
**UNIVERSITI PUTRA MALAYSIA**

***OPTIMIZATION OF PALM OIL EXTRACTION FROM DECANTER CAKE BY USING SOXHLET EXTRACTION***

**TIE HIENG ONG**

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**BACHELOR OF AGRICULTURAL AND BIOSYSTEMS ENGINEERING WITH HONOURS**

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**APPROVAL SHEET**

This thesis entitled **"Optimization of Palm Oil Extraction from Decanter Cake by Using Soxhlet Extraction"** prepared and submitted by **Tie Hieng Ong** as fulfilment of requirements for the degree of Bachelor of Agricultural and Biosystems Engineering with Honours has been examined and is recommended and acceptance.

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## DECLARATION

I hereby declare that this 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 Universiti Putra Malaysia or at any other institution.

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## ABSTRACT

Oil palm decanter cake (OPDC) is a solid biomass waste from palm oil mill where it is generated from a decanter. A proposed method for palm oil extraction was developed with the application of Soxhlet extraction through Response Surface Methodology (RSM). The main objective of this study was to determine the optimum of two parameters such as reaction time and solid to solvent ratio to achieve the maximum oil yield by Soxhlet extraction. The optimum parameters for laboratory scale of Soxhlet extraction designed by RSM were found to be at 4.923 hours of reaction time and solid to solvent ratio of 1:10. The proposed model design by RSM shows $R^2$ value of 0.776 where the experimental parameters were significant to the result. Considering the optimum parameters achieved based on three criteria stated such as minimum reaction time, minimum solvent ratio and maximum oil yield and the optimized data was employed for comparison of oil yield for OPDC without and with microwave pre-treatment. OPDC with microwave pre-treatment yielded 3.289g of palm oil which was higher than that of OPDC without microwave pre-treatment which yielded only 3.107g of palm oil. Physical chemical analyses on the extracted oil and biological analysis on OPDC were also determined for both samples. The result for specific gravity, L*a*b colour and refractive of extracted oil for OPDC without and with pre-treatment were 0.960 and 0.897; 58.8L*6.0a*29.3b and 58.7L*5.8a*29.0b; and 1.462 nd. and 1.462 nd. respectively. Biological analysis with SEM images indicated that OPDC with pre-treatment has more shrinkage on the surface after Soxhlet extraction compare to OPDC without pre-treatment. Chemical analysis by using FTIR showed that both oil sample with two major functional groups indicated the present of fatty acid in the oil sample. Result of this study revealed that RSM helps to optimize parameters in agricultural processing. The extracted oil from OPDC has potential of being used as biodiesel due to the present of ester.

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## LIST OF ABBREVIATIONS

| | |
|---|---|
| FFB | Fresh Fruit Bunch |
| POME | Palm Oil Mill Effluent |
| EFB | Empty Fruit Bunch |
| PMF | Palm Mesocarp Fibre |
| OPDC | Oil Palm Decanter Cake |
| HCWE | Hot Compressed Water Extraction |
| SFE | Supercritical Fluid Extraction |
| OER | Oil Extraction Rate |
| CPO | Crude Palm Oil |
| FFA | Free Fatty Acid |
| RSM | Response Surface Methodology |
| MPOB | Malaysia Palm Oil Board |
| RI | Refractive Index |
| SG | Specific Gravity |
| SEM | Scanning Electron Microscope |
| FTIR | Fourier Transform Infrared Spectroscopy |
| MCL | Material Characterization Laboratory |

# CHAPTER 1: INTRODUCTION

## 1.1 Background

Oil palm sector is one of the most major industries in Malaysia. Malaysia is the world second largest palm oil exporter in 2016 after Indonesia where the main export market is India with an intake of 2.83 million tonnes or 17.6% of total palm oil export (Kushairi.A, 2017). Aside from being one of the largest palm oil producer, Malaysia also generated profusion amount of industrial waste (Liew et al., 2014). The estimated generated waste from every ton of fresh fruit bunch (FFB) range from 0.6 to 0.8$m^3$ of palm oil mill effluent (POME), 22 to 23% of empty fruit bunch (EFB), 13.5% of palm mesocarp fibre (PMF) and 4-5% of oil palm decanter cake(OPDC) from a typical palm oil mill (Sahad *et al.*, 2014).

High production of biomass waste has negatively affected the total oil extraction rate (OER) of palm oil industry due to the losses of oil in the waste (Sahad *et al.*, 2014). The OER indicates the actual amount of oils extracted from FFB, as well as overall efficiency of palm oil mills. Hence approaches should be done to convert these biomass waste into another form of energy or usage. The current biomass residues are being used as fuel in the boiler or be converted into fertilizer. The solid biomass wastes were being used as main source of energy input for few palm oil mills to produce electricity and steam for palm oil production process such as sterilizer(Wu *et al.*, 2017). Few researchers have studied the suitability of OPDC as ruminant feed, plant fertilizer and composting material (Bakri, 2013; Sahad *et al.*, 2014). This study will discover the possibility of oil extracts as valuable feedstock rather than disposal on landfills due to present of oil in OPDC which provide more energy during burning process.

## 1.2 Problem Statement

Environmental issues are being concerned greatly in Malaysia as the palm oil mill can cause much environmental pollution from its waste such as oil palm decanter cake (OPDC). Huge amount of OPDC production requires a large land area for composting and this will cause pollution hazards such as soil and water pollution (Farhana.S, 2010). Most OPDC has been used as fertilizer, animal feed raw material for cellulose and polyose, bio-surfactant, biobutanol, bio-diesel and bio-oil productions over last decades (Kanchanasuta and Pisutpaisal, 2016). It is important for use to explore the process of extracting oil from oil palm at optimum output level as there are currently few extraction method available in the industry such as Hot Compressed Water Extraction (HCWE), Supercritical Fluid Extraction (SFE) and the conventional Soxhlet Extraction (laboratory scale) (Ameera and Arsad, 2016; Costa et al., 2018; Sarip et al., 2016).

Demand for biodiesel sector is increasing steadily (Szulczyk and Atiqur Rahman Khan, 2018). Biodiesel is a renewable vehicle fuel based on biomass. Few researchers showed that palm oil can be use as biodiesel for vehicle with diesel engine (Archer et al., 2018). This study will discover the functional group of extracted oil yield from OPDC as the major component for biodiesel is ester. Current palm oil industry is using sterilization on FFB to loosen the fruitlet from the bunch and to promote oil extraction. However there are few other pre-treatment that can be done on the fruits to enhance the process of palm oil extraction such as microwave and ultrasonic pre-treatment (Luque and Priego-Capote, 2010). Moreover, OPDC affect the total oil extraction rate (OER)of palm oil industry as there is still leftover oil in OPDC. Large production of OPDC will negatively affect the OER. Hence, it is such a waste for not fully utilise the leftover oil in OPDC.

## 1.3 Research question

i. What are the optimum parameters such as reaction time and solid to solvent ratio to obtain the highest oil yield from oil palm decanter cake (OPDC) by using Soxhlet extractor?

ii. How different in term of oil yield for OPDC with microwave pre-treatment and OPDC without pre-treatment?

iii. What is the biological characteristic of OPDC and physical, chemical characteristics of extracted oil from OPDC?

## 1.4 Aims and objectives

The primary focus of this study is to design an experimental work for extracting palm oil from OPDC by using Soxhlet extraction. Further work has also been taken to compare the performance of palm oil yield of Soxhlet extraction from OPDC with microwave pre-treatment and without pre-treatment.

This study embarks on the following specific objectives:

i. To determine the optimum of two operating parameters such as reaction time and solid to solvent ratio on the yield of palm oil extraction using Response Surface Methodology (RSM).

ii. To compare the yield of extracted oil for OPDC with microwave pre-treatment and without pre-treatment.

iii. To determine the biological analysis of OPDC and physical, chemical analyses of extracted oil.

## 1.5 Scope of research

Soxhlet extraction method was chosen due to the limitation of available laboratory equipment that is currently available in Engineering Faculty. The optimum parameters of Soxhlet extraction was investigated in this study were reaction time and solid to solvent ratio. Physical and chemical analysis of extracted oil from OPDC and the biological analysis of OPDC with and without microwave pre-treatment were investigated. The microwave pre-treatment was conducted based on method descried by Jason (Jason, 2017).

# CHAPTER 2 LITERATURE REVIEW

## 2.1 Introduction

Oil palm decanter cake (OPDC), empty fruit bunch (EFB) and palm mesocarp fibre (PMF) were the solid waste generated from palm oil mill. Oil palm decanter cake (OPDC) is a solid biomass waste from palm oil mill where it is generated from a decanter. Decanter cake was used to treat the underflow of the clarification tanks in the milling process where it separate the leftover crude palm oil (CPO) from the sludge before feeding into the purification tank and discharged from decanter as OPDC (Sahad *et al.*, 2014). Basically, OPDC is produced by the extraction of solids from palm oil sludge. Decanter cake sludge is in liquid form and is a product from centrifugal pump without going through decanter, while decanter cake solid is in solid form which is a product from decanter. The production rate of decanter cake amounts to 4 – 5 % weight of fresh fruit bunch processed. Fresh decanter cake contains over 70% moisture, while the dry matter contains oils, fibre and inorganic components. Moisture content of decanter cake should be reduced before Soxhlet extraction to increase the effectiveness of extraction (Ameera and Arsad, 2016). Figure 2.1 below showed the system of palm oil mill production and its output. Figure 2.2 showed the cross-section view of decanter and how it works.

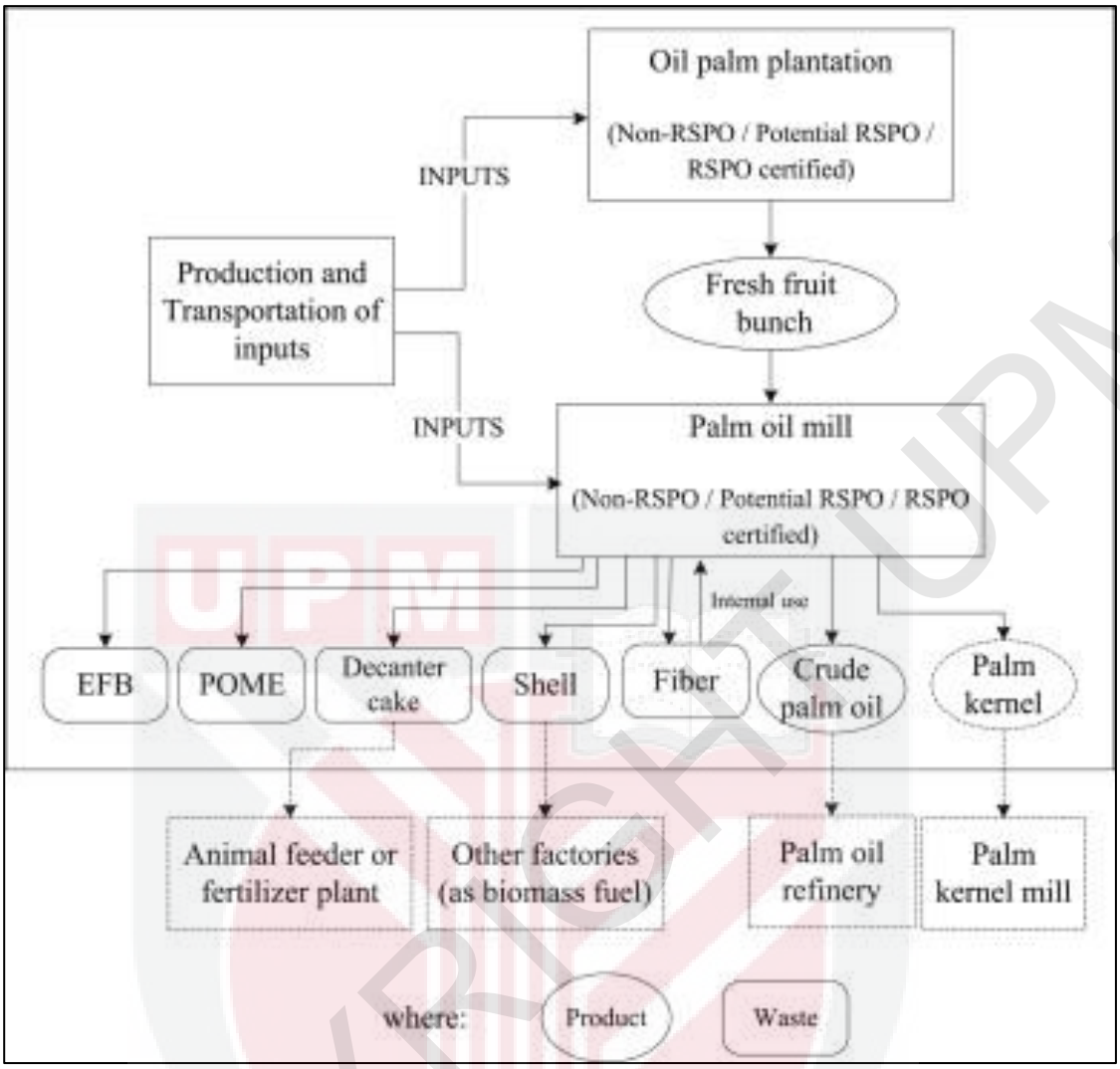



Figure 2.1: The system of palm oil mill production. Adapted from (Saswatetecha, 2015)

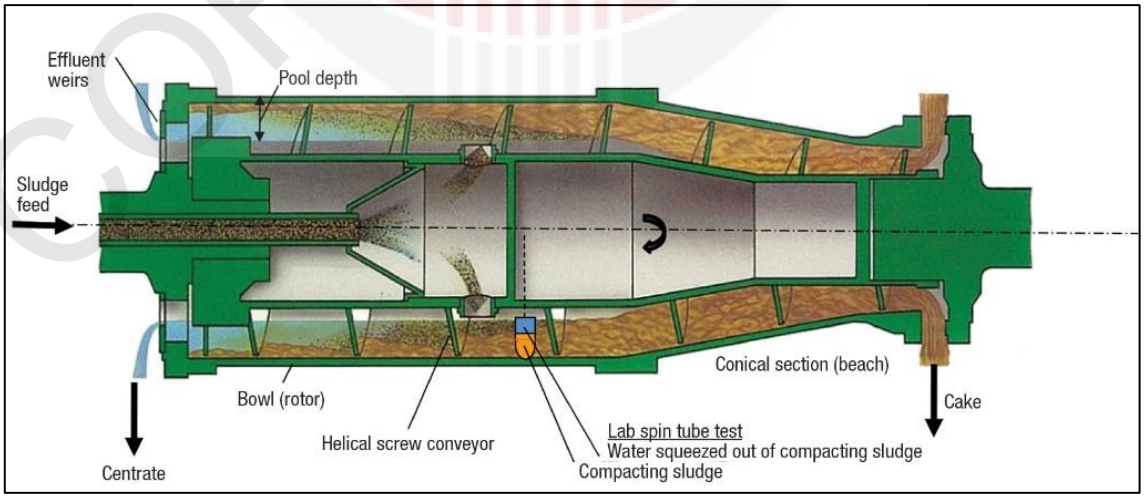



Figure 2.2: Cross section view of decanter. Adapted from (TFH, 2016)

## 2.2 Extraction Method

Crude palm oil (CPO) is obtained from mechanical screw press of oil palm mesocarp. The conventional palm oil milling process consists of: sterilization of fresh fruit bunches (FFB) for the termination of enzymatic hydrolysis of oil; stripping and digestion of fruits; screw press for the extraction of crude oil; screening of crude oil using vibrating mechanism; clarification of crude oil from water; and centrifugation and vacuum drying oil. However, there are few other methods have been introduced for oil extraction such as: Hot Compressed Water Extraction (HCWE), Supercritical Fluid Extraction (SFE) and Soxhlet Extraction as shown in Table 2.1 (Akanda *et al.*, 2012; Ameera and Arsad, 2016; Sarip *et al.*, 2016; Costa *et al.*, 2018).

Table 2.1: Oil Extraction by Different Method

| **Method** | **Sample** | **Operating Conditions** | **Yield (g-oil/g-dried sample)** | **Reference** |
|---|---|---|---|---|
| Hot Compressed Water Extraction (HCWE) | Palm Mesocarp | Palm mesocarp treated under pressure of 30, 40 and 50 bar with temperature ranging from 120 to 180 °C through semi-batch extraction of 10 min interval and total extraction time of 60 min. | 0.541 ± 0.005 | (Sarip *et al.*, 2016) |
| Supercritical Fluid Extraction (SFE) | Palm Kernal Cake | Supercritical-$CO_2$ Extraction of kernel cake at temperatures of (40 to 80) °C and pressures of (150 to 350) bar | 7.82 ± 0.28 | (Costa *et al.*, 2018) |
| Soxhlet Extraction | Vernonia cinereal Leaves | Soxhlet extraction of Vernonia cinerea leaves with extraction time (1–4 h), feed-to-solvent (1:10–1:25 g/mL) and ethanol concentration (20–80% v/v). | 10.01 ± 0.85 | (Alara, and Ukaegbu, 2018) |

### 2.2.1 Hot Compressed Water Extraction (HCWE)

HCWE is a favourable alternative technology which have proven to be successful in many applications (Sarip *et al.*, 2016). HCWE is promising and has potential to be applied for CPO extraction due to the positive result on oil and bioactive extraction. Nitrogen gas was passed through one of the ports of vessel and bubbled for 2 min to purge out the air and dissolved oxygen in water. The sample was heated based on the parameters set up then 500ml of fresh water was pumped into the extraction vessel for the next 10 minutes interval and the process was repeated. The schematic diagram of HCWE extractor is shown in Figure 2.3. HCWE was applied in the extraction of CPO from palm mesocarp where the operating conditions investigated were pressure of 30, 40 and 50 bar with varied temperature ranging from 120 to 180 °C through semi-batch extraction of 10 min interval and total extraction time of 60 min.

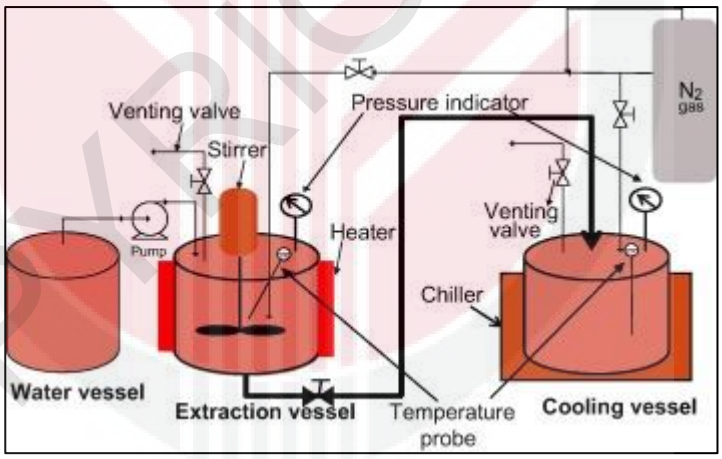



Figure 2.3: Schematic diagram of HCWE extractor. Adapted from *(Sarip* et al.*, 2016)*

Research showed that the maximum CPO extracted in the oil layer was 0.541 ± 0.005 (g-oil/g-dried mesocarp) with averaged FFA of 0.81 ± 0.08% at the optimum operating condition of 160 °C and 50 bar (Sarip et al., 2016).

### 2.2.2 Supercritical Fluid Extraction (SFE)

Supercritical-$CO_2$ referred to supercritical fluid extraction (SFE) that uses carbon dioxide ($CO_2$) as a solvent which is a nontoxic, inexpensive, non-flammable, and non-polluting supercritical fluid solvent for the extraction of natural products (Akanda *et al.*, 2012) Nearly 100% of oil can be extracted by using SFE and it is regarded as safe, with organic solvent-free extracts having superior organoleptic profiles. Research on using super critical fluid as solvent in palm oil extraction has been done. $CO_2$ has low critical temperature (31.1 °C) thus it is attractive for thermally labile food product while other solvents like ethane and propane are also used as supercritical fluids for the extraction of natural compounds. Ethane and propane have high solvating power enabling higher solubility of lipid components compared to SC-$CO_2$. However, ethane and propane are highly flammable and high cost hence these solvents are not widely used. Figure 2.4 showed the examples of substances used as supercritical solvents and its corresponding critical temperature and pressure.

A research on recovering the residual oil from industrial palm kernel cake by using SFE at temperatures of (40 to 80) °C and pressures of (150 to 350) bar (da Costa *et al.*, 2018). The SFE unit components are shown in Figure 2.5. Supercritical fluid was used in SFE where it can break up multi component mixture based on the different volatile capacities of each component used. SFE promoted detachment of the extract from the solvent by simple expansion. They stated that SFE with superior mass transfer distinctiveness will enable easier release of solutes. The experimental results showed that the highest yield on dry basis was (7.82 ± 0.28) %, obtained at 350 bar/80 °C.

| Gases | Critical Temperature (K) | Critical Pressure (MPa) |
|---|---|---|
| Carbon dioxide | 304.17 | 7.38 |
| Ethane | 305.34 | 4.87 |
| Methane | 190.55 | 4.59 |
| Ethylene | 282.35 | 5.04 |
| Propane | 369.85 | 4.24 |
| Nitrous oxide | 309.15 | 7.28 |
| Acetylene | 308.70 | 6.24 |
| Hydrogen | 33.25 | 1.29 |
| Nitrogen | 126.24 | 3.39 |
| Oxygen | 154.58 | 5.04 |
| Neon | 44.40 | 2.65 |
| Argon | 150.66 | 4.86 |
| Xenon | 289.70 | 5.87 |

Figure 2.4: Example of supercritical fluid used as solvent. Adapted from (Akanda et al., 2012)

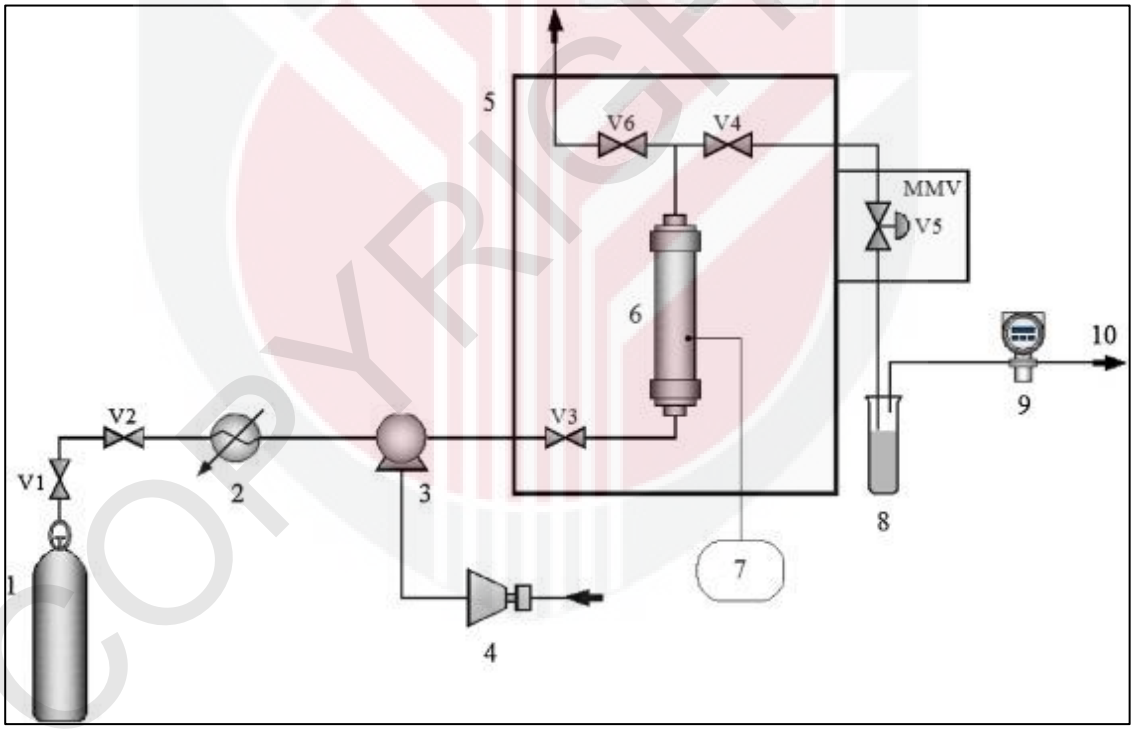



Figure 2.5: SFE unit components. 1. CO2 tank; 2. Cooling bath; 3. Pump; 4. Compressor; 5. Oven; 6. Extractor vessel; 7. Monitor; 8. Vial; 9. Flowmeter; 10. CO2 Outlet; V1 – V6 Flow control valves. Adapted from (Costa *et al.*, 2018)

### 2.2.3 Soxhlet Extraction

Soxhlet extraction is a conventional method used in the process of recovering phenolic compounds from plant matrix. Soxhlet extraction is the most commonly used method for extracting phenolic compounds as this extraction method has various advantages such as: low processing cost; simple operation; high performance; favourable for total recovery of extracts and less time and solvent consuming (Alara et al., 2018). During Soxhlet extraction, the solvent is heated to reflux, then the solvent vapour travelled up and condensed at condenser and drop back into the chamber. The chamber containing the solid material slowly fills with warm solvent. Desired compound dissolve in the warm solvent. After carrying out Soxhlet extraction, the mixture is then brought to rotary evaporator where the solvent is evaporated, and the left-over phenolic compound is weighted. Research on the extraction of phenolic compounds from Vernonia cinereal leaves through Soxhlet extraction with extraction time (1–4 h), feed-to-solvent (1:10–1:25 g/mL) and ethanol concentration (20–80% v/v) on the yield of extract.

The result of the study showed that the highest yields (10.01 ± 0.85 w/w) were obtained using extraction time of 2 h, feed/solvent of 1:20 g/mL and ethanol concentration of 60% v/v. Figure 2.6 showed the conventional Soxhlet extractor unit and its component.

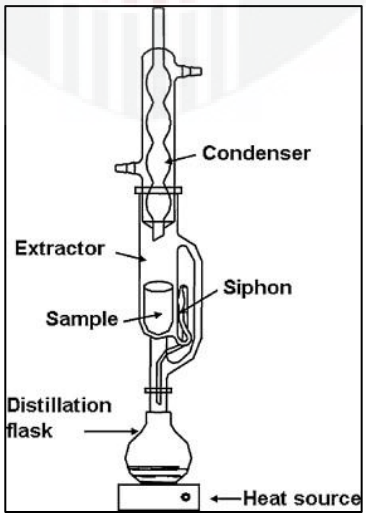



Figure 2.6 Conventional Soxhlet extractor. Adapted from (Luque and Priego, 2010)

## 2.3 Parameters Affecting Oil Extraction

In Soxhlet extraction, the sample is repeatedly brought into contact with fresh portions of extractant, which facilitates displacement of the transfer equilibrium. Meanwhile, the system remains at a relatively high temperature by effect of the heat applied to the distillation flask reaching the extraction cavity to some extent (Luque and Priego, 2010). Few parameters that can affect the oil extraction process in Soxhlet extraction are type of solvent, reaction time, solid to solvent ratio and temperature.

### 2.3.1 Type of Solvent

Different type of solvent can be used for Soxhlet extraction. A research on the effect of different solvent: n-hexane, methanol and acetone on the yield of oil extraction oil palm filter cake sludge. The result showed that methanol as solvent has the highest yield compare to acetone and n-hexane as shown in Table 2.2 (Ameera and Arsad, 2016). Methanol used in this research has the most polar solvent based on the polarity index. Each type of solvent has their own solvent polarity, vapour pressure and viscosity. Hence solvent with low viscosity can diffuse into the pore of sample easily to leach out the bioactive from the sample.

Table 2.2: Comparison on yield of oil extracted with different type of solvent used based on weight. Adapted from *(* Ameera and Arsad, 2016*)*

| **Type of Solvent** | **Operating Condition** | **Percentage of oil yield based on weight (%)** |
|---|---|---|
| n-Hexane | Soxhlet extraction of decanter cake at 350ml solvent with 4 hours of reaction time. | 13.33 |
| Methanol | | 66.64 |
| Acetone | | 33.33 |

### 2.3.2 Reaction time

The time of reaction in Soxhlet extraction affect the result in term of yield. Few researchers have studied the effect of reaction time in Soxhlet extraction on the yield of extraction (Ameera and Arsad, 2016; Alara et al., 2018; Hawthorne et al., 2000). Each study shows different result in the extraction yield. The yield of oil extracted will increase as the reaction increase and the yield become constant when the oil is fully extracted. However, Creencia et al. (2018) investigated that by using reaction time of 60, 90, 12, 150 minutes for extraction of oil from rubber seed, the highest yield is at 90 minutes with 10.09% of yield. Hence an optimum reaction time should be determined to achieve highest yield at shortest time.

### 2.3.3 Solid to Solvent ratio

As Soxhlet extraction is related to mass transfer, the amount of solvent and solid used is crucial as it affects the performance of Soxhlet extractor in term of oil yield from the sample. Research on the effect of solid to solvent ratio (1:10–1:25 g/mL) on extraction of phenolic compounds from Vernonia cinerea leaves through Soxhlet extraction was carried out (Alara et al., 2018) . Result showed 1:20 g/mL of solid to solvent ratio has the highest yield in oil extraction. High solid to solvent ratio increased the concentration gradient, hence it increases the rate of diffusion and this will allow greater extraction of solid by solvent. Experiments needed to be carried out to determine the optimum solid to solvent ratio that gives the best yield. In palm oil industry, it is more favourable to use least solvent that give the highest yield in oil extraction. Therefore, fixing the amount of solid as constant is preferable.

## 2.4 Types of Pre-treatment Can be Done Before Extraction

Pre-treatment is a crucial process in crude palm oil (CPO) production. Pre-treatment process in conventional palm oil extraction was usually involves heating oil palm fruits until the outer shell (or mesocarp) of the fruits becomes soft to facilitate separation and the extraction process (Pootao and Kanjanapongkul, 2016). Pre-treatment on the sample reduced the free fatty acid (FFA) content in the sample while the yield of oil extraction will increase (Osawa et al., 2007). The amount of FFA content should be based on the standard provided by Malaysia Palm Oil Board (MPOB) as shown in Table 2.3. Few types of physical pre-treatment can be done before the extraction process such as heating pre-treatment and ultra-sonic pre-treatment.

Table 2.3: Percent FFA content in CPO. Adapted from (MPOB, 2010)

| Fatty Acid | Name | Content in CPO (%) |
|---|---|---|
| C12:0 | Lauric Acid | 0.1 |
| C14:0 | Myristic Acid | 1.0 |
| C16:0 | Palmitic Acid | 43.7 |
| C16:1 | Palmitoleic Acid | 0.1 |
| C18:0 | Stearic acid | 4.4 |
| C18.1 | Oleic Acid | 39.9 |
| C18.2 | Linolenic Acid | 10.3 |
| C20.0 | Arachidic Acid | 0.3 |

### 2.4.1 Heating Pre-treatment

Sterilization is widely used to pre-treat oil palm fruit (Pootao and Kanjanapongkul, 2016). Prolonged sterilization offers a significant reduction in labour and manpower, it may require high investment and a long operation time. Heating through microwave oven is also one of the ideal heating pre-treatments. Microwave heating mechanized the transformation of alternating electromagnetic field energy into thermal energy by affecting the polar molecules of material. Between the attempts to improve Soxhlet performance, the most successful has been the use of microwaves, which has provided the wider variety of approaches (Luque and Priego, 2010). An experiment result showed that palm fruits that went through 10 minutes of 1000W microwave pre-treatment will result is higher yield of palm oil extraction (Jason, 2017). An extension in microwave pre-treatment time more than 10 minutes will result in higher FFA content in extracted oil.

### 2.4.2 Ultrasonic Pre-treatment

Ultrasonic application can be carried out by using ultrasonic transducer or piezoelectric. In general, the use of ultrasound as pre-treatment in essential oil extraction decrease by three times (mean average) the time of extraction. The use of ultrasound in low frequency can be employed to extract some compounds through molecular agitation, heating, micro-jets formation and cavitation phenomenon (Michelo et al., 2017). The vibration of ultrasonic waves causes morphology changes on the surface molecules of the sample where cavitation and breaking of pore occurs thus facilitate the oil extraction process.

## 2.5 Respond Surface Methodology (RSM)

Response surface methodology (RSM) enabled evaluation of the effects of many factors and their interactions on response variables (Tan *et al.,* 2009). The main advantage of using RSM is the reduced number of experimental runs needed to provide sufficient information for statistically acceptable results. RSM is very reliable where it can design a list of experiment runs based on the number of parameters provided.

### 2.5.1 RSM Software

The software used to run RSM is Design Expert Version 11. Design Expert Version 11 is the product of Stat-Ease, Inc where it provides many powerful statistical tools, such as:

i. Two-level factorial screening designs: Identify the vital factors that affect your process or product, so you can make breakthrough improvements.
ii. General factorial studies: Discover the best combination of categorical factors, such as source versus type of raw material supply.
iii. Response surface methods (RSM): Find the optimal process settings to achieve peak performance.

Design Expert 11 offers rotatable 3D plots to easily view response surfaces from all angles to have a clearer look on how each parameter affect the result. It also provides statistical analysis such as ANOVA and T test on how significant the effect of each parameter on the result.

# CHAPTER 3 MATERIALS AND METHOD

## 3.1 Summary of Experimental Flow

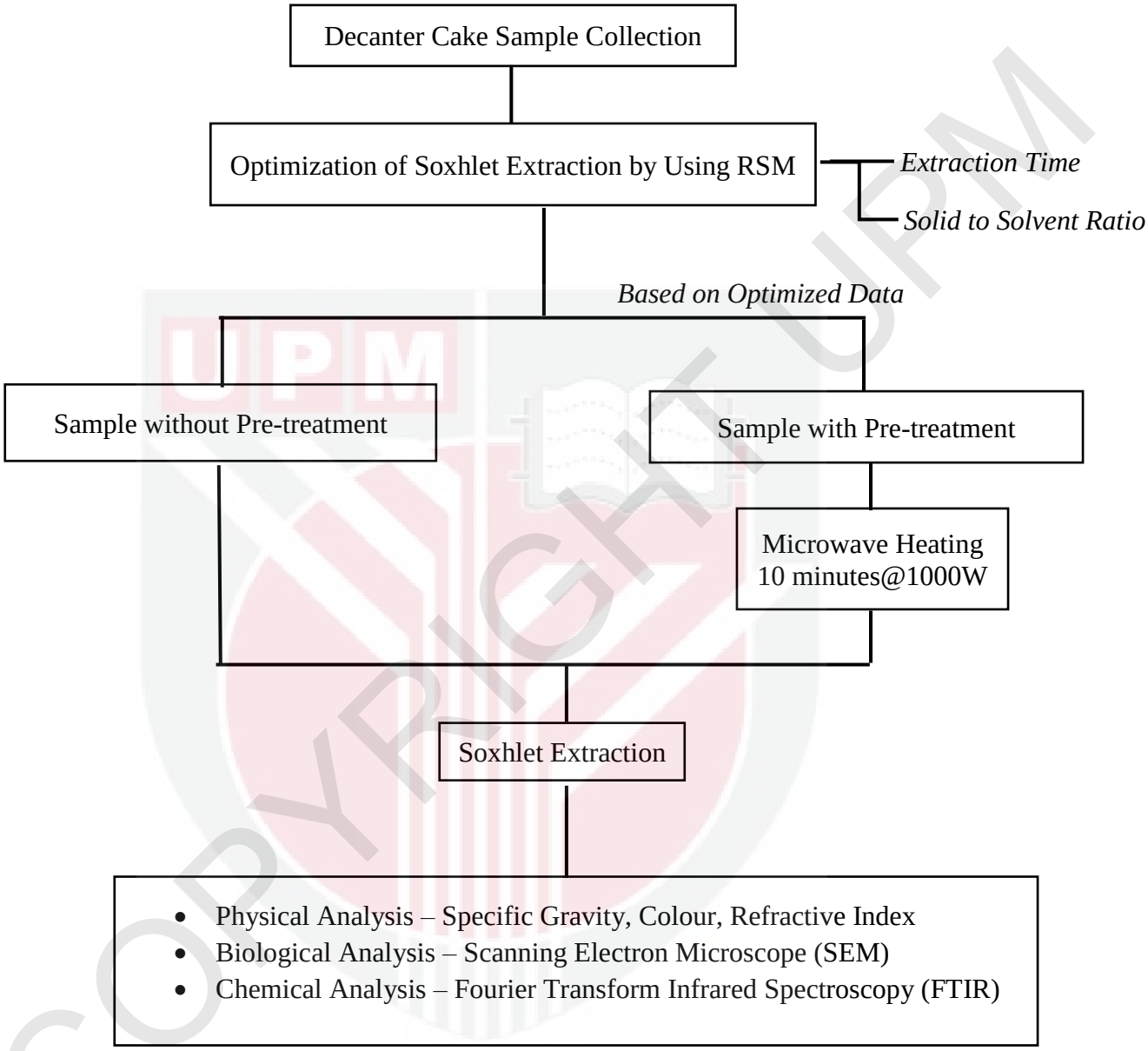



Figure 3.1: Flow diagram of palm oil extraction from OPDC

The summary of experimental flow for extraction palm oil from OPDC is as shown on Figure 3.1. The experimental work is started by sample collection, later RSM optimization for Soxhlet extraction for sample without pre-treatment and with microwave pre-treatment was conducted. Finally, physical, chemical analyses on extracted oil from both sample and biological analysis on OPDC were conducted.

### 3.2 Optimization of Experimental work using RSM

Design Expert Version 11 is an analysis software that carries out RSM was installed. Two parameters/ numeric factors: reaction time and solid to solvent ratio were set and keyed into the software as shown in Table 3.1. The unit of both parameters was set as reaction time (hr) and solid to solvent ratio(unitless). The high and low value of both parameters were set as shown in Table 3.1. The mass of OPDC solid was set at 20g.

Table 3.1: Numerical factors

| **Factor** | **Name** | **Units** | **Type** | **Minimum** | **Maximum** |
|---|---|---|---|---|---|
| A | Reaction Time | hr | Numeric | 4.00 | 8.00 |
| B | Solvent ratio | | Numeric | 10.00 | 13.00 |

The next step was to set the response for the experiment as shown in Table 3.2. This research has only one response which is yield of extracted palm oil and the unit is set as millilitre (ml).

Table 3.2: Response of the experiment

| Name | Units |
|---|---|
| Yield of extracted palm oil | ml |

The name of factors and their unit were checked before clicking the finish button. Design expert 11 generated 13 experiment runs as shown in Table 3.3. The experiment was conducted in triplicate. Table 3.3 showed the list of experiment designed by RSM.

Table 3.3: List of experiment runs

| Run | Factor 1 | Factor 2 | Response 1 |
|---|---|---|---|
| | A: Reaction Time(hr) | B: Solvent Ratio | Average Yield of extracted oil (ml) |
| 1 | 6 | 11.25 | |
| 2 | 3 | 11.25 | |
| 3 | 6 | 11.25 | |
| 4 | 6 | 11.25 | |
| 5 | 4 | 12.50 | |
| 6 | 6 | 11.25 | |
| 7 | 4 | 10 | |
| 8 | 8 | 11.25 | |
| 9 | 6 | 10 | |
| 10 | 6 | 11.25 | |
| 11 | 8 | 12.50 | |
| 12 | 8 | 10 | |
| 13 | 6 | 13 | |

Value for yield of oil in Table 3.3 was keyed into the software then 3 criteria were set as follow:

i. Minimal Reaction time
ii. Minimal Solvent
iii. Maximum Yield of Oil Extraction

By setting up these 3 criteria, Design Expert 11 formulated a list of optimized data that can achieve with the 3 criteria provided. The chosen optimized data with the highest desirability was tested by carrying out another experiment to confirm the oil yield.

### 3.3 Preparation of Raw Material

Figure 3.2: OPDC sample collected from Jengka Palm Oil Mill

Oil palm decanter cake (OPDC) was collected from Jengka Pahang Palm Oil Mill as shown in Figure 3.2. The OPDC was kept in cold storage room at the laboratory. n-Hexane (Baker Analyzed A.C.S Reagent) was obtained from the laboratory. The initial mass of OPDC was measured by using electronic balance. 45g of OPDC was then moulded in a rectangular cube by using hand. The sample was then dried in Oven Dryer (UN55) at 105 $^{o}$C for 24 hours as shown in Figure 3.3.

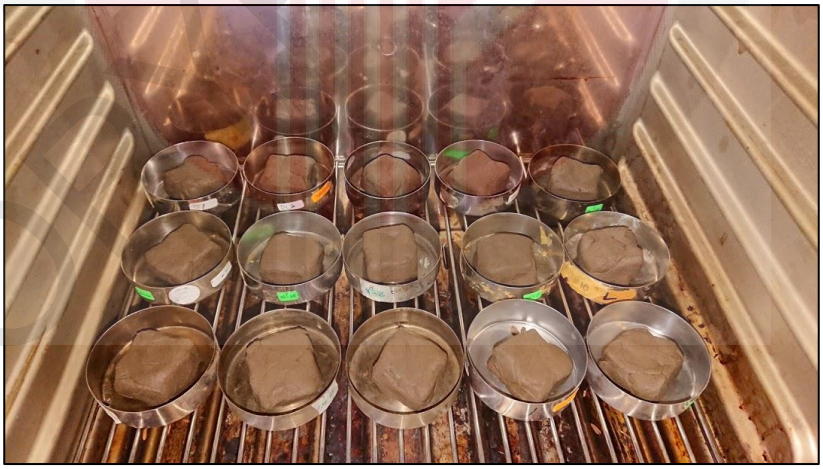


Figure 3.3**:** OPDC sample dried in oven dryer.

Figure 3.4: Dried OPDC sample

Figure 3.4 showed the dried OPDC sample after oven drying. The overall size of the dried OPDC is significantly reduced as the moisture is removed from it.

## 3.4 Set up of Soxhlet Extractor and Rotary Evaporator

### 3.4.1 Soxhlet Extraction Unit

Hexane has low boiling point (68°C); hence it is suitable for palm oil extraction by using Soxhlet extractor and rotary evaporator. 20g of dried OPDC was weighted and crushed into smaller pieces and placed in a cellulose extraction thimble CT30100 with internal diameter and length (30 x 100m). The thimble was then filled with cotton as stopper to prevent OPDC from leaking out during extraction. Figure 3.5 showed the diagram of FAVORIT Soxhlet Extractor. The amount of solvent used is based on the experimental data shown on Table 3.3 and the solvent was measured and filled into a bottom flask. The amount of solid used is fixed which is 20g and the solid to solvent ratio is (1 : x). x indicated the solvent ratio. The calculation of amount of solvent used is calculated by using Equation 1:

Amount of solvent used (ml)= 20g x solvent ratio -Equation (1)

Soxhlet extractor was set up. The thimble with sample was put inside the extraction chamber. The temperature of the system was set to 3 for pre-heating and later changed to 4 (approximately 70°C). The heating process was carried on for the parameters as shown in Table 3.2c. Soxhlet extraction was repeated 3 times for each parameter to obtain the average yield from the experiment.

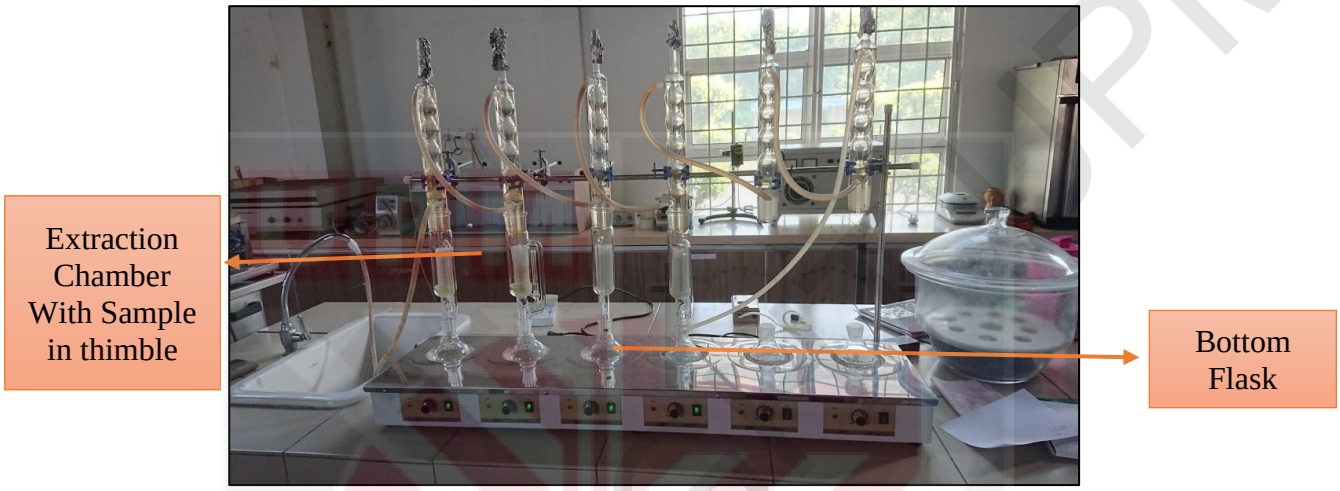



Figure 3.5 Diagram of Soxhlet extractor

### 3.4.2 Rotary Evaporator

After carrying out Soxhlet extraction, the bottom flask with extracted oil and hexane mixture was brought to rotary evaporator as shown in Figure 3.6. The temperature is set to 68°C; which is the boiling point of hexane. As hexane started to boil, hexane vapour travelled up to condenser and condensed into liquid form which later drop into the bottom flask of rotary evaporator. The left-over oil in the bottom flask was weighted and the value was filled into Table 3.3. Hexane was then collected and reused for the next experiment run.


Figure 3.6: Diagram of rotary evaporator

## 3.5 Pre-treatment of OPDC

Another experiment was carried out by using the optimized data obtained from RSM. Figure 3.7 showed Panasonic Microwave Oven (NN-J993) 1360W,240V used for this experiment. OPDC will undergo microwave heating for 10 minutes at 1000W following the method proposed by (Jason, 2017) as pre-treatment after regular oven dry as shown in Figure 3.8. After heating, the 20g of dried IPDC were used for Soxhlet extraction by using the optimized data.

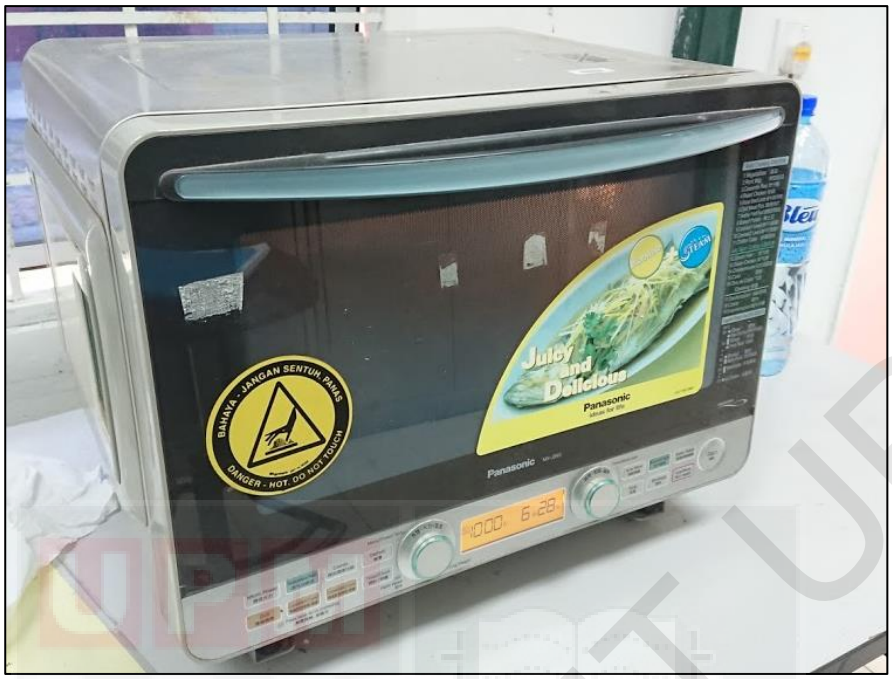



Figure 3.7: Panasonic Microwave Oven (NN-J993) 1360W,240V

Figure 3.8: OPDC sample heated in microwave oven

## 3.6 OPDC and Extracted Oil Analysis

### 3.6.1 Biological Analysis on OPDC

Biological analysis was carried out on OPDC without pre-treatment and OPDC sample with pre-treatment by using Scanning Electron Microscope (SEM) S-3400N where the SEM images at 300x magnification of the samples was taken digitally. The SEM experiment was carried out at Material Characterization Laboratory (MCL) Engineering Faculty UPM as shown in Figure 3.9. Scanning electron microscopy (SEM) is a good platform which is used to discover the mechanism behind the microscopic changes of morphological structure of the sample (Yue *et al.*, 2018). 4 decanter samples were tested as shown in the list as follow:

i. OPDC after oven dry.
ii. OPDC after Soxhlet Extraction (without pre-treatment)
iii. OPDC after microwave pre-treatment.
iv. OPDC after Soxhlet Extraction (with pre-treatment)

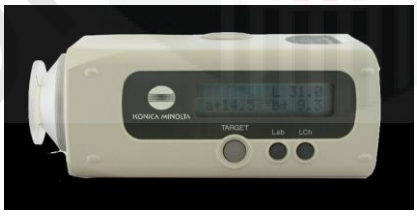

Figure 3.9: Diagram of SEM S-3400N

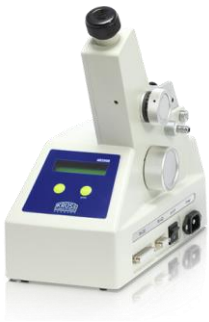
The surface of OPDC is non-conductive which is negative charge. SEM's electrons are also negative charge; hence a layer of gold coating was coated on the surface of each sample as gold is a conductive material that can absorb the electron blast by SEM.

### 3.6.2 Physical Analysis on Extracted Palm Oil

Physical analysis on extracted oil for sample with and without pre-treatment was determined by using the apparatus listed in Table 3.4. The temperature of oil samples was maintained at 40°C where the results were later compared with the standard values.

Table 3.4: Physical analysis on extracted oil.

| Physical Properties | Apparatus | Method |
|---|---|---|
| Specific Gravity, SG | Electronic balanced and 100ml measuring cylinder.  | An empty measuring cylinder was put on an electronic balance and the mass is set to 0. Extracted oil sample was then filled into the measuring cylinder until 6ml. The mass of oil sample was recorded. The density and SG of the oil were calculated by using the formula below:<br>Density (g/ml) = (mass of oil / volume)<br>SG = Density of Oil/ Density of Water |
| Colour | Colour Reader Cr-10  | Oil samples were put on a flat plate. Colour Reader CR-10 was set to measure LAB values. The LAB values of oil samples were measured by pressing the measure button. |

| | | |
|---|---|---|
| Refractive Index | Digital ABBE Refractometer  | The refractometer was calibrated by using distilled water by dropping few drops of distilled water on the surface of prism glass. The surface of prism glass was them wiped dry and few drops of oil sample was slightly dropped on it. The prism was closed and looked through the eye piece. The dial was adjusted to the place where the borderline was exactly on the centre of crosshair. The refractive index value was recorded. |

### 3.6.3 Chemical Analysis on Extracted Oil Sample.

Chemical analysis of extracted palm oil was carried out by using Fourier transform infrared spectroscopy (FTIR-Spectrum 100) as shown in Figure 3.10. FTIR is used to determine the functional group components from 2 extracted palm oil sample which is palm oil extracted from sample with and without pre-treatment. The IR spectra was determined over a wavenumber range from 650 - 4000 $cm^{-1}$. FTIR experiment was carried out at Material Characterization Laboratory (MCL) Engineering Faculty UPM. The pH value of oil sample was measured by using a pH meter.

Figure 3.10: Diagram of FTIR-Spectrum 100

# CHAPTER 4 RESULT AND DISCUSSION

## 4.1 Optimization of Soxhlet Extraction of OPDC by using RSM

The main parameters studied for Soxhlet extraction were reaction time and solvent to solid ratio. Table 4.1 below showed the experiment run result for RSM optimization.

### 4.1.1 List of Soxhlet Extraction Experiment runs for RSM

Table 4.1: List of experiment runs designed by RSM

| Run | Factor 1 | Factor 2 | Response 1 |
|---|---|---|---|
| | A: Reaction Time(hr) | B: Solvent Ratio | Average Oil Yield (ml) |
| 1 | 6 | 11.25 | 3.334 |
| 2 | 3 | 11.25 | 2.787 |
| 3 | 6 | 11.25 | 3.082 |
| 4 | 6 | 11.25 | 3.037 |
| 5 | 4 | 12.50 | 2.991 |
| 6 | 6 | 11.25 | 3.025 |
| 7 | 4 | 10 | 3.006 |
| 8 | 8 | 11.25 | 3.286 |
| 9 | 6 | 10 | 3.298 |
| 10 | 6 | 11.25 | 3.064 |
| 11 | 8 | 12.50 | 3.328 |
| 12 | 8 | 10 | 3.314 |
| 13 | 6 | 13 | 3.644 |

The amount of extracted oil yield with different parameters was shown on Table 4.1. The experiment run was conducted in triplicate to obtain the average oil yield. A longer reaction time and greater solvent ratio gave a higher oil yield. As depicted in Table 4.1, at 6 hours of reaction time and 13 solvent ratio, the yield of oil extracted is the highest

which is 3.644ml. Besides, the least extracted oil which is 2.991 ml was from experiment run 5 at 4 hours of reaction time and 12.5 solvent ratio. Figure 4.1 showed the oil yield in a bottom flask from rotary evaporator.

Figure 4.1: Sample oil yield

## 4.2 Data Optimization with RSM Software

The average oil yield data was keyed in the Design Expert Version 11 software, the optimization process was started. Table 4.2 below showed the criteria set up to obtain the optimized data. For optimized data, the software is set up to generate the minimum reaction time, minimum solvent ratio and maximize yield of oil extract.

Table 4.2: Constraints

| Name | Goal | Lower Limit | Upper Limit |
|---|---|---|---|
| A: Reaction Time | minimize | 4 | 8 |
| B:Solvent ratio | minimize | 10 | 13 |
| Yield of extracted oil | maximize | 2.787 | 3.644 |

There were total 10 solutions generated following the criteria set up where only solution with the highest desirability to achieve the criteria will be chosen to carry out confirmation test on oil yield. Table 4.3 showed the list of solutions generated by design Expert Version 11.

Table 4.3: List of solutions

| Number | Reaction Time | Solvent ratio | Yield of extracted oil | Desirability | |
|---|---|---|---|---|---|
| **1** | **4.923** | **10.000** | **3.179** | **0.706** | **Selected** |
| 2 | 4.942 | 10.000 | 3.182 | 0.706 | |
| 3 | 4.901 | 10.000 | 3.176 | 0.706 | |
| 4 | 4.983 | 10.000 | 3.187 | 0.706 | |
| 5 | 4.858 | 10.000 | 3.171 | 0.706 | |
| 6 | 4.838 | 10.000 | 3.168 | 0.706 | |
| 7 | 4.787 | 10.000 | 3.161 | 0.705 | |
| 8 | 5.352 | 10.000 | 3.229 | 0.699 | |
| 9 | 4.491 | 10.000 | 3.119 | 0.698 | |
| 10 | 4.417 | 10.000 | 3.108 | 0.695 | |

Based on Table 4.3, the chosen solution with 0.706 desirability was at 4.923 hours of reaction time and solvent ratio of 10 which is 200ml. The expected oil yield should be 3.179ml. To verify the yield, 3 confirmation tests were carried out based on the parameters in Solution 1. Solution 1 was chosen as it achieved the criteria which were lowest reaction time and lowest ratio which gives the highest oil yield. Table 4.7 showed the average oil yield based on solution 1 where the oil (3.107 ml) is lower than the expected oil yield (3.159 ml). The difference between theoretical oil yield and experimental oil yield was 0.072 ml. The percent error is considered low which is 2.26% as the desirability to achieve the criteria was 0.706 (70.6%).

### 4.2.1 ANOVA Test for Quadratic Model

A statistical analysis of variance (ANOVA) is performed to see either the process parameters are statistically significant. Finally, a confirmation test is conducted to verify the optimal process parameters obtained from the process parameter design.

Table 4.4: ANOVA Test for Response 1: Yield of Extracted Oil

| Source | Sum of Squares | df | Mean Square | F-value | p-value | |
|---|---|---|---|---|---|---|
| **Model** | 0.4550 | 5 | 0.0910 | 4.84 | 0.0311 | significant |
| A-Reaction Time | 0.1461 | 1 | 0.1461 | 7.77 | 0.0270 | |
| B-Solvent ratio | 0.0094 | 1 | 0.0094 | 0.4981 | 0.5031 | |
| AB | 0.0002 | 1 | 0.0002 | 0.0112 | 0.9187 | |
| $A^2$ | 0.0581 | 1 | 0.0581 | 3.09 | 0.1221 | |
| $B^2$ | 0.1143 | 1 | 0.1143 | 6.08 | 0.0430 | |
| **Residual** | 0.1315 | 7 | 0.0188 | | | |
| Lack of Fit | 0.0461 | 3 | 0.0154 | 0.7204 | 0.5899 | not significant |
| Pure Error | 0.0854 | 4 | 0.0213 | | | |
| **Cor Total** | 0.5865 | 12 | | | | |

Based on Table 4.4, the **Model F-value** of 4.84 implies the model is significant. There is only a 3.11% chance that an F-value this large could occur due to noise.

**P-values** less than 0.0500 indicate model terms are significant (Mushtaq *et al.*, 2015). In this case A, $B^2$ are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. Based on Table 4.4, the $B^2$ showed p value greater than 0.1000 which is not significant.

The **Lack of Fit F-value** of 0.72 implies the Lack of Fit is not significant relative to the pure error. There is a 58.99% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good so that a fit model is obtained.

Figure 4.2 showed that the levels of the yield predicted from the fitted empirical model are in line agreed with the observed values under the observed experimental conditions, with a sensibly high value of the coefficient of determination of 0.7758 ($R^2$) (Table 4.5) (Mushtaq *et al.*, 2015). The coloured dots showed the actual oil yield where the straight graph showed the predicted value. Based on Figure 4.2, there were few points fall below the expected value and few points were above the predicted value. The graph showed the consistent data that proven this experiment data is acceptable.

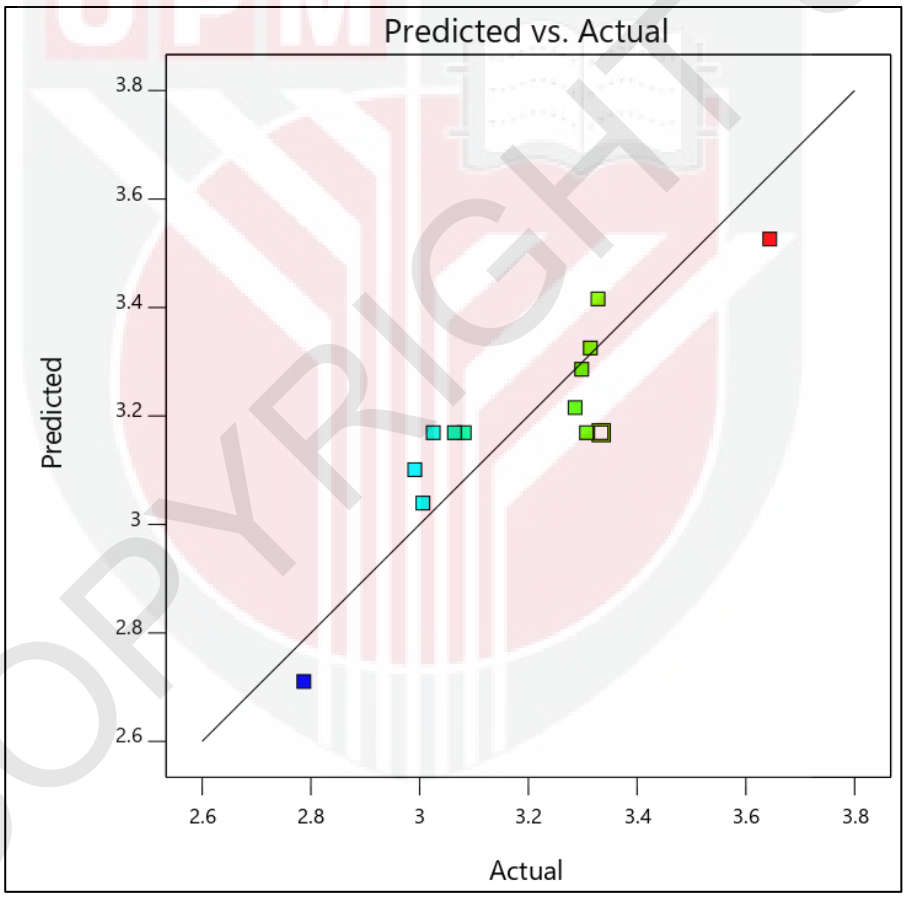



Figure 4.2: Graph of predicted vs actual value of oil yield

### 4.2.2 Fit Statistics

Table 4.5: Fit statistics analysis

| | | | | |
|---|---|---|---|---|
| **Std. Dev.** | 0.1371 | | **$R^2$** | 0.7758 |
| **Mean** | 3.19 | | **Adjusted $R^2$** | 0.6156 |
| **C.V. %** | 4.30 | | **Predicted $R^2$** | 0.0561 |
| | | | **Adeq. Precision** | 8.7604 |

Table 4.5 showed the fit statistics analysis for the experimental run. R-squared is a statistical measure of how close the data are to the fitted regression line. $R^2$ approaching 1 showed the model is completely fit while $R^2$ approaching 0 showed that the model cannot be used due to large variance differences (Drennan and Robert, 1996). The **Predicted $R^2$** of 0.0561 is not as close to the **Adjusted $R^2$** of 0.6156 as one might normally expect; i.e. the difference is more than 0.2. This may indicate a large block effect or a possible problem with the model and/or data. Things to consider are model reduction, response transformation, outliers, etc. All empirical models should be tested by doing confirmation runs.

**Adeq. Precision** measures the signal to noise ratio. A ratio greater than 4 is desirable (Liang et al., 2018). The ratio of 8.760 indicates an adequate signal.

### 4.2.3 Final Equation in Term of Actual Factor

Table 4.6: Final equation generated by RSM

| | |
|---|---|
| Yield of extracted oil | = |
| +14.19407 | |
| +0.353781 | Reaction Time (A) |
| -2.21912 | Solvent ratio (B) |
| +0.002900 | Reaction Time * Solvent ratio |
| -0.025944 | Reaction Time² |
| +0.099206 | Solvent ratio² |

Table 4.6 showed the final equation generated by RSM to calculate the oil yield based on parameters A and B. The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. The generated equation is as follow:

$$\text{Yield of Extracted Oil(g)} = 14.19407 + 0.353781(A) - 2.21912(B) + 0.0029(A*B) - 0.025944(A^2) + 0.099206(B^2) \quad \text{-Equation (2)}$$

### 4.2.4 3D surface plot for RSM

Figure 4.3 showed the 3D surface plot for yield of oil extract before optimization. The response surface plot showed the optimal condition between the variables such as reaction time and solvent ratio (Pirshahid et al., 2018) The red dots on the 3D plot showed the data above the predicted value while the pink dots showed data below predicted value. At the highest point of the 3D curve, the design point is below the expected value. This means the yield of oil extracted should be higher than the data obtained. To solve this problem, an optimized data is generated as shown in Table 4.3. Based on the confirmation test, even though the extracted oil yield is still below the predicted value, but the different is small. Figure 4.4 showed 3D plot for optimized data where there is a small flag on the highest point of 3D curve. That is the solution selected by the software.

In order to really get a feel for how the response varies as a function of the two factors chosen for display, 3D Surface was selected from the Graphs Toolbar and three-dimensional display of the response surface was shown.

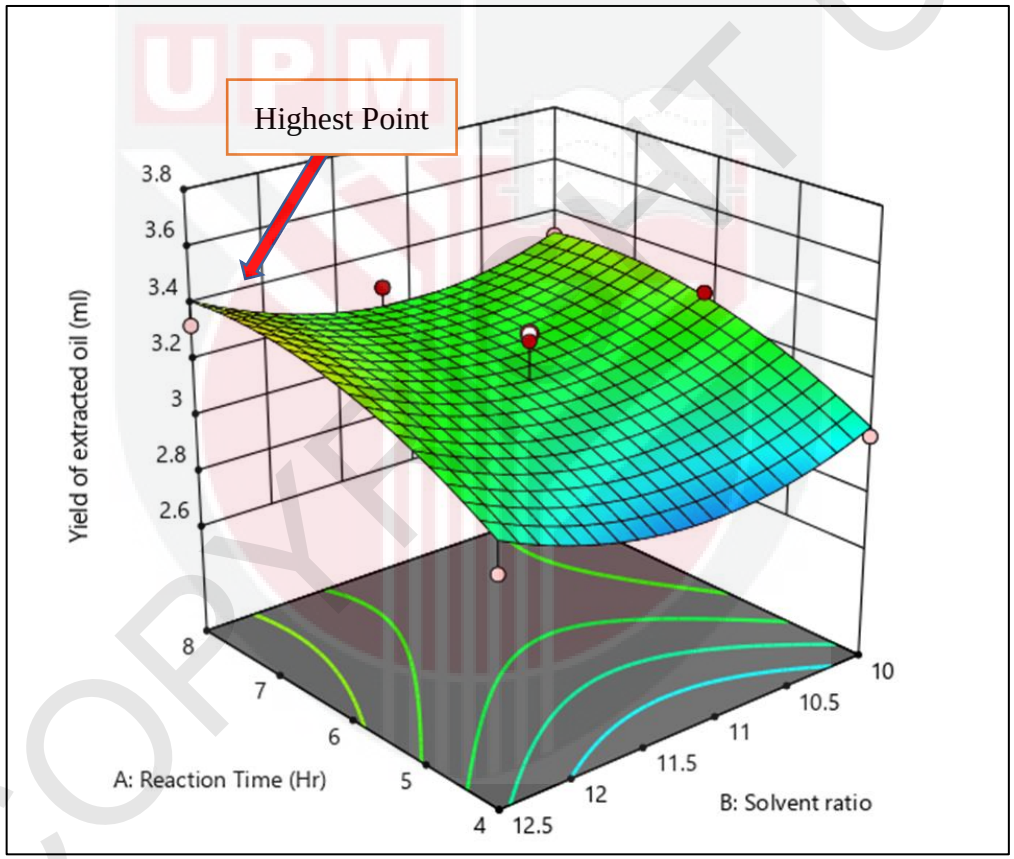



Figure 4.3: 3D surface plot for yield of oil extracted data

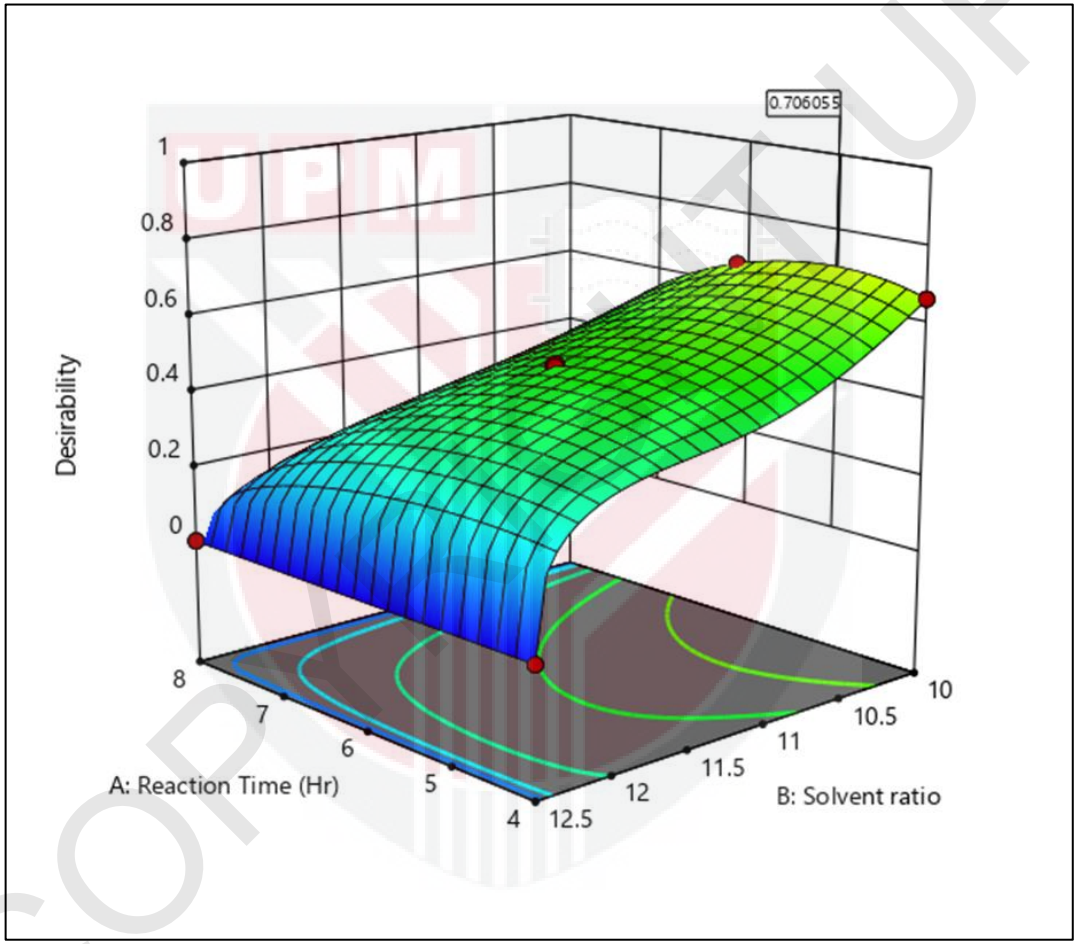



Figure 4.4: 3D surface plot for optimized data

## 4.3 Comparison of Oil Yield for Sample Without and With Pre-treatment

By using the optimized data as follow, the experiment was conducted triplicate to obtain the average oil yield for sample without and with microwave pre-treatment.

i. Sample = 20g

ii. Reaction time = 4.923 hr

iii. Solid to solvent ratio= 1:10 (200ml)

Table 4.7: Soxhlet extraction for sample without and with pre-treatment

| OPDC Sample | Average yield of extracted oil (ml) | Standard Deviation |
|---|---|---|
| Without Pre-treatment | 3.107 | 0.085 |
| With Pre-treatment | 3.289 | 0.047 |

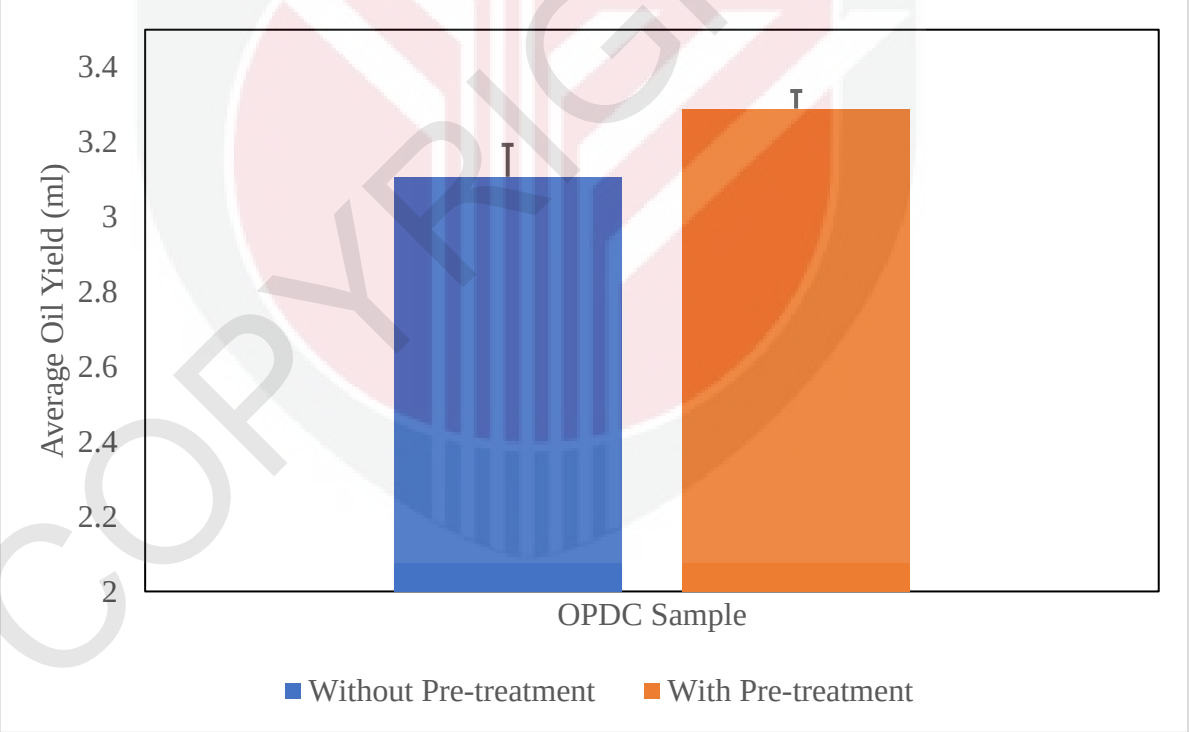



Figure 4.5: Comparison of average oil yield for both sample

The average oil yield for OPDC microwave pre-treatment was 3.289 ml at 4.923 hours of reaction time and solvent ratio of 10 (200ml) which was greater than OPDC sample without pre-treatment 3.107g as shown in Table 4.7 and Figure 4.5. The difference

between both yield value was 0.182g or 5.533% more oil yield compare to sample without pre-treatment. Microwave pre-treatment is different compare to regular oven drying. Microwave heating used electromagnetic wave that heats throughout the OPDC sample while oven drying heats up OPDC sample from outer surface only. Microwaves (MW) are a form of non-ionizing electromagnetic energy at frequencies ranging from 300 MHz to 300 GHz. This energy transmitted as wave can penetrate through biomaterials and interact with polar molecules into materials, such as water to generate heat. MV acts directly on molecules by ionic conduction and dipole rotation and thus only polar materials can be heated based on their dielectric constant (Sadeghi et al., 2017).

## 4.4 Physical Analysis on Extracted Oil

The physical properties (Specific gravity at 50°C, colour at 50°C and refractive index at 50°C) of extracted oil from OPDC with microwave pre-treatment and without microwave pre-treatment were carried out and the results were shown in Table 4.9, Table 4.10 and Table 4.11 respectively. The result was compared to the standard at 50°C as shown in table 4.8 below. These physical properties determined the quality of oil extract from OPDC.

Table 4.8: Physical properties of palm oil based on standard at 50°C. Adapted from (Koushki and Nahidi, 2008; Chinedu and Ebere, 2017)

| Physical Properties | Value |
|---|---|
| Density ($g/cm^3$) | 0.889 |
| Specific Gravity | 0.906 |
| Refractive Index (nd.) | 1.455 – 1.462 |

### 4.4.1 Specific Gravity

Specific Gravity (SG) is a term used to define the weight or density of a liquid as compared to the density of an equal volume of water at a specified temperature (Chinedu and Ebere, 2017). The results for the density and specific gravity (SG) are presented in Table 4.9.

Table 4.9: Density and SG of extracted oil at 50°C

| | Density ($g/cm^3$) | Specific Gravity |
|---|---|---|
| Sample without pre-treatment | 0.948 | 0.960 |
| Sample with pre-treatment | 0.886 | 0.897 |

Based on Table 4.9, the oil extracted from sample without pre-treatment has higher density ($0.948 g/cm^3$) and SG value (0.960) compared to sample with pre-treatment with density 0.886 $g/cm^3$ and SG value of 0.897. By comparing to the standard as shown in Table 4.8, oil extract from sample without pre-treatment had higher density and SG value. On the other hand, sample with pre-treatment has slightly lower density and SG values compared to standard. However, both oil samples had similar density and Sg compared to standard as shown in Table 4.8.

### 4.4.2 Colour Test

Colour and appearance are important quality parameter for oil. Any colour within visible range was represented with the aid of three-dimensional coordinates L, a and b. The axes L represents luminosity, with L=0 is black and L=100 is white. Axis a represent the position of colour between red and green where positive end of a value gives red, while negative end of a gives green colour. For b axis, it reflects the colour yellow at positive end and blue at negative end. The L,a,b values indicated the colour based on Figure 4.4a.

Table 4.10: Lab colour test data for oil sample

| Colour test | L | a | b |
|---|---|---|---|
| Sample without pre-treatment | 58.8 | 6.0 | 29.3 |
| Sample with pre-treatment | 58.7 | 5.8 | 29.0 |

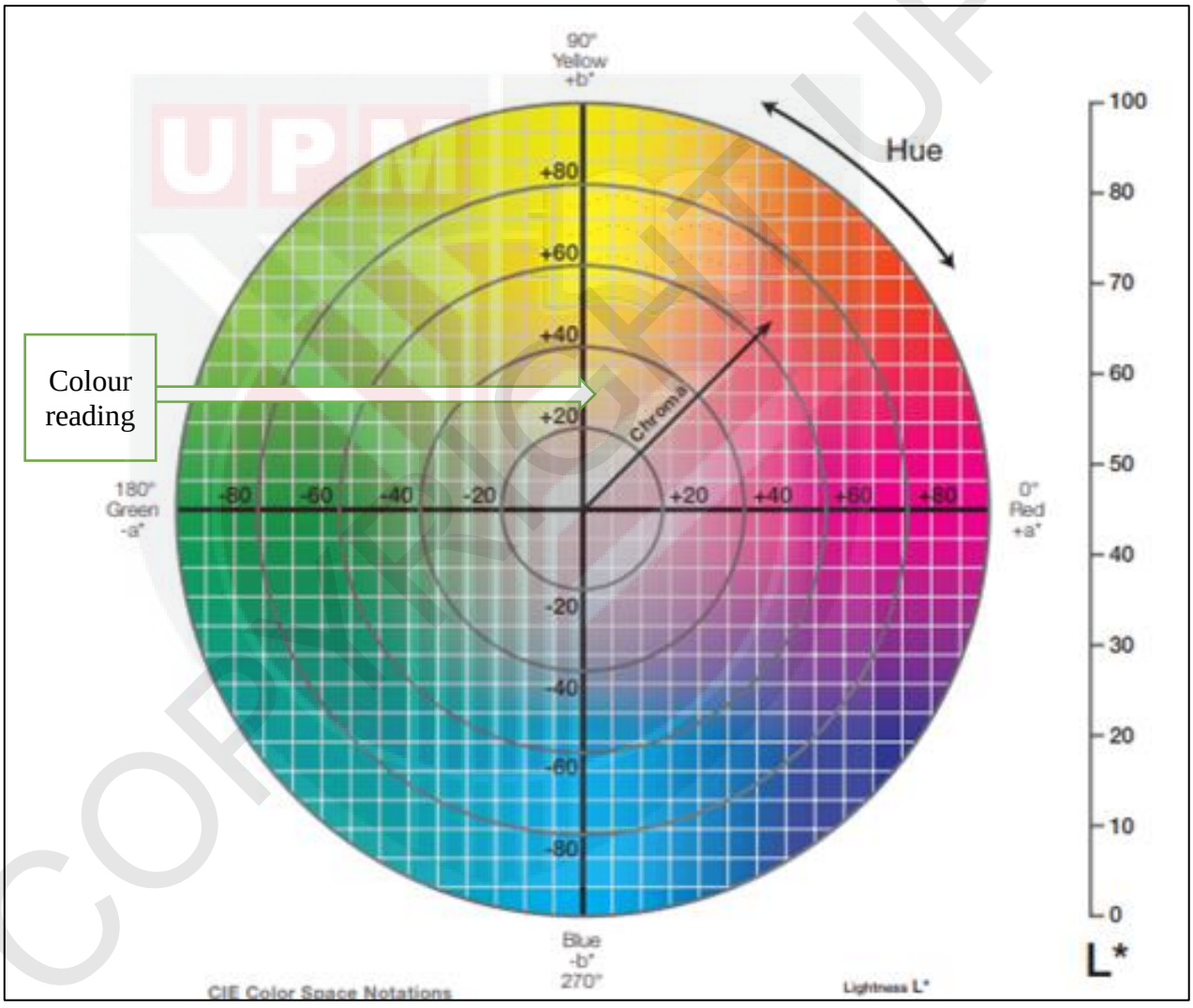



Figure 4.6: CIELAB Colour Chart. Adapted from (Paravina, 2018)

Figure 4.7: Oil sample at 50°C

Based on Table 4.10, both oil samples had similar L,a,b value. By referring to Figure 4.6 and Figure 4.7, the colour of oil sample was light yellowish colour at 50°C.

### 4.4.3 Refractive Index (RI)

Refractive index (RI) showed how much light bends when it travelled through the soil sample (Chinedu and Ebere, 2017). The value of RI for oil sample without and with pre-treatment from digital ABBE Refractometer was shown in Table 4.11.

Table 4.11: RI values of oil samples

| Experiment Run | RI (nd.) |
|---|---|
| | Average value |
| Sample without pre-treatment | 1.462 |
| Sample with pre-treatment | 1.462 |

Based on Table 4.11, the RI value of both samples are similar where the oil sample without pre-treatment has RI value of 1.462 which is slightly higher than sample with pre-treatment with RI value of 1.462. Both RI values fall within the standard as shown in Table 4.8. This proven the quality of oil extracted based on RI value is the same as the standard.

## 4.5 Biological Analysis on OPDC

The structure images of oil palm decanter (OPDC) without and with microwave pre-treatment were obtained by scanning electron microscope (SEM) before and after Soxhlet extraction as shown in Figure 4.8. Figure 4.8 (i-iv) showed SEM images of OPDC without pre-treatment before Soxhlet extraction; OPDC without pre-treatment after Soxhlet extraction; OPDC with pre-treatment before Soxhlet extraction and OPDC with pre-treatment after Soxhlet extraction respectively. SEM images showed the OPDC sample appeared to be shrink on the surface at 300x magnification.

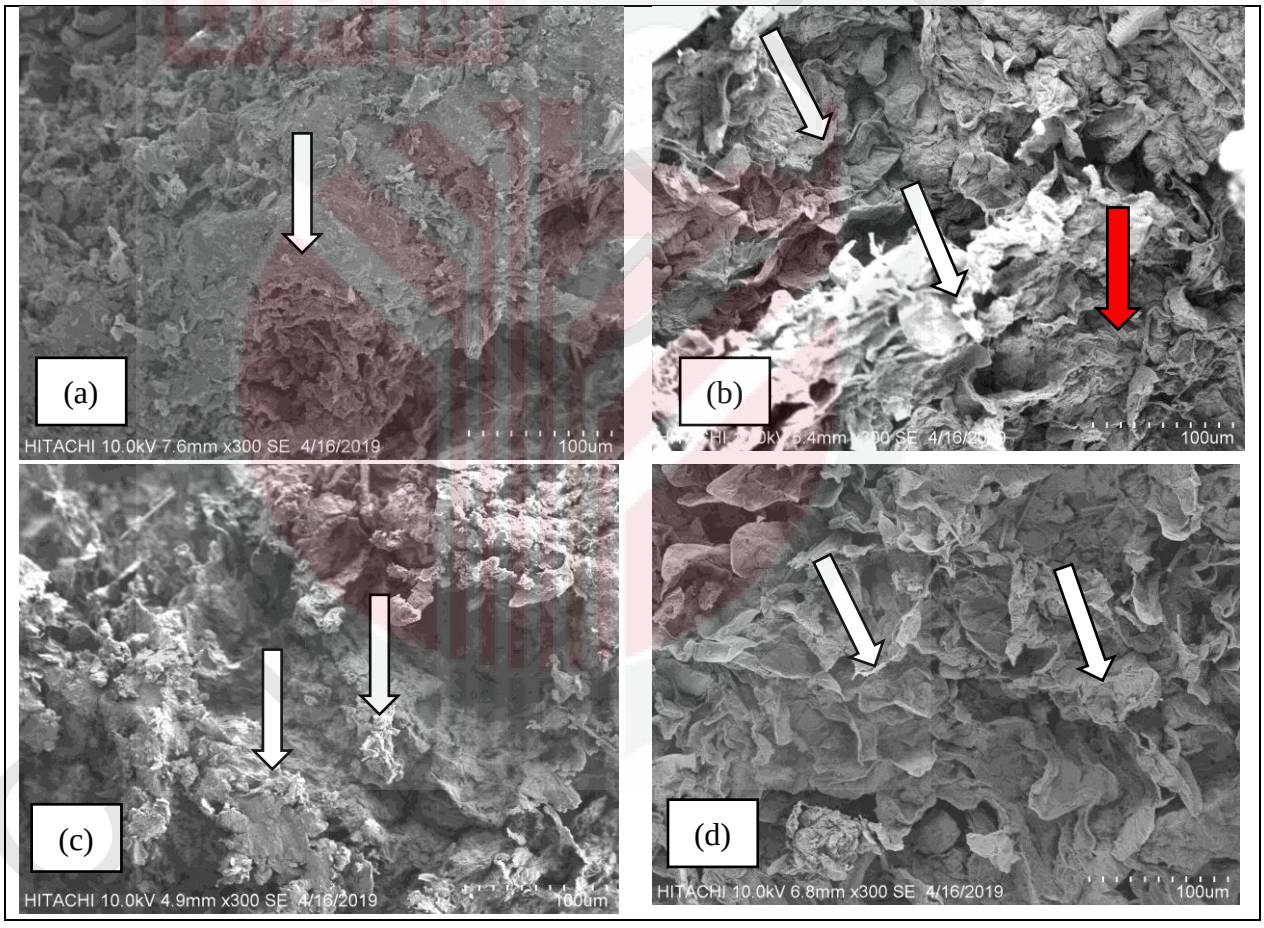



Figure 4.8: Scanning electron micrograph of OPDC; (a) SEM image of OPDC without pre-treatment before Soxhlet extraction; (b) SEM image of OPDC without pre-treatment after Soxhlet extraction; (c) SEM image of OPDC with pre-treatment before Soxhlet extraction; (d) SEM image of OPDC with pre-treatment after Soxhlet extraction

SEM images showed the morphological changes on the surface of the OPDC samples. The surface of OPDC sample without pre-treatment before extraction appear to be flatten and smoother as shown in Figure 4.8(a) compare OPDC with microwave pre-treatment before Soxhlet extraction as shown in Figure 4.8(c) where the surface has started to shrink. For sample after Soxhlet extraction, Figure 4.8(b) image showed a red arrow where the labelled part is still smooth and flatten compare to Figure 4.8(d) as the overall surface shown to be more shrunk. This showed that more oil was extracted for OPDC with microwave pre-treatment compare to OPDC without pre-treatment as stated in the result of oil yield comparison in section 4.3. In contrast, OPDC sample shown shrunk surface as the oil was extracted from the sample.

## 4.6 Chemical Analysis on Extracted Oil

The FTIR spectra of oil extracted for sample without and with pre-treatment by Soxhlet extraction are presented in Figure 4.9. The different pre-treatment method showed a relatively similar FTIR spectra pattern. FTIR test was carried out to determine the functional group of oil extract. The wavenumber of each functional group was referred to Figure 4.9.

| *Functional Group* | *Characteristic Absorption(s)* ($cm^{-1}$) |
|---|---|
| Alkyl C-H Stretch | 2950 - 2850 (m or s) |
| Alkenyl C-H Stretch<br>Alkenyl C=C Stretch | 3100 - 3010 (m)<br>1680 - 1620 (v) |
| Alkynyl C-H Stretch<br>Alkynyl C≡C Stretch | ~3300 (s)<br>2260 - 2100 (v) |
| Aromatic C-H Stretch<br>Aromatic C-H Bending<br>Aromatic C=C Bending | ~3030 (v)<br>860 - 680 (s)<br>1700 - 1500 (m,m) |
| Alcohol/Phenol O-H Stretch | 3550 - 3200 (broad, s) |
| Carboxylic Acid O-H Stretch | 3000 - 2500 (broad, v) |
| Amine N-H Stretch | 3500 - 3300 (m) |
| Nitrile C≡N Stretch | 2260 - 2220 (m) |
| Aldehyde C=O Stretch<br>Ketone C=O Stretch<br>Ester C=O Stretch<br>Carboxylic Acid C=O Stretch<br>Amide C=O Stretch | 1740 - 1690 (s)<br>1750 - 1680 (s)<br>1750 - 1735 (s)<br>1780 - 1710 (s)<br>1690 - 1630 (s) |
| Amide N-H Stretch | 3700 - 3500 (m) |

Figure 4.9: Table of IR Absorptions. Adapted from (Strouse, 1997)

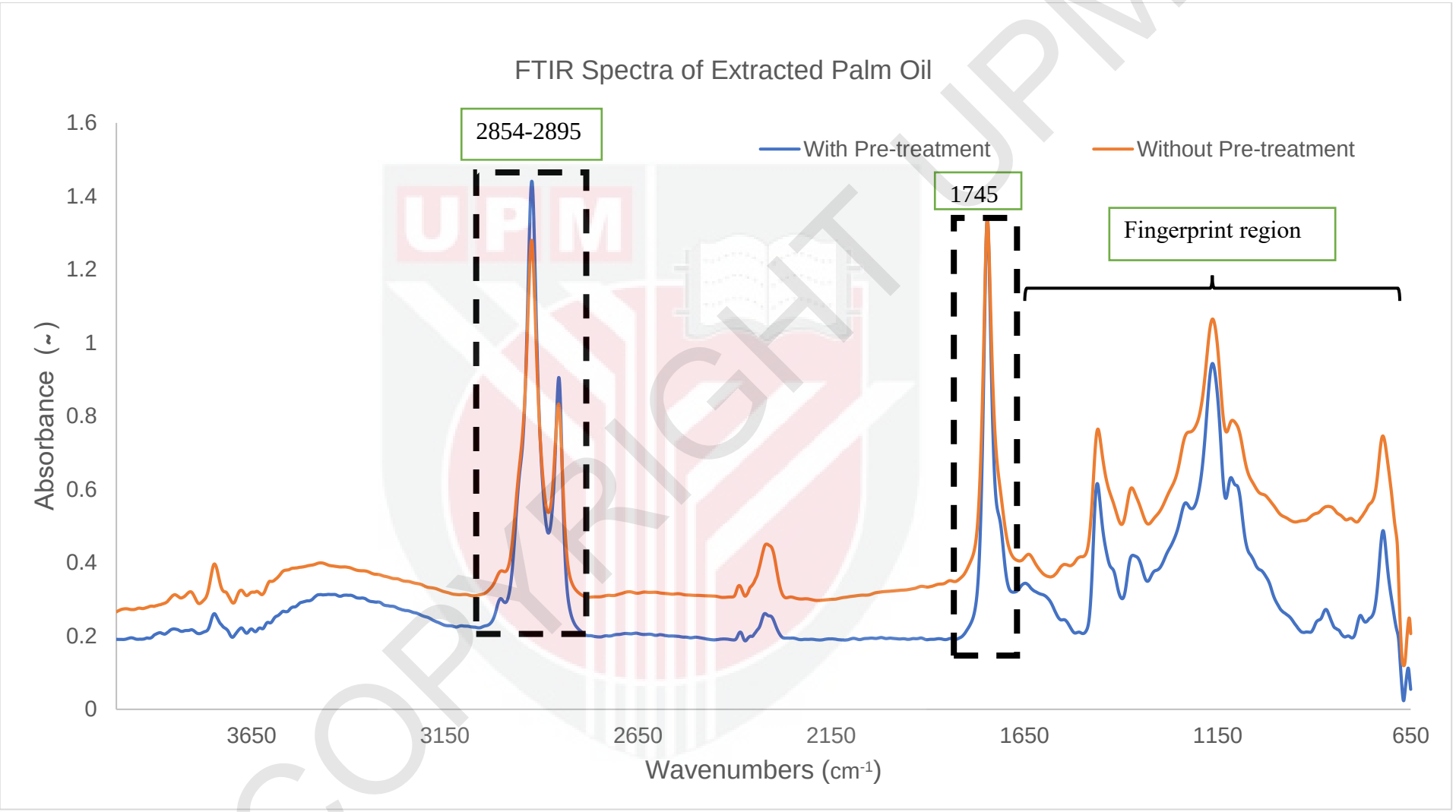



Figure 4.10: Overlay of FTIR Spectra for oil sample with and without pre-treatment for wavenumber between (650 to 4000 $cm^{-1}$)

Table 4.12: FTIR Spectra data for oils sample

| Oil Extract | pH | C-H Stretch | C=O |
|---|---|---|---|
| Without Pre-treatment | 5.690 | 2855-2925 | 1745 |
| With Pre-treatment | 5.315 | 2854-2925 | 1745 |

Functional group that absorb IR light at specific wavenumber will provide very specific peak at specific wavenumber. This information illustrates the kind of bonds in the compound. Table 4.12 showed two major peaks on FTIR Spectra of Figure 4.10. Both oil sample has very similar peaks where the 1st speak is at 2854 to 2925 $cm^{-1}$ and 2nd peak at 1745 $cm^{-1}$. By referring to Figure 4.9 the wavenumber of 1st peak is C-H alkene stretch while the wavenumber of 2nd peak referred to C=O stretch which is ester. Both oil samples were measured to be acidic where oil sample with pre-treatment showed higher acidic compared to oil extract without pre-treatment. This can be explained by referring to Figure 4.10 as the absorbance of 1st peak for sample with pre-treatment is much higher compared to sample without pre-treatment.

Ester is derived from carboxylic acid and alcohol ester is the main class of lipids which makes up the vegetable oil. Both functional groups indicated the present of fatty acid in the oil extract (Ameera and Arsad, 2016). Further analysis can be performed on oil extract by using Gas-chromatography to determine the component in oil extracts as palm oil contains oleic and palmitic acid the most which portray its oil quality.

The fingerprint region shown on the right side of Figure 4.10 within wavenumber (650 - 1500 $cm^{-1}$) is the region that is hard to analysis by FTIR due to stretching vibrations of functional groups. The stretching vibrations of a functional group vary within a narrow range (Ernest, 2015). This region usually contains a very complicated series of absorptions.

# CHAPTER 5 CONCLUSION AND RECOMMENDATION

## 5.1 Conclusion

The main aim of this study was to determine the optimum two parameters such as reaction time and solid to solvent ratio for extracting palm oil from oil palm decanter cake (OPDC) by using Soxhlet extraction. The optimum parameters for laboratory scale of Soxhlet extraction designed by Response Surface Methodology (RSM) were found to be at 4.923 hours of reaction time and solid to solvent ratio of 1:10.

The proposed model design by RSM shows $R^2$ value of 0.776 where the experimental parameters are significant to the result. Considering the optimum parameters achieved based on three criteria stated such as minimum reaction time, minimum solvent ratio and maximum oil yield and the optimized data was employed for comparison of oil yield for OPDC without and with microwave pre-treatment. OPDC with microwave pre-treatment yielded 3.289g of palm oil which was higher than that of OPDC without microwave pre-treatment which yielded only 3.107g of palm oil.

For physical analysis of extracted oil from OPDC without and with microwave pre-treatment, both data were similar. The result for specific gravity, L*a*b colour and refractive of extracted oil for OPDC without and with pre-treatment were 0.960 and 0.897; 58.8L*6.0a*29.3b and 58.7L*5.8a*29.0b; and 1.462 nd. and 1.462nd. respectively. Biological analysis with SEM images indicated that OPDC with pre-treatment had more shrinkage on the surface after Soxhlet extraction compare to OPDC without pre-treatment. This showed that more oil was being extracted from OPDC with microwave re-treatment. Lastly, the chemical analysis by using FTIR showed that both oil sample has two similar peaks on the FTIR Spectra where the 1$^{st}$ peak at the range of 2854 to 2925 $cm^{-1}$ was C-H alkene stretch and 2$^{nd}$ peak at 1754 which was C=O stretch ester. Both of these functional groups indicated the present of fatty acid in the oil sample.

## 5.2 Recommendations

More work can still be done regarding the extraction of palm oil from OPDC by using Soxhlet extraction through Response Surface Methodology (RSM) where the experimental data can be repeated even more times to obtain more consistent data with same parameters in order to achieve higher value of $R^2$ and desirability. Moreover, type of solvent can also be studied as one of the parameters that affect the yield of oil through Soxhlet extraction. Soxhlet extraction is a conventional method for oil extraction process, however other type of extraction method can be performed to compare the efficiency of both extraction method such as Supercritical Fluid Extraction (SFE). Other pre-treatment method such as ultrasonic pre-treatment on a laboratory scale can also be investigated to study its effect of on the oil yield.

Further chemical analysis on the component of extracted oil is suggested to be carried out by using Gas-chromatography (liquid) to study the detailed component of oil extract. Extracted oil contains ester which is the main class of lipid and has the potential to be used as biodiesel. Further approaches should be done to convert OPDC to another form of energy or usage.

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