UNIVERSITI PUTRA MALAYSIA

RHEOLOGICAL PROPERTIES AND OPTIMAL SPRAY DRYING PROCESS PARAMETERS OF PIPER BETLE L. (BETEL) LEAF EXTRACTS COATED WITH DIFFERENT EXCIPIENTS

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By

TEE LEE HONG

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

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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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Chair: Professor Luqman Chuah Abdullah, PhD
Faculty: Engineering

Piper betle L., more commonly known as betel or local name of *Sirih*, belongs to the family *Piperaceae*. Previous research has shown that the leaves of *P. betle* possess tremendous beneficial effects including antimicrobial, antioxidant, anti-diabetic, wound healing and gastro-protective properties. The presence of these beneficial properties indicates that leaf extract of betel has great potential for development into a wide range of health food supplements. However, there is a lack of research on the processing aspects to produce its bioactive component.

This research studied two main aspects including the rheological properties of betel leaves extract and optimization of spray drying process coated with three excipients which were xanthan gum, maltodextrin and lactose. The main purpose for studying the rheological properties of betel leaves is to prepare data that would be useful in designing processing equipment for betel leaves extract products. Spray drying was

chosen for easy handling and the preservation of bioactive compounds. The process parameters of spray drying were inlet hot air temperature, feed flow rate or pump flow rate and aspirator rate. The properties of dried powder produced were investigated in terms of particle size distribution, moisture content and bioactive component; hydroxychavicol (HC) content, hygroscopicity and powder yield. The experimental run and optimization work were designed using Box-Behnken method of Response Surface Methodology (RSM).

Proximate analysis on *Piper betle L.* freeze-dried extract was successfully conducted. The compositions consist of 16.45% moisture, 28.18% ash, 9.36% protein, 0.11% crude fiber, 0.16% crude fat and 45.74% carbohydrate. Rheology behavior of 10 °Brix and 12 °Brix concentration of betel leaves extract at temperature 5, 10, 20, 30, 40 and 50 °C was successfully investigated. Bingham Plastic Model and Casson model illustrated that betel leaves extract showed a plastic behavior. Power Law model was suitable to represent the rheology model of betel leaves extract.

The optimum operation conditions for the highest hydroxychavicol content with the lowest moisture content and the smallest particle size for xantham gum-coated powder were obtained at inlet drying temperature of 160 ˚C, the pump flow rate of 3 rpm, and airflow rate of 86%. The optimum responses of the process were: moisture content of 10.14%, particle size distribution of 7.37 μm, powder yield of 6.72 g, powder hygroscopicity of 26.76 g/100g dry powder and HC content of 383.19 ppm. For maltodextrin-coated spray drying process, the optimum processing parameters were as follow: the inlet drying temperature of 159.53˚C, the pump flow rate of 6.13

rpm, and airflow rate of 98.33%. The optimum responses of the process were: moisture content of 6.99%, particle size distribution of 5.48 μm, powder yield of 10.53 g, powder hygroscopicity of 28.88 g/100g dry powder and HC content of 229.33 ppm. While for lactose-coated spray drying process, the optimum processing parameters were as follow: the inlet drying temperature of 160 ˚C, the pump flow rate of 5.33 rpm, and airflow rate of 100%. The optimum responses of the process were: moisture content of 6.96%, particle size distribution of 6.58 μm, powder yield of 16.54 g, powder hygroscopicity of 16.58 g/100g dry powder and HC content of 80.23 ppm. Xantham gum was found out to be the most suitable carrier for the spray drying process of betel leaves extract as it contained the highest amount of HC content and showed better morphological properties which are more spherical in shape, more consistent in size and less shrinkage present on the surface of the particle.

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

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Oleh

TEE LEE HONG

November 2011

Pengerusi: Profesor Luqman Chuah Abdullah, PhD
Fakulti: Kejuruteraan

Piper betle L., lebih dikenali sebagai nama betel atau nama tempatan Sirih; tergolong dalam keluarga *Piperaceae*. Kajian sebelum ini menunjukkan bahawa daun sirih mempunyai kesan berfaedah termasuk antioksida, antimikrob, anti-diabetes, dan penyembuhan luka. Kehadiran sifat-sifat yang bermanfaat ini menunjukkan bahawa ekstrak daun sirih mempunyai potensi besar dalam pembangunan pelbagai produk.
Tetapi, penyelidikan dalam aspek-aspek pemprosesan untuk menghasilkan komponen bioaktifnya masih tidak mencukupi.

Penyelidikan ini merangkumi dua aspek utama termasuk kajian tentang sifat-sifat reologi ekstrak daun sirih dan pengoptimuman proses pengeringan semburan ekstrak daun sirih yang disalut dengan tiga agen lapisan iaitu xanthan gum, maltodextrin dan laktosa. Tujuan utama untuk mengkaji sifat-sifat reologi daun sirih adalah untuk menyediakan data yang berguna dalam mereka bentuk peralatan pemprosesan untuk

produk ekstrak daun sirih. Pengeringan semburan telah dipilih untuk mengendalimudahkan dan pemeliharaan sebatian bioaktif. Parameter proses pengeringan semburan kajian ini termasuk suhu udara panas, aliran kadar atau kadar aliran pam dan kadar penyedut. Serbuk yang dihasilkan telah dianalisis untuk kandungan sebatian aktif, Hydroxychavicol (HC), taburan saiz zarah, kandungan lembapan, keupayaan untuk menyerap air dan hasil serbuk. Larian eksperimen dan pengoptimuman telah direka bentuk menggunakan kaedah Box Behnken seperti yang disarankan oleh Response Surface Methodology (RSM).

Komposisi ekstrak sirih terdiri daripada kelembapan 16.45%, 28.18% abu, protein 9.36%, 0.11% serat mentah, 0.16% lemak dan karbohidrat 45.74%. Rheologi untuk 10 ° Brix dan 12 ° Brix ekstrak daun sirih pada 5 suhu, 10, 20, 30, 40 dan 50 °C telah berjaya disiasat. Bingham Model Plastik menggambarkan bahawa ekstrak daun sirih menunjukkan kelakuan plastik. Power law tidak sesuai untuk mewakili model rheologi daun sirih ekstrak. Casson model mendedahkan bahawa daun sirih ekstrak mempamerkan sifat plastik.

Keputusan yang diperolehi daripada RSM menunjukkan bahawa process parameter optima; suhu pengering 160 °C, aliran pam kadar 3 rpm, dan kadar aliran udara sebanyak 86%. Respons optima proses ialah: kandungan kelembapan 10.14%, saiz taburan zarah 7.37 µm, hasil serbuk 6.72 g, keupayaan menyerap air, 26.76 g/100g serbuk kering dan kandungan HC, 383.19 ppm. Untuk maltodextrin, parameter pemprosesan yang optima adalah seperti berikut: suhu pengering 159.53 °C, kadar aliran pam 6.13 rpm, dan kadar aliran udara dari 98,33%. Respons optima proses ialah: kandungan kelembapan 6.99%, saiz taburan zarah, 5.48 µm, hasil serbuk 10.53

g, keupayaan menyerap air, 28.88 g/100g serbuk kering dan kandungan HC, 229.33 ppm. Manakala bagi process bersalut dengan laktosa, parameter pemprosesan yang optima adalah seperti berikut: suhu pengeringan 160 °C, kadar aliran pam 5.33 rpm, dan kadar aliran udara sebanyak 100%. Respons optima proses ialah: kandungan kelembapan 6.96%, saiz taburan zarah 6.58 μm, hasil serbuk 16.54 g, keupayaan menyerap air, 16.58 g/100g serbuk kering dan kandungan HC, 80.23 ppm. Xantham gum didapati merupakan agent lapisan yang paling sesuai untuk proses semburan pengeringan daun sirih ekstrak. Dari segi morfologi, ia lebih berbentuk sfera,lebih konsisten dari segi saiz zarah dan tiada pengecutan hadir di permukaannya.

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I certify that an Examination Committee has met on 14th November 2011 to conduct the final examination of Tee Lee Hong on her Master of Science thesis titled "Rheological Properties and Optimization of Spray Drying Process of *Piper betle L.* (Sirih) Leaves Extract" in accordance with the Universities and University Colleges Act 1971 and the Constitution of Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

Members of the Thesis Examination Committee were as follows:

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Degree. 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 dully 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.

TEE LEE HONG

Date: 14 November 2011

TABLES OF CONTENTS