

**CHEMICAL INTERESTERIFICATION OF REFINED BLEACHED AND
DEODORISED PALM OLEIN WITH PALM METHYL ESTER**

By

THANG YIN MEE

**Thesis Submitted to the School of Graduate Studies, Universiti Putra
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Science**

July 2004

*For my wonderful papa and mama, brother, sisters & Bryan
with love....*

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

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Chairman : Professor Dzulkefly Kuang Abdullah, Ph.D.

Faculty : Science and Environmental Studies

Chemical interesterification using refined, bleached, and deodorised palm olein (POo) as feedstock with fractionated high iodine value palm methyl ester (ME) to produce liquid oil with high iodine value (IV) as well as high monounsaturation was studied. The interesterification has been conducted under different reaction conditions, including ratio of POo to ME, type of catalyst (Na versus NaOMe), catalyst dosage, reaction temperature, and quality of ME (high peroxide value versus low peroxide value) to develop optimum reaction conditions for maximum conversion to increase the unsaturation of the interesterified oil. The reaction is allowed to proceed for two hours before termination. Samples were taken frequently during the course of reaction and subjected to measurement of triacylglycerol (TAG) and diacylglycerol (DAG) distribution. The catalytic performance of interesterification was evaluated by determining changes in the TAG and DAG compositions by reverse-phase high performance liquid

chromatography (HPLC), and fatty acid composition by gas chromatography (GC).

After interesterification, the compositions of TAGs in the interesterified oil, such as dilinoleoyl oleoyl glycerol (OLL), dioleoyl linoleoyl glycerol (OLO), and tryoleoyl glycerol (OOO) increased from initial value of 0.4 mole % to 0.6-4.4 mole%, 1.7 mole % to 2.5-16.3 mole %, and 3.7 mole % to 5.2-21.0 mole%, respectively. While the dipalmitoyl linoleoyl glycerol (PLP) and dipalmitoyl oleoyl glycerol (POP) decreased after 2 hours reaction. Interesterified oils obtained contained about 2.0-9.7 mole % of PLP compared to 10.0 mole % in POo and about 6.7-28.2 mole % of POP compared to 30.3 mole % in POo. Meanwhile, the relative concentration of DAG was also changed after the reaction. The relative concentration of DAG of interesterified oils was in the range of 0.6 mole % to 16.6 mole % compared to 7.6 mole % in starting POo (P, O, and L represented palmitic, oleic, and linoleic acid, respectively)

An interesterified oil which simulates olive oil has been made from POo. The product obtained from interesterification of POo with low peroxide value methyl ester (LPVME) at optimum condition (POo/LPVME ratio of 1:1.4, using 0.1 % w/w NaOMe catalyst at 100°C) has been subjected to dry fractionation and compositions of the fractionated products have been determined by using HPLC and GC. The physical properties of the fractionated products have also been investigated using various standard techniques. Solid fat content (SFC) and

thermal behaviour of fractionated products have been investigated using nuclear magnetic resonance (NMR) and differential scanning calorimetry (DSC). Other methods, such as slip melting point (SMP), cloud point, crystallisation temperature, cold test, cold stability, and oxidative stability test have also been conducted. The physical properties of fractionated products have been compared with those of olive oil.

After fractionated, oleic acid content of the liquid fractions, interesterified oleins increased from 55.9 % before fractionation to 58.8-62.6 % and iodine value of 81.2-88.3. Generally, the products obtained from interesterification followed by fractionation contained higher oleic acid compared to that of starting POo (42.0 %). Therefore, interesterification process followed by fractionation has improved the unsaturation of a palm oil product as well as monounsaturated content, which is comparable to olive oil. Moreover, this product contained lesser DAG content compared to normal POo.

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

**INTERESTERIFIKASI KIMIA BAGI OLEIN MINYAK SAWIT DISULING
DILUNTUR DAN DINYAHBAU DENGAN METIL ESTER KELAPA SAWIT**

Oleh

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Julai 2004

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Interesterifikasi kimia bagi olein minyak sawit “disuling, diluntur, dan dinyahbau” (POo) dengan metil ester minyak sawit tak tepu (ME) untuk menghasilkan minyak yang mempunyai nilai iodin (IV) yang lebih tinggi, khususnya lebih tinggi dari segi monoketaktepuan telah dikaji. Interesterifikasi telah dijalankan dengan menggunakan pembolehubah-pembolehubah, seperti nisbah POo kepada ME, jenis mangkin (Na dan NaOMe), dos mangkin, suhu, dan kualiti ME (nilai peroksida tinggi dan nilai peroksida rendah) untuk mendapatkan keperluan optimum untuk meningkatkan ketaktepuan olein sawit. Tindak balas telah dijalankan selama dua jam sebelum tindak balas diberhentikan. Sampel telah dikeluarkan sepanjang tindak balas dan kandungan trigliserida (TAG) dan digliserida (DAG) sampel telah diuji. Perlaksanaan interesterifikasi bermangkin telah dinilai berdasarkan perubahan kepekatan TAG dan DAG dengan menggunakan kromatografi cecair prestasi tinggi fasa berbalik (HPLC) dan komposisi acid lemak dengan menggunakan kromatografi gas (GC).

Selepas interesterifikasi, kepekatan relatif bagi trigliserida-trigliserida yang merangkumi dilinoleoil oleoil gliserol (OLL), dioleoil linoleoil gliserol (OLO), dan trioleoil gliserol (OOO) telah bertambah daripada 0.4 % mol hingga 0.6-4.4 % mol, 1.7 % mol hingga 2.5-16.3 % mol, dan 3.7 hingga 5.2-21.0 % mol, masing-masing. Sementara itu, trigliserida-trigliserida yang terdiri daripada dipalmitoil linoleoil gliserol (PLP) dan dipalmitoil oleoil gliserol (POP) yang terdapat dalam POo telah berkurangan selepas interesterifikasi. Produk yang diperolehi mengandungi sebanyak 2.0-9.7 % mol PLP berbanding dengan 10.0 % mol dalam POo, dan sebanyak 6.7-28.2 % mol POP berbanding dengan 30.3 % mol dalam POo. Di samping itu, kepekatan relatif digliserida bagi POo juga didapati berubah selepas interesterifikasi. Kepekatan relatif digliserida bagi produk interesterifikasi telah diberi nilai di antara 0.6 % mol hingga 16.6 % mol berbanding dengan POo (7.6 % mol). Komponen: P, O, dan L mewakili asid palmitik, oleik, dan linoleik, masing-masing.

Percubaan untuk menghasilkan produk menyerupai minyak zaitun daripada interesterifikasi POo telah dijalankan. Sementara itu, produk daripada interesterifikasi antara POo dengan metil ester bernilai perosida rendah (LPVME) yang menggunakan parameter optimum (POo/LPVME dalam ratio 1:1.4 dengan menggunakan 0.1% w/w NaOMe pada 100°C) telah dijalankan secara pemeringkatan kontang. Komposisi produk pemeringkatan telah dijalankan dengan menggunakan HPLC dan GC. Di samping itu, ciri-ciri fizikal produk juga diuji dengan menggunakan pelbagai teknik. Kandungan pepejal lemak (SFC)

dan sifat termal telah dikaji dengan menggunakan resonans nuklear magnetik (NMR) dan perbezaan pengimbasan kalorimetri (DSC). Teknik lain, seperti takat lebur gelincir, takat mendung, takat penghabluran, ujian dingin, ketahanan dingin, dan ketahanan oksidatif telah diuji. Ciri-ciri fizikal produk pemeringkatan dibanding dengan minyak zaitun.

Selepas pemeringkatan, kandungan asid oleik bagi olein interesterifikasi telah bertambah daripada 55.9 % sebelum pemeringkatan kepada 62-58.8 % dan bernilai iodin 81.2-88.3. Secara relatif, produk daripada interesterifikasi berikutan pemeringkatan mengandungi kandungan asid oleik yang lebih tinggi daripada POo (42.0 %). Proses interesterifikasi berikutan pemeringkatan telah meningkatkan ketaktepuan produk berasaskan minyak sawit bukan hanya dari segi monoketaktepuan, yang mana ia sebanding dengan minyak zaitun. Tambahan, produk ini mengandungi digliserida yang lebih rendah berbanding dengan POo biasa.

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I certify that an Examination Committee met on 8 July 2004 to conduct the final examination of Thang Yin Mee on her Master of Science thesis entitled “ Chemical Interesterification of Refined Bleached and Deodorised Palm Olein with Palm Methyl Ester” 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.

THANG YIN MEE

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