## UNIVERSITI PUTRA MALAYSIA

# EFFECTS OF OIL-DISPERSED PHASE COMPOSITION AND SELECTED POLYSACCHARIDES ON THE PHYSICAL PROPERTIES AND STABILITY OF SOYBEAN-PALM KERNEL OLEIN BLEND OIL-IN-WATER EMULSIONS MODEL SYSTEM

**NOR HAYATI BINTI IBRAHIM**

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NOR HAYATI BINTI IBRAHIM

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By

**NOR HAYATI BINTI IBRAHIM**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia**
**in Fulfillment of the Requirements for the Degree of Doctor of Philosophy**

**September 2008**

***To my husband, family, teachers and lecturers***

Abstract of thesis presented to the Senate of University Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

# EFFECTS OF OIL-DISPERSED PHASE COMPOSITION AND SELECTED POLYSACCHARIDES ON THE PHYSICAL PROPERTIES AND STABILITY OF SOYBEAN-PALM KERNEL OLEIN BLEND OIL-IN-WATER EMULSIONS MODEL SYSTEM

By

**NOR HAYATI BINTI IBRAHIM**

**September 2008**

**Chairman : Professor Yaakob Bin Che Man, PhD**

**Faculty : Food Science and Technology**

An oil-in-water (O/W) emulsion is a system that consists of oil droplets dispersed in an aqueous continuous phase. Soybean oil (SBO) is commonly being used as oil-dispersed phases in many O/W-based food products. The products sometimes exhibit poor physical properties and stability against temperature fluctuations that can be attributed to a high unsaturation degree of SBO. These problems can be reduced by blending of SBO with more saturated oils such as palm kernel olein (PKO). However, crystallizing tendency of PKO at low storage temperatures may lead to partial droplet coalescence, causing destabilization of the emulsion. The use of certain polysaccharides however can reduce this problem and indirectly improve the overall emulsion properties. The objective of this research was to investigate the effects of oil-dispersed phase composition and selected polysaccharides on the physical properties and stability of SBO:PKO blend O/W emulsions model system. In the first stage of this study, the effect of palm kernel olein (PKO) incorporation on physical properties and stability of O/W emulsions was investigated. Soybean oil and blends of SBO:PKO at 10-40% PKO levels were used as dispersed phases (70% volume fraction) of egg yolk-stabilized O/W emulsions. The use of PKO caused a significant ($p < 0.05$) increase in droplet size but a significant ($p < 0.05$) decrease in rheological properties of the freshly prepared emulsions. With 10-30% PKO replacements, the emulsions were stable after storage at 25°C, most probably promoted by a significant content of C8-C12 fatty acids in PKO. With 30 and 40% PKO replacements, the emulsions were unstable after storage at 5°C due to high solid fat content (14-20%) which caused a severe partial coalescence. This was mainly evidence by increases in droplet equivalent surface mean diameter from 3.65-3.70 µm to 7.80-8.97 µm and decreases in emulsion yield stress from 1.72-1.82 Pa to 0.27-0.30 Pa after 30 days of storage. Throughout the storage, peroxide and anisidine values were found to be lower in the emulsions with PKO incorporated than in the emulsion with fully SBO. Under an accelerated oxidation condition (60°C, 12 days), a calibration model based on a Fourier-transform infrared spectral region (1800-1480 $cm^{-1}$) was developed to predict the peroxide value in oxidized emulsions over the range of 6-45 meq/kg.

In the second stage, physical properties and stability of emulsions as affected by the presence of individually 0.5% (wt/wt) xanthan gum (XG), carboxymethyl cellulose (CMC), guar gum (GG) and locust bean gum (LBG) were evaluated. A blend of SBO:PKO at 30% PKO level was used as a dispersed phase (40% volume fraction) of the emulsions. The microstructure of stored (5°C) XG emulsion showed the presence of partially coalesced droplets, explaining a large increase in its droplet size and the presence of 'free oil' after centrifugation at 3,500 rpm. However, partially coalesced droplets were not observed in stored CMC, GG and LBG emulsions and no ‘free oil’ could be separated under centrifugation force. The results support the ability of these polysaccharides in reducing partial coalescence by acting as a protective coating for oil droplets. Blends of XG, CMC and LBG were also prepared according to an augmented simplex-centroid mixture design with 10 points to investigate interaction effects of these polysaccharides on the emulsion rheological properties. The strongest synergistic effect was shown by ternary blends of XG:CMC:LBG at approximately 33-67% XG levels. Yield stress, apparent viscosity, elastic modulus and loss tangent responses were successfully fitted with a special quartic model ($R^2 > 0.89$). Hence, the mixture design with regression modelling approach was shown to be a valuable tool in better elucidating and predicting the interaction effects beyond the two-component blends.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai
memenuhi keperluan untuk ijazah Doktor Falsafah

# KESAN KOMPOSISI FASA MINYAK-TERSEBAR DAN POLISAKARIDA TERPILIH KE ATAS CIRI-CIRI FIZIKAL DAN KESTABILAN SISTEM MODEL EMULSI MINYAK-DALAM-AIR BERASASKAN ADUNAN MINYAK SOYA-OLEIN ISIRUNG SAWIT

Oleh

**NOR HAYATI BINTI IBRAHIM**

**September 2008**

**Pengerusi : Profesor Yaakob Bin Che Man, PhD**

**Fakulti : Sains dan Teknologi Makanan**

Emulsi minyak-dalam-air (O/W) ialah satu sistem yang terdiri daripada titisan-titisan minyak yang tersebar di dalam fasa akuas yang berterusan. Minyak soya (SBO) biasanya digunakan sebagai fasa minyak-tersebar di dalam kebanyakan produk makanan berasaskan emulsi O/W. Produk-produk berkenaan kadang-kala mempamerkan ciri-ciri fizikal dan kestabilan terhadap perubahan suhu yang kurang baik, yang boleh dikaitkan dengan darjah ketidaktepuan SBO yang tinggi. Masalah-masalah tersebut boleh dikurangkan dengan cara mengadunkan SBO dengan minyak yang lebih tepu seperti olein isirung sawit (PKO). Namun demikian, kecenderungan PKO untuk menghablur pada suhu penyimpanan yang rendah boleh membawa kepada percantuman separa titisan minyak, menyebabkan ketidakstabilan emulsi. Penggunaan polisakarida yang tertentu bagaimanapun boleh mengurangkan masalah berkenaan dan secara tidak lasungnya memperbaiki ciri-ciri keseluruhan emulsi. Objektif penyelidikan ini ialah untuk mengkaji kesan komposisi fasa minyak-tersebar dan polisakarida terpilih ke atas ciri-ciri fizikal dan kestabilan sistem model emulsi minyak-dalam-air berasaskan adunan minyak soya-olein isirung sawit. Di peringkat pertama kajian ini, kesan penggunaan olein isirung sawit (PKO) terhadap ciri-ciri fizikal dan kestabilan emulsi O/W telah dikaji. Minyak soya dan adunan-adunan SBO:PKO pada paras 10-40% PKO telah digunakan sebagai fasa tersebar (70% pecahan isipadu) beberapa emulsi yang distabilkan oleh kuning telur. Penggunaan PKO telah menyebabkan peningkatan dan penurunan yang signifikan ($p < 0.05$), masing-masing pada saiz titisan minyak dan ciri-ciri rheologi emulsi-emulsi tersebut. Dengan gantian 10-30% PKO di dalam fasa minyak tersebar, emulsi-emulsi berkenaan didapati stabil selepas disimpan pada suhu 25°C, kemungkinan disumbangkan oleh kandungan asid lemak C8-C12 yang signifikan di dalam PKO. Dengan gantian 30 dan 40% PKO di dalam fasa minyak tersebar, emulsi-emulsi berkenaan didapati tidak stabil selepas disimpan pada suhu 5°C, disebabkan oleh kandungan lemak pepejal yang tinggi (14-20%) di dalam fasa minyak yang tersebar telah menggalakkan percantuman separa titisan-titisan minyak yang ketara. Ini telah dibuktikan terutamanya oleh peningkatan pada min diameter ‘equivalent surface’ titisan minyak daripada 3.65-3.70 μm kepada 7.80-8.97 μm dan penurunan pada nilai ‘yield stress’ daripada 1.72-1.82 Pa kepada 0.27-0.30 Pa selepas 30 hari simpanan. Di sepanjang simpanan, nilai-nilai peroksida dan anasidina pada emulsi-emulsi yang mengandungi gantian PKO didapati lebih rendah berbanding dengan emulsi yang mengandungi SBO tulen. Di bawah keadaan pengoksidaan yang dipercepatkan (60°C, 12 hari), satu model kalibrasi telah dibina berasaskan spektrum-spektrum inframerah peralihan-Fourier pada julat gelombang 1800-1480 $cm^{-1}$, untuk meramal nilai peroksida pada julat 6-45 meq/kg yang terdapat di dalam emulsi yang teroksida.

Di peringkat kedua, kesan kehadiran 0.5% (berat/berat) gum xanthan (XG), karboksimetil sellulosa (CMC), gum guar (GG) dan gum kacang locust (LBG) (secara individu) ke atas ciri-ciri dan kestabilan fizikal emulsi telah dikaji. Adunan SBO:PKO pada paras 30% PKO telah digunakan sebagai fasa tersebar (40% pecahan isipadu) emulsi-emulsi tersebut. Selepas simpanan (5°C), mikrostruktur emulsi yang mengandungi XG menunjukkan kehadiran titisan-titisan minyak yang tercantum secara separa. Ini dapat menerangkan peningkatan pada saiz titisan minyak dan juga kehadiran 'minyak bebas' selepas pengemparan pada 3,500 rpm pada emulsi tersebut. Namun demikian, kehadiran 'minyak bebas' di bawah tekanan emparan dan titisan-titisan minyak yang tercantum secara separa tidak dapat dicerap pada emulsi-emulsi yang mengandungi CMC, GG dan LBG. Dengan ini, keputusan yang diperolehi dapat menyokong keupayaan polisakarida-polisakarida tersebut dalam mengurangkan proses percantuman separa dengan bertindak sebagai salutan pelindung kepada titisan-titisan minyak. Selanjutnya, adunan-adunan yang mengandungi XG, CMC dan LBG telah disediakan berpandukan rekabentuk percampuran 'augmented simplex-centroid' untuk mengkaji kesan interaksi polisakarida-polisakarida berkenaan ke atas ciri-ciri rheologi emulsi. Kesan sinergi yang paling kuat telah ditunjukkan oleh adunan pertigaan XG:CMC:LBG, pada paras XG secara kasarnya sebanyak 33-67%. Data-data 'yield stress', kelikatan, modulus elastik dan 'loss tangent' telah berjaya dipadankan dengan model 'special quartic' ($R^2 > 0.89$). Maka, rekabentuk percampuran bersama pemodelan regrasi ini merupakan satu pendekatan yang berguna bagi meramal dan memahami dengan lebih jelas kesan-kesan interaksi di kalangan polisakarida-polisakarida, selain daripada adunan-adunan dua komponen.

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I certify that an Examination Committee has met on 23rd September 2008 to conduct the final examination of Nor Hayati Binti Ibrahim on her Doctor of Philosophy thesis entitled "Effects of Oil-Dispersed Phase Composition and Selected Polysaccharides on Physical Properties and Stability of Soybean-Palm Kernel Olein Blend Oil-in-Water Emulsions Model System" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the student be awarded the relevant degree.

Members of the Examination Committee were as follows:

**Alejandro G. Marangoni, PhD**
Professor
Food and Soft Materials Science
University of Guelph, Canada
(External Examiner)

**Hasanah Mohd. Ghazali, PhD**
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia
(Internal Examiner 1)

**Zulkefly Kuang Bin Abdullah, PhD**
Professor
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner 2)

**HASANAH MOHD. GHAZALI, PhD**
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

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

**Yaakob Bin Che Man, PhD**
Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Chairman)

**Tan Chin Ping, PhD**
Lecturer
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Member)

**Nor Aini Idris, PhD**
Head
Food Technology & Nutrition Unit
Product Development & Advisory Services Division
Malaysian Palm Oil Board
(Member)

**HASANAH MOHD. GHAZALI, PhD**
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 19 December 2008

# DECLARATION

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

**NOR HAYATI BINTI IBRAHIM**

Date:

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