UNIVERSITI PUTRA MALAYSIA

# FORMATION AND CHARACTERISTICS OF ENGKABANG-BASED NANO-COSMECEUTICALS

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# FORMATION AND CHARACTERISTICS OF ENGKABANG-BASED NANO-COSMECEUTICALS

**By**

**SITI SALWA ABD GANI**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirement for the Degree of Doctor of Philosophy**

**July 2010**

DEDICATED TO:

My family: Abah & Emak, my siblings,

my nephews and nieces

For your unwavering support, love and what you mean to me.

Abstract of the thesis presented to the Senate of the Universiti Putra Malaysia in the fulfillment of the requirement for the degree of Doctor of Philosophy

**FORMATION AND CHARACTERISTICS OF ENGKABANG-BASED NANO-COSMECEUTICALS**

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Engkabang fat esters were synthesized from engkabang fat using Lipozyme® RM IM as a catalyst. The main composition of the fat esters were oleyl palmitate, oleyl stearate and oleyl oleate. The percentage yield was 93.67%. Phase behaviors of engkabang fat and engkabang fat esters were determined through the construction of ternary phase diagrams using nonionic surfactants. The increase of hydrophilic lipophilic balance (HLB) value of the surfactant gave larger homogenous and isotropic region in both ternary phase diagrams of Engkabang fat/Nonionic surfactant/Deionized water and Engkabang fat esters/Nonionic surfactant/Deionized water. Homogenous and isotropic regions were larger in the phase diagrams of engkabang wax esters compared to the phase diagrams of engkabang fat. Compositions of homogenous region from the ternary phase diagrams were selected as a pre-formulated cosmeceuticals

emulsions. Then, they were modified with the additions of solubilisant gamma, glycerin, xanthan gum and beeswax in an attempt to get stable formulations at high temperature.

Formulations F10 and E15 were chosen for further studies due to the stability at 45˚C. They were prepared using high shear homogenizer, followed by using high pressure homogenizer. Both formulations were stable at room temperature, at 45ºC and after undergoing thaw cycles test. The particle sizes of F10 and E15 after using high pressure were 115.75 nm and 148.41 nm, respectively. The zeta potential of F10 and E15 at 25ºC were -36.4 mV and -48.8 mV and the pH values were 5.59 and 5.81, respectively. The rheology of F10 and E15 showed pseudoplastic material with shear thinning properties. There were no bacteria and fungus growth in the samples. Short-term moisturizing effect on 20 subjects analyzed by means of Analysis of Variance (ANOVA), gave P-values of $7.35 \times 10^{-12}$ and $2.77 \times 10^{-15}$ for F10 and E15, respectively. The hydration of the skins increased after application of F10 and E15 with P-value below 0.05.

The formulations of encapsulated and non-encapsulated titanium dioxide using engkabang fat and esters were produced by emulsification method using high shear homogenizer. All the formulations were stable after undergoing thaw cycles test, at room temperature and 45ºC for three months except for Formulations F10-4A and F10-6A. The particle sizes of formulations were in

range 80 nm to approximately 400 nm. Surface charge measurements of formulations having values from -30 mV to -59 mV denoted the presence of stable dispersions.

The morphological characterization confirmed the encapsulations of titanium dioxide in the formulations. The rheology analysis of the formulations showed shear thinning property as when the shear rate increased, the viscosity decreased. Formulations containing Engkabang fat could be classified as the thixotropic materials, whereby formulations containing Engkabang fat esters could be classified as pseudo plastic materials under a non-Newtonian fluid. Increasing amount of $TiO_2$ gave higher pH values of the formulations and higher conductivity. The TGA thermograms showed three major weight losses due to the evaporation of water content, evaporation of water content in lattice structure and decomposition of oil phase. The stable formulations containing encapsulated of $TiO_2$ gave higher absorbance compared to the formulations containing non-encapsulated of $TiO_2$.

Abstrak tesis dikemukakan kepada Senat Universiti Putra Malaysia bagi memenuhi syarat untuk mendapatkan ijazah Doktor Falsafah

**PENGHASILAN DAN SIFAT-SIFAT EMULSI BERASASKAN ENGKABANG DALAM BIDANG NANO-KOSMESUTIKAL**

Oleh

**SITI SALWA ABD GANI**

**Julai 2010**

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Ester lemak engkabang disintesis daripada lemak engkabang menggunakan Lipozyme® RM IM. Komposisi utama daripada ester lemak adalah oleyl palmitate, oleyl stearate dan oleyl oleate. Peratusan penghasilan adalah 93.67%. Fasa perilaku lemak engkabang dan ester lemak engkabang ditentukan dengan menggunakan rajah tiga fasa menggunakan surfaktan tidak berion. Peningkatan nilai keseimbangan sifat suka air dan minyak (HLB) surfaktan memberikan fasa homogen dan isotropik yang lebih besar di kedua-dua wilayah diagram tiga fasa lemak engkabang / surfaktan tak berion / air dinyah ion dan lemak ester engkabang / surfaktan tak berion / air dinyah ion. Fasa homogen dan isotropik lebih besar dalam rajah tiga fasa ester lemak engkabang dibandingkan dengan rajah tiga fasa lemak engkabang. Fasa homogen dari diagram tiga fasa dipilih sebagai emulsi awal kosmesutikal.

Kemudian, formulasi itu diubahsuai dengan penambahan solubilisant gamma, glycerin, xanthan gum dan beeswax untuk mendapatkan formulasi yang stabil pada suhu tinggi.

Formulasi engkabang mengandungi lemak dan ester lemak engkabang, F10 dan E15, menggunakan mesin pengemulsi, diikuti dengan menggunakan mesin pengemulsi bertekanan tinggi. Kedua-dua formulasi tersebut stabil pada suhu bilik (25ºC), 45º C dan selepas menjalani ujian kitaran mencair. Saiz zarah F10 dan E15 selepas menggunakan mesin pengemulsi bertekanan tinggi ialah 115.75 nm dan 148.41 nm. Nilai zeta F10 dan E15 pada 25ºC adalah-36.4 mV dan-48.8 mV dan nilai pH 5.59 dan 5.81 untuk formulasi F10 dan E15. Sifat aliran F10 dan E15 menunjukkan ciri-ciri pseudo-plastik dengan sifat-sifat luncur menipis. Tiada pertumbuhan bakteria dan kulat dalam kedua-dua formulasi. Ujian jangka pendek kesan kelembapan kulit telah dijalankan ke atas 20 subjek dan dianalisis dengan menggunakan Analisis varians (Anova), memberikan nilai-P $7.35 \times 10^{-12}$ and $2.77 \times 10^{-15}$ bagi F10 and E15. Kelembapan kulit meningkat selepas penggunaan F10 dan E15 dengan nilai-*P* di bawah 0.05.

Formulasi mengandungi enkapsulasi dan tidak enkapsulasi titanium dioksida menggunakan lemak dan ester engkabang dihasilkan melalui kaedah pengemulsian menggunakan mesin pengemulsi bertekanan tinggi. Semua formulasi adalah stabil selepas menjalani ujian kitaran mencair, pada suhu bilik

dan 45ºC selama tiga bulan, kecuali formulasi F10-4A dan F10-6A. Saiz zarah formulasi adalah antara 80 nm ke 400 nm. Pengukuran cas permukaan formulasi menunjukkan nilai daripada -30 mV ke-59 mV mengesahkan kestabilan formulasi-formulasi tersebut.

Gambar TEM mengesahkan bahawa titanium dioksida berjaya diencapsulasi di dalam formulasi. Analisis sifat aliran formulasi-formulasi yang dihasilkan menunjukkan sifat-sifat luncur menipis dengan kelikatan menurun apabila kelajuan meluncur bertambah. Formulasi engkabang yang mengandungi lemak boleh diklasifikasikan sebagai bahan thixotropic dan formulasi mengandungi ester lemak engkabang boleh diklasifikasikan sebagai bahan pseudo plastik di bawah bendalir non-Newtonian. Peningkatan jumlah $TiO_2$ memberikan nilai pH dan. konduktiviti lebih tinggi pada formulasi. Gambar rajah haba TGA menunjukkan tiga pengewapan berlaku; pengewapan air, pengewapan air dalam struktur kisi dan penguraian fasa minyak. Formulasi stabil yang mengandungi titanium dioksida yang dienkapsulasi menunjukkan penyerapan UV yang tinggi berbanding dengan formulasi yang mengandungi titanium dioksida yang tidak dienkapsulasi.

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I certify that a Thesis Examination Committee has met on **1st July 2010** to conduct the final examination of **Siti Salwa Abd Gani** on her thesis entitled **"Formation and Characteristics of Engkabang-based Nano-Cosmeceuticals**" in accordance with Universities and Universities Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998.The committee recommends that the student be awarded the Doctor of Philisophy Degree.

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## DECLARATION

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

_____

**(SITI SALWA ABD GANI)**

Date:

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