

**ENZYMATIC SYNTHESIS OF PALM-BASED FERULATE ESTER  
AS SUNSCREEN AGENT**

**By  
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**Chairman: Professor Mahiran Basri, PhD**

**Faculty: Science**

The invention of modifying palm oils to produce sunscreen agent, ferulate ester (RBD-FE) had been investigated through transesterification reaction of RBD palm olein with ferulic acid (4-hydroxy-3-methoxy-cinnamate, FA) by enzymatic synthesis. Ferulic acid is a natural substance which has free radical scavenging effects and helps to prevent damage to our cells caused by UV light. However, incorporation of ferulic acid into cosmetic compositions is problematic due to its instability in aqueous solution and limited solubility in water in oil emulsions. Besides, currently used sunscreen agent in 90 percent of all sunscreen products throughout the world; octyl methoxycinnamate (OMC) is reported could be toxic to human skin.

This process has been studied as one approach in order to overcome these problems to provide a natural cosmetic ingredient with enhanced efficiency and stability. It combines the ultraviolet-absorbing properties of a cinnamate (ferulic acid) with the water-insoluble properties of a lipid (RBDPO) resulting in ultraviolet-absorbing lipids (RBD-FE) made from all natural ingredients under mild, solvent-free conditions to ensure a safe, clean and cosmetically acceptable product.

Enzyme screening revealed Novozym 435 to be the most efficient biocatalyst for the reaction and was chosen for optimization studies. The important parameters that may affect the synthesis of RBD-FE had been investigated. The corresponding optimal conditions for the transesterification reaction was time, 96 h; temperature, 40 °C; mole ratio of ferulic acid: RBDPO, 1:3; and amount of enzyme used, 0.10 g resulting in highest percentage of conversion (24.51%) to ultraviolet-absorbing lipids (RBD-FE) that works as a sunscreen agent.

Identities of product were verified using TLC, gas chromatography (GC), Fourier transform infrared spectroscopy (FT-IR), High Performance Liquid Chromatography (HPLC), GC-Mass Spectroscopy (GC-MS) and Nuclear Magnetic Resonance (NMR) Spectroscopy analyses. Thermal and oxidative stability of the RBD-FE were also evaluated. Later, palm-based ferulate ester was also formulated into cosmetic formulations to evaluate it as sunscreen agent followed by Sun Protection Factor (SPF) analysis.

RBD-FE showed higher thermal stability and better oxidative stability. Besides, it became an active ingredient in sunscreen formulation which acted as a broadband sunscreen agent which covers both UVA and UVB rays. It showed a wider light absorption peak between 290 and 400nm.

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

**SINTESIS BERENZIM ESTER FERULATE BERASASKAN MINYAKSAWIT  
SEBAGAI AGEN PENABIR SURIA**

Oleh

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Usaha untuk mengubahsuai minyak sawit untuk menghasilkan agen penabir suria, ester ferulate (RBD-FE) telah dikaji melalui proses penginteresteran antara olein sawit dan asid ferulik dengan menggunakan enzim. Asid ferulik adalah komponen semulajadi yang mempunyai kesan mengaut radikal bebas dan mencegah kerosakan ke atas sel-sel yang disebabkan oleh cahaya ultra-lembayung. Namun, penggunaan asid ferulik di dalam komposisi kosmetik adalah bermasalah disebabkan oleh ketidakstabilannya dalam larutan akueus dan keterlarutan terhad dalam emulsi air dalam minyak. Selain itu, agen penabir suria yang digunakan hampir 90 % di merata dunia; oktil metoksisinamat (OMC) dilaporkan adalah toksik ke atas kulit manusia.

Proses ini telah dikaji sebagai satu usaha untuk mengatasi masalah-masalah tersebut untuk menghasilkan satu bahan kosmetik yang semulajadi yang lebih berkesan dan stabil. Ia menggabungkan sifat penyerap ultra-lembayung dari sinamat dengan sifat kalis air lemak (RBD olein sawit), menghasilkan lemak penyerap ultra-lembayung (RBD-FE) yang diperbuat daripada bahan semula jadi dalam keadaan sederhana, lembut dan tanpa kehadiran pelarut untuk memastikan penghasilan produk yang selamat, bersih dan diterima secara kosmetik.

Penyaringan enzim menunjukkan bahawa Novozym 435 adalah biomangkin yang paling cekap dalam tindak balas tersebut lalu dipilih untuk kajian pengoptimuman. Parameter utama yang mungkin mengganggu sintesis RBD-FE telah dikaji. Tindak balas penderaman berlangsung dalam keadaan tanpa pelarut selama 96 jam pada suhu 40°C dengan menggunakan 0.1 g enzim Novozym 435 pada nisbah 1:1 bahan tindak balas, memberikan hasil peratusan tertinggi (24.51%) menghasilkan lemak penyerapan lembayung ultra-unggu (RBD-FE) yang bertindak sebagai agen penabir suria.

Identiti produk tersebut telah disahkan melalui teknik TLC, kromatografi gas (GC), spektroskopi inframerah tertransformasi-Fourier (FT-IR), Kromatografi Cecair perlaksanaan tinggi (HPLC), kromatografi gas-spektroskopi jisim (GC-MS) dan analisis resonans magnet nuklues (NMR). RBD-FE dikaji untuk mengetahui sifat-sifat seperti kestabilan haba, dan nilai peroksida. Selain itu, RBD-FE juga dimasukkan ke dalam formulasi barangan kosmetik untuk menilai sifatnya sebagai penabir cahaya matahari dan faktor pelindung matahari (SPF).

RBD-FE berasaskan minyak sawit menunjukkan kestabilan terhadap haba dan pengoksidaan. Malah, ia juga bertindak sebagai bahan aktif dalam formulasi krim penabir matahari dengan memberikan perlindungan yang baik dalam julat UVA and UVB. Ia menunjukkan puncak penyerapan cahaya yang luas antara 290 dengan 400nm.

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I certify that an Examination Committee has met on 9 May 2006 to conduct the final examination of Lim Sheo Kun on her Master of Science thesis entitled "Enzymatic Synthesis of Palm-Based Ferulate Ester as Sunscreen Agent" 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.

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**LIM SHEO KUN**

Date: 20 June 2006

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