## OPTIMIZATION OF PRESSED PALM MESOCARP FIBER RESIDUE OIL HEXANE EXTRACTION PLANT

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## **DEDICATION**

This thesis is dedicated to my grandmother, who raised me up and made me what I am today. Although she is no longer in this world, her words of wisdom and encouragements have always been in my mind and played a big part in helping me to overcome numerous obstacles and challenges to finish this thesis.

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### ABSTRACT

Operating hexane extraction plant to recover residue oil from pressed palm mesocarp fiber (PPMF) is one of the efforts done by oil palm millers in Malaysia and Indonesia in order to improve their overall oil extraction efficiency. Recovery is done in hexane extraction plant which consists of an extractor to extract residue oil from PPMF and distillation station to separate mixture of oil and hexane after the extraction process. However, oil yield of this plant is low whereby only up to 70 % of the available residual oil could be recovered. Besides, there is also a significant volume of hexane loss during distillation. Thus, an effort was made to optimize a commercial size hexane extraction plant to optimize its oil recovery with minimal hexane loss and least effect to oil quality and nutrient contents. The optimizations were done in phases whereby Phase 1 focused on optimization of extraction process by varying the extractor speed, hexane temperature and hexane flowrate and Phase 2 focused on optimizing the distillation process by varying flash evaporators, oil scrubber and oil dryer's temperature and pressure. Response surface methodology was used to optimize the extraction process with the experiments designed using Design Expert software. ANOVA shows p-value < 0.011 confirming the experiment model while all selected experiments factors were also found to be significant towards oil yield. The optimum condition for extraction process was 20 Hz extractor speed, 14 m<sup>3</sup>/hr hexane flowrate and 60 °C hexane temperature. Distillation process optimization was done using Aspen Plus software which was also cross-checked with actual running plant. The optimum condition for distillation process was as follows: Flash evaporator no. 1 at 80 °C and 380 mm Hg; Flash evaporator no. 2 at 130 °C and 420 mm Hg; Flash evaporator no. 3 at 148 °C and 510 mm Hg; Oil scrubber at 120 °C and 590 mm Hg; Oil dryer at 80 °C and 640 mm Hg. The above exercise resulted in increase of overall oil extraction efficiency by 18.8 % with no effect on hexane loss, oil quality and nutrient content. The improvement if expressed in monetary value translates to an additional income of RM 33,725 per month for the plant involved.

#### ABSTRAK

Pengoperasian loji pengekstrakan heksana untuk memperolehi semula minyak sisa daripada mesokarp buah sawit yang telah diperah (PPMF) adalah salah satu usaha yang dijalankan oleh kilang-kilang kelapa sawit di Malaysia dan Indonesia bagi meningkatkan kecekapan pengekstrakan minyak secara keseluruhan. Perolehan semula minyak sisa dijalankan di loji pengekstrakan heksana yang terdiri daripada pengekstrak untuk mengekstrak minyak sisa dari PPMF dan stesen penyulingan untuk mengasingkan campuran minyak dan heksana selepas proses pengekstrakan. Walau bagaimanapun, hasil minyak bagi loji ini adalah rendah di mana hanya 70 % daripada minyak sisa boleh diperolehi semula. Selain itu, jumlah kehilangan heksana yang ketara juga dilaporkan berlaku semasa penyulingan. Oleh itu, satu usaha telah dibuat untuk mengoptimumkan sebuah loji pengekstrakan heksana bersaiz komersial untuk mengoptimumkan perolehan semula minyak tanpa kesan terhadap kehilangan heksana, kualiti minyak dan kandungan nutriennya. Pengoptimuman telah dilaksanakan dalam dua fasa di mana Fasa 1 fokus kepada pengoptimuman proses pengekstrakan dengan mengubah kelajuan pengekstrak, suhu heksana dan kadar aliran heksana, manakala Fasa 2 fokus kepada pengoptimuman proses penyulingan dengan mengubah suhu dan tekanan penyejat kilat, penggahar minyak dan pengering minyak. Kaedah tindak balas permukaan telah digunakan untuk mengoptimumkan proses pengekstrakan dengan ujikaji yang direkabentuk menggunakan perisian Design Expert. Nilai p < 0.011 yang diperolehi daripada ANOVA mengesahkan model ujikaji yang direkabentuk, manakala semua faktor ujikaji yang dipilih didapati mempunyai kesan ketara terhadap hasil minyak. Operasi optimum untuk proses pengekstrakan ialah pada kelajuan pengekstrak 20 Hz, kadar aliran heksana 14 m<sup>3</sup>/jam dan suhu heksana 60 °C. Pengoptimuman proses penyulingan telah dilakukan menggunakan perisian Aspen Plus yang juga disahkan melalui ujikaji di loji. Pengendalian optimum untuk proses penyulingan adalah seperti berikut: Penyejat kilat no. 1 pada 80 °C dan 380 mm Hg; Penyejat kilat no. 2 pada 130 °C dan 420 mm Hg; Penyejat kilat no. 3 pada 148 °C dan 510 mm Hg; penggahar minyak pada 120 °C dan 590 mm Hg; Pengering minyak pada 80 °C dan 640 mm Hg. Hasil pengoptimuman yang diperolehi telah meningkatkan kecekapan pengekstrakan minyak secara keseluruhan kepada 18.8 % tanpa kesan ke atas kehilangan heksana, kualiti minyak dan kandungan nutrien. Penambahbaikan ini jika diterjemahkan dalam bentuk kewangan bermaksud penambahan pendapatan sebanyak RM 33,725 sebulan untuk loji yang tersebut.

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# LIST OF ABBREVIATIONS

CPO	-	Crude Palm Oil
FFA	-	Free Fatty Acid
MF	-	Mesocarp Fiber
PPMF	-	Pressed Palm Mesocarp Fiber
DOBI	-	Deterioration of Bleachability Index
MPOB	-	Malaysian Palm Oil Board
FFB	-	Fresh Fruit Bunches
POM	-	Palm Oil Mill
$CO_2$	-	Carbon Dioxide
RSM	-	Response Surface Methodology
CCD	-	Central Composite Design
IPA	-	Isopropanol Alcohol
NaOH	-	Sodium Hydroxide

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#### **CHAPTER 1**

#### **INTRODUCTION**

#### **1.1 Background of Study**

As at end of December 2016, among all oils and fats produced and consumed globally, palm oil accounts for 29% and 30% respectively while Malaysia accounts for 30% of world palm oil production second to Indonesia at 54%. For the same period, Malaysia has contributed about 37% to the total palm oil traded throughout the world while Indonesia has contributed 53%. (MPOC, 2019). Being the 2<sup>nd</sup> largest producer and exporter of palm oil, Malaysia has an important role to play in fulfilling growing global need of palm oil with increasing world population. Thus, sustaining operation of palm oil mill is very important and always have been a biggest challenge among palm oil millers in the country.

Production of palm oil starts at palm oil mill with extraction of crude palm oil (CPO) from oil palm fruitlets' mesocarp via mechanical pressing using twin screw press. This CPO will then further be treated at palm oil refineries to produce RBD olein and stearin which are raw materials for many downstream products such as cooking oil, margarine, confectionaries, pharmaceutical products and cosmetic products. CPO gaining lots of popularity among all of the oil and fats in the world due to its high oil yield per planted area, high thermal stability and also contain many essential nutrients that do not have in the other edible oils in the world.

Extraction of CPO using twin screw press machine is the only technology available now in the palm oil industry which deemed to be the most economic and efficient method so far. A trial was conducted earlier to extract the CPO using solvent (hexane) however not feasible due to its high operating cost mainly to purchase hexane. Besides the above, the other major reason the idea was abandoned was due to high toxicity of hexane which can cause detrimental effect to both peripheral and central nervous system of the industrial workers whom exposed to them for a stipulated period (Huang, 2008).

CPO extraction methods have been evolving from the day it was commercially produced by West Africans way before 1914 (Berger and Martin, 2000). From a simple machine, i.e. cylinder fitted with manually operated beaters upgraded to a Duchshcer press made of cylindrical cage of wooden slats and a ram on a screw thread turned manually by means of long bar forcing the ram on the fruits. This was then upgraded further to use perforated cylindrical metal cage instead of wooden slats. The development continues with the introduction of hand-operated hydraulic press by Stork in 1959 capable of processing 600 -1,000 pounds of fruit with 80% of oil recovery. This was vastly used and its hydraulic mechanism motorized later. Concurrently Colin expeller which was introduced much earlier in 1930 which consist of perforated cylindrical cage, fitted with spiral screw or worm operated manually through a gear gain highest popularity in Cameroon initially and later on spread all over the world. Today's modern-day twin-screw press is actually originated from Colin expeller which has been upgraded and improved over many years through continuous research and development from various palm oil producers and machinery manufacturers.

However, even the most advance twin screw press available now could only provide up to a maximum 96% oil recovery efficiency. According to Rusnani, Abdul and Choo (2012), oil residue in pressed palm mesocarp fiber (PPMF) after pressing with screw press ranges between 5 - 8% on dry weight basis or 3 - 5% on wet basis and this is very much affected by the milling process. This residue oil has been regarded as acceptable loss by the palm oil millers. The PPMF with residue oil burnt in a biomass boiler as fuel without any further recovery of oil. The loss of 4% residue oil compute to a total national loss of RM 1.25 billion/yr. based on 97,825,268 MT of fresh fruit bunches (FFB) processed in the country in the year of 2018 (Malaysian Palm Oil Board). Quite recently this practice has been changed with the introduction of solvent extraction plant to the palm oil industry to recover residual oil from PPMF.

With this plant, millers could process their oil laden PPMF after twin screw press to recover the residual oil. Due to its attractive investment returns and shorter payback periods many big players from the palm oil industry in the country and Indonesia such as Sime Darby, KL-Kepong (KLK), FELDA and FELCRA has adopted this technology with total of 18 numbers of such solvent extraction plants operating so far in our country as at end of December 2018.

### **1.2 Problem Statement**

Recovery of residue oil from PPMF is trending now among palm oil millers throughout Malaysia and Indonesia. Besides giving added revenue to the owner through additional oil recovery, PPMF oil is also found to be rich in numerous nutrients such as carotenoids, vitamin E (tocopherol and tocotrienols), sterols and coenzyme Q10 which could be extracted and further processed to become pharmaceutical products. So far recovering of PPMF residue oil using solvent (hexane) extraction method is the only technology that is commercially available. The technology which was invented and patented by Messrs. Eonmetall Technology Sdn. Bhd. uses food grade hexane for extraction. Extraction of residual oil from PPMF was done inside a band conveyor extractor using hexane and the extracted oil was purified with series of heating, evaporation and condensation to separate the PPMF oil and hexane mixture also called as "miscella".

Although the technology has been accepted by many palm oil millers, there are few weaknesses that could not be solved till now. The yield of PPMF oil from the hexane extraction plant was not achievable as promised earlier by the technology provider. This has affected the profitability of the investors thus seriously affecting marketability of this plant in palm oil industry. One of the possible reasons for this is due to the lower extraction temperature which is maintained around 52- 55°C so as to avoid hexane evaporation as hexane has to be in liquid state at all times during

extraction so as to allow penetration into PPMF and solute the oil within. However current operation temperature is way too low against the boiling point of hexane at 68 – 69°C and thus it still can be increased. This is because at higher solvent temperature solubility of extract into solvent will increase thus overall oil extraction will also increase as reported by Taher et al (2014). Besides extraction temperature, the other reason that could probably affect extraction rate is volume of solvent used for extraction as reported by Zhao et al (2009) and also contact time between solvent and feed material during extraction process as reported by Ghitescu et al (2015). Currently  $7m^3$ /hr of hexane sprayed onto the PPMF with total contact time of 3 hours. However, these rates are still adjustable but limited to  $14m^3$ /hr of hexane flow and 6 hours of contact time due to fixed size of hexane storage tank and extractor drive system at the plant.

Besides the 3 factors, 2 other possible factors for the lower oil yield are initial moisture content in PPMF which is preferred to be as low as possible as stated by Rusnani et al (2012) and the size of feed material which is also preferred to be as small as possible to increase contact surface area (KMEC Engineering, 2019). However, these two factors are fixed at the particular PPMF residue oil hexane extraction plant being studied in this research study due to plant constraints. PPMF that comes out from the mechanical screw press after pressing at palm oil mill is directly conveyed into the hexane extraction plant without any facility for pre-treatment. Installation of a dryer or muncher to dry and further cut this PPMF to make it more fine and drier before entering the plant requires major revamp of the whole setup of the mill and hexane extraction plant. Currently the moisture content in PPMF ranges between 45 - 50% on mass while PPMF lengths varies between 1 cm - 9 cm. Figure 1.1 and 1.2 show the condition of the PPMF when entering into hexane extraction plant.



Figure 1.1 Palm mesocarp fiber being expelled out from the mechanical screw press after pressing at the palm oil mill.



Figure 1.2 Conditions of the PPMF from the mechanical screw press that will be conveyed into the hexane extraction plant.

Besides lower oil yield, hexane loss during distillation process is also another area of concern in this study. This is because significant volume of hexane is consumed at this plant for the extraction process and some of the hexane is lost during the distillation process. The technology provider has set loss of 4 litres of hexane per every tonne of PPMF fiber processed as guaranteed performance for this plant and this is easily achieved so far. However, this 4 litres/mt of hexane would translate to a loss of RM 38,412 every month for this plant and if this could be reduced, significant financial benefit could be realized by the plant. Apart from the oil yield and hexane loss, quality of final PPMF oil is also another major concern in the industry. The oil is found to be with high content of free fatty acid (FFA) and high oxidation level indicated by its low deterioration of bleachability index (DOBI). These two off qualities require high refining costs at palm oil refineries to rectify which make this oil not acceptable by the palm oil refineries. The most possible reason for these off qualities is because the oil in mesocarp fiber (MF) is subjected to continuous high temperature and moisty environment due to contact with live saturated steam during digestion process at the mill and distillation process at the hexane extraction plant. Presence of iron in PPMF oil which is mostly in the form of iron filings also partly contributed to the oxidation of oil in the PPMF. These iron filings usually come from the wear and tear part of the machineries. Due to the off qualities and unacceptance by the palm oil refineries, most of this PPMF oil is sold as animal feed to the poultries and farms.

The current extraction temperature is at a maximum temperature of 55°C only so no further detrimental effect towards oil quality is expected here. However, distillation temperature is ranges between 80 to 136°C which is higher than that at the mill and this may contribute to further deterioration of FFA and DOBI of the PPMF oil. Further oxidation due to the presence of iron in the oil has been taken care at this plant whereby all sections of the plant which comes in contact with the PPMF and PPMF oil are constructed using stainless steel material. Thus, there will be no free iron to act as an oxidative agent to promote oxidation process. Therefore, potential threat to the oil quality is only expected from the distillation temperature.

PPMF oil is also highly nutritious and found to be a rich source of carotenoids, vitamin E (tocopherol and tocotrienols) and sterols as reported by Yuen et al (1996). These nutrients if extracted and processed to become pharmaceutical products have potential to replace many inorganic or chemical based medicines. It is a big loss to give away this special oil as animal feed rather than make use of its nutrients for humans. Therefore, it would be very beneficial to the palm oil industry and our country if the nutrient content of this oil is preserved during the distillation process of the miscella.

### **1.3** Significance of the study

To the best of my knowledge, no studies have been conducted so far to improve or optimize the hexane extraction plant concerning on the oil yield, hexane loss, oil quality and nutrient contents. Operation of all available plants so far were done according to the initial procedures outlined by the technology provider upon handover of the plant. Hopefully at the end of this study the plant could be optimized to produce high yield PPMF oil with lowest hexane loss possible, minimum deterioration to the oil quality and minimum loss on nutrient content.

#### **1.4** Objective of the Research

Main objective of this study is to optimize the PPMF residue oil extraction plant to obtain highest oil yield with lowest hexane loss, minimum effect to oil quality and nutrients loss. For this the following sub-objectives were outlined:

- i. To identify all factors in the hexane extraction plant that potentially could affect the PPMF oil yield, hexane loss, PPMF oil quality and nutrient contents
- ii. To model/design experiments on the effect to PPMF oil yield, hexane loss,PPMF oil quality and nutrient contents by varying the identified factors
- iii. To execute the designed experiments, analyze the collected data and finally optimize the plant based on outcome of this project study.

### 1.5 Scope of Study

This project focuses to optimize a commercial running PPMF residual oil hexane extraction plant to achieve high oil yield with lowest hexane loss, minimum effect to oil quality and minimum nutrient content loss. Scope of this project study was limited to two sections of the plant namely extraction and distillation as highlighted in Figure 1.3. Optimization studies were conducted in two phases whereby Phase 1 meant to optimize extraction section followed by Phase 2 to optimize distillation section. Series of studies with varying operating parameters of the extraction and distillation process were conducted to determine optimum operating parameters that could achieve the objective of this study.

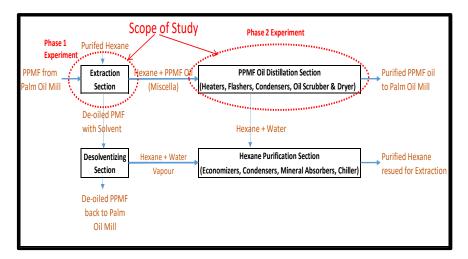


Figure 1.3 Scope of Study - Pressed Palm Mesocarp Fiber Residue Oil Hexane Extraction Plant

Desolventizing section mainly functions to evaporate residue hexane in PPMF after the extraction process using high-pressure steam at 184°C and 10 barg and this section was not considered to be part of this project study. This is because the boiling point of hexane is around 68°C and with such high temperature steam definitely all residual hexane will be evaporated and recovered.

#### 1.5.1 Phase 1 optimization

In Phase 1 the extraction section is optimized because this is where the residual oil is extracted from PPMF and any inefficiencies here could affect PPMF oil yield. Extraction section consist of an electric driven mesh band conveyor type continuous extractor and hexane spraying system equipped with heating jacket. Hexane are sprayed onto PPMF that travelling on the band conveyor to extract residual oil within PPMF. A steam jacket that wraps hexane pipeline allows temperature of sprayed hexane adjusted. Total length of the extractor is 23.8 m and capable of conveying maximum 13.5 mt of PPMF per hour.

Variable parameters of this section which potentially could affect the oil yield are extractor speed, hexane temperature and hexane flowrate which were being maintained at 18Hz, 52°C and 8m<sup>3</sup>/hr before this research study. Design Expert software was used to design experiments to analyze these parameters to check on their interaction towards oil yield. Upon successful analysis, Response Surface Methodology (RSM) was used to optimize the extraction section's oil yield.

### 1.5.2 Phase 2 optimization

Phase 2 optimization was meant to optimize distillation section which functions to separate mixture of PPMF residual oil and hexane which is also called miscella. Distillation section consist of 3 units flash evaporators, 1-unit oil stripper, 1unit oil dryer and 2 units of heaters to heat up miscella temperature before evaporation and stripping. The section can process up to 9,000 litres of miscella per hour. Phase 2 optimization was performed after successful Phase 1 optimization. Variable operating parameters from distillation section that potentially could affect the PPMF oil yield and hexane loss were identified at first. The identified parameters were flash evaporators' temperature and pressure, oil stripper's temperature and pressure and oil dryer's temperature and pressure. The initial operating temperature and pressure of these parameters before optimization were as follows:

- i. Flash evaporator  $1 80^{\circ}$ C and 300 mmHg
- ii. Flash evaporator 2 125°C and 360 mmHg
- iii. Flash evaporator 3 136°C and 500 mmHg
- iv. Oil stripper  $-120^{\circ}$ C and 590 mmHg
- v. Oil dryer  $-80^{\circ}$ C and 640 mmHg

The complete distillation section was simulated in Aspen Plus software and its variable parameters mentioned (i - v.) were varied to find optimum parameters that gives optimum oil yield with minimum hexane loss.

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