UNIVERSITI PUTRA MALAYSIA

DRYING CHARACTERISTICS OF PIPER BETLE LINN LEAVES USING VACUUM AND HEAT PUMPS DRYING METHODS

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By

PUTERI FARAH WAHIDA BINTI MGT AHMAD

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

June 2013

This thesis is specially dedicated to
my beloved husband,
my wonderful and supportive parents and siblings,
and
my in laws.

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

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June 2013

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Piper betle L. belongs to genus Piper of Piperacea family. It is locally called as sirih and betel in English. It is deep green heart shaped of leaves. The vine is dioeciously where male and female plants are different, shade perennial root climber. It has been proved by scientific findings of therapeutic properties in betel leaves that bioactive extract of this plant has significant potential in developing into herbal products. However, lack of literature in processing of this herbal extract found. There are two active phytochemicals found in betel leaves namely hydroxychavicol (HC) and eugenol (EU) which contribute to various beneficial bioactivities. Lab-scale synthesis of these phytochemicals has been reported but the technical information for its mass production is not available. The objectives of this study are to investigate the drying behavior of vacuum drying and heat pump drying of betel leaves on drying kinetics and to evaluate the effects of drying parameters on quality of dried betel leaves. In order to investigate the behavior of betel leaves, vacuum drying was chose to shorten the drying time by lowering the boiling point of water to evaporate at low temperature. Heat pump was chosen due to low relative humidity of the dried air use in heat pump system at low temperature.

As for vacuum drying method, temperatures were set at range from 30 to 70˚C and the vacuum pressure of 0, 38, and 76cmHg. The optimum drying condition for vacuum drying process was determined at temperature of 70˚C under vacuum pressure of 76cmHg. The drying kinetics was successfully modeled using Handerson & Pabis model. For heat pump drying of betel leaves, temperatures were set as 30, 35 (ambient), and 39˚C. The results shows drying characteristics values at temperature of 39˚C and was modeled using Page model.

The assessment of color for dried betel leaves were found that at vacuum pressure plays insignificant changes to the color of dried betel leaves. Color of dried betel leaves generally dependent on the drying temperature applies. The quality of color of dried betel leaves lost as the temperature increased. Same observation was found for dried betel leaves using heat pump drying method. Phytochemical compounds contained in the dried betel leaves were analyzed using high performance liquid chromatography (HPLC) and found that when using vacuum drying process there was significant loss for both EU and HC. For heat pump drying process, EU and HC loss dependent on the temperature applied during drying process.

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

CIRI-CIRI PENGERINGAN DAUN SIRIH PIPER BETLE LINN KERING MENGGUNAKAN KAEDAH PENGERINGAN CANGGIH

Oleh

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Pengerusi : Professor Luqman Chuah Abdullah, PhD
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Piper betle L. kepunyaan genus Piper dan keluarga Piperacea. Di Malaysia, ia dikenali sebagai sirih dan 'betel' dalam Bahasa Inggeris. Ia adalah daun yang berwarna hijau dan berbentuk hati. Penemuan saintifik telah membuktikan hartanah terapeutik dalam ekstrak daun sirih mempunyai potensi yang besar dalam membangunkan produk herba. Walau bagaimanapun, bahan rujukan dalam pemprosesan ekstrak herba jarang ditemui. Terdapat dua fitokimia aktif yang ditemui dalam daun sirih iaitu hydroxychavicol (HC) dan eugenol (EU) yang menyumbang kepada pelbagai manfaat bioaktiviti. Kebanyakan sintesis fitokimia hanya terhad di dalam makmal dan tiada penemuan dilaporkan untuk sintesis secara besar-besaran.

Objektif kajian ini adalah untuk menyiasat tingkah laku pengeringan ke atas kinetic pengeringan daun sirih di bawah keadaan vakum dan pam haba untuk menilai kesan kualiti daun sirih kering melalui pelbagai pemboleh ubah pengeringan. Dalam usaha untuk menyiasat tingkah laku pengeringan daun sirih, pengeringan vakum dipilih untuk memendekkan masa pengeringan dengan menurunkan takat didih air untuk menyejat pada suhu rendah manakala pam haba telah dipilih kerana penggunaan kelembapan relatif yang rendah dalam udara kering suhu yang rendah.

Untuk kaedah vakum pengeringan, suhu telah ditetapkan adalah dalam lingkungan 30˚C hingga 70˚C dan tekanan vakum 0, 38, dan 76cmHg. Keadaan optimum pengeringan untuk proses pengeringan vakum telah ditentukan pada suhu 70˚C di bawah tekanan vakum 76cmHg. Kinetik pengeringan telah berjaya dimodelkan menggunakan model Handerson & Pabis. Untuk pam haba pengeringan daun sirih, suhu telah ditetapkan sebagai 30, 35 (suhu persekitaran), dan 39˚C. Keputusan menunjukkan ciri-ciri pengeringan pada suhu 39˚C adalah yang tertinggi dan telah dimodelkan menggunakan model Page.

Penilaian warna daun sirih kering pada tekanan vakum memainkan perubahan yang tidak ketara kepada warna daun sirih kering. Warna daun sirih kering umumnya bergantung pada suhu pengeringan yang dikenakan. Kualiti warna daun sirih kering hilang apabila suhu meningkat. Pemerhatian yang sama telah dilihat bagi daun kering sirih menggunakan kaedah pengeringan pam haba.

Sebatian fitokimia yang terkandung dalam daun sirih kering dianalisis menggunakan kromatografi cecair berprestasi tinggi (HPLC) dan mendapati bahawa apabila menggunakan proses pengeringan vakum terdapat kehilangan kualiti yang ketara bagi kedua-dua sebatian EU dan HC. Untuk proses pengeringan pam haba, EU dan HC menurun bergantung kepada suhu yang digunakan semasa proses pengeringan.

ACKNOWLEDGEMENT

In the name of Allah, the Most Gracious and the Most Merciful
Alhamdulillah, all praise to Allah for the strengths and His blessing in completing this thesis. Special appreciation dedicated to my supervisor, Professor Dr. Luqman Chuah Abdullah, for his supervision, patience, motivation and immense knowledge throughout my master study. His guidance helped me in all the time of research and writing of this thesis. Not forgotten, I would like to thank the rest of my co-supervisors; Dr. Pin Kar Yong, Professor Dr. Ir. Law Chung Lim, Professor Dr. Ir. Thomas Choong Yaw Shean for their encouragement, insightful knowledge and comments related to my research.

My sincere thanks to staffs from Forest Research Institute Malaysia (FRIM), especially to Tuan Haji Abdul Rashih for his technical assistant and kind-knowledge sharing. My appreciation is also extended to the laboratory technician, Mr. Mohd Zahiruddin Daud from Department of Process and Food Engineering for his helped and assistance.

Last but not least, my deepest gratitude goes to my beloved parents, parents in law and all family members for their endless encouragement and prayers. To my one and only husband, Mohd Johari Abdul Malik, I would like to express my appreciation for his love, understanding and care during the preparation of this thesis. To friends who indirectly contributed in this research, your kindness means a lot to me. Thank you.

I certify that a Thesis Examination Committee has met on **7 June 2013** to conduct the final examination of Puteri Farah Wahida binti Mgt Ahmad on her thesis entitled "Drying Characteristics of *Piper betle* L. using Vacuum and Heat Pump Drying Methods" in accordance with the Universities and University Colleges Act 1971 and the Constituent of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The committee recommends that the student be awarded the Master of Science.

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INTRODUCTION

1.1 Herbal industry in Malaysia

Currently, plants are the preferred sources by the world market as raw material in healthcare products formulation. There is a growing trend of consumers moving from synthetic drugs to herbal cures which are believed to cause lesser side effects. The herbal related market includes herbs which are used in different types of products which are herbal medicines, nutritional supplement, food and food additives and cosmetics ingredients. The herbal industry in Malaysia has reached the value of RM5 billion with an annual growth of 15 to 20 percent (Bernama, 2008).

Most of herbal products are produced by adding a certain amount of active phytochemical extract from the medicinal plants. Active phytochemical extract is referred as extract that possesses therapeutic activities (WHO, 2000). There is a wide range of naturally-occurred phytochemicals found in bioactive extracts. These phytochemicals provide balanced and synergic effects which contribute to the beneficial properties of the extracts. This is the crucial part that is lacking in the single-compound synthetic drugs.

The Malaysian herbal manufacturers have commercialized several herbal products formulated from various medicinal plants such as Kacip Fatimah (*Labisia pumila*), Tongkat Ali (*Eurycoma lonngifolia*) and Misai Kucing (*Orthorsiphon stiminues*) to meet the local demands. Table 1.1 listed some of the most popular medicinal plants and their herbal products that are available in local market and widely accepted by local consumer (Pin, 2009).

Herbal products is known for their remarkable safety profile throughout time and history Although allergic reactions have been recorded for a number of herbs, the general safety profile of many herbs are well recognized. Farnsworth (1993) has concluded that the side effects or toxic reactions associated with herbal medicines in any form are rare. However, consumers should have initiative to investigate the benefits and side effects of the product before use. Soepadma (1992) has reported that there are about 1200 species of medicinal plants found in Malaysia's forest. This lead to a great possibility to develop and produce more beneficial herbal extracts that could be formulated into commercial products.

Table 1.1. Popular medicinal plants and their products in Malaysia

Local name	Scientific name	Product
Tongkat Ali	*Eurycoma longifolia*	Energy drinks Health supplement
Kacip Fatimah	*Labisia pumila*	Herbal tea Coffee
Pegaga	*Centella asiatica*	Health supplement Coffee
Misai Kucing	*Orthosiphon aristatus*	Herbal tea

Mas cotek	*Ficus deltoidea*	Herbal tea
Asam keping /Asam gelugor	*Garcinia atroviridis*	Weight maintenance supplement Herbal tea
Dukung anak	*Andrographis paniculata*	Health supplement
Habbatus Sauda	*Nigella sativa*	Essential oil Health supplement
Neem leaves	*Azadirachta indica*	Health supplement
Mahkota dewa	*Phaleria macrocarpa*	Coffee Health supplement
Mengkudu	*Morinda citrifolia*	Health supplement Juice
Manjakani	*Quercus infectoria*	Health supplement Coffee Juice

(information gathered from local market survey)

1.2 Herbal processing

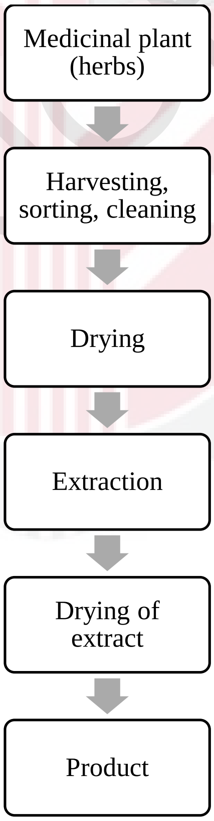

Figure 1.1. The process involved in herbs production

The process involved in producing good quality of herbal product is shown in Figure 1.1. The key processes are drying, extraction, and drying of extract (Ramlan *et al.*,2000). Drying is basically known as preservation process of any product by lowering the amount of moisture content in the materials. Most of the drying techniques apply heat on the raw materials which might cause degradation or loss of quality. The major consideration in herbs drying is to preserve the active phytochemicals of the medicinal plant by shorter the drying time and lowering the drying temperature.

1.3 *Piper betle* Linn

Piper betle L. belongs to genus Piper of Piperacea family. It is locally called as sirih and betel in English. It is deep green heart shaped of leaves. The vine is dioeciously where male and female plants are different, shade perennial root climber. The betel leaves was origin from Malaysia and traditionally used for chewing in their natural raw condition along with many other ingredients like sliced areca nut and slaked lime (Guha 2006).

Figure 1.2. *Piper betle* L. (betel) leaves

Guha (2006) has reported that betel leaves was consumed by about 15 to 20 million people in India. Betel leaves has been cultivated traditionally in India on about 55000 ha with an annual production worth about Rs 9000 million (about RM600 million). Compared to other herbs in Malaysia such as Tongkat Ali, betel leaves has a fast growth rate which mature within 15 to 30 days and are harvested 1 to 4 times every month while Tongkat Ali needs to regrown once harvested because the part used is its root.

1.4 Problem statement

Recently, a lot of studies showed that betel leaves have good potential as supplements in health care and medicinal uses. It has been proved by scientific findings of therapeutic properties in betel leaves that bioactive extract of this plant has significant potential in developing into herbal products. However, lack of literature in processing of this herbal extract found. There are two active phytochemicals found in betel leaves namely hydroxychavicol (HC) and eugenol (EU) which contribute to various beneficial bioactivities of betel leaves including antioxidant, antibacterial, anticancer, and anti-inflammatory. Lab-scale synthesis of these phytochemicals has been reported but the technical information for its mass production is not available. These phytochemicals are mostly obtained from plants. EU which is a type of essential oil has been used in many applications like cosmetics and fragrance.

In publication of (Guha 2006), betel leaves has reported to be spoiled due to dehydration, fungal infection, dechlorophyllation. Since betel leaves is very perishable pants, India estimated the wastage if betel leaves causes about 10% loss per annum which is equivalent to RM60 million.

Drying is the most effective method to preserve the wastage of betel leaves and the quality of it. Few parameters of the drying process should be taken as a consideration in order to preserve the betel leaves such as air temperature, air velocity, relative humidity, and the material to be dried. Most of the drying processes involved high temperature to shorten the drying time, in fact; high temperature caused degradation of valuable compounds contained in plant materials including betel leaves.

1.5 Objectives

1. To investigate the drying behavior of vacuum drying and heat pump drying of betel leaves on drying kinetics.
2. To evaluate the effects of drying parameters on quality of dried betel leaves.

1.6 Scope of research

The scope of this research will cover the two different drying methods which involve vacuum drying and heat pump drying. In order to investigate the behavior of betel leaves, vacuum drying was chose to shorten the drying time by lowering the boiling point of water to evaporate at low temperature. Heat pump was chosen due to low relative humidity of the dried air use in heat pump system at low temperature.

Both drying methods will cover the drying kinetics of the drying process, moisture diffusivity, color and phytochemical quality of dried betel leaves. Drying of raw material is the first step of the processing which has a strong influence on the quality of the extract produced. The effects of drying temperature and pressure used on the quality of betel leaves and drying kinetics were studied in order to determine the optimum drying condition.

The effectiveness of drying methods was evaluated by studying the kinetics. The qualitative and quantitative results were the guiding outline in determining the feasible drying parameters of the drying methods.

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