UNIVERSITI PUTRA MALAYSIA

SQUALENE RECOVERY FROM PALM FATTY ACID DISTILLATE USING PRESSURE SWING AND CONVENTIONAL SUPERCRITICAL CARBON DIOXIDE EXTRACTION

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FSTM 2011 15

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

November 2011

DEDICATION

To my dear husband, Mohd Helmi Ali for his support and affectionate caring each moment in my life, especially throughout my study, specially dedicated to my sons, Ahmed Ilhan Thaqif and Ahmed Ilhan Zayd, my father Suleiman Bin Mohidonni, my late mother, Zainah Bt Mohd Ali, my parents in law, Ali Othman and Mintan Amat, for spiritual support and doa, that I owe them each moment of my life.

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

SQUALENE RECOVERY FROM PALM FATTY ACID DISTILLATE USING PRESSURE SWING AND CONVENTIONAL SUPERCRITICAL CARBON DIOXIDE EXTRACTION

By

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November 2011

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Squalene is a natural lipid compound and has a good potential as an antioxidant, hence an excellent choice to be used as functional ingredient. The objectives of this study were to find the optimum conditions for squalene extraction from palm fatty acid distillate (PFAD) using two processes of supercritical carbon dioxide (SC-CO_2) extraction, such as conventional extraction and pressure swing process and find the better process for squalene recovery. Response surface methodology (RSM) with central composite design (CCD) was used to determine optimum squalene recovery from PFAD using SFE. For both processes, extraction were set at temperatures ranged from 40 to 60 °C and pressure from 200 to 400 bar for a 90 min dynamic extraction period. For pressure swing process, holding time was set from 20 to 40 min. Fourteen and twenty runs were carried out for conventional extraction and pressure swing techniques, respectively. Squalene was quantified utilizing reverse phase high performance liquid chromatography (RP-HPLC). Comparison of the

performance of the two processes was made based on total lipid extract and squalene concentration in the extract. Besides that, comparison was also made based on the squalene solubility study of both techniques. The results showed that optimum SFE conditions for conventional extraction process were at 200 bar and 50 °C, which resulted in total lipid extract and squalene concentration of 13.87±0.52 % w/w and 418.31±18.40 ppm respectively. Meanwhile for pressure swing process, optimum condition were at 230 bar, 40 °C and 20 min holding time, which yielded a total lipid extracts and squalene concentration of 19.19±2.47 % w/w and 368.28±18.10 ppm respectively. The experimental value agreed with that of predicted value at the optimal conditions. The results show that pressure swing process was the more effective than conventional extraction in producing higher total lipid extracts. However, squalene concentration from the extracts was found to be the higher in conventional extraction even though the pressure swing process gave better efficiency in terms of total lipid extracts. The solubility study shows that the squalene solubility in SC-CO_2 was higher using conventional extraction (18.1207 kg/kg) compared to pressure swing process (15.2374 kg/kg) since the main objective is the attainment of highest squalene concentration, hence, it can be concluded that SFE with conventional extraction is more desirable than pressure swing process.

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

PENEMUAN SQUALENE DARIPADA SULINGAN ASID LEMAK SAWIT MENGGUNAKAN PENGEKSTRAKAN TEKANAN BERAYUNAN DAN KONVENSIONAL KARBON DIOKSIDA LAMPAU GENTING

Oleh

NORHIDAYAH BTE SULEIMAN

November 2011

Pengerusi : Prof. Madya Badlishah Sham Baharin

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Squalene merupakan komposisi lemak semulajadi dan mempunyai potensi yang baik sebagai antioksida, maka satu pilihan yang baik untuk dijadikan sebagai sebatian fungsian. Objektif kajian ini adalah untuk mendapatkan keadaan optimum bagi proses pengekstrakan squalene daripada sulingan asid lemak sawit dengan menggunakan teknik bendalir lampau genting karbon dioksida (SC-CO_2) yang berbeza iaitu teknik konvensional dan tekanan yang berayunan dan juga untuk mendapatkan teknik yang terbaik bagi pengekstraksan squalene. Metodologi permukaan sambutan (RSM) dengan rekabentuk komposit berpusat (CCD) digunakan untuk menentukan nilai optimum ekstraksi squalene daripada sulingan asid lemak sawit menggunakan SC-CO_2. Bagi kedua-dua teknik, ekstraksi telah dijalankan pada suhu 40 hingga 60 °C dan tekanan dari 200 hingga 400 bar selama

90 min. Bagi teknik tekanan berayunan, masa tetap disetkan dari 20 hingga 40 min. Empat belas dan dua puluh kali eksperimen dijalankan dan kandungan squalene ditentukan menggunakan kromatografi cecair prestasi tinggi berfasa terbalik (RP-HPLC). Perbandingan dibuat berdasarkan kuantiti ekstraksi lemak dan juga kepekatan squalene daripada ekstraksi yang diperoleh. Selain itu, perbandingan juga dibuat berdasarkan ciri dapat larut bagi kedua-dua teknik. Keputusan menunjukkan keadaan optimum bagi teknik konvensional adalah 200 bar dan suhu 50 °C, dimana ekstraksi lemak yang diperoleh adalah 13.87±0.52 % w/w dan kepekatan squalene adalah 418.31±18.40 ppm. Sementara bagi teknik tekanan berubah-ubah pula, keadaan optimumnya adalah pada 230 bar, 40 °C dan masa tetap 20 min, dimana menghasilkan ekstraksi lemak sebanyak 19.19±2.47 % w/w dan kepekatan squalene adalah 368.28±18.10 ppm. Nilai eksperimen bersamaan dengan nilai yang diramal pada keadaan optimum. Keputusan menunjukkan teknik tekanan berayunan merupakan teknik yang efektif dalam pengekstrakan ekstraksi lemak. Walaubagaimanpun, kepekatan squalene yang diperoleh dari ekstraksi adalah lebih tingggi menggunakan teknik konvensional. Penyelidikan ke atas kelarutan squalene juga menunjukkan bahawa kelarutan squalene adalah lebih tinggi dengan menggunakan teknik konvensional (18.1207 kg/kg) berbanding dengan teknik tekanan berayunan (15.2374 kg/kg). Oleh kerana, objektif utama adalah pencapaian kepekatan squalene tertinggi, maka, dapat disimpulkan yang SFE dengan teknik konvensional adalah lebih diperlukan daripada teknik tekanan berayunan.

ACKNOWLEDGEMENT

Alhamdullillah, first of all, I would like to express my utmost thanks and gratitude to Allah S.W.T., the Sustainer, the most Gracious and most Merciful, who has given me the chance and capability to complete this research. My salawat and salam is addressed to His righteous messenger, prophet Muhammad s.a.w.

I would like to express my deepest gratitude to my supervisor, Associate Professor, Badlishah Sham Baharin, for his valuable suggestions, constructive criticisms, guidance and patience throughout the research and special appreciation and gratitude is extended to Associate Professor, Dr. Md Zaidul Islam Sarker and Dr. Seyed Hamed Mirhosseini for the constructive suggestions, tremendous encouragement and constant guidance.

To my beloved lab mates and friends, thanks a lot for your help, suggestions, comments and moral support in the improvement and completion of this thesis.

Special thanks to all staffs of Faculty of Food Science and Technology for their contributions towards the success of this research. Last but not least, thanks to everybody that was involved and contributed to the success of this research.

Approval examiner

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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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

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Date: 24 November 2011

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