UNIVERSITI PUTRA MALAYSIA

EFFECTS OF MICROWAVE-VACUUM DRYING ON DRYING KINETICS AND QUALITY OF ORTHOSIPHON ARISTATUS (BLUME) MIQ. LEAVES AND EURYCOMA LONGIFOLIA JACK ROOTS

HADA MASAYU ISMAIL @ DAHLAN

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By

HADA MASAYU BINTI ISMAIL @ DAHLAN

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

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DEDICATION

This thesis is specially dedicated to

my husband, parents, children

and

my late son who passed away on 17th June 2013

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Master of Science

EFFECTS OF MICROWAVE-VACUUM DRYING ON DRYING KINETICS AND QUALITY OF *Orthosiphon aristatus* (BLUME) MIQ. LEAVES AND *Eurycoma longifolia* JACK ROOTS

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HADA MASAYU BINTI ISMAIL @ DAHLAN

November 2016

Chairman : Associate Professor Mohd. Nordin Ibrahim, PhD
Faculty : Engineering

Orthosiphon aristatus (OA) is known locally as misai kucing; belongs to the family Lamiaceae. Previous studies have shown that the leaves of OA have beneficial effects including anti-oxidant, anti-hypertensive and diuretic effect. Another herb, *Eurycoma longifolia* (EL) is well-known for its local name as tongkat ali; belongs to the family Simaroubaceae. Previous research has shown that the roots of EL have beneficial effects including anti-malarial, anti-bacterial and anti-pyretic.

The presence of the properties that are beneficial in both herbs have shown that extracts from these materials have great potential in the development of various healthcare products. However, research in the area of processing for the production of bioactive compounds still insufficient.

This research studied the effects of microwave-vacuum drying (MVD) techniques of OA leaves and EL roots and determined the optimum drying condition based on drying kinetics and phytochemicals content. Optimum condition of convection oven drying obtained at 45°C for the leaves and 60°C for the roots were selected and used as the control drying method in the study.

The MVD experimental runs and optimization work were designed using Central-composite of Response Surface Methodology (RSM). Thirteen experiment points were obtained from RSM with microwave-power ranged from 150 – 300 W and vacuum pressure ranged from15 – 50 kPa. Experiments were carried out at continuous and intermittent modes. The effects of the two variables on phytochemicals of OA leaves and EL roots were investigated. Quality indicator selected for the leaves and the roots were Rosmarinic acid (RA) and Eurycomanone (EUR), respectively. At intermittent mode, three pulsation ratio (PR) of 2, 3 and 4 were studied. Three thin-layer drying models namely Page, Midili and Logarithmic were applied to fit the experimental data in order to select the best model to represent MVD process for both type of herbs.

The optimum MVD conditions at continuous mode for the highest RA content of OA leaves were obtained at 300 W microwave power and 50 kPa vacuum pressure. The optimum responses of the process were: RA 556.28 mg/L and drying time 17.14 minutes. Whereas for optimum MVD conditions at intermittent mode, PR 3 was found to be the most suitable condition with 700.39 mg/L of RA concentration in the dried leaves. On the other hand, for EL roots, the optimum MVD conditions at continuous mode for the highest EUR content were obtained at 150 W microwave power and 50 kPa vacuum pressure. The optimum responses of the process were: EUR 7.92 mg/L and drying time 18.64 minutes. Whereas for optimum MVD conditions at intermittent mode, PR 4 was found to be the most suitable condition with 3.58 mg/L of EUR concentration in the dried roots.

Midili model was found to be the most suitable model to describe the MVD process of both types of herbs with root mean square error (RMSE) ranged from 0.9953 – 0.999 for OA leaves and 0.9666 – 0.999 for EL roots.

MVD at 300 W microwave power and 50 kPa vacuum pressure, at intermittent mode, with PR 3 was found out to be the most suitable drying method for the microwave-vacuum drying technique for OA leaves as it contained the highest amount of RA. While, for EL roots, MVD at 150 W microwave power and 50 kPa vacuum pressure, at continuous mode was found out to be the most suitable drying method for the microwave-vacuum drying technique for EL roots however it contained less amount of EUR as compared to EUR from control drying method.

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

KESAN PENGERINGAN SECARA VAKUM-GELOMBANG MIKRO TERHADAP KINETIK PENGERINGAN DAN KUALITI HERBA DAUN *Orthosiphon aristatus*(BLUME) MIQ. DAN AKAR *Eurycoma longifolia* JACK

Oleh

HADA MASAYU BINTI ISMAIL @ DAHLAN

November 2016

Pengerusi : Profesor Madya Mohd. Nordin Ibrahim, PhD
Fakulti : Kejuruteraan

Orthosiphon aristatus (OA) lebih dikenali dengan nama tempatannya misai kucing; tergolong dalam keluarga Lamiaceae. Kajian sebelum ini menunjukkan bahawa daun OA mempunyai kesan berfaedah termasuk anti-oksida, anti-hipertensi dan kesan diuretik.

Manakala *Eurycoma longifolia* (EL) pula terkenal dengan nama tempatannya tongkat ali; tergolong dalam keluarga Simaroubaceae. Kajian sebelum ini menunjukkan bahawa akar EL mempunyai kesan berfaedah termasuk anti-malaria, anti-bakteria dan anti-piretik.

Kehadiran sifat-sifat yang bermanfaat dalam kedua-dua herba ini menunjukkan bahawa ekstrak daun OA dan akar EL mempunyai potensi besar dalam pembangunan pelbagai produk kesihatan. Walaubagaimanapun, penyelidikan dalam aspek pemprosesan untuk penghasilan komponen bioaktifnya masih tidak mencukupi.

Objektif kajian ini adalah untuk mengkaji kesan teknik pengeringan secara vakum-gelombang mikro (MVD) pada herba daun OA dan akar EL, serta untuk menentukan kondisi pengeringan optimum berdasarkan kinetik pengeringan dan kandungan fitokimia herba tersebut. Kondisi optimum pengeringan secara oven konveksi yang diperolehi pada suhu 45°C untuk daun OA dan 60°C untuk akar EL telah dipilih dan digunakan sebagai kaedah kawalan di dalam penyelidikan ini.

Jumlah eksperimen MVD dan kerja optimasasi telah direka menggunakan rekabentuk komposit-pusat daripada Metodologi Permukaan Respons (RSM). Tiga belas eksperimen telah diperolehi daripada RSM dengan kuasa gelombang mikro di antara 150 - 300 W dan tekanan vakum di antara 15 - 50 kPa. Eksperimen telah dijalankan pada mod berterusan dan tidak-berterusan. Kesan kedua-dua pembolehubah, iaitu kuasa gelombang mikro dan tekanan vakum, terhadap kandungan fitokimia daun OA dan akar EL telah dikaji. Petunjuk kualiti Asid rosmarinik (RA) dan Eurycomanone (EUR) telah dipilih masing-masing untuk daun OA dan akar EL. Bagi kajian yang melibatkan mod tidak-berterusan, tiga ratio (PR) telah dikaji iaitu ratio 2, 3 dan 4. Tiga model pengeringan iaitu Page, Midili dan Logarithmic telah digunakan untuk menyesuaikan data eksperimen bagi memilih model pengeringan yang terbaik untuk mewakili proses MVD kedua-dua jenis herba.

Kondisi optimum MVD pada mod berterusan telah diperolehi pada kuasa gelombang mikro 300 W dan 50 kPa tekanan vakum untuk daun OA. Pada kondisi ini, kandungan RA adalah yang tertinggi. Respons optimum yang diperolehi adalah: RA 556.28 mg / L dan masa pengeringan 17.14 minit. Manakala PR 3 didapati paling sesuai untuk kondisi optimum MVD pada mod tidak-berterusan, dengan kepekatan RA sebanyak 700.39 mg / L dalam daun OA. Sebaliknya, bagi akar EL, kondisi optimum MVD pada mod berterusan telah diperolehi pada kuasa gelombang mikro 150 W dan 50 kPa tekanan vakum. Pada kondisi ini, kandungan EUR adalah yang tertinggi. Respons optimum yang diperolehi adalah: EUR 7.92 mg / L dan masa pengeringan 18.64 minit. Manakala PR 4 didapati paling sesuai untuk kondisi optimum MVD pada mod tidak-berterusan, dengan kepekatan RA sebanyak 700.39 mg / L dalam akar EL.

Model pengeringan Midili didapati paling sesuai untuk menggambarkan proses MVD bagi kedua-dua jenis herba dengan purata-ralat-punca-kuasa (RMSE) antara 0.9953 - 0.999 untuk daun OA dan 0.9666 - 0.999 untuk akar EL.

Melalui kajian yang telah dijalankan ini, didapati kaedah pengeringan yang paling sesuai untuk daun OA menggunakan teknik MVD adalah pada kondisi pengeringan 300 W kuasa gelombang mikro dan 50 kPa tekanan vakum, pada mod tidak-berterusan, PR 3. Ini adalah kerana kepekatan RA adalah paling tinggi pada kondisi ini. Manakala, untuk akar EL, didapati kaedah pengeringan yang paling sesuai menggunakan teknik MVD adalah pada kondisi pengeringan 150 W kuasa gelombang mikro dan 50 kPa tekanan vakum, pada mod berterusan. Namun, kepekatan EUR yang diperolehi adalah kurang berbanding kaedah kawalan.

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Insya Allah.

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

Mohd. Nordin Ibrahim, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Rabitah Zakaria, PhD
Senior Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Pin Kar Yong, PhD
Research Officer
Herbal Technology Centre
Innovation and Commercialisation Division
Forest Research Institute Malaysia
(Member)

ROBIAH BINTI YUNUS, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) were adhered to.

Signature: __________________________________
Name of Chairman of Supervisory Committee: Associate Professor Dr. Mohd. Nordin Ibrahim

Signature: __________________________________
Name of Member of Supervisory Committee: Dr. Rabitah Zakaria

Signature: __________________________________
Name of Member of Supervisory Committee: Dr. Pin Kar Yong

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LIST OF ABBREVIATIONS

MVD	Microwave-vacuum drying
PR	Pulsing ratio
OA	*Orthosiphon aristatus*
EL	*Eurycoma longifolia*
RA	Rosmarinic acid
EUR	Eurycomanone
RSM	Response Surface Methodology
b_o	Regression coefficient of intercept term
b_1	Linear regression coefficient
b_2	Linear regression coefficient
b_{11}	Squared regression coefficient
b_{22}	Squared regression coefficient
b_{12}	Interaction regression coefficient
Y	Response of RSM
T	Absolute temperature
HPLC	High Performance Liquid Chromatography
UPLC	Ultra Performance Liquid Chromatography
RMSE	Root mean square error

CHAPTER 1

INTRODUCTION

1.1 Herbal Industry in Malaysia

The prospect of herbal and spices industry in Malaysia is very promising and anticipated to produce new source of wealth for the nation. Issues that need to be addressed by the industry includes technology application not optimum, insufficient supply of raw materials, inconsistent quality of raw materials, limited cultivation area, lack of focus on high-value herbal products and lack of investments from large companies (Mohd Hafizudin, 2015).

Thus, the industry must be revived as Malaysia has been blessed with plentiful natural resources and massive number of species with medicinal potential. In Malaysia, there are market growth per annum of 10 to 15% for pharmaceutical and herbal remedies, and 20% for health and functional food (Zurinawati, 2004). Resulting from this growing market, tremendous effort has been carried out in order to discover highly valuable plants and turn it into finished products.

1.2 Herbal Postharvest Processing

Herbal postharvest processing refers to primary processing following harvesting process including cooling, cleaning, sorting, drying, grinding and packaging. It is important to produce herbal products with good quality and conforms to market regulations and guidelines in order to penetrate into the global market. The goals in postharvest processing are:

i) to improve quality and value of herbs
ii) reduce postharvest losses
iii) optimize herbal processing to maintain shelf life

Initial postharvest drying conditions are critical to avoid moisture loss and slow down undesirable chemical changes. It is an oldest preservation technique to maintain shelf-life with regards to physical appearance, taste, flavour and texture. Drying refers to the thermal processes that aimed to reduce moisture content of fruits and vegetables, and it is one of the time and energy consuming processes in the food industry (Puligundla *et al.*, 2013). Combination methods such as sensorial, biochemical, mechanical and colorimetric measurements are involved in drying, but focused technology is on the biochemical marker and fingerprint methods as indices for quality control (Li *et al.*, 2008).

1.3 Problem statements

Orthosiphon aristatus (OA) and *Eurycoma longifolia* (EL) are valuable medicinal plants that have significant potential in the production of healthcare products and food supplements in Malaysia. The plants were listed under the national herbal species under the Agriculture National Key Economic Area (NKEA) initiative for the herbal sub-sector.

In herbal processing, dehydration is the most crucial part because this step is part of the product development process for powdering, tea making, extraction, capsuling and tabletting. Fresh harvested plants and semi-processed plants were used as starting material. In Malaysia, most of the commercial products derived from OA are in a form of herbal tea processed from the dried leaves. Tea processing involves particle size reduction of the dried leaves, packaging, sterilization and quality control. On the other hand, commercial products derived from EL are in a form of capsules and 3-in-1 beverages. Manufacturing these products involve dried materials as input that are microbial-free to ensure the products are safe to be consumed.

Convection oven drying is widely used in herbal industry for herbal drying. The drawback in this drying method are time-consuming (Arslan & Musa, 2010), quality degradation due to heat exposure (Jaloszyński *et al.*, 2008; Lin *et al*., 2010; Giri & Prasad, 2007) and unavailability of standard drying parameters to maintain product quality.

Applying microwave energy to food material seems to be applicable approach for coping with disadvantages in conventional drying. When microwave energy applies to food materials, heat is generated within the product. Temperature of the product increase rapidly resulting in faster water removal than conventional drying. This contributes to saving time and energy. However, microwave drying can caused product damage caused by excessive heating which may be due to poorly controlled heat and mass transfer (Ramaswamy & Marcotte, 2006). Two strategies were applied for drying food materials using microwave technology. The strategies using vacuum in the dryer to lower drying temperature and application of microwave energy in a pulsed manner to maximize drying efficiency since continuous heating does not accelerate the rate of water removal when critical moisture content is reached.

Microwave drying assisted with vacuum had reported previously to be used successfully for the dehydration of various fruits, vegetables and aromatic herbs including rosemary (Calín-Sánchez *et al.*, 2011), edamame (Qing-guo *et al.*, 2006), grapes (Clary *et al.*, 2007), potato (Bondaruk *et al.*, 2007), tomato (Abano *et al.*, 2013), green peas (Chauhan & Srivastava, 2009), carrots (Cui *et al*., 2005), mushrooms (Giri & Suresh, 2007), apples (Han *et al.*, 2010) and berries (Mitra & Meda, 2009). However, its application in medicinal herbs is still scarce. The dryer also not exist commercially, mostly are laboratory scale and research done tailored to the product. The present study was to access the beneficial of this drying technology and investigate its performance to OA leaves and EL roots in term of their drying kinetics and phytochemicals quality.

1.4 Objectives

The objectives of the study were:

- To determine the effects of microwave-vacuum drying techniques on drying kinetics and phytochemicals content of OA leaves and EL roots.
- To determine the optimum drying condition based on phytochemicals content of OA leaves and EL roots.

1.5 Scope of Research

The scope of this research work covered drying process for the leaves of *Orthosiphon aristatus* (OA) and roots of *Eurycoma longifolia* (EL) using microwave-vacuum drying (MVD) technique. The influence of two variables from MVD namely microwave powers ranging from 150 to 300 W and vacuum pressures ranging from 15 to 50 kPa were studied based on drying kinetics and selected phytochemicals quality. The design of the experiments and process optimization was done using Response Surface Methodology (RSM) from Design-Expert software, a tool for process optimization. Modes of drying for MVD were at continuous and intermittent. At intermittent mode, pulsing ratios of 2, 3 and 4 were applied.

The selected phytochemicals for OA leaves and EL roots were Rosmarinic acid (RA) and Eurycomanone (EUR), respectively. The quality of the dried leaves and roots based on these phytochemicals at different drying condition was quantified using liquid chromatography. Three thin-layer drying models namely Page, Midili and Logarithmic were chosen to fit the experimental data in order to select the best model to represent MVD process for both type of herbs.

Convection oven drying was selected as control drying method. Drying temperatures applied for OA leaves were 45, 50 and 55°C, while for EL roots were 40, 50, 60 and 70°C. Optimum drying temperatures based on phytochemicals content were selected and used in the study.

1.6 Structure of the Thesis

The thesis comprises five chapters. Firstly, Chapter 1 introduces the research problem as well as outlines towards the thesis conclusions. Chapter 2 provides literature review in which the theoretical, knowledge sharing as well as the hypotheses arising from knowledge developed during the literature review. Chapter 3 describes and explains the method used and data collection in this study. Chapter 4 summarizes the results applying the method in this study. Finally, Chapter 5 contains the conclusions about the hypotheses and research issues based on the results of Chapter 4 and their implications for practice as well as future research.

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Hada Masayu Ismail, Mohd. Nordin Ibrahim, Rabitah Zakaria, Pin Kar Yong & Mohd. Radzi Ahmad. (2016) **.** Effects of convection oven drying on drying kinetics and quality of *Eurycoma longifolia* Jack. Submitted to Bioresource Technology Journal.

Journal to be Submitted

Mohd. Nordin Ibrahim, **Hada Masayu Ismail**, Pin Kar Yong & Rabitah Zakaria, Mohd. Radzi Ahmad. (2016) **.** Effects of microwave-vacuum drying on drying kinetics and quality of *Eurycoma longifolia* Jack. Food Research.

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