

**ASSOCIATION BEHAVIOUR AND PHYSICOCHEMICAL PROPERTIES
OF DIHYDROXYSTEARIC ACID AND OCTYL DIHYDROXYSTEARATE,
AND THEIR APPLICATIONS IN COSMETICS**

By

MARISOL HAINNY BINTI MOHAMED SOPIAN

**Thesis Submitted to the School of Graduate Studies,
Universiti Putra Malaysia, in Fulfilment of the Requirement for the
Degree of Master of Science**

June 2006

DEDICATIONS

Specially goes to:

My dad, Mohd Sopian Jusoh

My mom, Maria Abdullah

My Husband, Sheik Azrif Bux

My Brother, Mohd Bayani Affendy

' Thank you for the support and strength'

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

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Chairman : Professor Anuar Kassim, PhD

Faculty : Science

The phase behaviour in ternary system had been studied at 85°C under visual observation by polarizing light and they were confirmed by polarization microscope. It was found spherulite and needles type of crystal for DHSA and Octyl dihydroxystearate respectively. The polymorphs were typically distinguished by differences in their X-ray spectra using X-ray diffraction spectroscopy. Polymorph DHSA and Octyl dihydroxystearate in MCT exist as beta prime (β') and beta (β). The results also showed that mixture of DHSA/ Octyl dihydroxystearate in different type of third component (MCT and ethanol) may possess only stable β' form, only stable β form or both β and β' form.

A rheological analysis showed the deformation and flow behaviour of DHSA / Octyl dihydroxystearate and MCT respectively. From hysteresis value, the correlation between the crystal polymorph and decreasing of ratio DHSA plays an important role with the structure breakdown. Instability of polymorph gave vary and unpredictable viscosity value to the mixture of DHSA and Octyl dihydroxystearate. Study of frequency sweep gave idea viscoelastic properties of DHSA and Octyl dihydroxystearate in MCT. Most samples gave result of storage modulus was always higher than the loss modulus ($G' > G''$). Loss modulus higher than storage modulus ($G'' > G'$) for 30:70 of DHSA/ Octyl dihydroxystearate at 85% MCT.

Their needle and spherulite microstructures, which confer a brittle or smooth feel, were useful in the formulation of concealer stick. The concealer sticks were prepared by adding DHSA, Octyl dihydroxystearate, and MCT as main components combined with fillers, pigment, binders and fragrance. The concealer stick with different ratios of DHSA and Octyl dihydroxystearate gave different results in its stability. The stability test included breaking point, heat test, hardness test, slip melting point, softening point and humidity.

Formulation at 20:80 DHSA/ Octyl dihydroxystearate was chosen as the best formulation due to the physical properties previously which consist of β polymorph densely packed spherulite crystal. When compared to commercial, this formulation showed a better result in terms of softness and strength effect of the sticks.

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

**KELAKUAN PENYEKUTUAN DAN SIFAT FIZIK-KIMIA ASID
DIHIDROKSISTEARIK DAN OKTIL HIDROKSISTEARAT DAN
PENGGUNAANNYA DALAM KOSMETIK**

Oleh

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Pengerusi : Profesor Anuar Kassim, PhD

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Kajian sistem tiga fasa telah dilakukan pada suhu 85°C dengan menggunakan cahaya berkutub dan mikroskop berkutub. Dua jenis kristal wujud bagi asid dihidroksistearik (DHSA) dan Oktil dihydroksistearat iaitu sperulit dan jejarum. Polimorf DHSA dan Oktil dihidroksistearat dapat ditentukan oleh pembelauan X-ray. Keputusan menunjukkan polimorf asid dihidroksistearik (DHSA) dan Oktil dihidroksistearat dalam MCT wujud sebagai beta prime (β') and beta (β). Campuran pelbagai nisbah asid dihidroksistearik (DHSA) dan Oktil dihidroksistearat dalam kepekatan bahan ketiga (MCT dan etanol) mungkin wujud dalam β' atau β sahaja dan mungkin juga β dan β' .

Kajian reologi menunjukkan sifat aliran dan pencacatan bagi asid dihidroksistearik (DHSA) dan Oktil dihidroksistearat. Daripada nilai histerisis,

hubung kait antara polimorf dan pengurangan nisbah DHSA memainkan peranan yang penting dalam pemecahan struktur. Ketidakstabilan polimorph turut memberikan nilai kepekatan yang tidak tersusun dan susah dijangka. Kajian terhadap pergerakan frekuensi memberikan ciri viskoelastik bagi asid dihidroksisterik (DHSA) dan Oktil dihidroksistearat. Kebanyakannya menunjukkan modulus 'simpan' lebih tinggi daripada kehilangan modulus ($G' > G''$), Tetapi bagi sampel DHSA dan Oktil dihidroksistearat 30:70 (85% MCT) menunjukkan kehilangan modulus lebih tinggi berbanding modulus 'simpan' ($G'' > G'$).

Mikrostruktur sperulit dan jejaram yang memberikan ciri halus dan rapuh amat sesuai untuk kegunaan dalam konseler. Formulasi konseler ini disediakan dengan menggunakan DHSA, Oktil dihidroksistearat dan MCT sebagai bahan asas bersama dengan pigmen, pengisi, bahan pengikat campuran dan wangian. Keputusan menunjukkan kepelbagaian dalam ujian kestabilan. Ujian kestabilan tersebut adalah ujian titik pemecahan, ujian tahan haba, ujian kekerasan, ujian titik kelembutan, ujian titik pengelinciran pencairan dan ujian kelembapan.

Formulasi bagi 20:80 DHSA/ Oktil dihidroksistearat telah dipilih sebagai formulasi yang terbaik berasaskan kepada sifat kimia-fiziknya yang terdiri daripada sperulit β kristal yang padat. Apabila dibandingkan dengan dengan produk komersial ianya lebih baik dari segi struktur yang lebih lembut dan kuat pada konselar tersebut.

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I certify that an Examination Committee met 16th June 2006 to conduct the final examination of Marisol Hainny Binti Mohamed Sopian on her Master of Science thesis entitled “Association Behaviour and Physicochemical Properties of Dihydroxystearic Acid and Octyl Dihydroxystearate and Their Application in Cosmetic” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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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 UPM or other institutions.

MARISOL HAINNY BINTI MOHAMED SOPIAN

Date: 18 December 2006

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