FRACTIONATION, CHARACTERISATION AND OPTIMISATION OF REFINED, BLEACHED AND DEODORISED PALM OIL USING PROGRESSIVE FREEZE CONCENTRATION

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This thesis is especially dedicated to my loving and wonderful husband Nazrizal Zainal Abidin and my handsome, cheerful and beloved son, Muhammad Nazheef, as well as my beloved parents and sibling. You are all my strength and inspiration to make me able to overcome all the challenges and trials to complete this thesis.

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ABSTRACT

The aims of this experimental work were to study the influence of operating condition on progressive freeze concentration (PFC) of refined, bleached and deodorised palm oil (RBDPO) using coil crystalliser and to determine the optimum condition based on the response of effective partition constant (K) and triglyceride (TG) of olein. The operating condition of circulation flowrate (CF, 2000 to 3000 mL/min), coolant temperature (CTemp, 24 to 29 °C), circulation time (CTime, 40 to 60 minutes), and initial iodine value (IIV, 50 to 55 wijs) could affect the efficiency of RBDPO fractionation process where mass and heat transfer study and economic analyses were carried out. The results indicated that CF of 2800 mL/min, CTemp of 28 °C, CTime of 60 minutes, and IIV of 55 wijs gave high value of iodine value (IV) and low value of slip melting point (SMP) for olein in the range of 56.97 to 55.84 wijs and 21.57 to 23.04 °C, respectively. The optimization process found that the operating parameter of CF, CTemp and CTime were not significant as indicated by the p value of less than 0.05 from the Pareto chart, meanwhile IIV was significant. Furthermore, the optimum condition for low K and high olein's TG found using software assisted response surface methodology (RSM) were in the following range: 2502 to 2537 mL/min for CF, 26 to 27 °C for CTemp, 53 to 54 minutes for CTime, and 56.8 to 56.9 wijs for IIV. For energy and cost analysis, it was found that the fractionation of RBDPO through PFC is cheaper than the conventional fractionation as it has been calculated that the energy usage and production cost for PFC are 0.025465 kW/hr/kg and RM0.03/kg of RBDPO respectively. All the analyses supported the objectives of this study and gave enough evidence of good performance and quality of olein as well as low cost of operation in PFC fractionation.

ABSTRAK

Tumpuan kajian ini adalah untuk mengkaji pengaruh keadaan operasi ke atas pemekatan pembekuan progresif (PFC) kepada pemeringkatan minyak kelapa sawit yang ditapis, diluntur dan dinyahbau (RBDPO) dengan menggunakan penghablur lingkararan dan bagi menentukan nilai optimum berdasarkan tindak balas dari pemalar pemisahan berkesan (K) dan trigliserida (TG) bagi olein. Keadaan operasi bagi kadar aliran (CF, 2000 to 3000 mL/min), suhu penyejuk (CTemp, 24 to 29 ℃), masa edaran (CTime, 40 to 60 minutes), and nilai iodin permulaan (IIV, 50 to 55 wijs) boleh mempengaruhi kecekapan kepada pemeringkatan RBDPO di mana berat dan pemindahan haba serta ekonomi analisa telah dikaji. Hasilnya didapati bagi nilai CF pada 2800 mL/min, CTemp pada 28 °C, CTime pada 60 minit dan IIV pada 55 wijs memberikan nilai yang tinggi untuk nilai iodine (IV) dan nilai rendah untuk takat lebur gelincir (SMP) bagi olein, masing-masing pada julat 56.97 hingga 55.84 wijs dan 21.57 hingga 23.04 °C. Proses pengoptimuman pula mendapati bahawa parameter CF, CTemp dan CTime tidak penting sebagaimana yang ditunjukkan oleh nilai p iaitu kurang daripada 0.05 yang diperolehi daripada carta Pareto, manakala IIV adalah penting. Sementara itu, nilai optimum untuk K yang rendah dan TG olein yang tinggi adalah diperolehi menggunakan perisian kaedah tindakbalas permukann (RSM) adalah dalam julat 2502 hingga 2537 mL/min untuk CF, 26 hingga 27 °C untuk CTemp, 53 hingga 54 minutes untuk CTime, dan 56.8 hingga 56.9 wijs untuk IIV. Bagi tenaga dan analisa kos, didapati bahawa sistem PFC adalah lebih murah berbanding sistem pemeringkatan konvensional di mana penggunaan tenaga dan kos adalah 0.025465 kW/hr/kg dan RM0.03/kg of RBDPO, masing-masing. Kesemua keputusan analisis yang diperolehi menyokong objektif yang dikaji dan memberikan bukti yang kukuh untuk prestasi dan kualiti yang baik untuk olein serta kos operasi yang rendah bagi pemeringkatan PFC.

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LIST OF ABBREVIATIONS

PORIM	-	Palm Oil Research Institute of Malaysia
RCOC	-	Refiner's Certificate of Competency
PORAM	-	Palm Oil Refiners Association of Malaysia
RBDPO	-	Refined, Bleached and Deodorized Palm Oil
SFC	-	Suspension Freeze Concentration
SSHE	-	Scrape Surface Heat Exchanger
PFC	-	Progressive Freeze Concentration
TG	-	Triglyceride
SMP	-	Slip Melting Point
IV	-	Iodine Value
RSM	-	Response Surface Methodology
LDL	-	Low Density Lipoprotein
POP	-	1,3-dipalmitoyl-2-oleoyl-glycerol
POO	-	1-palmitoyl-2,3-dioleoyl-glycerol
POS	-	1-palmitoyl-2-oleoyl-3-stearoyl- glycerol
SF	-	Solid Fat
CP	-	Cloud Point
MT	-	Metric Tonnes
CPO	-	Crude Palm Oil
PME	-	Palm Oil Methyl Ester
FFA	-	Free Fatty Acid
HPLC	-	High Performance Liquid Chromatography
FC	-	Freeze Concentration

VOCs	-	Hazardous Volatile Compound
CC	-	Coil Crystalliser
RWB	-	Refrigerated Water Bath
SC	-	Suspension Crystallization
DOE	-	Design of Experiment
CCD	-	Central Composite Design
GC	-	Gas Chromatography
RWB	-	Refrigerated Water Bath
FID	-	Flame Ionisation Detector
GLC	-	Gas-Liquid Chromatography
ECN	-	Equivalent Carbon Number
TSD	-	Temperature Step Down
THU	-	Temperature Hold Up

LIST OF SYMBOLS

K	-	effective partition constant
$\dot{Q}_{LC}$	-	energy demand
U_o	-	overall heat transfer coefficient
B	-	beta
CO_2	-	carbon dioxide
C_L	-	concentration of liquid
C_s	-	concentration of solid
V_L	-	volume of liquid
A	-	alfa
T	-	time
G	-	grams
A	-	area (cm)
%	-	percentage
U	-	rate of crystal front
μ	-	rate of advance of crystal front
RMSE	-	ratio of solute error
$\dot{m}$	-	mass flowrate
C_{Fs}	-	final solute concentration
C_H	-	solute concentration
C_1	-	initial solutes concentration
M	-	mass
v_1	-	initial volume
v_2	-	final volume

N	-	number of experiment data
ΔH_m	-	enthalpy of mixture
$\dot{Q}_{app}$	-	heat requirement
K	-	ratio of total amount
°C	-	temperature
W_{pred}	-	Weight of predicted

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CHAPTER 1

INTRODUCTION

1.1   Research Background

Palm oil is richly edible and an important member of vegetable oil group besides coconut oil, palm kernel, or olive oil which are used as raw material in food or non-food industries. The oil palms (*Elaeis or macaw-fat*) are very well-known worldwide as a raw material for various products such as soap, frying oil, biodiesel, edible fats in the confectionery, powder detergent, ice cream, mayonnaise, pomades, candle and etc.

Different types of oil can be extracted from the oil palm fruit such as crude palm oil, RBD palm oil, palm stearin, palm olein, fractionated palm olein and palm mid-fraction. Out of these, Malaysia's most exported oil is palm oil and palm olein. Globally, Malaysia has the capacity to produce and consistently export best palm oil in a large quantity. Oil palms are now grown in tropical countries as an agricultural crop due to it is ability to act as a high yielding source of edible and technical oil. Most of them with a minimum 1600 mm/year of rainfall, geographically within 10° of the equator

Palm Oil Research Institute of Malaysia (PORIM) is an institute responsible in monitoring and carrying out researches on the quality of palm oil in Malaysia. PORIM constantly launch assessment programmes to evaluate and ensure those palm oil refiners are capable of producing high quality products and certify them with Refiner's Certificate of Competency (RCOC). Other than that, Palm Oil Registration and Licensing Authority (PORLA) plays a role in ensuring that Malaysia's refiner and their products fulfil the standards and specifications set. They are also responsible for inspections at the port so that the products to be exported satisfied the Palm Oil Refiners Association of Malaysia (PORAM) specifications. In the nutshell, Malaysia's palm oil is globally traded in more defined quality.

Future prospect of palm oil developments and demands are highly encouraging. There is no doubt that there will be more sectors, especially in Malaysia, trying to discover and develop new technologies to enhance the productivity and quality of oil at low cost. Moreover, this is supported by the fact that there is increasing number of population in major consuming nations, especially in Asia. There will absolutely be additional demands and increase per capita use of edible oil of palm oil (Murphy, 2009).

Centuries ago, a traditional method is used to separate palm oil component. However, in 1960s, the industries tried to apply new simpler techniques of separation and yet yielding a better purified products. One of the most important steps in palm oil processing is the separation of olein from Refined, Bleached and Deodorized palm oil (RBDPO) to produce edible oil that will not solidify at room temperature. Conventionally, this process is carried out through fractionation.

There are three types of fractionation, which are dry fractionation, detergent fractionation, and solvent fractionation. Among these, dry fractionation is the most common. It involves crystallisation of olein at its freezing/melting temperature. Dry fractionation is comparatively better than the other two. Hence, it is mostly applied in Malaysia. Solvent and detergent fractionation require greater capital investments (for

particular chemicals) (Hamm, 2005) whereas dry fractionation only requires crystalliser, filter and washing unit. However, there is a drawback in dry fractioning in which it is relatively inferior in separating process compared to the other two techniques.

The dry fractionation process resembles another chemical engineering process called Suspension Freeze Concentration (SFC). This is one of the freeze concentration methods. Generally, SFC is a process of crystallising substance with higher melting/freezing point in the mixture, by bringing down the temperature of the mixture/solution to the melting point while stirring. This will then leave behind the substance with lower melting/freezing temperature in the form of mother liquor and the substances of higher melting/freezing point as solid form.

Dry fractionation or SFC system initially starts with Scrape Surface Heat Exchanger (SSHE) in crystalliser, producing small crystals. The process continues with a crystallisation process using a stirred tank crystalliser. The sample is separated into solid and liquid form according to the melting point of olein and stearin which are the major component in RBDPO. Then, buffer tank collects those crystals and liquid. Next, filtration process starts using filter press and vacuum filter. Finally, the washed olein is recycled back to yield highly purified oil and remove the impurities as shown in Figure 1.1;

Another method of freeze concentration other than SFC is Progressive Freeze Concentration (PFC). PFC method is being planned to be an alternative to SFC. It is a method in which a single ice will be formed on a cooling plate. Thus, it makes the separation of ice crystal from the concentrated raw material much easier than the common suspension crystallization method, where smaller ice crystals are formed.
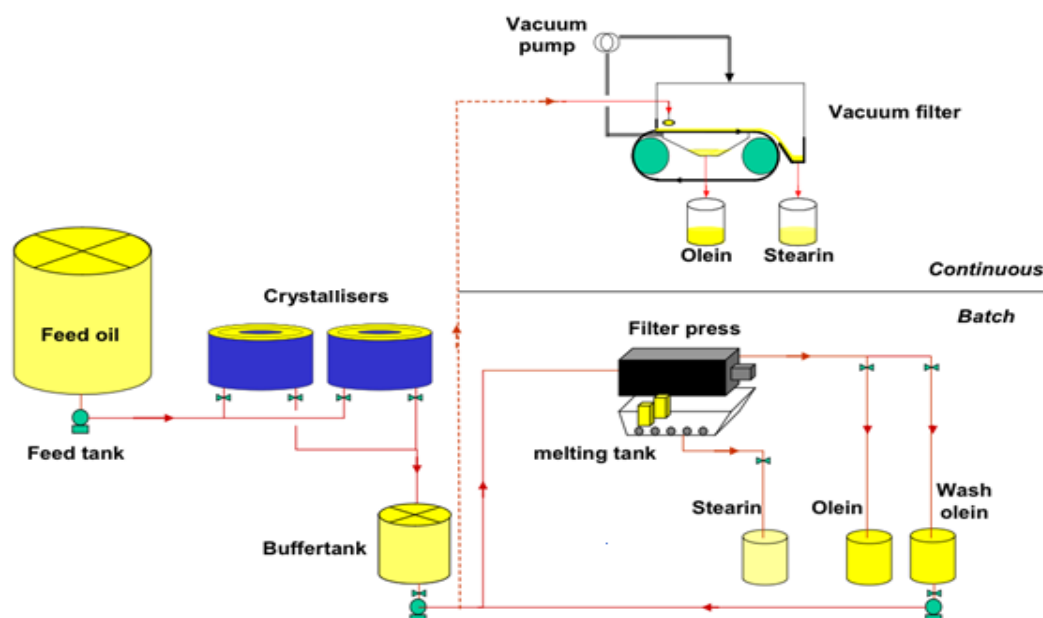
Figure 1.1: Dry fractionation of edible oil

The advantages of PFC are easier separation of crystal from the mother liquor because PFC formed in single solid crystal while SFC formed in small seed crystal. In addition, PFC method does not use SSHE to generate the crystal and it can reduce the capital cost of the plant process .SSHE itself contribute approximately 30% of capital cost investment in the process. SSHE is the most expensive heat exchanger and it is compulsory in SFC process. Different characteristics between SFC and PFC are clearly shown in Figure 1.2 below:
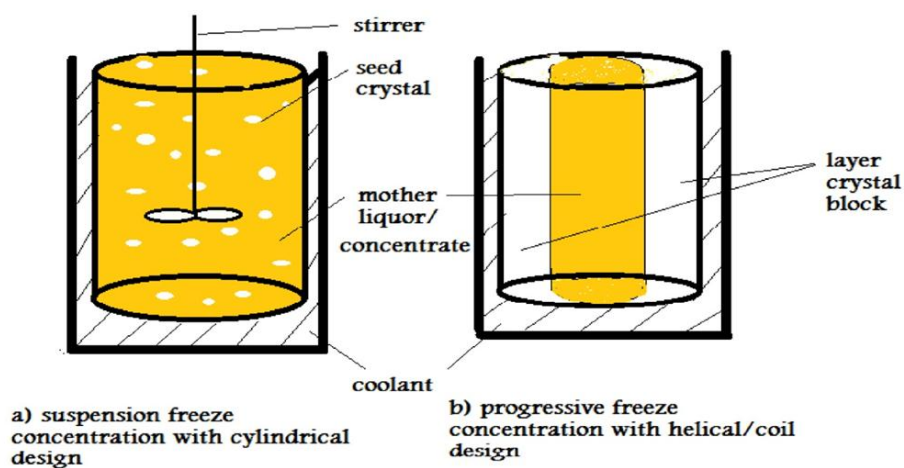
Figure 1.2: Different characteristic SFC and PFC

1.2   Problem Statement

As mentioned earlier, dry fractionation is a method to separate RBDPO involving numerous equipment such as SSHE, filter press or vacuum filters and washing units. This complicated system is in principle similar to SFC. Although SFC can be classified as efficient, the numerous unit operations and SSHE increases the capital cost in setting up the facility.

The other type of Freeze Concentration process, PFC, produces crystals of the liquid with higher melting point (stearin) as a block/layer on cooled surface. The formation of the crystal prevents contamination of the lower melting point substances (olein) in the crystal lattice, thus ensuring production of high purified olein. The target liquor would be circulated on a cooled surface to encourage formation of highly pure crystal block/layer.

As the crystals are in the form of blocks, the separation from the mother liquor would be easier, solely by draining the mother liquor off of the crystalliser. Seeding equipment is not needed as the seeding layer could merely be formed by circulating the pure substance with higher melting point in the crystalliser prior to the separation process. The reduction of operating units reduces the capital cost involved in developing such facility.

However, the capacity of PFC in replacing SFC is still an undergoing research PFC is somehow not yet favourable in the industry because of its slow productivity. There are many designs that have been previously developed in PFC, mainly focusing on the crystalliser, in order to increase its efficacy. One of them was developed by Jusoh *et al.*, (2008a) where their PFC technique uses coil crystalliser as the main component for crystallization.

The main question is whether or not this PFC process could be engaged in palm oil fractionation, which has never been investigated before. It is very interesting to know whether the designed crystalliser is able to perform a highly efficient separation process for olein from RBDPO and to see whether the PFC process is more effective to be used in fractionation of olein and stearin.

1.3   Objective of the study

The objective of this research is to reduce the cost of processing and increases the quality of olein as edible oil by using PFC, a method to replace the current conventional system of dry fractionation, which can be categorized as SFC. It also determines the most optimum parameter in PFC system in producing the best quality of palm oil out of separation and purification process. The objectives of this research are:-

i.   To study the performance of PFC integrated with a stainless steel coil crystalliser for fractionation of RBDPO.

ii. To determine the optimum condition for separating RBDPO using this PFC method system.

iii. To carry out economic analysis of PFC system.

iv. To carry out mass and heat transfer analysis of the PFC system of olein or oil.

1.4   Scope of Research

In order to achieve the objectives and discover the efficacy of PFC system applied in RBDPO fractionation process, these listed scopes have been implemented:

i)   Performance of PFC will be evaluated using effective partition coefficient, *K*, and triglyceride (TG) content of olein which are influenced by four parameters: circulation flow rate (2000 to 3000 mL/min), circulation time (40 to 65 min), coolant temperature (24 to 29 °C) and initial iodine value (50 to 55 wijs) of RBDPO. The optimum condition for PFC can be finalised by response surface methodology (RSM) from STATISTICA software using those four parameters it will give two readings, which are K value and TG content of olein.

ii)   The economic value will be evaluated by calculating energy utilization or energy demand using $\dot{Q}_{LC}$ formula. this will interpret the capital investment for the processes.

iii)   General heat transfer coefficient will be calculated by using formula of overall heat transfer coefficient U_o whereas quality of oil will be measured by iodine value (IV), slip melting point (SMP) and TG content of olein.

1.5   Research Contribution

This research will give a positive impact to the separation and purification of RBDPO by freeze concentration method. It is because freeze concentration is known

to produce the best product but at a high cost. Further studies show that progressive freeze concentration can reduce the cost due to simplified and less of equipment required. In this case, Malaysia's Palm Oil Industries has the solution on how to get high quality products from RBDPO at the lowest cost. In addition, another benefit that comes from this research is less usage of thermal energy, and electricity. Hence, this process is more environmental-friendly without hazardous chemicals or without waste as it will produce the best quality sample.

1.6 Thesis Layout

This thesis has seven chapters. Chapter 1 introduces the background of the study which is about palm oil, commercial of the plant and a peak on methods in separating and purifying olein and stearin out of Refined, Bleached, and Deodorised Palm Oil (RBDPO). Chapter 2 gives an idea about the process of palm oil in general, describing new methods which are still under research, the efficacy of parameter proposed, difference between earlier and latest methods,. Chapter 3 describes details on the method involved in this research by means of PFC crystalliser and crystallising involves production of a high quality oil especially olein and stearin. Meanwhile, Chapter 4 describes four parameters, which are circulation flow rate, coolant temperature, circulation time and initial iodine value. This chapter also explains the result and compares them with result of previous research the. Next, Chapter 5 demonstrate 4 parameters used in order to discover the optimum condition for this PFC system and the values based on two responses which are K value and TG content of olein. In Chapter 6, mass and heat transfer study, energy consumption, and cost analysis would be further explained and support the preference for this system. Last but not the least, the conclusion and idea that emerge from this research will be mentioned in Chapter 7.

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