

# **UNIVERSITI PUTRA MALAYSIA**

## PRODUCTION AND CHARACTERISATION OF DIACYLGLYCEROLS FROM VARIOUS EDIBLE OIL DEODORISER DISTILLATES BY LIPASE-CATALYSED ESTERIFICATION

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By

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Diacylglycerols (DAG) are minor acylglycerols (< 10% w/w) found in edible oils and fats. Recently, 1,3-DAG has been found to prevent and manage obesity. Consequently, the novel industrial production process of 1,3-DAG was patented worldwide (US 2001/0004462) by Kao Corporation, Japan. In this work, an alternative process to produce 1,3-DAG was discovered. The process comprised of esterifying free fatty acids from edible oil deodoriser distillates with glycerol by using 1,3-position specific lipases. Deodoriser distillates from the processing of palm, soybean, canola and corn oils were used.

The effects of esterification reaction parameters such as the source of 1,3position specific lipase, reaction time, lipase concentration, reaction temperature, total fatty acid to glycerol molar ratio, water and molecular sieves contents were conducted. *Rhizomucor miehei* lipase (Lipozyme<sup>®</sup> RM IM) was found to be the best performing lipase. The reaction time required for optimum production of DAG is at 6 h for palm oil deodoriser distillate, and 4 to 5 h for soybean, canola and corn oil deodoriser distillates. The following reaction parameters resulted in optimum yield of DAG: 10% (w/w) of Lipozyme<sup>®</sup> RM IM, reaction temperature of 65  $^{\circ}$ C, total fatty acid to glycerol molar ratio of 2.5: 1, total absence of water in the substrates, and the presence of molecular sieves of 30% (w/w). DAG yield of 60 to 72% and DAG purity of 82 to 85% were obtained.

DAG produced from the deodoriser distillates and their various blends were characterised for their fatty acid and DAG compositions, iodine values (IV), thermal profiles, and slip melting points (SMP). The analytical results indicated that DAG produced from palm oil deodoriser distillate had lower degrees of unsaturation and, therefore, had higher melting points compared with that from soybean, canola and corn oil deodoriser distillates. DAG produced from soybean oil deodoriser distillate had the highest degree of unsaturation.

In the DAG produced from soybean, canola, and corn oil deodoriser distillates,  $\beta$ -sitosterol, campesterol and stigmasterol were the dominant phytonutrients, while tocotrienols were the major phytonutrient in the DAG produced from palm oil deodoriser distillate. The phytosterol and vitamin E compositions in the DAG produced from the various blends varied according to the proportion of the distillate used.

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

#### PENGHASILAN DAN PENCIRIAN DIASILGLISERIDA DARIPADA SULINGAN PENYAHBAU DENGAN MENGGUNAKAN ESTERIFIKASI BERMANGKIN LIPASE

Oleh

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Diasilgliserida (DAG) boleh didapati secara semulajadi dalam kuantiti yang kecil (< 10% b/b) di dalam kebanyakan minyak dan lemak yang boleh dimakan. Baru-baru ini, 1,3-DAG telah didapati boleh mencegah dan mengawal obesiti. Dengan itu, satu proses penghasilan 1,3-DAG secara industri telah dipatenkan di seluruh dunia (US 2001/0004462) oleh syarikat Kao Corporation, Jepun. Dalam kajian ini, satu proses alternatif untuk menghasilkan 1,3-DAG telah ditemui. Proses ini melibatkan esterifikasi asid lemak yang terdapat pada sulingan penyahbau dan gliserol dengan menggunakan lipase khusus posisi 1,3. Sulingan penyahbau daripada pemprosesan minyak-minyak kelapa sawit, kacang soya, kanola dan jagung telah digunakan.

Kajian tentang kesan parameter tindakbalas esterifikasi seperti sumber lipase khusus berposisi 1,3, masa tindakbalas, kepekatan lipase, suhu tindakbalas, nisbah molar jumlah asid lemak kepada gliserol, kandungan air dan penapis molekul telah dilakukan. Lipase *Rhizomucor miehei* (Lipozyme<sup>®</sup> RM IM) merupakan lipase yang terbaik. Masa tindakbalas yang diperlukan untuk penghasilan DAG yang optima adalah 6 jam untuk sulingan penyahbau minyak kelapa sawit, dan 4 hingga 5 jam untuk sulingan penyahbau minyak-minyak kacang soya, kanola dan jagung. Kajian ini juga mendapati bahawa parameter tindakbalas berikut menyebabkan penghasilan DAG yang optima: Lipozyme<sup>®</sup> RM IM 10% (b/b), suhu tindakbalas 65 <sup>o</sup>C, nisbah molar jumlah asid lemak kepada gliserol pada 2.5: 1, ketidakhadiran air pada substrat, dan kehadiran 30% (b/b) penapis molekul. Hasil DAG antara 60 hingga 72% dan tahap ketulenan DAG di antara 82 hingga 85% telah didapati.

Komposisi asid lemak dan DAG, nilai iodin, profil terma, dan takat lebur telah digunakan untuk mencirikan DAG yang dihasilkan daripada sulingan penyahbau dan campuran sulingan. Hasil kajian menunjukkan bahawa DAG yang dihasilkan daripada sulingan penyahbau minyak kelapa sawit mengandungi tahap ketidaktepuan yang rendah dan, oleh itu, mempunyai takat lebur yang lebih tinggi berbanding dengan DAG yang dihasilkan daripada sulingan penyahbau minyakminyak kacang soya, kanola dan jagung. DAG yang dihasilkan daripada sulingan penyahbau minyak kacang soya mempunyai tahap ketidaktepuan yang tertinggi.

Dalam DAG yang dihasilkan daripada sulingan penyahbau minyak-minyak kacang soya, kanola dan jagung,  $\beta$ -sitosterol, kampesterol dan stigmasterol adalah fitonutrien yang utama, manakala tokotrienol adalah fitonutrien yang utama di dalam DAG yang dihasilkan daripada sulingan penyahbau minyak kelapa sawit. Komposisi fitosterol dan vitamin E dalam DAG yang dihasilkan daripada campuran sulingan didapati berubah mengikut nisbah sulingan yang digunakan.

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