT OF BIOACTIVE MINOR COMPONENTS FROM CPO AND PPFMO BY SEQUENTIAL ADSORPTION-DESORPTION TECHNIQUE

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April 2018

Chair : Mohd Noriznan Mokhtar, PhD
Faculty : Engineering

The effort to separate or enrich bioactive compounds such as Vitamin E, phytosterols, squalene, and carotenoids from natural resources has been made to fulfil the increasing demands of the global market. Crude palm oil (CPO) which extracts from the palm oil fruit mesocarp fibre, is the product of palm oil mill, while, palm-pressed mesocarp fibre oil (PPMFO) is the residual oil of palm-pressed mesocarp fibre, a solid biomass which produced after CPO extraction in palm oil mill. Numerous studies reported on the presence of bioactive compounds as the minor components in CPO and PPMFO. In order to fulfil the market demand of these minor components, attempts such as supercritical fluid extraction and molecular distillation were develop to separate the minor components from plant source. However, limitation are present in current separation process. Therefore, in this study, interest is focusing on the enrichment efficiency of the minor components from CPO and PPMFO through a proposed sequential adsorption-desorption technique. Initially, static adsorption-desorption test was carry out to evaluate the adsorption and desorption efficiency using six commercial mesopores adsorbents which represented different polarity towards the minor components that obtained from CPO. Non-polar adsorbents (Diaion HP20 and Sepabeads SP850) showed better adsorption-desorption efficiency than polar adsorbent (silica gel and Florisil) and weak polar adsorbent (Diaion HP2MG and Amberlite XAD-7HP) due to the similar polarity between the minor components and adsorbent. Diaion HP20 was selected as the best adsorbent resulting of the economic price and it can be stored at room temperature compared with Sepabeads SP850. Then, CPO was adsorbed by Diaion HP20 and placed into a Soxhlet extraction system to perform sequential adsorption-desorption process study. Three different organic solvent were used in the sequential adsorption-desorption process study to investigate the effect of desorption solvent and desorption time towards the enrichment efficiency of minor components. After obtaining the best process parameters, the sequential adsorption-desorption process were repeated by subjecting PPMFO as the feedstock. Under the same process parameters, Vitamin E, phytosterols, and squalene from CPO were obtained in the 1st fraction using methanol with desorption time of 4 h that gave enrichment factor (EF) of 3.4, 3.9, and 1.8, respectively, which slightly higher than those minor components obtained from PPMFO, 1.2, 1.8, and 1.4,

respectively. Meanwhile, the carotene obtained from both CPO and PPMFO was enriched in the 3rd fraction by using *n*-hexane with an enrichment factor of 1.1 and 1.5, respectively. In conclusion, the obtained result revealed the efficiency of the proposed sequential adsorption-desorption technique to enrich the minor components from CPO and PPMFO.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENGAYAAN SEBATIAN BIOAKTIF KOMPONEN MINOR DARIPADA CPO AND PPMFO OLEH TEKNIK PENJERAPAN-NYAHJERAPAN BERTURUTAN

Oleh

PHOON KAH YEE

April 2018

Pengerusi : Mohd Noriznan Mokhtar, PhD
Fakulti : Kejuruteraan

Usaha giat untuk memisah atau memperkaya sebatian bioaktif daripada sumber berasaskan semulajadi dilaksanakan untuk memenuhi permintaan pasaran dunia yang semakin meningkat. Minyak kelapa sawit (CPO) diperoleh dari sabut mesokarpa buah kelapa sawit dan merupakan produk kilang minyak kelapa sawit, manakala minyak perahan sabut mesokarpa kelapa sawit (PPMFO) adalah minyak sisa dari perahan sabut mesokarpa kelapa sawit, di mana ia merupakan biojisim pepejal selepas hasilan CPO di kilang minyak kelapa sawit. Kebanyakan kajian menyatakan kehadiran sebatian bioaktif sebagai komponen kecil dalam CPO dan PPMFO. Percubaan seperti esktraksi cecair superkritikal dan *penyulingan molekul dilakukan* untuk memisah komponent kecil daripada tumbuh-tumbuhan bagi memenuhi permintaan pasaran terhadap komponen kecil. Namun demikian, batasan wujub dalam proses pemisahan semasa. Oleh itu, kajian ini menumpukan terhadap kecekapan pengayaan komponen minor dalam CPO dan PPMFO melalui teknik jujukan jerapan-nyahjerapan yang dicadangkan. Pada permulaan, ujian jerapan-nyahjerapan statik dijalankan untuk menilai kecekapan jerapan dan nyahjerapan terhadap komponen kecil dalam CPO menggunakan enam mesopori penyerap komersial di mananya mewakili kekutuban yang berbeza. Penyerap tak berkutub (Diaion HP20 and Sepabeads SP850) menunjukkan kecekapan jerapan-nyahjerapan yang lebih baik berbanding dengan penyerap berkutub (silica gel dan Florisil) dan berkutub lemah (Diaion HP2MG and Amberlite XAD-7HP) kerana kekutuban yang sama antara komponen kecil dengan penyerap. Diaion HP20 dipilih sebagai penjerap terbaik berdasarkan harga yang rendah dan boleh disimpan pada suhu bilik berbanding dengan Sepabeads SP850. Selepas itu, CPO dijerap oleh Diaion HP20 dan letak dalam sistem *ekstraksi Soxhlet* untuk mengkaji proses jerapan-nyahjerapan berturutan. Proses jerapan-nyahjerapan berturutan menggunakan tiga pelarut organik berbeza dan seterusnya mengenalpasti kesan pelarut dan masa terhadap kecekapan pengayaan komponen minor tersebut. Proses jerapan-nyahjerapan berturutan diulangi dengan menggunakan PPMFO sebagai bahan mentah selepas mendapati proses parameter yang sesuai. Dalam proses parameter yang sama, Vitamin E, phytosterols, dan squalene daripada CPO diperolehi dalam pecahan pertama menggunakan metanol dengan masa nyahjerapan, 4 jam dan memberi faktor pengayaan (EF) masing-masing

sebanyak 3.4, 3.9 dan 1.8, dimana lebih tinggi berbanding dengan komponen minor yang diperolehi daripada PPMFO, masing-masingnya ialah 1.2, 1.8 dan 1.4. Sementara itu, karotenoid daripada CPO dan PPMFO diperkaya pada pecahan yang ketiga dengan mengguna *n*-heksana dengan masing-masing memberi faktor pengayaan sebanyak 1.1 dan 1.5. Kesimpulannya, keputusan yang diperolehi menunjukkan kecekapan teknik jerapan-nyahjerapan yang dicadangkan untuk memperkayakan komponen kecil daripada CPO dan PPMFO.

ACKNOWLEDGEMENTS

This thesis becomes a reality with the direct and indirect support from many individuals. I would like to convey my gratefulness to the following person.

First and fore most, I would like to express my gratitude and thank to my main supervisor, Dr. -Ing. Mohd Noriznan Mokhtar for his guidance, support, supervision and advice. I thank to Dr. Grrace Ng Hui Suan for her inspiration and support for my courage to pursue Master study. I sincerely thank to my co-supervisor, Dr. Rabitah Zakaria and Dr. Yim Hip Seng for the motivation and guidance.

Sincerely appreciate to the non-academic staff of Department of Process and Food Engineering especially En Zahir for his assistance. Many thanks to all my friends in Universiti Putra Malaysia and UCSI University for lending their help during my Master study.

Last but not least, my special appreciation to my beloved family for their moral support, love and understanding throughout my Master study.

Thank you.

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

CPO	Crude palm oil
EF	Enrichment Factor
EFB	Empty fruit brunch
FFAs	Free fatty acids
FFB	Fresh fruit brunch
IPA	Isopropyl ethanol
MAGs	Monoacylglycerols
DAGs	Diacylglycerols
TAGs	Triacylglycerols
R^2	Correlation coefficients
HPLC	High performance liquid chromatography
GC	Gas Chromatography
LDL-C	Low density lipoprotein cholesterol
MSTFA	N-methyl-N-trimethylsilyl-trifluoroacetamide
PI	Polarity index
PPMFO	Palm-pressed mesocarp fibre oil
ROS	Reactive oxygen species
RNS	Reactive nitrogen species
SFE	Supercritical fluid exraction

CHAPTER 1

INTRODUCTION

1.1 Background

The demand of bioactive compounds, especially those extracted from natural resources is increasing globally due to the rise of safety concern, higher efficacies, and lower production cost (Ofori-Boateng & Lee, 2013; Puah, Choo, Ma, & Chuah, 2005). Compared with the petroleum synthetic, natural resources bioactive compounds are present in a scarce amount. It is advisable to select ready available natural resources which contain a high concentration of bioactive compounds to increase the separation yield and further reduced the separation cost.

African oil palm (*Elaeis guoneensis)* fruits produces two types of distinctive vegetable oils: crude palm oil (CPO) and palm kernel oil (PKO). CPO, a reddish orange colour oil which extracted from the fruit mesocarp through mechanical screw pressing technique (Mba, Dumont, & Ngadi, 2015). CPO represent the major product in oil palm industry by occupies about 88% of the total palm oil production (Abdullah & Sulaiman, 2013). CPO can be used for edible purposes such as cooking oils and margarine. (Mba et al., 2015) While, PKO, a light yellow colour oil which extracted from the kernel can be applied in oleochemicals such as detergents and soap (Abdullah & Sulaiman, 2013; Basiron, 2007; Sambanthamurthi, Sundram, & Tan, 2000).

Palm oil becomes the major source by contributing 33% (66 million tonnes) of 200 million tonnes of world oils and fats production in 2014 (Malaysian Palm Oil Board (MPOB), 2015). While, in Malaysia, oil palm industry served as the fourth largest economy contributor to give over RM 80 billion (about 8%) to gross national income (Malaysian Innovation Agency, n.d.). Since the first commercial oil palm estate setup at Tennamaran estate, Selangor in 1917, the oil palm plantation area in Malaysia increased drastically. Until 2014, the oil palm plantation area in Malaysia reached to 5.39 million hectares, which recorded a production of 19.67 million tonnes of palm oil (Abdullah & Sulaiman, 2013; Awalludin, Sulaiman, Hashim, & Nadhari, 2015). In addition, Malaysia becomes the world’s second palm oil producer and exporter after Indonesia since 2008 (Abdullah & Sulaiman, 2013).

Oil palm industry generates substantial income to Malaysia’s economy but in returns, it generates the largest amounts of biomass waste within agriculture sector. A palm tree produces 10% of oil yield and the remaining 90% is the biomass (Ofori-Boateng & Lee, 2013). Oil palm industry generated 83 million tonnes dry biomass in the year 2010 and is estimated will reach to 110 million by 2020 (Malaysian Innovation Agency, n.d.). Palm-pressed mesocarp fibre is one of the solid biomass waste which produced after mechanical screw-pressing CPO extraction process in palm oil mill. It accounts 7% (about 5.8 million tonnes) in the solid biomass waste of Malaysia palm oil industry in the year 2010. This amount is expected re ach to 8.8 million tonnes in the year 2020 (Malaysian Innovation Agency, n.d.). This creates an issue for oil palm industry on how to valorise the palm-pressed mesocarp fibre for value added end-uses. Currently, palm-pressed mesocarp fibre is utilized as the fuel for sterilization and electricity generation in palm oil mill operation (Abdullah & Sulaiman, 2013). Lau, Choo, Ma, and Chuah (2008) commented that approximately 5-7% residual oil was trapped in the palm-pressed mesocarp fibre after the mechanical screw-pressing. This residual fibre oil is known as palm-pressed mesocarp fibre oil (PPMFO).

1.2 Problem statements

The demand of bioactive compounds, especially those extracted from natural resources is increasing globally due to the rise of safety concern, higher efficacies, and lower production cost (Ofori-Boateng & Lee, 2013; Puah et al., 2005). Compared with the petroleum synthetic, natural resources bioactive compounds are present in a scarce amount. It is advisable to select ready available natural resources which contain a high concentration of bioactive compounds to increase the separation yield and further reduced the separation cost.

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1.3 Hypothesis

Based on review previous studies, hypothesis could be made:

1) The performance of an adsorbent in adsorb and desorb of Vitamin E, phytosterols, carotenoids, and squalene can be characterize based on the its surface polarity (Li & Chase, 2010; Soto, Moure, Domínguez, & Parajó, 2011).
2) The concentration of Vitamin E, phytosterols, carotenoids, and squalene that obtained from CPO and PPMFO can be increased through the proposed sequential adsorption-desorption process (Gunawan, Kasim, & Ju, 2008; Latip, Baharin, Man, & Rahman, 2001).

1.4 Objectives

1) To characterize different adsorbents in adsorb and desorb Vitamin E, phytosterols, carotenoids, and squalene that obtained from CPO.
2) To evaluate the proposed sequential adsorption-desorption process based on the enrichment efficiency of Vitamin E, phytosterols, carotenoids, and squalene that obtained from CPO an+++d PPMFO.

1.5 Scope of work

This thesis concerned on the strategy to enrich the concentration of the Vitamin E, phytosterols, carotenoids, and squalene using the proposed sequential adsorption-desorption process in a Soxhlet extraction system. Vitamin E, phytosterols, carotenoids, and squalene were obtained from CPO source. Static adsorption-desorption test was used to compare the adsorption and desorption efficiency of minor components among six selected commercial adsorbents. The best adsorbent was further used in the sequential adsorption-desorption process study. Three oil fractions were produced after the sequential adsorption-desorption process. Different desorption solvents and times of the 1st fraction were tested to increase the enrichment factor of Vitamin E, phytosterols, and squalene. While different desorption solvents of the 2nd fraction was also done to increase the enrichment factor of carotenoids. Then, study was repeated with PPMFO as the feedstock, performed at the best condition which obtained from the CPO study.

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