

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

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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 °C), 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.

## TABLE OF CONTENTS

CHAPTER	TITLE	PAGE
	DECLARATION	ii
	DEDICATION	iii
	ACKNOWLEDGEMENTS	iv
	ABSTRACT	v
	ABSTRAK	vi
	TABLE OF CONTENTS	vii
	LIST OF TABLES	xii
	LIST OF FIGURES	xv
	LIST OF ABBREVIATIONS	xviii
	LIST OF SYMBOLS	xx
	LIST OF APPENDICES	xxii
<b>1</b>	<b>INTRODUCTION</b>	<b>1</b>
	1.1 Research Background	1
	1.2 Problem Statement	5
	1.3 Objective of the Study	6
	1.4 Scope of Research	7
	1.5 Research Contribution	7
	1.6 Thesis Layout	8
<b>2</b>	<b>LITERATURE REVIEW</b>	<b>9</b>
	2.1 Introduction	9
	2.2 Palm Oil	10

2.2.1	Palm Oil Composition	11
2.2.2	Chemical Properties of Palm Oil	13
2.2.3	Physical Properties of Palm Oil	14
2.2.4	Uses and Benefit of Palm Oil	15
2.2.5	Future Prospect for Palm Oil	17
2.3	Palm Oil Refining Method Process	19
2.3.1	Physical Refining	21
2.3.2	Chemical Refining	23
2.4	Fractionation of Refined, Bleached and Deodorised Palm Oil (RBDPO)	24
2.4.1	Dry Fractionation	26
2.4.2	Detergent Fractionation	28
2.4.3	Solvent Fractionation	29
2.5	Quality of RBDPO	30
2.6	Fundamental of Crystallization	30
2.6.1	Crystallization from Solution	31
2.6.2	Crystallization from Melt	31
2.7	Crystallization Fractionation by Freeze Concentration	32
2.7.1	Suspension Freeze Concentration (SFC)	35
2.7.1.1	Crystal Growth	35
2.7.1.2	Nucleation	36
2.7.1.3	Supersaturation	36
2.7.2	Progressive Freeze Concentration (PFC)	37
2.8	Comparison between SFC and PFC	38
2.9	Heat and Mass Transfer	40
2.10	Energy Consumption and Cost Advantages	41
2.11	Effect of Operating Condition for Fractionation of RBDPO	42
2.11.1	Effect of Circulation Flowrate	42
2.11.2	Effect of Coolant Temperature	43
2.11.3	Effect of Circulation Time	43
2.11.5	Effect of Initial Iodine Value	44
2.12	Optimization Process	44
2.12.1	Response Surface Methodology (RSM)	45
2.12.2	Design of Experiment (DOE)	46
2.12.3	Central Composite Design (CCD)	46



<b>3</b>	<b>MATERIALS AND METHODOLOGY</b>	<b>47</b>
3.1	General Research Activity	47
3.2	Experiment Flowchart	48
3.3	Raw Materials and Equipment	50
3.4	Preliminary Screening Process and Experimental Set-up	55
3.5	Experimental Work Procedure	56
3.6	Investigation of Effect of Operating Condition	58
3.6.1	Effect of Circulation Flowrate	60
3.6.2	Effect of Coolant Temperature	60
3.6.3	Effect of Circulation Time	60
3.6.4	Effect of Initial Iodine Value	61
3.7	Analysis of Quality Palm Oil and Efficiency of Fractionation	61
3.7.1	Effective Partition Coefficient, K-value	62
3.7.2	Yield	63
3.7.3	Slip Melting Point (SMP)	63
3.7.4	Iodine Value (IV)	65
3.7.5	Triglyceride (TG) Content	66
3.7.6	Free Fatty Acid Composition	67
3.8	Optimization Process	68
3.8.1	Design of Experiment (DOE)	71
3.8.2	Central Composite Design (CCD)	73
3.8.3	Evaluation of Fitted Model	76
3.8.4	Determination of Optimal Condition	77
3.9	Mass and Heat Transfer Coefficient	77
3.10	Energy Utilities and Economic Analysis	79
 <b>4</b>	 <b>EFFECT OF OPERATING CONDITION ON PROGRESSIVE FREEZE CONCENTRATION OF RBDPO</b>	 <b>81</b>
4.1	Introduction	81
4.1.1	Effect of Circulation Flowrate	83
4.1.2	Effect of Coolant Temperature	89
4.1.3	Effect of Circulation Time	94
4.1.4	Effect of Initial Iodine Value	98

<b>5</b>	<b>OPTIMIZATION PROCESS OF THE PARAMETER CONDITION</b>	<b>102</b>
5.1	Introduction	102
5.2	Experimental Design	103
5.3	Optimization of the System Efficiency According the K-Value	105
5.3.1	Model Adequacy Check	107
5.3.2	Response Surface Contour Plot	114
5.3.2.1	Effect and Interaction of CTemp ( $X_2$ ) and CF ( $X_1$ )	115
5.3.2.2	Effect and Interaction of CTime ( $X_3$ ) and CF ( $X_1$ )	117
5.3.2.3	Effect and Interaction of IIV ( $X_4$ ) and CF ( $X_1$ )	118
5.3.2.4	Effect and Interaction of CTime ( $X_3$ ) and CTemp ( $X_2$ )	120
5.3.2.5	Effect and Interaction of IIV ( $X_4$ ) and CTemp ( $X_2$ )	122
5.3.2.6	Effect and Interaction of IIV ( $X_4$ ) and CTime ( $X_3$ )	124
5.3.3	Optimum Operating Condition	126
5.4	Optimization of the System Efficiency According the TG of Olein	127
5.4.1	Model Adequacy Check	129
5.4.2	Response Surface Contour Plot	135
5.4.3	Optimum Operating Condition	139
<b>6</b>	<b>MASS AND HEAT TRANSFER STUDY, ENERGY CONSUMPTION AND COST ANALYSIS</b>	<b>141</b>
6.1	Mass and Heat Transfer Study	141
6.2	Mass Transfer Analysis	142
6.2.1	Calculation of Mass Flowrate Condition (2800 mL/min)	143
6.2.2	Overall result of Mass Balance for PFC Process	144
6.3	Overall Heat Transfer Coefficient	146

6.4	Energy Consumption and Cost Analysis	153
<b>7</b>	<b>CONCLUSION AND RECOMMENDATION</b>	156
7.1	General Conclusion	156
7.2	Recommendation of Future Work	158
	<b>REFFERENCES</b>	160
	Appendices A-C	171-183

## LIST OF TABLES

<b>TABLE NO.</b>	<b>TITLE</b>	<b>PAGE</b>
2.1	Composition of CPO	11
2.2	All fats in edible oils	12
2.3	Triacylglycerol composition of palm oil	13
2.4	Triglycerides of palm oil products (carbon no.)	14
2.5	Fatty acid composition of palm oil products	14
2.6	Physical composition of palm oil products	15
2.7	Example of basic application of palm oil and palm kernel	17
2.8	Production of Malaysia palm oil	18
2.9	Comparative costs of production of selected oils and fats	41
2.10	Cost production of olein by dry fractionation	42
3.1	PORAM standard specification for process palm oil	51
3.2	Experimental equipment and its specification	51
3.3	Specification of USB TC-08 temperature data acquisition tool.	53
3.4	Varied and constant variables of operating condition	59
3.5	An initial 2 <sup>4</sup> factorial design	71
3.6	Design of experiment for independent variables	72

3.7	Central composite design of independent variables	74
3.8	Correlation between coded and real variables values	75
3.9	ANOVA Table	76
4.1	Operating condition to investigate the effect of circulation flowrate	84
4.2	Operating condition to investigate the effect of coolant temperature	90
4.3	Operating condition to investigate the effect of circulation time	94
4.4	Operating condition to investigate the effect of initial IV	98
5.1	Experimental Design with coded and actual value	104
5.2	Response K-Value for 4 parameter condition run	106
5.3	K-Value of experimental and predicted for each run	109
5.4	ANOVA for K-value	112
5.5	Regression analysis for K-value	113
5.6	Optimum range of operating condition	127
5.7	Response of TG in olein for 4 parameter condition run	128
5.8	TG of olein as experimental and predicted data for each run	130
5.9	ANOVA for TG of olein	133
5.10	Regression analysis for TG of olein	134
5.11	Summary of the model for the effect and interaction between two factors of operating condition	139
5.12	Optimum range of operating condition	140

6.1	Overall mass balance of the PFC process	145
6.2	Calculation of $U_o$	151
6.3	Calculation of energy consumption and costing for cooling section	155

## LIST OF FIGURES

<b>FIGURE NO.</b>	<b>TITLE</b>	<b>PAGE</b>
1.1	Dry fractionation of edible oil	4
1.2	Different characteristic SFC and PFC	4
2.1	Chemical and physical refining routes	20
2.2	RBDPO fractionation process into RBD olein and RBD stearin	22
2.3	Integration of dry fractionation process in refining cycle	23
2.4	Integration of chemical refining routes	24
2.5	Dry fractionation of RBDPO	27
2.6	Apparatus for conventional PFC	37
2.7	Apparatus for new design PFC	38
2.8	Tubular ice system for scale-up of PFC	39
2.9	In general of CCD with consist of $2^k$ points	46
3.1	Experiment flowchart	49
3.2	Peristaltic pump and water bath	52
3.3	NIR Analyser	52
3.4	Gas Chromatography (GC)	54
3.5	Stainless Steel Coil Crystalliser (CC)	54
3.6	Schematic drawing for experimental set-up	56

3.7	The coil crystalliser (CC)	57
3.8	A steps of RSM application	70
3.9	Illustration of mass balance	77
4.1	Solid stearin formed on the inner wall of CC	82
4.2	Product of palm olein and palm stearin	83
4.3	SMP and IV for olein at different circulation flowrates	85
4.4	SMP and IV for stearin at different circulation flowrates	86
4.5	K-value, TG and yield of olein at different circulation flowrates	87
4.6	SMP and IV for olein at different coolant temperature	91
4.7	SMP and IV for stearin at different coolant temperature	92
4.8	K-value, TG and yield of olein at different coolant temperature	93
4.9	SMP and IV for olein at different circulation time.	95
4.10	SMP and IV for stearin at different circulation time	96
4.11	K-value, TG and yield of olein at different circulation time	97
4.12	SMP and IV for olein at different initial IV	99
4.13	SMP and IV for stearin at different initial IV	99
4.14	K-value, TG and yield of olein at different initial IV	100
5.1	Predicted vs. observed value for response K-value	110
5.2	Residual plot of quadratic model for the response K-value	110
5.3	Pareto Chart on K-value	114



5.4	Contour plot of CTemp against CF	115
5.5	Surface plot of K-value against CTemp and CF	116
5.6	Contour plot of CTime against CF	117
5.7	Surface plot of K-value against CTime and CF	118
5.8	Counter plot of IIV against CF	119
5.9	Surface plot of K-value against IIV and CF	120
5.10	Contour plot of CTime against CTemp	121
5.11	Surface plot of K-value against CTime and CTemp	122
5.12	Contour plot of IIV against CTemp	123
5.13	Surface plot of K-value against IIV and CTemp	124
5.14	Contour plot of IIV against CTime	125
5.15	Surface plot of K-value against IIV and CTime	126
5.16	Predicted vs observed value for the response TG of olein	131
5.17	Residual plot of quadratic model for the response TG of olein	132
5.18	Pareto Chart on TG of olein	134
5.19	2D contour plot for all possible combinations of factors to affect TG of olein	137
5.20	3D surface plot for all possible combinations of independent variable to affect TG of olein	138
6.1	Mass balance of condition flowrate 2800 mL/min	143
6.2	Temperature profile of the heating process in the PFC system	147
6.3	Temperature profile of the cooling process in the PFC system	148
6.4	$U_o$ at different time depending on coolant temperature applied	152

## 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
$\mu$	-	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
$\Delta H_m$	-	enthalpy of mixture
$\dot{Q}_{app}$	-	heat requirement
$K$	-	ratio of total amount
$^{\circ}\text{C}$	-	temperature
$W_{pred}$	-	Weight of predicted

**LIST OF APPENDICES**

<b>APPENDIX</b>	<b>TITLE</b>	<b>PAGE</b>
A1	Typical Convection Coefficients to Estimate Thermal Transfer	172
A2	Technical Information of Palm Oil Properties	173
B	F Distribution table	175
C1	Sample of TG (IV=50wijs)	178
C2	Sample of TG (IV=55wijs)	180
C3	FFA Composition of Palm Olein	182
C4	FFA Composition of Palm Stearin	183

## **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 its 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

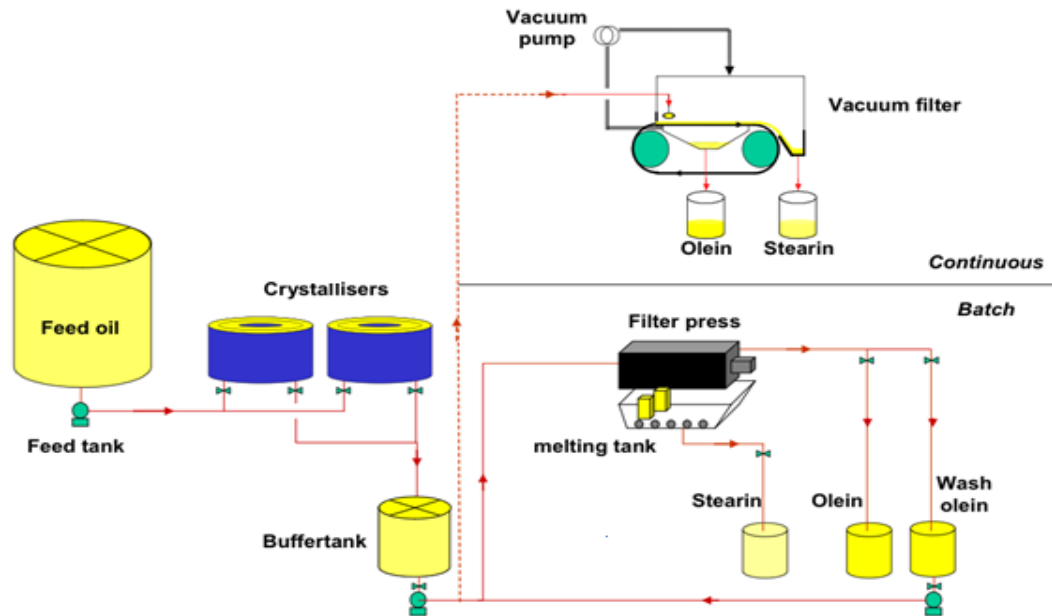


particular chemicals) (Hamm, 2005) whereas dry fractionation only requires crystalliser, filter and washing unit. However, there is a drawback in dry fractioning in which it is relatively inferior in separating process compared to the other two techniques.

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.

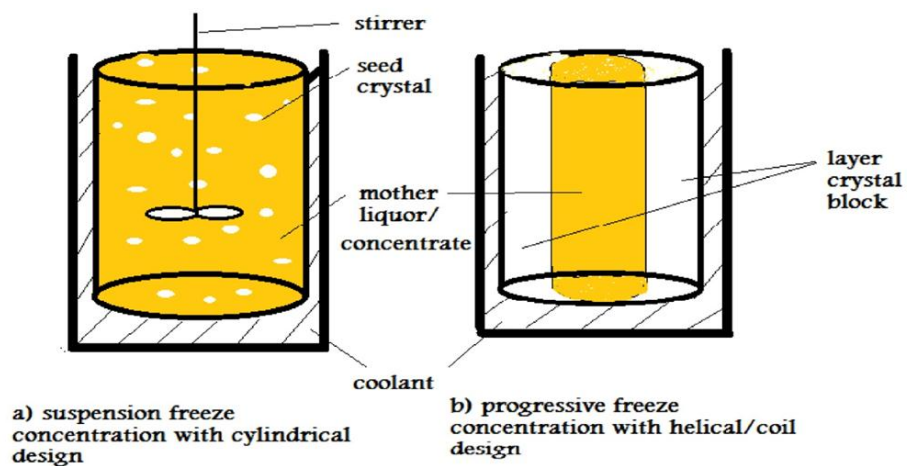
Dry fractionation or SFC system initially starts with Scrape Surface Heat Exchanger (SSHE) in crystalliser, producing small crystals. The process continues with a crystallisation process using a stirred tank crystalliser. The sample is separated into solid and liquid form according to the melting point 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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