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EFFECTS OF POLYGLYCEROL ESTERS ON PALM OLEIN FRACTIONATION

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By

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EFFECTS OF POLYGLYCEROL ESTERS ON PALM OLEIN FRACTIONATION

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Chairperson : Professor Ir. Thomas Choong Shean Yaw, PhD
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Palm olein fractionation is an important process to produce palm super olein which has iodine value higher than 56 for market access to temperate countries. The process is, however, energy consuming and costly due to its lengthy cooling time in crystallization. Polyglycerol esters are introduced in the palm olein fractionation to enhance the process by reducing the crystallization time. This study investigated the effects of 0.1% (w/w) – 0.9% (w/w) polyglycerol esters on palm olein fractionation from the aspects of crystal nucleation, crystal growth and product quality. Palm olein (iodine value 56) with 0.1% (w/w), 0.3% (w/w), 0.5% (w/w), 0.7% (w/w) and 0.9% (w/w) polyglycerol esters additives were cooled to its desired crystallization temperature at 13ºC in 335 minutes using the focused beam reflectance measurement. Crystal nucleation and growth during the crystallization were monitored in the focused beam reflectance measurement where the in-line changes of crystal size distribution during the crystallization were analysed. The product quality of the palm olein fractionation was analysed using iodine value test and triacylglycerol composition. Our research findings from focused beam reflectance measurement showed that polyglycerol esters promote crystal nucleation and less formation of undesired large crystals were observed during the palm olein crystallization. Polyglycerol esters expedited the crystal nucleation as compared with the control without polyglycerol esters. The product analysis found that polyglycerol esters prevent olein entrainment in the solid fraction after filtration where the content of 1,3-dipalmitoyl-2-oleyl-glyerol in the solid fraction increased while its 1-palmitoyl-2,3-oleyl-glycerol content decreased. The optimal dosage that offered best performance was 0.3% (w/w) polyglycerol esters which gave formation of the most medium crystals and best palm mid fraction product with lowest iodine value and highest content of 1,3-dipalmitoyl-2-oleyl-glycerol. These results imply that polyglycerol esters are a nucleation enhancing and crystal growth retarding additive in palm olein crystallization at 0.3% (w/w) dosage.

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

KESAN-KESAN ESTER POLIGLISEROL TERHADAP PROSES PEMERINGKATAN OLEIN SAWIT

Oleh

YOONG JUN HAO

November 2017

Pengerusi : Profesor Ir. Thomas Choong Shean Yaw, PhD
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Pemeringkatan olein sawit merupakan satu proses yang penting untuk menghasilkan super olein sawit dengan nilai iodin yang lebih tinggi daripada 56 bagi memenuhi keperluan pasaran di negara-negara beriklim sejuk. Proses ini melibatkan kos yang tinggi dan memerlukan banyak tenaga disebabkan oleh tempoh penyejukan semasa penghabluran yang panjang. Ester poligliserol digunakan dalam proses pemeringkatan olein sawit bagi mempercepatkan proses tersebut dengan mengurangkan tempoh penghabluran. Kajian ini menyelidik kesan 0.1% (w/w) – 0.9% (w/w) ester poligliserol ke atas pemeringkatan olein sawit dari pelbagai aspek termasuklah penukleasan and pertumbuhan hablur serta kualiti produk. Olein sawit (nilai iodin 56) dengan 0.1% (w/w), 0.3% (w/w), 0.5% (w/w), 0.7% (w/w) dan 0.9% (w/w) ester poligliserol tambahan disejukkan sehingga mencapai suhu penghabluran pada 13ºC dalam masa 335 minit menggunakan pengukuran kepantulan alur terfokus. Penukleasan dan pertumbuhan hablur dipantau melalui pengukuran kepantulan alur terfokus di mana perubahan taburan saiz kristal dalam talian semasa proses penghabluran dianalisa. Kualiti produk pemeringkatan olein sawit dianalisis dengan menggunakan pengujian nilai iodin dan kandungan triasilgliserol. Hasil kajian daripada pengukuran kepantulan alur terfokus menunjukkan bahawa ester poligliserol menggalakkan penukleasan hablur dan mengurangkan pembentukan hablur bersaiz besar yang tidak diingini dalam proses penghabluran. Ester poligliserol mempercepatkan penukleasan hablur berbanding dengan kawalan tanpa ester poligliserol. Analisa produk menunjukkan bahawa ester poligliserol mengurangkan pemerangkapan olein dalam pecahan pepejal selepas proses penapisan di mana kandungan 1,3-dwipalmitoyl-2-oleyl-gliserol dalam pecahan pepejal meningkat manakala kandungan 1-palmitoyl-2,3-oleyl-gliserol berkurang. Dos optimum yang memberi prestasi terbaik ialah 0.3% (w/w) ester poligliserol di mana ia membantu dalam pembentukan hablur bersaiz sederhana paling banyak dan produk pecahan tengah sawit yang terbaik dengan nilai iodin terendah dan kandungan 1,3-dwipalmitoyl-2-oleyl-gliserol tertinggi. Keputusan ini menunjukkan bahawa ester poligliserol adalah aditif

yang menggalakkan penukleasan hablur dan merencatkan pertumbuhan hablur dalam proses penghabluran olein sawit dengan penambahan 0.3% (w/w) ester poligliserol.

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For any errors or inadequacies that may remain in this work, of course, the responsibility is entirely my own.

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

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

CPO	Crude palm oil
BOB	1,3-bihenoyl-2-sn-oleoyl-glycerol
DAGs	Diacylglycerols
FAC	Fatty acid composition
FBRM	Focused Beam Reflectance Measurement
FFA	Free fatty acid
GC	Gas Chromatography
GRAS	Generally Recognized as Safe
HPLC	High Performance Liquid Chromatography
IV	Iodine Value
Lc	Large crystal with mean size from 100–251 μm
MAG	Monoacylglycerols
Mc	Medium crystal with mean size from 29–86 μm
PGE	Polyglycerol esters
PMF	Palm mid fraction
POP	1,3-dipalmitoyl-2-oleoyl glycerols
POO	2,3-dioleoyl-1-palmitoyl glycerols
PLP	1,3-dipalmitoyl-2-linoleoyl glycerols
PLO	1-palmitoyl-2-linoleoyl-3-oleoyl glycerols
PPP	Tripalmitoyl-glycerol
RBD	Refined, bleached and deodorized
RBDPL	Refined, bleached and deodorized palm olein
Sc	Small crystal with mean size from 10–23 μm

SOS	1,3-distearic-2-oleoyl glycerol
TAGs	Triacylglycerols
XLc	Extra large crystal with mean size from 293–1000 μm
XSc	Extra small crystal with mean size from 1–5 μm

CHAPTER 1

INTRODUCTION

1.1 Palm Olein Fractionation

Oil palm produces two types of oil, namely crude palm oil from mesocarp and crude palm kernel oil from the kernel. Palm oil consists mainly of glycerides and small portions of non-glyceride minor components i.e. phytonutrients. ln order to render the oils for different applications, these glycerides need to be modified into palm olein (liquid fraction of palm oil which contains more low melting triacylglycerols (TAGs)) and palm stearin (solid fraction of palm oil which contains more high melting TAGs) with different iodine values (IV).

In Malaysia, crude palm oil is refined to produce the refined, bleached and deodorised (RBD) palm oil. The RBD palm oil is then separated into RBD palm olein (RBDPL) and RBD palm stearin using dry fractionation. RBDPL has been widely used as cooking oil in most of the countries around the world. In 2016, Malaysia exported 7.03 million tonnes of RBD palm olein, accounting for 43.8% of total export of palm oil products to the world's market (MPOB, 2016). However, RBD palm olein encounters solidification issues in cold-temperature countries e.g. China, Europe, United States and Australia. This is because palm oil has higher content of saturated fatty acids which attributed to its higher melting point as compared to other soft oils e.g. soybean and rapeseed oils. RBD palm olein solidifies at cold temperature below 24ºC which causes many consumers refrain from using the oil as cooking oil. Particularly in countries such as China, one of the major markets of Malaysian palm oil accounting for 1.28 million tonnes of RBD palm olein export, palm cooking oil is often perceived as an 'unhealthy" oil due to its unpleasant appearance similar to lard when it is solidified at cold temperature lower than 24ºC (MPOB, 2016). The solution to address the solidification issue of RBD palm olein is important to secure the market of palm oil, which is mainly sold as cooking or frying oil in the world's market, including China.

Fractionation is a process that separates high melting triacylglycerols (TAGs) from low melting TAGs of oils and fats (Metin and Hartel, 2005). This is a more natural process as compared to other fat modification processes such as interesterification and hydrogenation. Removing some amounts of higher melting components from oil and fat will cause the remaining oil to be more liquid such that it can be used at lower temperatures as a liquid oil and it can also be blended with other liquid oil to further its cold stability characteristic (Deffense, 1985). The dry fractionation involves two major parts i.e. crystallization and filtration. A controlled crystallization plays pivotal role in dry fractionation process (Metin and Hartel, 2005). In palm oil fractionation, the control of lipid crystallization to obtain the desired number, size distribution, polymorph, and dispersion of the crystalline phase is important.

In order to cater to the market needs in the temperate countries, dry fractionation of RBDPL has been carried out by the industry to produce palm super olein. The main advancements have been multiple fractions to obtain fractions with varying properties for specific solid and liquid applications and the introduction of various filtration technologies culminating in the high-pressure press of today. The liquid-fraction products from the dry fractionation, palm super olein, can be used as low-melting point cooking oils to cater for cold-temperate countries. Palm super olein with low melting point does not solidify at low temperature and it can be used in house-hold as cooking oils. On the other hand, solid fraction from fractionation of RBDPL can be used to produce specialty products with high value addition such as ice cream premix powders, cocoa butter equivalent, bakery fats, chocolate fillings for bread and cookies, chocolate coatings, margarine and shortenings. (Bangun, 2009; Niu et al., 2015; Ji et al., 2014; Liu et al., 2014; Ji et al., 2013). Figure 1.1 shows the fractionation processes and products of palm oil.

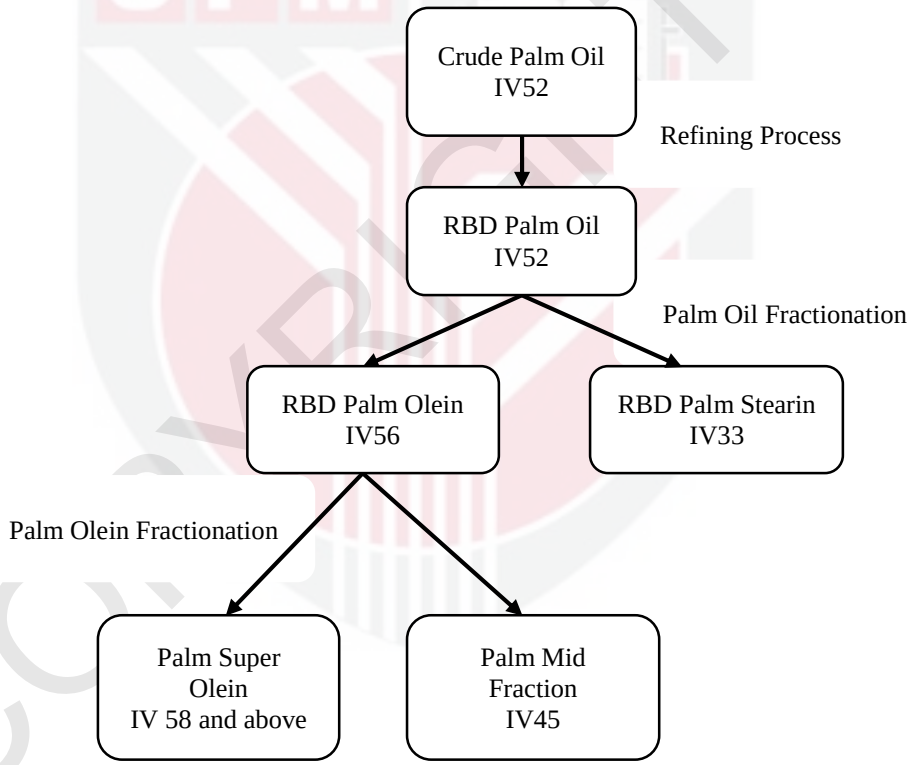

Figure 1.1 : Fractionation and Products of Palm Oil

1.2 Problem Statement

Dry fractionation is the simplest and cheapest process and no auxiliary agents such as solvents or detergents are introduced during processing. However, dry fractionation is less efficient than solvent or detergent fractionation processes (Timms, 2005), mainly due to the olein entrainment in the solid fraction after the filtration of dry fractionation process. The difficulty of having the suitable crystal size made the olein fractionation very challenging. Large crystals have stronger crystal attachment surface but however it will cause olein entrainment if the crystals are too large. Olein entrainment reduces when crystal sizes reduce. In this connection, many researches have been carried out to improve the fractionation efficiency by reducing production time and enhancing the crystallization techniques to avoid olein entrainment especially for multiple stages of fractionation. This will help the palm oil industry to enhance its production output in the fractionation process. Palm olein fractionation is an expensive process as it involves long hour of fractionation which accounted for increased cost of production. Polyglycerol esters (PGE) was introduced as additive agent for enhancing the palm oil fractionation process. Previous study showed that adding PGE promotes formation of more uniform crystals which helped increase yield of the palm olein products from the palm oil fractionation process at 24ºC (Kuriyama et al., 2011). The effects of PGE on crystal growth and nucleation in palm oil fractionated was studied by Saw et al, 2017 using the focused beam reflectance measurement (FBRM). Study confirmed that palm oil crystals were more even in size when PGE was added (Saw et al., 2017). In light of the possible findings in the previous studies, it is hypothesized that PGE additive can be one of the possible options to enhance the crystallization which will reduce the fractionation time and promote formation of even-sized palm olein crystals. The effects of PGE additive on the crystallization of palm olein require studies to determine its optimum dosage and crystallization mechanism. Currently studies on PGE are limited and there is no available data on the effects of PGE additive on palm olein crystallization (Saw et al., 2017; Sakamoto et al., 2005).

1.3 Scope of Works

This project will determine the suitable dosage of PGE among 0.1% (w/w), 0.3% (w/w), 0.5% (w/w), 0.7% (w/w) and 0.9% (w/w) which has the lowest olein entrainment in the products and forms crystals in more uniform size. This study will analyse the effects of PGE on nucleation and crystal growth in palm olein crystallization using FBRM to determine the possible dosage which is able to form uniform size crystals. IV and TAG of the products from the palm olein fractionation, namely palm super olein and palm mid fraction (PMF), will be analysed to investigate olein entrainment. In general, PGE affects the crystal formations during crystallization process and the final product qualities.

1.4 Objectives of the Study

The following objectives are set for this study: -

a) To study effects of PGE on nucleation and crystal growth in palm olein fractionation;
b) To produce uniform-sized crystals using PGE;
c) To study effects of PGE on the iodine values and TAG of the fractionation products; and,
d) To identify the most suitable dosage of PGE for palm olein fractionation.

1.5 Chapters Summary

The structure of the thesis and its chapters are summarized as follows:

Chapter 2: Literature Review

This chapter discusses the fundamental reaction and theory of palm oil fractionation, including the first and secondary fractionation. It also discusses the products of fractionation, phase behaviour of palm oil fractionations, nucleation and crystal growth. In addition to the fractionation, the chapter also describes the background and previous works on PGE.

Chapter 3: Materials and Methods

In this chapter, a short description on the materials and methodology is given. The chapter explained the specification and operation of the equipment used in the study. In addition, the chapter also discussed the measurement and analytical methods, and the experimental design for the study.

Chapter 4: Results and Discussion

The experimental results and findings from the study were charted and showed in this chapter. The experimental findings were mainly the effects of PGE on fractionation, where the nucleation and crystal growth during crystallization were studied and discussed, and analysis on the fractionation products (IV and TAG of palm super olein and PMF). The crystallization mechanism and optimum PGE dosage are also discussed towards end of this chapter.

Chapter 5: Conclusions and Recommendations

This chapter concludes the overall experimental findings from the study. The chapter also discusses future works' recommendations according to the current findings.

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