UNIVERSITI TEKNOLOGI MARA

RAPID EXPANSION SUPERCRITICAL SOLUTION (RESS) CARBON DIOXIDE AS A GREEN TECHNOLOGY FOR GINGER RHIZOME SOLID OIL PARTICLES FORMATION

NUR AIN BINTI MOHD ZAINUDDIN

Thesis submitted in fulfilment of the requirements for the degree of **Master of Science**

Faculty of Chemical Engineering

March 2016

CONFIRMATION BY PANEL OF EXAMINERS

I certify that a Panel of Examiners has met on 30th December 2015 to conduct the final examination of Nur Ain Binti Mohd Zainuddin on her Master of Science thesis entitled "Rapid Expansion Supercritical Solution (RESS) Carbon Dioxide As A Green Technology For Ginger Rhizome Solid Oil Particles Formation" in accordance with Universiti Teknologi MARA Act 1976 (Akta 173). The Panel of Examiners recommended that the student be awarded the relevant degree. The panel of Examiners are as follows:

Prof Ir. Dr. Jailani Salihon, PhD Professor, Faculty of Chemical Engineering Universiti Teknologi MARA (Chairman)

Dr. Azil Bahari Bin Alias, PhD
Deputy Dean (Research, Industrial Linkage and Alumni)
Faculty of Chemical Engineering
Universiti Teknologi MARA
(Internal Examiner)

Prof Madya Dr.Mohd Azizi Bin Che Ku Yunus, PhD Associate Professor Faculty of Chemical Engineering Universiti Teknologi Malaysia (External Examiner)

SITI HALIJJAH SHARIFF, PhD

Associate Professor Dean Institute of Graduates Studies Universiti Teknologi MARA Date:

AUTHOR'S DECLARATION

I declare that the work in this thesis/dissertation was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

Name of Student : Nur Ain Binti Mohd Zainuddin

Student I.D. No : 2013822038

Programme : Master of Science (EH 750)

Faculty : Chemical Engineering

Thesis Title : Rapid Expansion Supercritical Solution (RESS)

Carbon Dioxide As A Green Technology For

Ginger Rhizome Solid Oil Particles Formation

Signature of Student :

Date : March 2016

ABSTRACT

Ginger (Zingiber officinale, Rosc.) contains high antioxidant bioactive constituents. Nowadays, ginger extract received great attention due to the increasing application of natural antioxidant in pharmaceutical products. Many studies acknowledged the high amount of natural antioxidant content in ginger as a valuable component to replace synthetic antioxidant that can be used in anticancer activity for cancer treatment. Most of the previous researchers used Supercritical Fluid Extraction (SFE) and conventional extraction technique such as steam distillation and solvent extraction method in ginger extraction. However, the conventional technique for ginger extraction required organic solvent, long extraction time, produce toxic residual and waste water that can be harmful to human and environment. SFE, solvent extraction and steam distillation for ginger extraction produced ginger extract in oil form that need to undergo conventional technique for particle formation. Recent developments in extraction technology allowed extraction of bioactive components in herb to produce solid oil particle instead of liquid oil that need to undergo further conventional techniques for particles formation. Therefore, in this study a clean extraction method that is Rapid Expansion Supercritical Solution (RESS) technology using supercritical carbon dioxide as a solvent (RESS-CO₂) was introduced for ginger powder extraction to produce solid oil particle of bioactive components. The extraction was conducted within a range of temperature (40, 45, 50, 55, 60, 65 and 70°C) and pressure (3000, 4000, 5000, 6000 and 7000psi), at a constant flowrate (24 ml/min) for 40 minutes extraction. The extract obtained was characterized using Scanning Electron Microscopy (SEM) and ImageJ Program for solid oil particle size determination and Gas chromatography Mass Spectrometry (GCMS) for component identification. The smallest solid oil particle size 2.22 µm was obtained at temperature 40°C, and pressure 5000psi. The highest extraction yield 2.41% was achieved at temperature 65°C, and pressure 6000psi. Analysis of the smallest solid oil particle using GCMS showed that the detected components were Gingerol and β -Elemene, Farnesene, Caryophyllene, Farnesol, Borneol, Thujopsene, Aromadendrene, Bergamotene, Nerolidol, α -copaene, cis- α -copaene-8-ol, Decanal, and 2-undecanone. Experimental work for the whole range of temperature and pressure consumed more cost and time. Thus, in this study, experimental particle size data was modeled by using Artificial Neural Network (ANN) for particle size prediction. A multilayer feedforward back-propagation ANN model was developed where two inputs variables (extraction temperature and pressure) and one output (particle size) with 35 experimental data points. An optimal ANN model consists of 1 hidden layer with 7 hidden neurons with the lowest mean square error (MSE) of 0.00031 and regression coefficient (R) of 0.99721 showed that good training and good performance agreement between ANN model predicting data and experimental data.

ACKNOWLEDGEMENTS

In the name of ALLAH, the most Gracious, the most Merciful. Alhamdulillah, all praises to ALLAH the Almighty who help me and gave me strength, inspiration and patience to complete this long and challenging research journey successfully. My gratitude and thanks go to my supervisor Dr.Norhuda Binti Ismail, for her continuous moral support, guidance, valuable time, ideas, prayer and assisting me throughout my difficulties in this project. Special thanks and appreciation also goes to my very supportive co-supervisor Miss Sitinoor Adeib Binti Idris and Dr. Siti Halimah Binti Sarijo for their willingness in motivating and helping me to complete this journey.

My Appreciation goes to the staffs at Faculty of Chemical Engineering, Mr. Mohd Yazid Yusof, Mr.Mohd Faeez Sarulan, Mr. Amin Fafizullah Omar and Mr.Rizuan Hamzah for their assistance and guidance in setting up and operate the laboratory equipment, assistant science officer at Faculty of Dentistry, Mr. Hizwan Nizam Abd Rahman for guidance to perform analysis using Scanning Electron Microscopy (SEM), and assistant science officer at Faculty of pharmacy Mr. Mohamed Rahimi Sulaiman and staff at Faculty of Applied Science, Mdm Sahida for her guidance to perform the analysis by using Gas Chromatography Mass Spectrometry (GCMS). Thank you for all the support, patience and ideas in assisting me with this project.

I also would like to express my thankful to the Ministry of Education Malaysia and Skim Tenaga Pengajar Muda UiTM for the financial support received.

My appreciation goes to all Faculty of Chemical Engineering postgraduates' students and to my former co-workers at Notion Venture Sdn.Bhd, Meru, Selangor for moral support and encouraging me to complete this project.

Finally, this thesis dedicated to my husband, Mohd Firdaus Bin Ismail, my son, Muhammad Izz Danish Rizqin, my parents, Mohd Zainuddin Bin Awang and Mariah Binti Latif, my father in law, Ismail Bin Hussin and to all my brothers for their continuous support and prayers. This piece of victory is dedicated to all of you. Alhamdulilah.