UNIVERSITI PUTRA MALAYSIA

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By

SAMI SAADI

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

December 2010

DEDICATION

This thesis is dedicated to my beloved parents, brothers, and sisters; to all members of my big family SAADI. It also goes to teachers, scientists, researchers, and all seekers for knowledge.

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

CHARACTERIZATION OF PALM-BASED BINARY FAT BLENDS AND DEVELOPMENT OF MARGARINE FROM THE BLENDS

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Chairman : Associate Professor Abdul Azis Ariffin, PhD

Faculty : Food Science and Technology

The main objectives of this study were: (1) to determine the effect of stearin excess and small dose of monoacylglycerol (an emulsifier) on thermal behavior, solid fat content and microstructure properties of PO based margarine fats, and (2) to assess the changes in the physicochemical and rheological properties of water-in-oil (w/o) emulsion systems during the storage time taking *multipurpose margarine* (MPM) as an example.

The ability of palm oil (PO) to crystallize as beta prime polymorph has made it as an attractive option for the production of margarine fat (MF). Palm stearin (POs) expresses similar crystallization behavior, and is considered one of the best substitutes of hydrogenated oils due to its capability to impart the required level of plasticity and body to the finished product. Normally, POs is blended with PO to reduce the melting point at

body temperature (37°C), Lipid phase, formulated by PO and POs in different ratios, were subjected to an emulsification process and the following analyses were done: triacylglycerol (TAG), solid fat content (SFC) and thermal behavior. In addition, iodine value (IV), fatty acid composition (FAC) and rheological properties including viscosity (η) and shear stress (τ) were determined in before emulsification process (BEP), while the microstructure properties including size and number of crystals were also determined for experimental and commercial margarine fats (EMF and CMF) after emulsification process (AEP).

Results showed that blending and emulsification at POs level over 40% (w/w) were significantly changed the physicochemical and microstructure properties of EMF as compared to CMF, resulting in a desirable PPO/POP% of less than 36.1%. SFC at 37 ºC, crystal size, crystal number, crystallization and melting enthalpies (ΔH) were 15%, 5.37µm, 1425 crystal/µm^2, 17.25 J/g and 57.69 J/g, respectively. Differential scanning calorimetry (DSC) analysis of blends indicated significant effect on temperature transition; peak sharpness and enthalpy at POs level more than 40% (w/w). The continuous incorporation of POs in the fat matrix system of lipid phase of BEP and AEP showed the apparition of new peaks at high temperature level ranging from 50 to 56 °C. These peaks were attributed to the higher melting compounds of TAGs and the occurrence of polymorphic transition. The pNMR showed the formation of eutectic systems at POs over 40% (w/w), resulting in low level of SFC of less than 15% at body temperature (37 °C). All data reported indicate that the formation of granular crystals in margarine fat was dominated by high melting TAG namely PPO/POP, whilst the small dose of monoacylglycerol that is used as emulsifier slowed the emergence of undesirable polymorph in EMF as compared to CMF.

The assessment of binary blend behavior of PO/POs based water-in-oil (w/o) emulsions during the storage time showed significant changes for total physicochemical and rheological properties of (w/o) emulsion types, resulting in SFC at 28 °C, consistency, softness and storage modulus (G') of less than 25%, 16 Kg f/cm^2, 30 mm ease of cone penetration and 15x10^4 Pa, respectively. These data provide an indication on the weakness structure network and low workability force of the MPM models over 40% (w/w) of POs. Examination microscopy of the images revealed that the incorporation of PS levels more than 40% (wt./wt.) have the ability to readily transform beta prime polymorph (β') to beta (β), while the reduction of PS over 40% (wt./wt.) slowed down the emergence of β polymorph. Meanwhile, PV and FFA of 2 Meq O_2/Kg and 0.35% respectively, screened excellent oxidative stability and high resistance against acidity and rancidity of multipurpose margarine (MPM). This oxidative stability in MPM models during the storage time may explain by the presence of an optimal average of saturated bonds that made them to be chemically more stable against oxidative deterioration.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah master sains

PENCIRIAN CAMPURAN LEMAK BERASASKAN KELAPA SAWIT SECARA DEDUA DAN PENGHASILAN MARJERIN DARIPADA CAMPURAN TERSEBUT

Oleh

SAMI SAADI

Dicember 2010

Pengerusi : Profesor Madya Abdul Azis Ariffin, PhD

Falkuti : Sains dan Teknologi Makanan

Objektif utama kajian ini adalah untuk: (1) menentukan kesan dos stearin berlebihan dan dos monoasigliserol (pengemulsi) yang sedikit terhadap perilaku terma, kandungan lemak pepejal dan sifat struktur mikro sistem yang berasaskan PO (lemak mentega), dan (2) menentukan perubahan sifat fisikokimia sistem emulsi air-dalam-minyak (w/o) sepanjang waktu simpanan *(Contoh: mentega serbaguna)*.

Keupayaan PO untuk menghablur membentuk polimorf beta primer telah menjadikannya sebagai pilihan yang menarik untuk penghasilan lemak marjerin (MF). POs menunjukkan sifat penghabluran yang sama, dan ini dianggap sebagai salah satu pengganti minyak terhidrogen yang terbaik disebabkan keupayaannya untuk memberikan tahap kekenyalan beserta bentuk jasad yang dikehendaki kepada produk

akhir. Biasanya, POs diadun dengan PO untuk mengurangkan takat lebur kepada suhu badan (37°C), Fasa lipid, dirumuskan dari nisbah PO dan POs yang berbeza, akan melalui proses emulsi. Analisis sebelum dan selepas proses pengemulsian dijalankan seperti berikut: TAG, SFC, perilaku terma. Sebagai tambahan, nilai iodin (IV), komposisi asid lemak (FAC), dan sifat reologi termasuk kadar kelikatan(η) dan juga daya tekanan selari (τ) telah ditentukan sebelum proses emulsi (BEP), manakala keadaan struktur mikro termasuk saiz dan jumlah kristal juga telah ditentukan untuk marjerin kajian (EMF) dan marjerin komersial (CMF) selepas proses emulsi (AEP).

Keputusan kajian menunjukkan bahawa pencampuran dan pengemulsian pada kadar PS yang melebihi daripada 40% (w/w), secara signifikannya mengubah sifat fisikokimia dan mikro EMF berbanding dengan CMF, menyebabkan kandungan PPO/POP yang diperlukan, SFC pada 37 ºC, saiz kristal, kuantiti kristal, kristalisasi dan peleburan entalpi (ΔH) yang kurang daripada 36,1%, 15%, 5.37 µm, 1425 kristal/µm^2, 17.25 J/g dan 57.69 J/g, secara masing-masing. Kesan sebaliknya diperoleh apabila kadar PS melebihi 40% (w/w). Analisis Kalorimetri Pengimbasan Kebezaan (DSC) menunjukkan kesan campuran yang signifikan ($p < 0.05$) terhadap suhu peralihan; ketajaman puncak dan entalpi pada aras PS yang melebihi 40% (w/w). Penggunaan PS yang berterusan dalam sistem matrik lemak bagi fasa lipid BEP dan AEP telah menunjukkan kewujudan puncak baru pada suhu tinggi antara 50- 56 °C. Puncak ini boleh dikaitkan dengan takat lebur kompaun TAG yang lebih tinggi serta kejadian transisi polimorfik. Keputusan pNMR pula menunjukkan pembentukan sistem eutektik pada PS/POs melebihi 40% (w/w), yang seterusnya menghasilkan kadar SFC yang rendah iaitu kurang daripada 15%

pada suhu badan (37 °C). Keputusan resonansi magnetik nukleans berdenyut (pNMR) menunjukkan penurunan signifikan kepada SFC bagi adunan yang mengandungi PS kurang daripada 40% (w/w), menjadikan kandungan SFC yang rendah kurang daripada 15% pada suhu badan (37°C). Semua data ini telah menunjukkan bahawa pembentukan kristal granular dalam lemak marjerin adalah didominasi oleh TAG bertakat lebur tinggi iaitu PPO/POP, sedangkan dos kecil monoasigliserol digunakan sebagai pengemulsi agar melambatkan kadar kelajuan penghabluran.

Penilaian untuk perilaku campuran dedua PO/POs bagi emulsi minyak dalam air (w/o) sepanjang masa simpanan telah menunjukkan perubahan signifikan ($p < 0.05$) pada keseluruhan sifat fisikokimia dan reologi bagi jenis emulsi (w/o) dengan penghasilan SFC pada 28°C, konsistensi, kelembutan dan modulus penyimpanan (*G'*) yang kurang daripada 25%, 16Kg f/ cm^2 dan 30 mm kemudahan bagi penyerapan kon dan $15x10^4$ Pa, masing-masing. Data ini telah menunjukkan jaringan struktur yang lemah dan daya tenaga yang kurang bagi model MPM yang melebihi 40% (w/w) PS/POs. Pemeriksaan di bawah mikroskop terhadap imej-imej menunjukkan bahawa penambahan POs yang lebih daripada 40% (wt./wt.) mempunyai keupayaan untuk menukarkan polimorf beta prima (β') kepada beta (β), manakala pengurangan POs lebih daripada 40% (w/w) akan melambatkan ketulenan polimorf β. Sementara itu, PV dan FFA daripada 2 Meq O_2/Kg dan 0.35% masing-masing, menggambarkan kestabilan oksidatif dan ketahanan yang tinggi terhadap keasidan dan ketengikan MPM sewaktu penyimpanan. Kestabilan oksidatif dalam model MPM sewaktu proses penyimpanan ini boleh diterangkan oleh kehadiran purata ikatan tepu optimal yang membuatkan ia lebih stabil secara kimia dari kerosakan oksidatif.

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I certify that a Thesis Examination Committee has met on 3 December 2010 to conduct the final examination of Sami Saadi on his thesis entitled "Characterization of Palm-Based Binary Fat Blends and Development of Margarine from the Blends" in accordance with the Universities and University 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 Master of Science.

Members of the Thesis Examination Committee were as follows:

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of Supervisory Committee were as follows:

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DECLARATION

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

SAMI SAADI

Date: 3 December 2010

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