# SEPARATION TECHNIQUES OF CRUDE PALM OIL AT CLARIFICATION AREA VIA OPTIMUM PARAMETERS

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#### **OPTIMUM PARAMETERS**

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# SEPARATION TEHCNIQUE OF CRUDE PALM OIL AT CLARIFICATION AREA VIA OPTIMUM PARAMETERS

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A thesis submitted in fulfillment of the requirement for the award of the degree of Bachelor of Chemical Engineering

Faculty of Chemical and Natural Resources Engineering Universiti Malaysia Pahang

APRIL 2009

## DECLARATION

I declare that this thesis is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature	:
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Date	: 29 APRIL 2009

DEDICATION

To my beloved ones; my mother, father, siblings, and all my dearest friends

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In preparing this thesis, I was in contact wth many individual, academicians, researchers as well as industrial peolple. They have contributed toward my understanding and thoughts. In particular, I wish to express my sincere appreciation to my main supervisor Tn Hj. Mohd Noor Bin Nawi and co-supervisor En. Wan Zurizak Bin Wan Daud for encourangement, guidance, critic, friendship, understanding and so on. Without their continued support and interest, this thesis would not be the same as presented here. I also would like to thank staffs in Kilang Sawit LCSB, Lepar for their kindly help for sample preparation.

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## ABSTRACT

The purpose of clarification is to separate the oil from its entrained impurities such as water, cell debris, fibrous material and non oily solid (N.O.S). In this study, optimum parameter such as temperature, effect of impeller, residence time and separation technique of crude palm oil is at clarification stage is determined. By using correct separation technique and condition, mill can maximize oil production and reduce oil losses. In this experimental, CPO are heated at 50°C, 60°C, 70°C, 80°C, 90°C, 110°C and 120°C for optimum temperature determination using water shaker batch. Experimentally, the results proved that by using 95°C for heating, CPO took the shortest time for separation. Difference size beakers also tested while separation. As larger the surface area of beaker, shorter time is taken for CPO separation. Otherwise the volume of CPO is directly proportional to the separation time. The present of impeller also enhances the separation of oil and sludge thus reduced the oil content in underflow while the pure oil obtained is increased.

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## ABSTRAK

Penjernihan berfungsi untuk memisahkan minyak daripada bendasing seperti air, debris, fiber dan pepejal bukan minyak. Dalam kajian ini, optima parameter seperti suhu, kesan pemutaran, masa penahanan dan teknik pemisahan minyak kelapa sawit mentah ditentukan. Dengan menggunakan kaedah pemisahan yang betul, kilang dapat memaksimumkan pengeluaran miyak sawit dan mengurangkan kehilangan minyak. Dalam eksperimen ini, minyak sawit mentah di panaskan menggunakan pemanas air seragam pada suhu 50°C, 60°C, 70°C, 80°C, 90°C, 110°C dan 120°C untuk menentukan suhu optima. Keputusan menunjukkan pada suhu 95°C, masa untuk pemisahan diambil adalah paling singkat. Semakin besar luas permukaan bikar, semakin singkat masa diambil untuk pemisahan, manakala semakin banyak isipadu minyak sawit mentah semakin lama masa diambil untuk pemisahan. Kehadiran pemutar juga boleh menggalakkan pemisahan minyak dan enap cemar sepertimana kurang kandungan minyak di saluran bawah tangki penjernihan dan lebih banyak minyak tulen diperolehi.

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## **CHAPTER 1**

## **INTRODUCTION**

#### 1.0 Introduction

Palm oil is extracted from the mesocarp of the fruit of the palm *Elaeis guineensis*. There are a few varieties of this plant but Tenera, which is a hybrid of the Dura and the Pisifera, present abundantly through out the whole Peninsular. Productivity of plantations is high (typically 15 t/ha of fresh fruit bunches in Ivory Coast, 20-30 t/ha in Malaysia and Indonesia).

From the fruit two distinct types of oils are produced palm oil and palm kernel oil. Both are edible oils but with very different chemical composition, physical properties and applications. Each palm fruit produces about 90% palm oil and 10% palm kernel oil. The mesocarp comprises about 70 - 80% by weight of the fruit and about 45 - 50% of this mesocarp is oil. The rest of the fruit comprises the shell, kernel, moisture and other non fatty fiber. The extracted oil is known as crude palm oil (CPO) which until quite recently was known as the golden commodity.

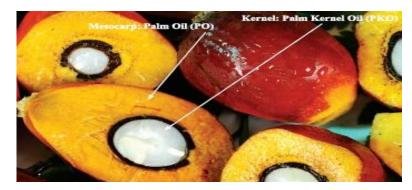


Figure 1.1 Palm Oil Fruitlets

The most important for oil palm mill is to maximize oil recovery to fulfill the demand. Global consumption of major oils and fats has been increasing over the last few years, driven by growing consumer demand, especially in the developing world. Usage of vegetable edible oils also increasing which it is uses in replacing animal fats in foods, feeds and other non food applications. Palm oil is the fastest growing segment of the world edible oil production base, growing from less than 6 million MT in 1983/1984 to more than 27 million MT in 2002/2003. In the five-year period 1999/2000 to 2002/2003, palm oil production increased at an average of 9.5% per year. In comparison, the total supply of oils and fats only grew at an average annual growth rate of about 4% in the same period, to a total of 122 million MT.

Fresh fruit from plantation need to be sent quickly to the mill for processing to obtain crude palm oil. There are several stages of processing the extraction of palm oil from fresh fruit bunches. These include reception, sterilization, bunch stripping, digestion, oil extraction and finally clarification and purifications. (Figure 1.2)

### 1.01 Reception

Fresh fruit arrives from the field as bunches or loose fruit. The fresh fruit is normally weighing on a scale so that quantities of fruit arriving at the processing site may be checked. Large installations use weighbridges to weigh materials in trucks. The quality standard achieved is initially dependent on the quality of bunches arriving at the mill. The mill cannot improve upon this quality but can prevent or minimize further deterioration.

### 1.02 Sterilization

Sterilization or cooking means the use of high-temperature wet-heat treatment of looses fruit. Cooking normally uses hot water; Sterilization uses high pressurized steam (120 to 140°C at 40psi) that subject to freshly harvested fruit bunches with a minimal delay to inactivate the lipolytic enzymes that cause oil hydrolysis and fruit deterioration. The cooking action serves several purposes. Fruit cooking weakens the pulp structure, softening it and making it easier to detach the fibrous material and its contents during the digestion process.

- The high heat is enough to partially disrupt the oil-containing cells in the mesocarp and permits oil to be released more readily.
- The moisture introduced by the steam acts chemically to break down gums and resins. The gums and resins cause the oil to foam during frying. Some of the gums and resins are soluble in water. Others can be made soluble in water, when broken down by wet steam (hydrolysis), so that they can be removed during oil clarification. Starches present in the fruit are hydrolyzed and removed in this way.

## **1.03 Bunch Stripping**

At this stage, the separating of fruits from bunch by mechanical stripping using rotary drum.

#### 1.04 Digestion

Sterilized and separated fruit then undergo digestion. Digestion is the process of releasing the palm oil in the fruit through the rupture or breaking down of the oil-bearing cells. The digester commonly used consists of a steam-heated (80°C-90°C) in cylindrical vessel fitted with a central rotating shaft carrying a number of beater (stirring) arms. Digestion helps to reduce the viscosity of the oil, destroys the fruits' outer covering (mesocarp) and loosening the mesocarp from the nuts.

## 1.05 Oil Extraction

The crude oil is extracted from the digested fruit mash using screw press without breakage the kernel. The extracted liquid and nuts are discharged from the screw press. The extracted oil contains amount of water, solids and dissolved impurities that must be removed. The fiber particles from the pressed crude oil are first removed by passing the oil over a vibrating screen. Here, sand and dirt are allowed to settle. Water is removed by settling or centrifuging and finally by vacuum drying.

## 1.06 Clarification

Clarifying and drying of oil. The main point of clarification is to separate the oil from its entrained impurities. The fluid coming out of the press is a mixture of palm oil, water, cell debris, fibrous material and 'non-oily solids'. Because of the non-oily solids the mixture is very thick (viscous). Hot water is therefore added to the press output mixture to thin it. The dilution (addition of water) provides a barrier causing the heavy solids to fall to the bottom of the container while the lighter oil droplets flow through the watery mixture to the top when heat is applied to break the emulsion (oil suspended in water with the aid of gums and resins). Water is added in a ratio of 3:1. The diluted pass through a screen to remove coarse fibers. The screened mixture is boiled from one or

two hours and then allowed to settle by gravity in the large tank so that the palm oil, being lighter than water, will separate and rise to the top. The clear oil is decanted into a reception tank. This clarified oil still contains traces of water and dirt. To prevent increasing FFA through autocatalytic hydrolysis of the oil, the moisture content of the oil must be reduced to 0.15 to 0.25 percent. Re-heating the decanted oil in a cooking pot and carefully skimming off the dried oil from any engrained dirt removes and any residual moisture. Continuous clarifiers consist of three compartments to treat the crude mixture, dry decanted oil and hold finished oil in an outer shell as a heat exchanger.

## 1.07 Oil Storage

In large-scale mills the purified and dried oil is transferred to a tank for storage prior to dispatch from the mill. Since the rate of oxidation of the oil increases with the temperature of storage the oil is normally maintained around 50°C, using hot water or low-pressure steam-heating coils, to prevent solidification and fractionation. Iron contamination from the storage tank may occur if the tank is not lined with a suitable protective coating. Small-scale mills simply pack the dried oil in used petroleum oil drums or plastic drums and store the drums at ambient temperature.

## 1.08 Kernel recovery

The residue from the press consists of a mixture of fiber and palm nuts. The nuts are separated from the fiber by hand in the small-scale operations. The sorted fiber is covered and allowed to heat, using its own internal exothermic reactions, for about two or three days. The fiber is then pressed in spindle presses to recover second grade (technical) oil that is used normally in soap-making. The nuts are usually dried and sold to other operators who process them into palm kernel oil. The sorting operation is usually reserved for the youth and elders in the village in a deliberate effort to help them earn some income. Large-scale mills use the recovered fiber and nutshells to fire the steam boilers. The super-heated steam is then used to drive turbines to generate electricity for the mill. For this reason it makes economic sense to recover the fiber and to shell the palm nuts. In the large-scale kernel recovery process, the nuts contained in the press cake are separated from the fiber in a depricarper. They are then dried and cracked in centrifugal crackers to release the kernels. The kernels are normally separated from the shells using a combination of winnowing and hydrocyclones. The kernels are then dried in silos to a moisture content of about 7 percent before packing.

During the nut cracking process some of the kernels are broken. The rate of FFA increase is much faster in broken kernels than in whole kernels. Breakage of kernels should therefore be kept as low as possible, given other processing considerations.

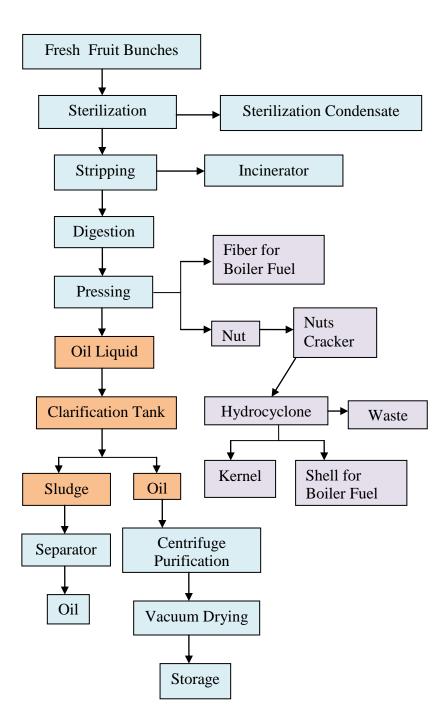


Figure 1.2 A Block Flow Diagram of The Palm Oil Mill Process

From Figure 1.2, orange block shows the clarification and purification process area that involves in this research.

### **1.1 Problem Statement**

The problem now at oil palm industry are high cost in turbine generate energy that use to heat up the settling tank. Other problem is time taken to finish the separation of CPO is quite long that usually take 1 hour, thus lower the production of purify palm oil with higher ratio of oil compare to sludge and non oily solid. In other hand employee that handle the processing of CPO can be decrease hence save the production cost. Using high temperature for CPO separation also will increasing equipment maintenance cost and create such harmful condition to mill workers.

Palm oil mill also faces challenge to maximize the oil collection in crude oil tank using optimum temperature supply from steam coil, so the clear oil can be skimmed out directly to oil tank without undergoing separation in clarifier tank as shown in Figure 3.1.

We also need to minimize the oil content pass through clarifier tank because there are some disadvantages. The disadvantages occur when crude oil pass through the 10.5 meter in height clarifier tank, worker need to climb to the high tank to adjust the adjustable level valve for clear oil skimming. The adjusting operation need to be done at least every one hour. Otherwise, the oil become thicker downward in the clarifier tank and remixes with the sludge. It will cause a lot of oil-sludge mixed discharge to underflow and then to the sludge tank. Longer time needed when the oil is reclarify in clarifier tank after have been separated with sludge and solid in centrifuge. (Figure 3.1)

## 1.2 Objectives

- To maximize oil production in oil palm mill.
- To determine optimum temperature, optimum residence time and effect of impeller in separation of crude palm oil (CPO) for separation to oil, sludge and non oily solid at clarification process area.

### **1.3** Scopes of study

To achieve the objective, scopes have been identified in this research. The scopes of this research are listed as below:-

- (I) Identification of sludge oil separation problem.
- (II) Identification of the right equipment for separation technique.
- (III) Determination of optimum temperature
- (IV) Determination of optimum residence time.
- (V) Determination of stirrer in clarification tank

With constant feed rate, diameter and height of CPO in tank.

#### **1.4** Rationale and Significance

The rationale for determine the optimum temperature and residence time of crude palm oil in settling tank while clarification process is to save energy and time while provide the heat.

The result from survey of 5 crude palm oil factories showed that electricity is the dominant source of energy for production process. Total energy consumption of all electric machines used in the production process is average 14.46 kWh/ton FFB. Palm oil mills in Thailand operate on cogeneration system using biomass residue as fuel in the boiler. The boiler produces high pressure and temperature steam, which expands in a backpressure steam turbine and produces enough electric power for the internal needs of the mill. The exhaust steam from the turbine goes to an accumulator which distributes the steam to various processes in the mill. The electricity used in this mill is distributed among 2 sources; turbine generator in factory and purchase from government supplier. The electricity generated in the factory is about 77.7 % of total electricity consumption. The power plant in the crude palm oil mill incorporates water tube boiler with a steaming capacity of 20 to 30 ton of steam/ hour. Fiber is used as fuel for generation of power in factory and used to supply the domestic purpose (3.3% of total electricity consumption).

Fuel used in the production process consists of 0.12 liters diesel oil/ ton FFB. Diesel oil is uses for diesel generator for start up boiler.

Thus, by knowing the optimum temperature in separation CPO, employer can save cost for energy supplying and reduce number of employee. We also may observe the effect of agitator in separation crude palm oil to oil, sludge and non oily solid. The impeller with speed of 5-10 rpm enhances the separation process.

## **CHAPTER 2**

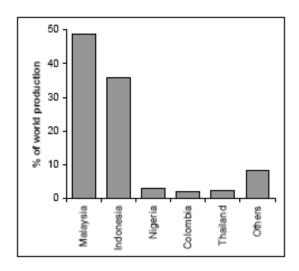
#### LITERATURE REVIEW

#### 2.0 Definition of Palm Oil.

Palm oil is produced from the oil palm, primarily Elaeis guineensis, which originated in West Africa, but has adapted extremely well to other tropical lowland regions. The largest producer of palm oil is Malaysia, accounting for approximately 49% of global production. Indonesia ranks second, accounting for another 36%. Nigeria follows a distant third, with 2.9%. Oil palm plantations exist on a much smaller scale in several other African countries and in Central and South America (e.g., Colombia, Ecuador and Costa Rica). SE Asia is thus by far the main palm oil producing region accounting for in excess of 85% of world production (Figure 2.1). This is produced from over of 6 million hectares of plantation which represents nearly 80% of the world total oil palm plantation area. Between 1999/00 and 2002/03 Malaysian production of palm oil grew at 8.5% per year, whilst Indonesian production growth outstripped the world average, growing at 14.7% per year. (*IIED, ProForest, Rabobank, 2004*).

Palm oil contains an equal proportion of saturated and unsaturated fatty acids. It's particularly rich in the saturated palmitic acid (44%), with substantial amounts of the monounsaturated oleic acid (40%), and smaller amounts of polyunsaturated fatty acids (10%).

End users of palm oil are firstly producers of margarines, shortenings, cooking oils etc., such as Unilever and Vandemoortele, and secondly the users of such products. These are primarily found in the bakery business, confectionery, ice cream, snacks, the noodle industry and sectors using frying products.



Source: Oil World 2003 Oil World Annual. Mielke, Hamburg. **Figure 2.1** World Production of Palm Oil by Country in 2002-2003

## 2.1 Definition of Crude Palm Oil (CPO).

Crude Palm Oil (CPO) accounts for 21% of the global oils and fats supply, and 26% of the global vegetable oil supply. In comparison, soybeans yield 0.4 to 0.5 tones per hectare, and account for 25% of global oils and fats supply and 31% of global vegetable oil supply. Palm oil is the highest yielding oil crop per hectare. One hectare of oil palm yields 15–30 tones of fresh fruit, giving 2 to 7 tones of CPO, as well as PKO (Palm Kernel Oil) that is extracted from the kernels. Average production per tree is about 10 to 12 fruit bunches per year, each weighing between 20 and 30 kg. The harvested FFB are transported by truck from the plantation to the mill.

Crude palm oil has three main components, which are a mixture of oil and water, oil in water emulsions and water in oil emulsions. In clarifying station, the crude palm oil is separated into pure oil and sludge (*Stork*, 1960). An approximate average composition of screw pressed crude palm oil might be 64% oil, 24% water and 12% non oil solid (*Maycock*, 1987).

The properties of crude palm oil are shown in Figure 2.2. Examination of a sludge sample revealed the presence of oil droplets of sizes varying from less than 1  $\mu$ m. The difference in specific gravity between sludge oil is practically constant at 0.1 throughout the temperature range from 40°C to 100°C (*Stork, 1960*).

The largest solid impurity to be separated is the fiber and the smallest is the cellular debris. Due to the high ratio of solids to oil and the low ratio of water to solids in the crude oil from a screw press, water is added in order to enhance the settling efficiency. Experimental results show that the viscosity increases with the amount of water added up to 50% dilution. Beyond this point, the viscosity continuously falls with higher dilution but less steeply.

Crude palm oil is actually a mixture of about 92 to.95% oil, 4-5% oil soluble, up to 5% free fatty acids (FFA) and about 0.5% water, water soluble and solids. Generally the oil is made up of triglyceride (94-97%), diglyceride (2-3.5%) and monoglyceride (0.3-0.5%).(*Abdul Aziz, 2000*).

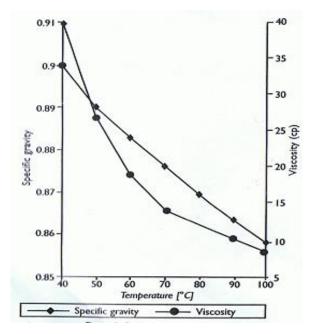


Figure 2.2 Crude Palm Oil Properties

Palm oil is a dark yellow to yellow-red oil (high carotene content) of vegetable origin obtained by pressing or boiling the flesh of the fruit of the oil palm. Palm oil differs from palm kernel oil, the latter being obtained from the kernels of the oil palm.

### 2.1.1 Chemical composition

Palm oil and palm kernel oil are composed of fatty acids, esterified with glycerol just like any ordinary fat. Both are high in saturated fatty acids, about 50% and 80%, respectively. The oil palm gives its name to the 16 carbon saturated fatty acid palmitic acid found in palm oil; monounsaturated oleic acid is also a constituent of palm oil while palm kernel oil contains mainly lauric acid. Palm oil is the largest natural source of tocotrienol, part of the vitamin E family. Palm oil is also high in vitamin K and dietary magnesium.

Napalm derives its name from naphthenic acid, palmitic acid and pyrotechnics or simply from a recipe using naphtha and palm oil.

The approximate concentration of fatty acids (FAs) in palm oil is as follows: (*Ang, Catharina Y.W., 1999*)

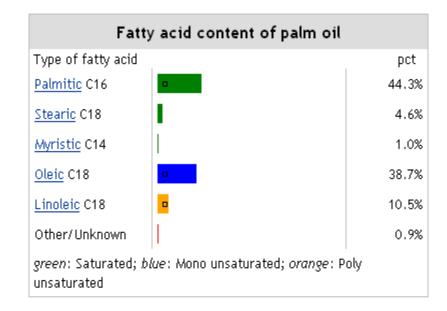


Figure 2.3 Fatty Acid Content of Palm Oil

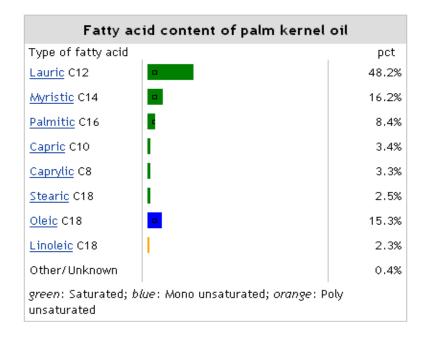


Figure 2.4 Fatty Acid Content of Palm Kernel Oil

Fatty acids are saturated and unsaturated aliphatic carboxylic acids with carbon chain length in the range of C6 up to C24. An example of a fatty acid is palmitic acid

CH3 - (CH2)14 - COOH

Figure 2.5 and 2.6 summarize the formation of storage oil of the palm fruit mesocarp cells.

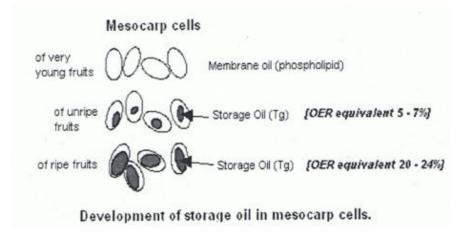


Figure 2.5 Development of storage oil in mesocarp cells.

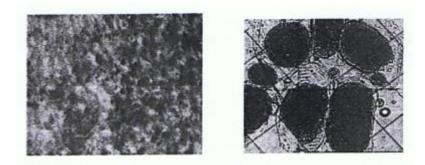


Figure 2.6 Young (left) and ripe (right) fruit mesocarp cells with oil globules.

Storage oil (Figure 2.6) also termed as oil globule or body occupies more twothird of volume of the cells.

Triglyceride (Tg) is a very stable molecule but only as long as it is confined within the cells. All mesocarp cells, in addition to having storage oil, contain lipase(hydrolytic enzyme). this enzyme has very specific function, i.e., breaking the triglyceride molecule back to fatty acids and glycerol through a process called hydrolysis. however this enzyme is heat liable. at above 80°C the enzyme is destroyed. Early deactivation of the enzyme will result in higher triglyceride recovery.

Harvesting and rough handling of harvested bunch especially at he platform and ramps will damage the cellular components of the fruits. When damage oil will be hydrolyzed to fatty acids and glycerol (Abdul Aziz and Tan, 1989).

In a complete hydrolysis (Figure 2.7 and 2.8), all the 3 fatty acids of the triglyceride are dislodged. the released fatty acid is termed as free fatty acid(FFA). In an incomplete hydrolysis one or two acids may be dislodged. the resultant products are glycerol with one fatty acid still attached (Figure 2.8).

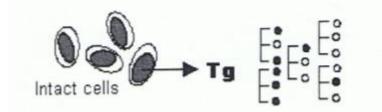


Figure 2.7 Storage oil of intact cells is purely triglyceride (Tg)

Storage oil released from broken cells contains the triglycerides (tg) and hydrolyzed products of Tg, namely diglycerides (Dg), Monoglycerides (Mg), free fatty acids (FFA) and glycerols. The amount and type of free fatty acids depend on the degree and type of damage experienced by the fruits. All cellular damages are attributed to physical actions.

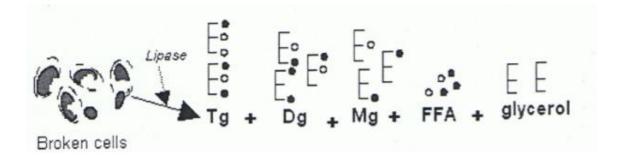


Figure 2.8 Traces the formation of di and monoglycerides, FFA and glycerol.

Free fatty acids (FFA). are the products of hydrolysis of TG and/or Dg and/or Mg. The FFA of CPO include the saturated lauric, myristic, palmitic and stearic acid ant the unsaturated oleic, linoleic and linolenic acid. (Figure 2.9)

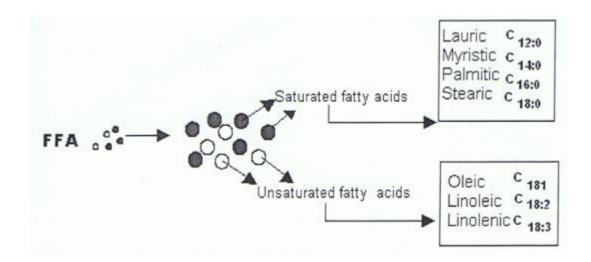


Figure 2.9 The hydrolyzed free fatty acid of CPO.

The presence microbial and moisture in crude oil allows hydrolysis. Nonetheless CPO must be allowed to have some moisture. Moisture gives some buffering effect to the oil to abstain from other chemical reactions. However its potent effect is only possible in the presence of moisture.

Glycerol esterified to two fatty acid molecules is called diglyceride, Whilst glycerol with one fatty acid is monoglycerides. The free fatty acids, formed via hydrolysis process, consist both the saturated and unsaturated fatty acids(Figure 2.9).

Whilst saturated fatty acids are very stable, the unsaturated fatty acids are easily oxidized. These oxidation products include the peroxides, ketones and aldehydes.(Figure 2.10).

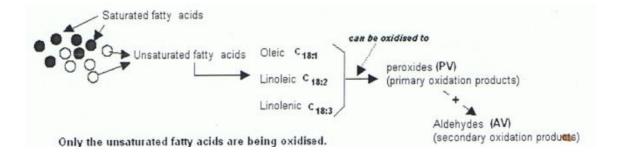


Figure 2.10 Only the saturated fatty acids are oxidized.

The best quality CPO is one that contains the highest amount of triglycerides. Tryglycerode (Appendix D) is a molecule made up of glycerol holding 3 fatty acids. The variable fatty acids (both saturated and unsaturated acids) thats constitute the triglyceride molecule. (Table 2.1)., permits each triglyceride to attain its specific physical (melting and boiling points) properties (Figure 2.11). Stearic-stearic-stearic (SSS) or stearic-palmitic-stearic (SPS) or palmitic-palmitic-palmitic (PPP) trygliceride is solid at room temperature, whilst oleic-oleic-oleic (OOO) or palmitic-oleic-oleic (POO) or oleic-linleic-oleic (OLO) triglyceride is liquid. Stearic- oleic-stearic (SOS), Palmitic-palmitic-oleic-oleic-oleic linleic-oleic (POP) and Lauric-oleic-lauric (LOL) will be in semi solid. Significant quantity of Dg and Mg in the oil will affect the efficiency of crystallization of the designated triglyceride.

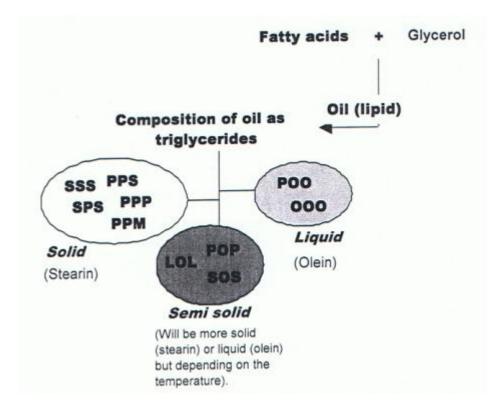


Figure 2.11 Summarize of the effect of cooling on the different triglycerides in palm oil.

<b>Table 2.1</b> The unsaturated acid mono and diglycerides with high melting will crystallize
if cooled below 20°C.

		Melting Point(Centrigrade)			
Fatty acid	Carbon No.	as acid	as 1-Mg	as 1,3-Dg	as (Tg) triglyceride
Lauric	12	44.2			46.4
Myristic	14	54.4			57.0
Palmitic	16	62.9	77	76.3	63.5
Stearic	18	69.6	81.5	79.4	73.1
Oleic	18:1	16.3	35.2	21.5	5.5
Linoleic	18:2	-6.5	15.7	-2.6	-13.1
Linolenic	18:3	-12.8			-24.2

## 2.2 Life Cycle Inventory (LCI)

Inventory data collected over of 3 months from each mill is divided into environmental inputs and output. the data is then extrapolated to quantify the inputs from the environment and outputs to the environment for every one ton of CPO produced to create LCI as shown in Table 2.2 and 2.3. Using LCI data which consists of input and output of the system boundary for every 1 ton of CPO produces, the Life Cycle Impact Assessment (LCIA) is conducted using the Simapro Software version 6.0. The software is an European software with European Data. However, the software is generic and Malaysian data has been input to conduct the study. (*Vijaya S, Ma A.N, Choo Y.M & Nik Meriam N.S*)

Mill	А	В	С	D	E	F
Туре	Private	Plantation	Private	Plantation	Plantation	Plantation
Processing						
Capacity (t/hr)	40	45	90	20	70	30
Fuel flow rate (t/hr)	5.60	4.88	9.00	2.10	13.00	3.20
Steam output (t/hr)	19.0 Vickers	25.0 Mechmar	35.0 Vickers	11.0 IBAE	50.0 Vickers	15.0 Allied
Boiler type ENVIRONMENTAI	Hoskins		Hoskins		Hoskins	Equipment
Fresh Fruit Bunch						
(t) Power	5.34	5.27	5.39	4.93	4.87	5.07
consumption from Turbine (kWh)	106.73	105.48	107.71	98.59	97.36	101.38
Power consumption from Grid (kWh) Diesel consumption	0.18	2.95	0.10	2.00	0.00	1.99
for Mill (L) Diesel consumption	1.29	2.92	0.38	2.17	4.51	0.00
for vehicles in mill (L)	2.60	0.84	2.10	0.28	0.68	0.15
Boiler fuel Mesocarp fibre(t)						
Shell (t)	0.52	0.47	0.49	0.43	0.55	0.57
Shell (t)	0.22	0.20	0.05	0.11	0.36	0.24
Boiler water consumption (L)	2.54	3.43	2.12	2.79	3.50	3.83
Steam input for sterilization (t) Steam input to	3.47	3.43	3.50	3.20	3.16	3.29
turbine (t)	2.54	3.43	2.12	2.79	3.50	3.83

# Table 2.2 Inventory Data of 6 Mills-Environmental Inputs for Every 1 t of CPO Produced

Bolded rows indicate renewable energy or recycled materials

Table 2.3 Inventory Data of 6 Mills-Environmental Outputs for Every 1 t of CPO Produced

Mill	А	В	С	D	E	F
ENVIRONM	ENTAL OUTP	UTS				
Mesocarp	0.12	0.16	0.16	0.16	0.04	0.04
Fibre (t)						
Shell (t)	0.15	0.17	0.32	0.24	0.00	0.11
EFB (t)	1.23	1.21	1.24	1.13	1.12	1.17
POME(m3)	3.20	3.16	3.23	2.96	2.92	3.04
Methane from POME pond (m <sup>3</sup> )	58.27	57.59	58.81	53.83	53.16	55.35
CO <sub>2</sub> from	31.38	31.01	31.67	28.99	28.62	29.81
POME pond (m <sup>3</sup> )	51.50	51.01	51.07	20.77	20102	27.01
Boiler ash (t)	0.02	0.02	0.02	0.01	0.01	0.02
Flue Gaseous						
from stack Particulate						
Matter (kg)	0.11	0.14	0.56	0.06	0.45	0.14
CO (kg)	0.06	0.06	0.11	0.02	0.14	0.05
CO <sub>2</sub> (kg)	56.87	56.05	114.59	11.56	161.20	40.05
SO <sub>x</sub> (kg)	0.001	0.001	0.002	0.0004	0.003	0.001
NO <sub>x</sub> (kg)	0.12	0.09	0.20	0.04	0.20	0.07
RECYCLED	WASTE					
EFB	Shredded& used as fuel	Mulching	Mulching	Mulching	Mulching	Mulching
POME	Treated & sold as fertilizer	Treated and used as fertilizer	Treated & discharged	Treated and used as fertilizer	Treated and used as fertilizer	Treated and used as fertilizer
Excess						
Mesocarp fibre & shell	Sold as fuel	Sold as fuel	Sold as fuel	Sold as fuel	•	Sold as fuel
Boiler ash	Land application	Land application	Land application	Land application	Land application	Land application

## 2.3 Water Consumption

More than 60 % of water used is for cooling system in turbine generator, the rest of 40 % is for steam boiler. Steam from boiler is first applied to turbine generator for electricity, and then supplied to sterilization, digestion, kernel dryer tank, hot water tank, and clarification tank and oil storage tank. Cooling water is also reuse in production process.

## 2.3.1 Use of Process Water

Water supply is required for the following process:

- 1. Boiler makes up water.
  - During power generation, steam from boiler is supplied to turbine generator.
  - During sterilization, live steam from boiler is applies to autoclave in order to facilitate the stripping of fruit.
  - During digestion, live steam from boiler is pumped to vessel to facilitate homogenization.
  - Heating purpose, live steam from boiler is supply to steam coil in oil settling tank, hot water tank, oil storage tank and kernel dryer tank in order to control temperature.
- 2. Cooling water for the turbine is used to generate electricity from burning fiber.
- 3. During extraction, hot water is used to remove oil from fiber
- 4. During remove large particle from oil in desander step, hot water is use to clean surface of vibrating screen .
- 5. During oil recovery from sludge, as a dilution water for sludge to adjust the concentration of sludge before go to separator.
- 6. Miscellaneous usage such as seal water pump and factory and machine cleaning.

Table 2.4 shows the detailed water consumption at 3 factories which different in oil recovery technology and degree of wastewater recycle in production process.

- Factory A install decanter and separator for oil recovery from sludge and recycle waste water from sterilizer in production process .
- Factory C install decant or for oil recovery from sludge.
- Factory E installs separator for oil recovery from sludge. Separator consume large amount of water because it is common practice to add hot water during centrifugation.

Process stream	Flow rate (m <sup>3</sup> /ton FFB)		
	Factory A	Factory C	Factory E
Boiler make up water (steam) • Sterilizes	0.40	0.54	0.75
• Digester	0.15 0.02	0.25 0.09	0.15 0.06
Other	0.23	0.20	0.54
Water from cooling system in turbine is reuse in	0.85	0.56	0.55
<ul> <li>Screw press</li> <li>Vibrating screen</li> <li>Separator</li> <li>Factory cleaning</li> <li>Domestic purpose and other</li> </ul>	(0.08) (0.005) 0.10 0.08 0.68	- 0.004 - 0.08 0.49	0.08 0.44 0.02
Water use in production process Total water consumption	0.58 1.20	0.62 1.10	1.30 1.30

 Table 2.4 Breakdown of Water Inputs of Selected Factories (Chao, C.C. 1999)

Note: ( ) sterilize condense wastewater reuse in production process.

Oil is immiscible in water. Thus, a mixture of sludge and oil tends to separate into two layers. The force separating the two elements is the difference in the specific gravity between the two elements. A settling time of at least 1hour is now always allowed and the temperature of 85°C is often considered to be suitable. Approximately

70% to 75% of the oil settles easily but the balance cannot be separated by dilution and static settling (*Mongana*, 1955). The most important task in designing a sedimentation process is recognizing the form of settling which is taking place in the suspension under examination (*Rushton et al.*, 1996).

## 2.4 Solid liquid separator

(Wakeman, R.J and Tarleton, E.S, 2007)

# 2.4.1 The selection of solid-liquid separation equipment

The selection of solid-liquid separator equipment is dependent on different factors, and the choice is so very wide, that is it clearly impossible to give a formula whereby one can select the right piece of equipment without any factor of error

#### 2.4.1.1 Sedimentation or filtration?

With sedimentation one must remember that substantially the only driving force is difference in specific gravity between the phases. The apparent difference can be increased by, for instance, applying a centrifugal force or by increasing the mass of individual particles through flocculation. Sedimentation is a process which from the onset can have limiting factors which are beyond the design control. Sedimentation systems in other hand tend to be relatively cheap and ideally for continuous and certainly automatic operation as in palm oil mill.

Filtration on other hand, give an almost unlimited amount of design control since it relies on a man-made septum, so chosen that it will retain those particles which must be separated. Filtration system however far less suitable for continuous production, sometimes not even for automatic production, and tend to be more expensive per volume treated then sedimentation system. (*L. Svarovsky*, 2000)

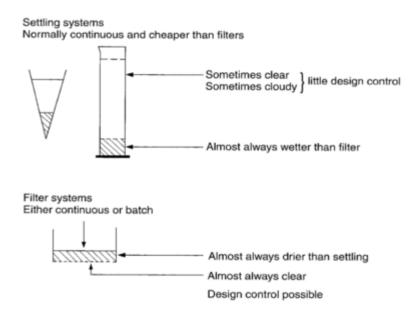


Figure 2.12 Schematic Illustration of Settling and Filtration

## 2.4.2 Gravity thickener and clarifiers

Gravity thickeners and clarifiers represent a class of solid walled separator, where gravitational forces are used to raise the concentration of a suspension through sedimentation to produce a thickened sludge with a clear liquid as overflow. The rate of sedimentation should be as high as reasonably possible to both increase throughput and reduce floor area. The cross-sectional area plan of a thickener controls the time available for sedimentation and is important in determining clarification capacity. The physical depth of a separator controls sludge thickening time and is an important parameter in determining thickening capacity.

#### 2.4.2.1 Circular basin thickener

*Typical uses*: Larger scale thickening and deliquoring of solids from relatively dilute suspension.

*Typical particle size and feed concentration range:* 0.1-500 µm and <20% w/w.

A circular thickener comprises a relatively shallow, open top cylindrical tank with either a flat bottom shaped in the form of an inverted cone (see Figure 2.13). The feed mixture is gently and continuously introduced to the feed well in which exists a pool of settling suspension along with any additional coagulant or flocculants. With settling and thickening proceeding, clear liquid (the overflow) is removed via an annular weir at the top of the unit and solids sludge (the underflow) is removed from a 'well' at the bottom.

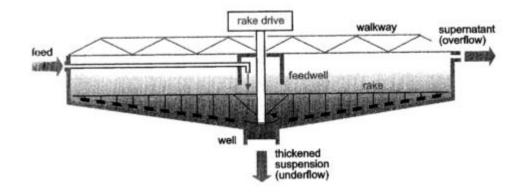


Figure 2.13 Circular Basin Thickener Showing Rakes, Drive, Head and Walkway

Slowly rotating arms (or rakes) mounted on a central drive head aid the thickening process by directing thickened solids towards the well for subsequent discharge, and by creating channels for release of further liquid from the sludge. The construction and form of the rake are important design parameters as is the rating of the motor in the central drive head which must be capable of moving the rake through the thickened solids both during normal operation and during start-up of the rake after stoppage.

Tanks with diameter smaller than 25 m are usually form from steel and have flat bottoms with rake arms at an angle less than 10°.

Continuous discharge of solids from a gravity settling tank can be achieved without mechanical aid is the tank is shaped so that the sludge is flows naturally towards the discharge port. This required steep-sided conical vessels; the angle of the cone generally 40° and 60°, and thus the diameter of a settling tank is invariably rather less than a thickener. A diaphragm baffle is located near the base to prevent arching of solids across the outlet port.

## 2.4.2.2 High capacity thickener

*Typical uses* : Separation of rapid settling solids where available space is at premium.

High capacity thickeners work on a broadly similar principle to conventional thickeners. By high correct use of high molecular weight, fast-acting flocculants, however, large flocs that sediment very quickly can be generated to allow thickeners with high solids handling capacities and relatively small floor areas. Although high capacity thickeners have found many used, particularly when significant amounts of fines are present, they do not represent a replacement for conventional thickeners as flocculants usage is notably higher and thus more costly.

Typical particle size and feed concentration range: 0.1-300  $\mu m$  and <15% w/w.

The circular class of high capacity thickener is similar in general form to a conventional circular basin thickener, but cylinder diameters are limited to between 4 and 18 m (see Figure 2.14). Units are constructed from steel and include a cylindrical top portion, an inverted cone bottom section and an angled rake system mounted a central drive head. Suspension throughputs are limited to about 4000 m<sup>3</sup> h<sup>-1</sup>.

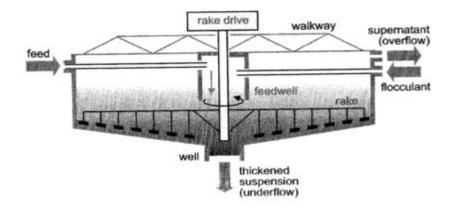


Figure 2.14 Sectional View of the Circular High Rate Thickener

## 2.4.2.2.2 Deep cone

Typical particle size and feed concentration range: 0.1-300  $\mu m$  and < 15 % w/w.

The deep cone thickener shown is broadly similar in form to a conventional thickener, but the sides of the inverted cone have a much steeper angle, in the region of 37°. Units are available with diameters up to 15 m to process suspensions at throughputs ad high as

70 m<sup>3</sup> h<sup>-1</sup>. A paddle or rake system rotating at speeds between 0.25 and 2 rpm is usually added to aid the thickening process and facilitate final sludge concentration up to 70% w/w. Although deep cone thickeners are relatively cheap to install and occupy a small floor area, the operating cost can be higher. The flocculants required to promote efficient settling and operation are generally expensive, and high power input maybe needed to maintain the stirring action of the paddle through pastes that are characteristic of high viscosity underflows.

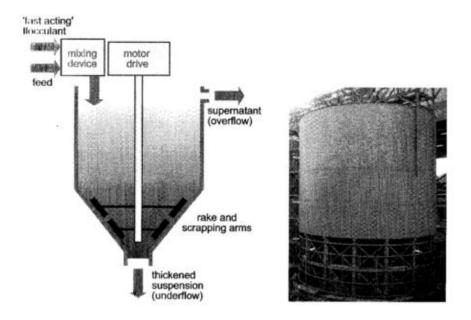


Figure 2.15 Schematic Representation of a Deep Cone Thickener and Inset Photograph (Door-Oliver Eimco)

## 2.4.2.3 Clarifiers

*Typical uses*: recovery of clear liquor from dilute suspension. *Typical particle size and feed concentration range*: 1-50 µm and < 15 % w/w.

The primary duty of clarifier is to remove relatively small amounts of solid from more dilute suspension to yield a clarified liquid overflow and a generally unwanted solids sludge underflow. The basic single-pass unit shown schematically in Figure 2.9. During normal continuous operation the feed moves in a horizontal direction through the flocculator towards the settlement basin at throughput of  $1.5-3 \text{ m}^3\text{h}^{-1}$ . While in the basin the solids undergo almost unhindered settling to the bottom of the basin and clarified liquids discharged over a weir.

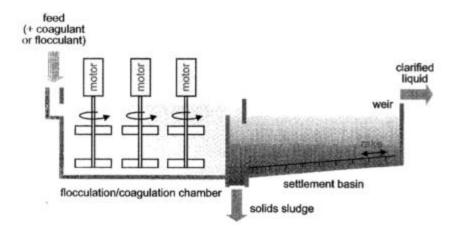
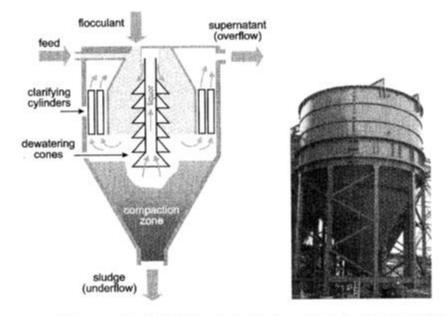


Figure 2.16 Schematic of a Single-Pass Rectangular Basin Clarifier

To accelerate flocculation and help removal of excessive fines, recirculation of some sediment solids to the incoming feed is sometimes used. The recirculation may be either internal or external to the clarifier and often results in higher throughput, albeit at the expense of greater mechanical complexity. Example of internal recirculation shown in Figure 2.17. The rake system commonly found in other thickeners and clarifier is unnecessary.

# Figure 2.17



Schematic of an E-Cat high-rate clarifier (sometimes referred to as a thickener clarifier) showing the internal circulation of liquors. Inset photograph with permission from Dorr-Oliver Eimco.

The potential benefits of clarifiers that incorporate recirculation are offset by the short residence time of liquid and strictly limited solids storage capacity. Both of these require a unit to be carefully controlled in order to prevent an overload due to an excess of incoming material.



Figure 2.18 Clarifier Tank (O.P.C., Cameroon)



Figure 2.19 Clarifier Tank (Nova Technologies Ltd., Nigeria)

## 2.4.3 Dilution of crude oil

In pilot plant, clarification was carried out in settling 220 liters capacity. The conclusions drawn from experiments carried out by N Ravi Menon is summarized below.

## (a) Capacity.

The 150 working volume of 220 litres capacity settling tank can cope with 500kg of dilutes crude oil per hour (100% dilution with water). The crude oil settles for approximately 18 min.

## (b) Separation of NOS

Almost total separation is achieved since only 1.5% of the impurities find their way into the oil. The average moisture and dirt content of the continuously settled oil is 1.3% and 0.06% respectively. This would not be suitable in an industrial process.

## (c) Oil recovery.

Approximately 60% of the oil is recovered.

## (d) Interface.

At times the interfacial layer tends to increase and occupy the space intended for clean oil (density close to that of oil). At that point, the crude oil feed is reduced slightly after a few minutes, enough oil has separated to enable continuous settling to take place again. The hourly capacity of 500kg allows for the slowing down of the feed.

#### (e) Removal of sludge.

The output is steady. The composition of sludge is constant. Oil content ranges from 11 % to 13% and NOS from 4% to 6.5% (12 analyses).

## 2.4.3.1 Clarification of Wet Process Crude Oil

A lot of difficulties were experienced, probably as a result of the high colloid content of crude oil. The efficiency does not exceed 65% to 70%.

## 2.4.3.2 Clarification of Centrifuge Crude Oil.

The process offers no difficulties. A single static separation leads to an efficiency of 94% to 95%. This may be increased to 96.5% level by second separation. There seems to be no advantages in carrying out further separation.

The very fast settling of centrifuge or discontinuous press crude oil is a consequence of the low content in dirt, colloids and other solids suspension. Among the latter, fruit pectin deserve special attention.

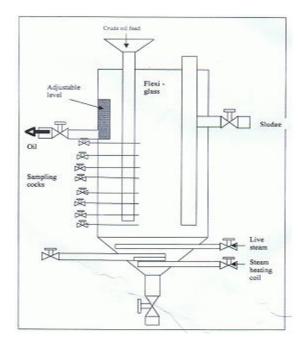


Figure 2.20 Experimental Continuous Settling Tank

# 2.4.3.3 Factor Affecting Settling

Many factor that had been observed can affecting settling such as by screening we can improves the settling of crude oil because of the coalescence of globules. By subjecting crude oil to high frequency vibrations also may increase to settling ability.

Boiling also important in improving settling of crude oil. The crude oil must be ensured that it is actually boils if we used steam injection. Some emulsions break only at boiling temperature and remain stable up to 90°C.

#### 2.4.4 Clarification Tank Simulation

The clarifier is an industrial plant in which the concentration of a suspension is increased purely by sedimentation. Clarifier function is to produce the formation of a clear liquid. In palm oil mills, the clarification tanks serve the purpose of separating from the sludge, the free oil and the coarse oil droplets, which rise to the surface fairly quickly. The most important task in designing a sedimentation process is recognizing the form of settling taking place in the suspension. Dilute sedimentation is unpractical in the mills due to the large settling area needed. When the concentration of the particles become so high or the settling area is inadequate such that the particles no longer settle as individuals. The settling behavior also changes with increasing concentration and rapidly going through a transition stage. Transition stages occur, time for leading to hinder settling is reaching. Shearing force has to be introduced into the crude oil to reduce the apparent viscosity with minimum turbulence.

Consolidation (compression deliquoring phase) and compression occurs to some extent as solids reach the base of the vessel and the layers of particles build up. We can see the underlying one become pack more closely together then achieving a higher concentration. The presence of an inclined surface in a sedimentation system has the effect of increasing the settling rate of particulates. Thus, the bottom of the clarification tank should be conical in shape. Tank height will increase with increased retention time, while the settling surface area needed is proportional to the feed rate. The length of feeding pipe is determined based on the velocity of the rise of 30µm oil droplets. Generally, the clarification of 1:1diluted crude palm oil at 85°C - 90°C with a stirring rate of 5rpm 3hr of retention time.

#### **2.4.4.1 Dilute sedimentation**

The general equation of motion of a small particle in a liquid can be obtained by applying Newton's second law of motion to the physical situation. In gravity sedimentation, the dominant force is due to the gravitational acceleration acting on the buoyant mass of particle (*Rushton et al., 1996*). An oil droplet of volume v in the sludge undergoes a vertical upward force corresponding to where is the density of the sludge. The weight of the droplet itself is vpg where  $\rho$  is the density of the oil. Thus, the force pressing the droplet upward, *F*, is the difference between the vertical pressure and the weight. If the droplet is assumed to be spherical having a diameter *x*, then

$$F = [(\pi x^3)/6](\rho - \rho_s)g \tag{1}$$

In dilute sedimentation, the Reynolds numbers of the particles are usually low. Stokes solved the Navier-Stokes equation, which describes the behaviour af an infinitesimal element of an incompressible fluid experiencing only gravity as the body force, by assuming the inertia term to be neglible and produced the relation of the drag force, *D*, on the particle as:

$$D=3\ \mu x u \pi \tag{2}$$

Where  $\mu$  is the dynamic viscosity of the sludge and u is the settling velocity of the oil droplet. After the initial acceleration period, which is usually short, the terminal settling velocity,  $u_t$ , is obtained from the equation below:

$$ut = x^2 (\rho - \rho s) / (18\mu)$$
 (3)

Relative settling speeds for various extraction methods has been studied and shown in Table 2.5 (*Maycock*, 1987).

Method of extraction	Relative speed of settling ms <sup>-1</sup>
Centrifugal	8.5
Hydraulic press	7.5
Screw press	0.5
Wet process	2.0

 Table 2.5 Relative Settling Speed For Various Extraction Methods

The design method for dilute sedimentation is based on the principle of providing enough residence time for the separation to take place. The process can be described most simply by the Camp-Hazen ideal continuous sedimentation tank model, which equates the residence time derived from the vertical settling motion of the particle and the horizontal plug flow movement brought about by the drag of the liquid. Thus, for residence time, *t*, the vertical equation is:

$$t = H/u_t \tag{4}$$

and the horizon equation is:

$$t = A H/Q \tag{5}$$

where *H* is tank depth , *A* its area, *Q* the volumetric flow rate of liquid through the tank and  $u_t$  is the terminal velocity of the particle. By solving the simultaneous Equation (4) and (5), yield:

$$A = Q/u_t \tag{6}$$

For a 30 t per hour palm oil mill, the oil extraction rate (OER) is assumed as 0.2 and dilution ratio of 1:1 to oil with crude palm oil (CPO) content of 80.15% oil, 13.55% moisture and 6.3 % solids. The mass flow rate feed to the clarification tank is 13.49 t per hour. CPO mixture is assumed to have same density as water, and from Table 2.5 for

screw press, the relative speed of settling,  $u_t$  is 0.5 m per hour. From Equation (7) above, the settling area need is

$$A = 13.49/0.5 = 26.98 \text{ m}^2$$

and the diameter of the clarification tank, D can be determined as shown:

$$\pi D2/4 = 26.98$$
  
 $D = 5.8611$  m

As the tank diameter is too big, the dilute sedimentation is an unpractical approach for the mills.

#### 2.4.5 Separation by centrifugal sedimentation.

Centrifugal sedimentation is based on a density difference between solids and liquids, the particles are subjected to centrifugal forces which make them move radials through the liquid either outwards or inwards, depends on whether they are heavier or lighter than the liquid. Centrifugation may thus be regarded as an extension of gravity sedimentation to finer particles sizes and it also separate emulsions of gravity sedimentation to finer particles sizes and it can also separate emulsions which are normally stable in the gravity field.

Substances having different densities will separate by gravity. Oil is lighter than water and easily forms a separate upper layer. The effect of gravity noticeable by centrifugation. For example by rotating the mixture to be separated in a container. Centrifugation is often used in oil mills and refineries to separate impurities and water from oils and fats and also fat crystals from fat slurry after addition of a surface-active agent

## 2.4.5.1 Factor Affecting Centrifugal Separation

The major physical properties of feed material affecting centrifugal separation are as follow:

- particle size and shape
- differential density between solid particle and he liquids
- concentration of particles
- liquid viscosity

Sludge solids in suspension are combination of granular and fibrous particles. By classic theory, granular and fibrous particle are considered to settle terminal velocity, independent of one another, without changing in shape and size. Like gravity settling, centrifugal sedimentation or separation is dependent on the differential density between the particle and liquid; the greater the differential, the faster the rate of settling.

Particle distribution or concentration is interrelated with particle size and shape, and more generally effect the concentration of the sediment or centrifugal cake to an appreciable extent. The nature and compressibility of the solids also substantially affect the concentration of the cake.

## 2.4.5.2 Machine Affecting Centrifugation

Four main machine variables affecting centrifugation are:

- bowl design
- bowl speed
- pool volume
- relative conveyer speed

#### (a) Bowl design

Increasing the bowl length is one way to improve clarification in a bowl of fixed diameter in a bowl of fixed diameter as this increases settling area and retention time without affecting the centrifugal force. However, mechanical design parameter limit the L/D ratio of the bowl to prevent excessive bowl and gearbox stresses. Most of decanters have bowl L/D ratio of 2:5 to 3:5. A shallow conical angle is preferred to handle soft solids from the palm oil sludge in order to reduce the slippage force responsible for bringing back the solids into the pool.

#### (b) Bowl speed

Centrifuge normally operates in the range of 500-3000 times the force of gravity. When the bowl speed is increased, an improved capture of solid generally results as more fines are trapped and compacted into the sludge cake. The cake solids will become drier or wetter at high bowl speeds depends entirely on the nature of the fine particles in the sludge being dewatered. In general, as the sludge speed is increased, moisture content will increase in sludge that contain significant levels of fines and moisture content will decrease in those that have a heavier and coarser makeup.

When the speed of the bowl is raised past its optimum value, slippage forces on the dewatering beach increasingly prevent the lighter and more poorly compacted sludge solids from being discharged with the sludge cake. These low density solids are discharged with the centrate, reducing solid capture and lowering centrate quality.

## (c) Relative conveyer speed.

The differential speed between the bowl and the scroll is responsible for transporting cake solids out of the centrifuge. By raising the differential speeds, the sludge residence time is lowered and the clarification volume is increased.

These changes result in the sludge cake being wetter and the centrifuge being able to handle a greater level of solid throughput.

#### (d) Pool volume.

Making the pool deeper in the decanter increases the liquid residence time and improves the clarification process and capture of solid particles. In addition, a deeper pool produces a wetter sludge cake by shortening the available beach surface area and decreasing the time for drainage.

Too shallow of pool may cause high linear velocities to be active near the interface of the liquid and solid layers. This turbulence acts to re suspend the non settling fines captured in the compact solids, causing them to be discharge with the centrate. A shallow pool will expose most of the drainage deck area increase dewatering time on the beach results in the discharge cake becoming drier.

#### 2.4.5.3 Process Variables Affecting Centrifugation

#### (a) Sludge/crude oil feed rate.

Crude oil feed rate is the parameter that most influence in liquid solid retention time. Increasing feed rate to decanter or increasing of sludge throughput may result decreasing liquid phase quality and improving cake solid concentration. At high feed rate, residence time of liquid in the bowl is decreasing resulting only coarse solids to be discharged as cake while fines solids become lost to the concentrate. With less throughput, thinner cake is form and make it can be drained more easily. Too low feed rate resulting very dry cake solid but the weakness is it may cause increasing in wear and abrasion on convey or scroll.

## (b) Sludge/crude oil temperature.

Increase the temperature will result decreasing in sludge/crude oil viscosity and faster the settling rate.

# (c) Solid characteristic

Solid characteristic may affect the clarification and dryness of cake. The more discrete, fibrous and granular particles, more easy the solid to dewater. Sludge that having small particle size and low density are more difficult to dewater because increasing in surface area.

## **CHAPTER 3**

# METHODOLOGY

## 3.1 Material

The substances used in this research are crude palm oil in palm mill diluted with 50% water and hexane as separation agent.

# 3.2 Apparatus/Equipment

Equipment used in this research is water bath shaker, oil bath, analytical balance, Mermet oven, clarifier tank beside others apparatus such as 100ml measuring cylinders, test tube beaker 50 ml, beaker 100 ml, beaker 200ml, clamp and stopwatch. Description of equipments use as follow:

• Crude tank oil - 8ft height x 16ft long x 8ft width.

- Adjustable tank temperature supply by steam coil.

- Clarifier tank 4.5m in diameter.
  - 10.5m in height.
  - Built in with nonadjustable speed stainless steel agitator from 5rpm to 10rpm.

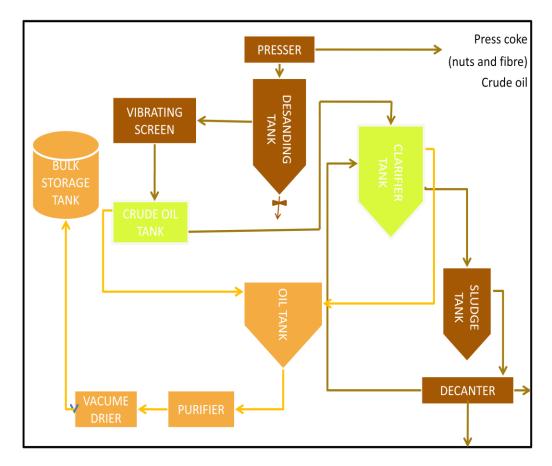


Figure 3.1 Clarifications and Purification Process Area

Notes: \_\_\_\_ Indicates clear oil.

→ Indicates crude palm oil.



Figure 3.2 Shaking Water Batch



Figure 3.3 Oil Batch



Figure 3.4 Clarifier Tank for Experiment Effect of Impeller to CPO Separation

## **3.3** Experimental Works

The related experimental works involved in order to achieve maximum oil production from the crude palm oil are divided into four major sections:

- i. Sample Preparation (crude palm oil heated in water batch shaker and oil batch)
- Composition of oil(x), sludge and non oily solid measurement for difference dilution factor(%).
- iii. CPO separation time(t) measurement using difference temperature.
- iv. CPO separation time(t) measurement using difference beaker size.
- v. Determination of oil composition in underflow of clarifier tank.

## **3.4 Sample Preparation**

The crude palm oil sample is taken from Kilang Sawit LKPP Corporation Lepar before it is being diluted with hot water in crude oil tank.

#### **3.5** Experiment 1: Crude palm oil heating to significant temperature.

To take cognizance of the significant temperature effects on the separation process in crude oil tank, it is useful to make a comparison of process operated at shaking water batch used temperatures of 50°C, 60°C, 70°C, 80°C, 90°C and using oil batch at 100°C, 110 and 120°C. By using 50% of factor dilution, sample is placed in a beaker and is heated for every temperature difference. The next step is observation of CPO separation at each temperature used and the time for complete separation is taken. After getting the best range of CPO separation which takes the lowest time, the separation will be operated within the best range to get the optimum temperature. The process is repeated till 3 times for every temperature and the mean is calculated. Graph time separation versus temperature is plotted. Figure 3.4 below indicates the flow diagram of the method used.

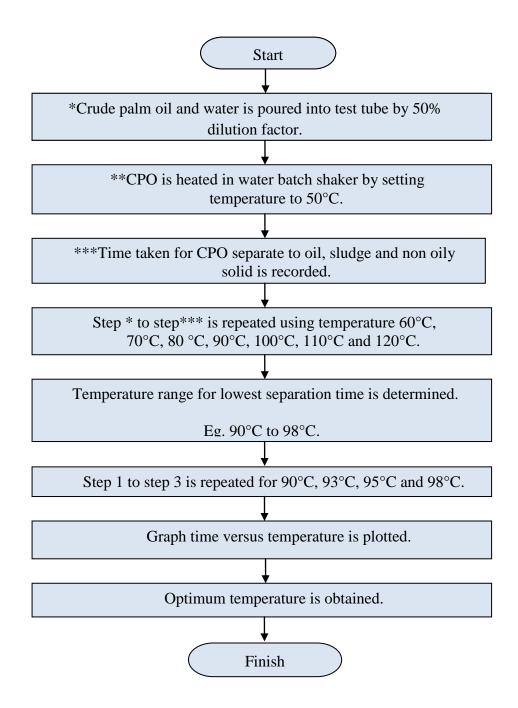


Figure 3.5 Flow Diagram of Experiment 1

## **3.6** Experiment 2: Crude palm oil heating to significant factor dilution.

The crude palm oil is heated at 95°C in water shaking batch using difference factor dilution from 10%, 20%, 30%, 40%, 50%, 60%, 70%, 80% and 90% as shown in Table 3.1. If using 10% factor dilution, crude palm oil will be diluted with 10% of water in 90% CPO. The total of mixture CPO and water is 40ml using a constant beaker size. Then, composition of oil and time for separation for every factor dilution is taken and the experiment will be repeated three times to take the mean. The process is repeated till 3 times for every temperature and the mean is calculated. Table oil recovery for every factor dilution and graph time separation versus factor dilution is plotted. Figure 3.5 below indicates the flow diagram of the method used.

	Composition (%)			
Beaker	Crude Palm Oil (mL)	Water(mL)		
1	10% (36.0mL)	90% (4.0mL)		
2	20% (32.0mL)	80% (8.0mL)		
3	30% (28.0mL)	70% (12.0mL)		
4	40% (24.0mL)	60% (16.0mL)		
5	50% (20.0mL)	50% (20.0mL)		
6	60% (16.0mL)	40% (24.0mL)		
7	70% (12.0mL)	30% (28.0mL)		
8	80% (8.0mL)	20% (32.0mL)		
9	90% (4.0mL)	10% (36.0mL)		

Table 3.1 : Sample of Crude Palm Oil with Difference Dilution Factor

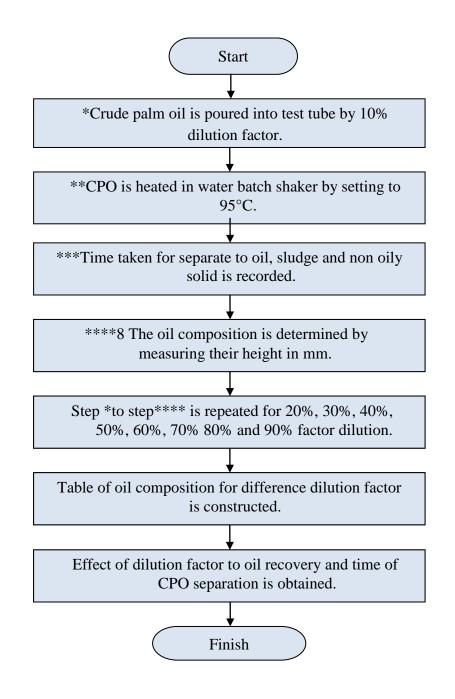


Figure 3.6 Flow Diagram of Experiment 2

#### 3.7 Experiment 3: Crude palm oil heating to significant beaker size.

The constant crude palm oil is heated at 95°C in shaking water batch using 50% factor dilution in difference beaker size . The beaker used is test tube, beaker 50 ml, beaker 100 ml, beaker 200ml. Time for CPO separation for every beaker is taken using stopwatch and the graph time versus size beaker is plotted. Figure 3.6 below indicates the flow diagram of the method used.

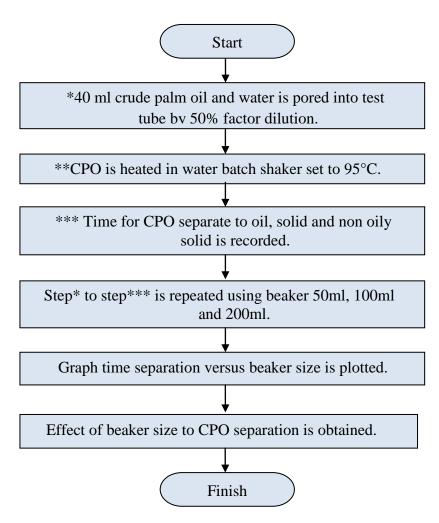


Figure 3.7 Flow Diagram of Experiment 3

## **3.8** Experiment 4: Crude palm oil heating to significant volume.

The crude palm oil is heated at 95°C in shaking water batch using 50% factor dilution in difference volume of CPO. The beaker used is test tube, beaker 50 ml, beaker 100 ml, beaker 200ml. Time for CPO separation for every beaker is taken using stopwatch and the graph time versus volume is plotted. Figure 3.7 below indicates the flow diagram of the method used.

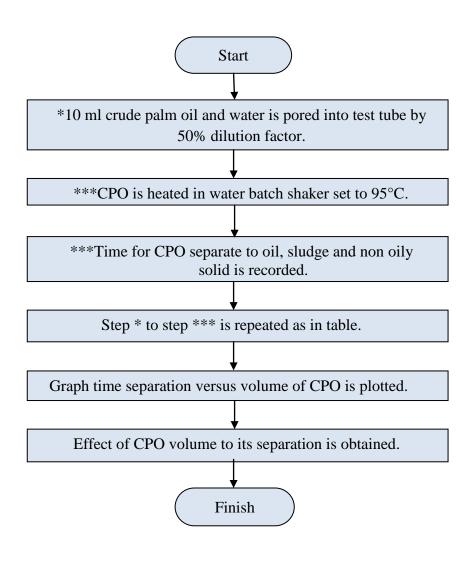


Figure 3.8 Flow Diagram of Experiment 4

## **3.9** Experiment 5: Effect of Impeller in Clarifier Tank.

Figure 3.8 below indicates the flow diagram of the method used.

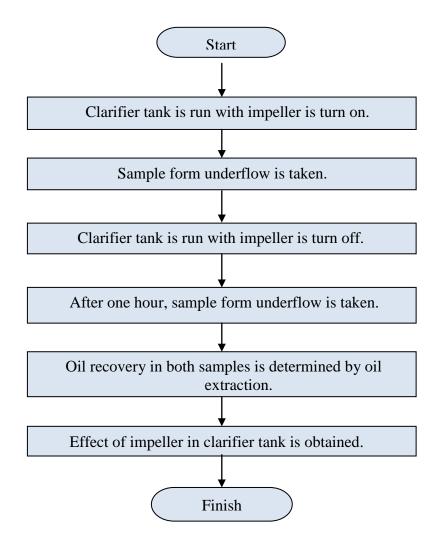


Figure 3.9 Flow Diagram of Experiment 5

In this experimental, two sample is used by using crude palm oil which is collected from underflow of vertical clarifier tank. First sample is taken while impeller in clarifier tank is on. Second sample is taken after one hour impeller in clarifier tank is turn off. Then oil both of the sample is being extracted to determine the oil composition. Below shown how oil extraction procedures.

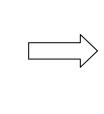
### 3.9.1 Oil Extraction

Both samples are put in dish and weight of sample is measured using analytical balance. The sample is being dried in Mermet oven about 24 hours to eliminate all the moisture and water in sample. Then the sample is put in dessicator to cool the sample to room temperature and eliminate the moisture by silica gel in the dessicator. After cool, again the sample is weight to determine the weight of sample after dried. The next step, the sample from dish is collected using spatula and cotton. It is very important to make sure no sample residue left in the dish and all will be weight. Both sample and cotton is put in difference pericarp and they are place in extractor glass. Hexane is pour into round bottom flask and placed on heating mantel. Water tap through condenser and extractor power is on. Oil is absorb to hexane and it make the hexane color turn to yellowish. Extraction process is continued till no yellowish hexane color in Liebeg Condenser. Pure hexane is removed from the Liebeg Condenser when the hexane rise the maximum level that mark on Liebeg. The extraction process is over when no hexane left in Liebeg condenser. The oil recovery that collected in round bottom flask is weight and oil weight is determined by get the difference empty round bottom flask to round bottom flask containing oil. Table of oil recovery calculation is build to determined difference of oil recovery in underflow clarifier tank before and after impeller turn off. Figure 3.9 below indicates the flow diagram of the method used.



1. Sample

placed in dish and be weight.





2. Sample drying.



4. Dried sample collection.

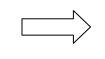




3. Sample cooling in dessicator.



5. Dried sample placed in pericarp to place in Liebeg Condenser





6. Sample extraction

Figure 3.10 Flow Diagram of Oil Extraction



Figure 3.11 Hexane as extraction medium



Figure 3.12 Round bottom flask place in heating mantel containing hexane at first and after extraction finish, only oil left here.



Figure 3.13 Yellowish color present by hexane containing oil in Liebeg condenser

#### **CHAPTER 4**

### **RESULTS AND DISCUSSION**

#### 4.1 Crude Palm Oil Heating to Significant Temperature.

Figure 4.1 below show crude palm oil before and after separation by heating at 95°C with 50% dilution factor. After separation oil rise to the top, sludge at the center and some in the bottom.



Figure 4.1 Crude Palm Oil Before (left) and after Separation by Heating

Figure 4.2 show oil composition after being separate using centrifugal machine. The sample taken from crude oil tank in factory heated at 95°C using 30% to 50% dilution factor. From the figure we can observe oil composition at the top follow by emulsion, water and sludge.

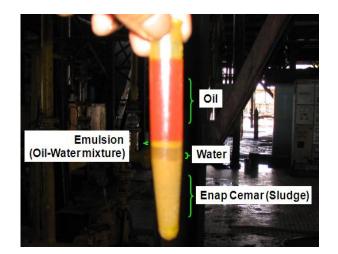


Figure 4.2 Oil Compositions after Being Separate Using Centrifugal Machine

Figure 4.3 below shown graph crude palm oil separation time with difference temperature. Using temperature 50°C, CPO take longest time which is 30.79 minutes to separate 40ml CPO dilute with 50% water. At this temperature energy in warm water give slow non oily solid movement to separate from oil. Time separation decreasing extremely using 50°C to 70°C cause as temperature decreasing the viscosity of CPO also decreasing make it easier to separate to oil and sludge. The force separating oil and sludge is the difference in the specific gravity between the two elements.

The separation time keep decreasing till the temperature used is 90°C. The crude oil also must be ensured that it is actually boils because some emulsions break only at boiling temperature and remain stable up to 90°C. Boiling is very important to improve the settling of crude oil. But by setting the water shaking batch to 100°C for heating, the separation time show an increasing in separation time. It happened because water used in dilution start boiling at this temperature. The effect from water boil is air bubble from water is rise to the top and too high colloid occurred between content of crude oil. Oil that starting to separate from sludge and oil remix because of the colloidal resulting separation failed after CPO separate at minutes 7.09. By using 110°C for heating,

although the time for separation decreasing to 5.89 minutes from 7.094 minutes using 110°C, separation is failed because at minutes 7.52 the water and air bubbles that already separate from oil rise to the top. Using temperature 120°C for heating, separation is failed cause at minutes 4.08 oil and waters both boil and no clear separation composition oil, water and non oily solid obtained.

	Separation Time (min)							
Temperature								
(°C)	Trial 1	Trial 2	Trial 3	Average				
50	31.95	30.53333	29.88333	30.78889				
60	19.05	22.13333	19.95	20.37778				
70	8.066667	8.2	7.9	8.055556				
80	6.766667	6.45	7.316667	6.844444				
90	4.766667	5.216667	3.3	4.427778				
100	6.683333	7.1	7.5	7.094444				
110	4.533333	4.533333 6.583333 5.65 5.588889						

 Table 4.1 Time Separation at Difference Temperature.

Figure 4.3 below show average CPO separation time by using difference temperature for heating.

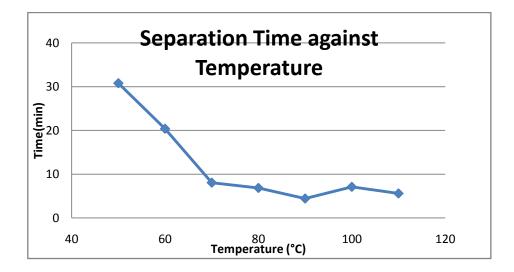


Figure 4.3 Graph Time Taken for Separation in Difference Temperature

From figure 4.4 above, we can observe at 90°C, the separation time become lower. To determine optimum time, the temperature range 90°C, 93°C, 95°C and 98°C used for separation and the result shown in Table 4.2. From the experiment by using 95°C the separation time lowest and we can conclude that optimum time for CPO separation to oil, water and non oily solid is about 95°C.

	Separation Time (min)						
Temperature							
(°C)	Trial 1	Trial 2	Trial 3	Average			
90	4.766667	5.216667	3.3	4.427778			
93	5.633333	6.4	4.016667	5.35			
95	3.4	4.433333	4.233333	4.022222			
98	6.566667	5.266667	6.016667	5.95			
100	6.683333         7.1         7.5         7.094444						

 Table 4.2 Time Separation at Difference Temperature.

Figure 4.4 shows separation time using temperature 90°C, 93°C, 95°C, 98°C and 100°C as value from average time in table 4.2.

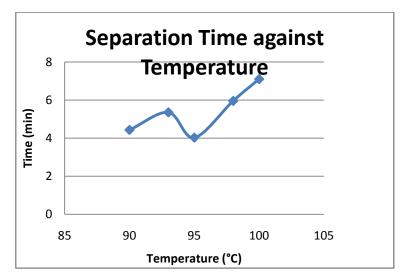


Figure 4.4 Graph Time Taken for Separation in Difference Temperature

#### 4.2 Crude Palm Oil Heating to Significant Dilution Factor.

Figure 4.5 below showing crude palm oil after separation used 10%, 20%, 30%,40%, 50%, 60%,70%, 80% and 90% dilution factor.

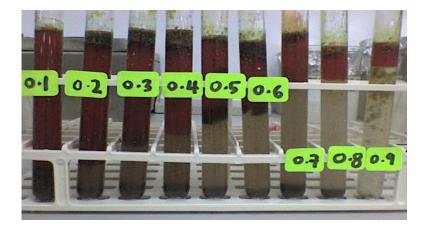


Figure 4.5 Crude Palm Oil after Separation for Difference Dilution Factor

Table 4.3 below show oil recovery when after separation CPO at 95°C in various dilution factor from 10% to 90%. At 10% dilution factor, 90 percent CPO will dilute with 10 percent water producing 82.1 percent oil. The other component from CPO is non oily solid come from empty fruit bunch and fruit let fibers and debris. For the following factor dilution, oil recovery decreasing when we use larger dilution factor. In production of oil in palm mill, dilution factor in range 30 percent to 50 percent should be used because some reason. The oil recovery is 40 to 50 percent and its give balance water and CPO consumption as shown in Table 4.3. The water used also will be reduce if the oil content in empty fruit bunch is increase cause of rainy day. If using too low dilution factor, which is about 10 percent to 20 percent, there is no clear and enough distance between separated clear oil water and non oily solid to remix with oil is high. Water boil also too less for enhance CPO separation and bring non oily solid to bottom. By using high dilution factor from 70 percent to 95 percent, it will result low oil recovery. For example, using 90 percent factor dilution, 10 percent of CPO dilute in 90 percent hot

water but only 4.1 percent of oil is recover. The consumption of water is too high while the oil recovery is too low. The cost will increase as need more hot water and energy use to raise up the water temperature to 95°C. This is not effective to oil palm mill as their objective is to maximum oil production and save cost.

	Oil Recovery (100%)				
Dilution Factor					
(%)	Trial 1	Trial 2	Trial 3	Average	
10	0.804124	0.851064	0.808081	0.821089	
20	0.691489	0.677083	0.647059	0.671877	
30	0.552083	0.56701	0.326603	0.481899	
40	0.479167	0.49	0.484211	0.484459	
50	0.414894	0.419355	0.395833	0.410027	
60	0.326087	0.262295	0.218182	0.268855	
70	0.072917	0.393939	0.202247	0.223034	
80	0.006211	0.181818	0.252747	0.146926	
90	0.002242	0.010727	0.11236	0.041776	

Table 4.3 Oil Recovery at Difference Dilution Factor

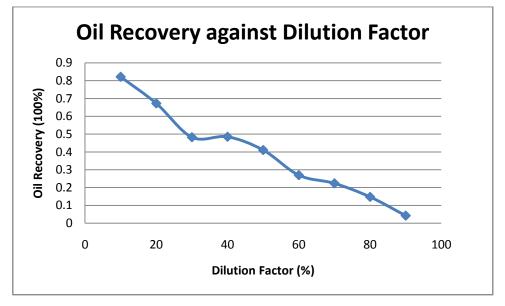


Figure 4.6 Graph Oil Recovery versus Difference Dilution Factor

Table 4.4 below show time taken for CPO separation with difference factor dilution. The separation time in range 7 to 10 minutes is nonlinear as increasing of factor dilution. From this value, we can observe that factor dilution did not give a specific effect in CPO time separation.

	Separation Time (min)			
Dilution Factor				
(100%)	Trial 1	Trial 2	Trial 3	Average
10	7.233333	8.15	7.566667	7.65
20	8.316667	8.033333	9.566667	8.638889
30	8.983333	9.316667	9.483333	9.261111
40	9.316667	7.15	4.9	7.122222
50	9.166667	9.75	10.41667	9.77778
60	8.333333	8.783333	9.716667	8.944444
70	9.5	10	10.38333	9.961111
80	8.233333	7.75	7.983333	7.988889
90	6.716667	7.233333	7.233333	7.061111

Table 4.4 Separation Time at Difference Dilution Factor

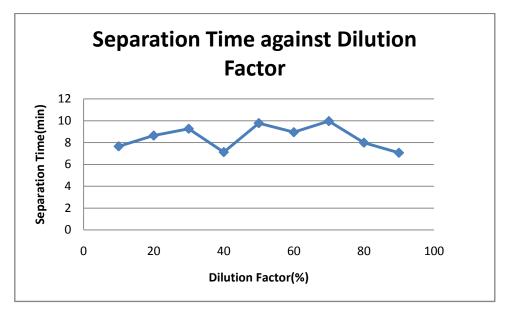


Figure 4.7 Graph Separation Time versus Difference Dilution Factor

#### 4.3 Crude Palm Oil Heating to Significant Beaker Size

Figure 4.6 below show crude palm oil after separation using difference beaker size and heated at 95°C. The volume is constant for every beaker used which is 40ml by using 50% factor dilution. From the figure we can observed the height of same volume of CPO is differ because the of the area. As the area smaller the height is higher.

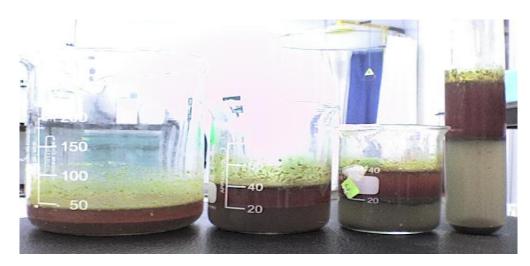


Figure 4.8 Crude Palm Oil after Separation for Difference Beaker Size

The time for separation is decreasing when separation occur in larger surface area as shown in Table 4.5. It happened because heat distribution become faster in larger surface area as the larger surface outer beaker is being heat in water batch shaker. In this experiment, beaker with 7.3 mm diameter and 5.7305 mm<sup>2</sup> surface area is used. To get circular surface area  $\pi$  (D<sup>2</sup>/4) is used. The movement of oil to the top while separate from water and non oily solid also become faster because the colloidal area decreasing using large beaker. By using smaller test tube which having 2.3 mm diameter and 1.8055 mm<sup>2</sup> in this experiment, the height of CPO increase and this may cause oil need longer time to rise to the top. the colloidal and resistance between non oily solid, oil and water increasing as they are compact in smaller surface area and this will resulting longer separation time.

		Separation Time (min)			
Beaker	Beaker				
Diameter	Surface Area				
(mm)	(mm²)	Trial 1	Trial 2	Trial 3	Average
2.3	1.8055	6.566667	7.35	7.45	7.122222
4.3	3.3755	5.15	6.516667	5.25	5.638889
5	3.925	6.066667	5.116667	5.066667	5.416667
7.3	5.7305	4.583333	3.25	4.916667	4.25

**Table 4.5** Separation Time using Difference Beaker Size.

Figure 4.7 below show as the surface area increase, time separation of CPO decreasing.

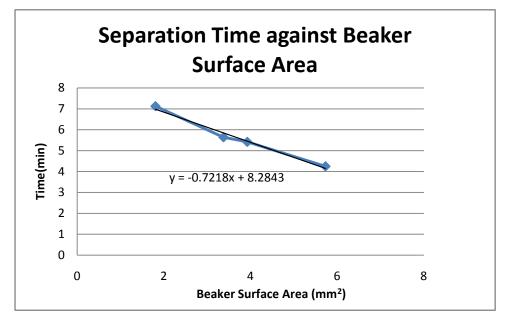


Figure 4.9 Graph Separation Time against Beaker Surface Area

### 4.4 Crude Palm Oil Heating to Significant Surface Area

Figure 4.8 below show crude palm oil after separation for difference volume of CPO in difference beaker size. Difference beaker size used to place difference volume of CPO as larger amount of CPO need larger beaker. The height of CPO is constant which is 300mm. The heating used 95°C hot water in water shaking batch using 50% dilution factor.

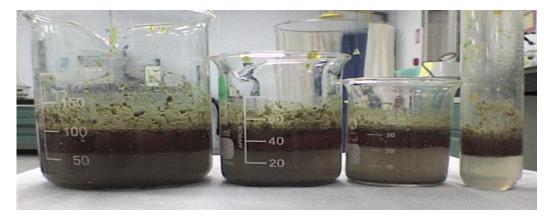


Figure 4.10 Crude Palm Oil after Separation for Difference Volume of CPO in Difference Beaker Size

From this experimental result shown in Table 4.6, 10 mm<sup>3</sup> CPO will separate to oil non oily solid and water in 3.4 minutes. While 36 mm<sup>3</sup>, 52 mm<sup>3</sup> and 94 mm<sup>3</sup> CPO take 7.69, 8.46 and 12.65 minutes respectively. It show larger volume of CPO need longer time to separate as increasing in colloidal while separation. Larger amount of oil contents need to separate themselves from sludge resulting longer time.

	Separation Time (min)					
CPO Volume (mm <sup>3</sup> )	Trial 1 Trial 2 Trial 3 Average					
10	2.4	3.566667	4.35	3.438889		
36	6.633333	8.116667	8.333333	7.694444		
52	7.35	8.016667	10	8.455556		
94	9.883333	13.38333	14.68333	12.65		

 Table 4.6 Separation Time using CPO Volume.

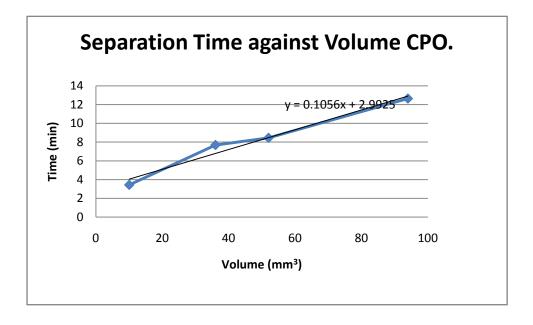


Figure 4.11 Graph Separation Time against Crude Palm Oil Volume

#### 4.5 Effect of Impellers in Clarifier Tank

By using impeller, it will enhance separation of crude palm oil. Table 4.7 below show oil recovery before and after impeller in clarification tank is turn off. From the value oil content in underflow clarification tank before impeller turn off is only 5.59% while after impeller turn off the oil content is increase to 14.14%. It shows that without impeller, oil take longer time to separate from sludge thus take longer time to rise to the top. It has also been experimentally shown that the use of shearers in the clarification tank tends to give cleaner pure oil and lower oil content in sludge.

Weight in Gram	Vertical clarifier (Before	Vertical clarifier (After
	impeller turn off)	impeller turn off)
Weight of Sample	9.9741	5.6362
Weight of Moisture Loss	8.4725	4.7583
Weight of Oil	0.5576	0.7973
Moisture	84.94 %	84.42 %
N.O.S	15.06 %	15.58 %
Oil Content	5.59%	14.14 %
Dry Matter	37.12 %	90.8 %

**Table 4.7** Table Oil Recovery at Underflow Clarifier Tank

Below show formula used to calculate value in Table 4.7:

Weight of oil = Weight of flask and oil – weight of flask

$$Moisture = \frac{Weight of moisture lost}{Weight of sample} X 100\%$$

N.O.S = 100 - Moisture

$$Oil Content = \frac{Weight of oil}{Weight of sample}$$

Dry Matter =  $\frac{\text{Weight of oil}}{\text{N.O.S}}$ 

Figure 4.10 below show sludge taken form underflow clarifier tank. Sludge in underflow supposed to have low oil content compare to sludge because most oil is rise to the top, skim and send to pure oil tank. From figure below we can observe that sample taken before impeller is turn off. The color of sludge is darker. Mean most oil separated from non oily solid such as fiber and debris. The non oily solid sediment to the bottom and will be remove once a week via scour valve. From sample taken after impeller turn off (left), the sample color lighter mean sludge mix with oil and water because no shear force given to enhance oil separation.



Figure 4.12 Separation of Sludge After (left) and Before (right) Impeller Turn Off in Vertical Clarifier

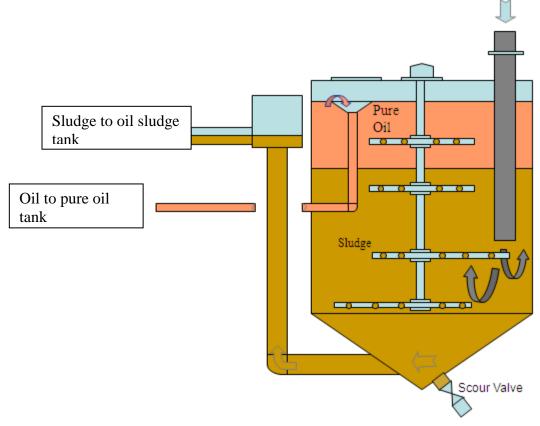


Figure 4.13 Vertical Clarifier

#### **CHAPTER 5**

#### CONCLUSION

#### 5.0 Conclusion

The purpose of this project is to determine optimum temperature optimum residence time and effect of impeller in clarification tank for separation of crude palm oil (CPO) to oil, sludge and non oily solid at clarification process area. This optimum condition used in order to maximize oil production in oil palm mill.

From the obtained result, the optimum temperature is 95°C by using 30% to 50% dilution factor. Water is used in dilution because of oil miscible in water, non chemical substance and safe to human body as palm oil used in food. In CPO separation, as temperature increase the time for separation is decreasing because viscosity and specific gravity of CPO also decreasing. The force separating the two elements is the difference in the specific gravity between the two elements between oil and sludge.

Difference in crude palm oil volume gives effect to residence time. Larger the volume time taken for separation increasing. As the larger tank use, it can place larger capacity of crude palm oil and the time for separation increasing.

Impeller will enhance separation of crude palm oil in clarifier tank by shear force acts by the impeller will produce cleaner pure oil and a lower oil content in sludge in underflow tank.

### 5.1 Recommendation

- (I) To further establish this method of separation, it is necessary to develop advanced analytical method for the qualitative and quantitative analysis of CPO rather than taking the separation time by observation. The development of real-time techniques such as spectroscopy is useful in confirmation of identification CPO separation time should be considered in future.
- (II) Experiment should run in room temperature because crude palm oil easily turn to solid in air conditioner lab. In future study, determination of optimum temperature for CPO separation should be done in real mill size crude oil tank to observe the real separation phenomena with CPO inlet flowrate.
- (III) The need to redesign the clarification would help to increase oil collection. Shape of impeller may give effect to sludge and oil clarification. Difference shape of impeller may give difference efficiency to crude palm oil separation and should be considered in future study.
- (IV) The ideal dilution for press liquor is zero dilution as this produces the least effluent and results in minimum oil losses. In real oil palm mill, the clarifier should be routinely desluged according to procedures established by the mill. All the de-oiling tanks should preferably be of stainless steel. Cement tanks shall not be used as the walls will collapse over time.
- (V) Multiple testing needed to get more accurate and precise reading since the time for separation is taken by manual and observation.

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### **APPENDICES**

## **APPENDIX A**

## Safety Data for Hexane



## **1. Product Identification**

Synonyms: Hexanes,Normal Hexane; Hexyl Hydride; Hexane 95% CAS No.: 110-54-3 (n-hexane) Molecular Weight: 86.18 Chemical Formula: CH3(CH2)4CH3 n-hexane Product Codes: J.T. Baker: 9262, 9304, 9308, N168 Mallinckrodt: 5186

## 2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	Hazardous
Hexane	110-54-3	85 - 100%	Yes
Methylcyclopentane	96-37-7	1 - 2%	Yes
Trace amount of Benzene(10 ppm)	071-43-2	*	No

## 3. Hazards Identification

#### **Emergency Overview**

## DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.

Health Rating: 3 - Severe (Life) Flammability Rating: 3 - Severe (Flammable) Reactivity Rating: 1 - Slight Contact Rating: 2 - Moderate Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES; CLASS B EXTINGUISHER Storage Color Code: Red (Flammable)

#### **Potential Health Effects**

The health hazards addressed are for the major component: n-hexane.

#### Inhalation:

Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Greater exposure may cause muscle weakness, numbness of the extremities, unconsciousness and death.

#### **Ingestion:**

May produce abdominal pain, nausea. Aspiration into lungs can produce severe lung damage and is a medical emergency. Other symptoms expected to parallel inhalation.

#### **Skin Contact:**

May cause redness, irritation, with dryness, cracking.

#### **Eye Contact:**

Vapors may cause irritation. Splashes may cause redness and pain.

#### **Chronic Exposure:**

Repeated or prolonged skin contact may defat the skin and produce irritation and dermatitis. Chronic inhalation may cause peripheral nerve disorders and central nervous system effects.

### **Aggravation of Pre-existing Conditions:**

Persons with pre-existing skin disorders or eye problems or impaired respiratory function may be more susceptible to the effects of the substance. May affect the developing fetus.

## 4. First Aid Measures

## Inhalation:

Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.

### **Ingestion:**

Aspiration hazard. If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Get medical attention immediately.

### **Skin Contact:**

Remove any contaminated clothing. Wipe off excess from skin. Wash skin with soap and water for at least 15 minutes. Get medical attention if irritation develops or persists. **Eve Contact:** 

Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

### Note to Physician:

BEI=2,5-hexadione in urine, sample at end of shift at workweeks end, 5 mg/g creatine. Also, measure n-hexane in expired air. Analgesics may be necessary for pain management, there is no specific antidote. Monitor arterial blood gases in cases of severe aspiration.

## **5. Fire Fighting Measures**

## Fire:

Flash point: -23C (-9F) CC Autoignition temperature: 224C (435F) Flammable limits in air % by volume: lel: 1.2; uel: 7.7

Extremely Flammable Liquid and Vapor! Vapor may cause flash fire. Dangerous fire hazard when exposed to heat or flame.

## **Explosion:**

Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Contact with oxidizing materials may cause extremely violent

combustion.Explodes when mixed @ 28C with dinitrogen tetraoxide. Sensitive to static discharge.

## **Fire Extinguishing Media:**

Dry chemical, foam or carbon dioxide. Water may be ineffective.

## **Special Information:**

In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode. Water spray may be used to keep fire exposed containers cool. Vapors can flow along surfaces to distant ignition source and flash back. Vapor explosion hazard exists indoors, outdoors, or in sewers.

## 6. Accidental Release Measures

Ventilate area of leak or spill. Remove all sources of ignition. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Use non-sparking tools and equipment. Collect liquid in an appropriate container or absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combustible materials, such as saw dust. Do not flush to sewer! If a leak or spill has not ignited, use water spray to disperse the vapors, to protect personnel attempting to stop leak, and to flush spills away from exposures. US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

J. T. Baker SOLUSORB® solvent adsorbent is recommended for spills of this product.

## 7. Handling and Storage

Protect against physical damage. Store in a cool, dry well-ventilated location, away from direct sunlight and any area where the fire hazard may be acute. Store in tightly closed containers (preferably under nitrogen atmosphere). Outside or detached storage is preferred. Inside storage should be in a standard flammable liquids storage room or cabinet. Separate from oxidizing materials. Containers should be bonded and grounded for transfers to avoid static sparks. Storage and use areas should be No Smoking areas. Use non-sparking type tools and equipment. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.

## 8. Exposure Controls/Personal Protection

#### **Airborne Exposure Limits:**

N-Hexane [110-54-3]: -OSHA Permissible Exposure Limit (PEL): 500 ppm (TWA) -ACGIH Threshold Limit Value (TLV): 50 ppm (TWA), Skin other isomers of hexane -ACGIH Threshold Limit Value (TLV): 500 ppm (TWA),1000ppm (STEL) Ventilation System:

A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

#### **Personal Respirators (NIOSH Approved):**

If the exposure limit is exceeded and engineering controls are not feasible, wear a supplied air, full-facepiece respirator, airlined hood, or full-facepiece self-contained breathing apparatus. Breathing air quality must meet the requirements of the OSHA respiratory protection standard (29CFR1910.134).

#### **Skin Protection:**

Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

### **Eye Protection:**

Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

## 9. Physical and Chemical Properties

Appearance: Clear, colorless liquid. Odor: Light odor. Solubility: Insoluble in water. Specific Gravity: 0.66 pH: No information found. % Volatiles by volume @ 21C (70F): 100 Boiling Point: ca. 68C (ca. 154F) Melting Point: ca. -95C (ca. -139F) Vapor Density (Air=1): 3.0 Vapor Pressure (mm Hg): 130 @ 20C (68F) Evaporation Rate (BuAc=1): 9

## 10. Stability and Reactivity

## **Stability:**

Stable under ordinary conditions of use and storage. Heat will contribute to instability.
Hazardous Decomposition Products:
May produce acrid smoke and irritating fumes when heated to decomposition.
Hazardous Polymerization:
Will not occur.
Incompatibilities:
Strong oxidizers.
Conditions to Avoid:
Heat, flames, ignition sources and incompatibles.

### **11.** Toxicological Information

N-Hexane: Oral rat LD50: 28710 mg/kg. Irritation eye rabbit: 10 mg mild. Investigated as a tumorigen, mutagen and reproductive effector.

\Cancer Lists\			
Ingredient	NTP Known	Carcinogen Anticipated	IARC
Category 			
Hexane (110-54-3)	No	No	
None			
Methylcyclopentane (96-37-7)	No	No	
None			
Trace amount of Benzene (10 ppm) (071-43-2)	Yes	No	1

## **12. Ecological Information**

#### **Environmental Fate:**

When released into the soil, this material may biodegrade to a moderate extent. When released into the soil, this material is not expected to leach into groundwater. When released into the soil, this material is expected to quickly evaporate. When released into water, this material may biodegrade to a moderate extent. When released to water, this material is expected to quickly evaporate. When released to water, this material is expected to quickly evaporate. When released to water, this material is expected to quickly evaporate. When released into the water, this material is expected to quickly evaporate. When released into the water, this material is expected to have a half-life between 1 and 10 days. This material has an estimated bioconcentration factor (BCF) of less than 100. This material has a log octanol-water partition coefficient of greater than 3.0. This material is not expected to significantly bioaccumulate. When released into the air, this material is expected to be readily degraded by reaction with photochemically produced hydroxyl radicals. When released into the air, this material is expected to have a half-life between 1 and 10 days.

### **Environmental Toxicity:**

No information found.

## **13. Disposal Considerations**

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved incinerator or disposed in a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal

disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

## **14. Transport Information**

**Domestic (Land, D.O.T.)** 

-----

Proper Shipping Name: HEXANES Hazard Class: 3 UN/NA: UN1208 Packing Group: II Information reported for product/size: 215L

International (Water, I.M.O.)

\_\_\_\_\_

**Proper Shipping Name:** HEXANES **Hazard Class:** 3 **UN/NA:** UN1208 Packing Group: II **Information reported for product/size:** 215L

## **15. Other Information**

NFPA Ratings: Health: 1 Flammability: 3 Reactivity: 0

### Label Hazard Warning:

DANGER! EXTREMELY FLAMMABLE LIQUID AND VAPOR. VAPOR MAY CAUSE FLASH FIRE. HARMFUL OR FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT. AFFECTS THE CENTRAL AND PERIPHERAL NERVOUS SYSTEMS.

### **Label Precautions:**

Keep away from heat, sparks and flame.

Keep container closed.

Use only with adequate ventilation.

Wash thoroughly after handling.

Avoid breathing vapor or mist.

Avoid contact with eyes, skin and clothing.

#### Label First Aid:

Aspiration hazard. If swallowed, vomiting may occur spontaneously, but DO NOT INDUCE. If vomiting occurs, keep head below hips to prevent aspiration into lungs. Never give anything by mouth to an unconscious person. Call a physician immediately. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes. In all cases call a physician.

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### **APPENDIX B**

## Safety Data for Palm oil

#### 1.General

Chemical Name: Not available. Chemical Formula: Not available.

#### 1. Hazards Identification

**Potential Acute Health Effects:** Hazardous in case of ingestion. Slightly hazardous in case of eye contact (irritant), of inhalation.

#### **Potential Chronic Health Effects:**

Hazardous in case of ingestion. Slightly hazardous in case of inhalation. CARCINOGENIC EFFECTS: Not available. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available.

#### 2. First Aid Measures

**Eye Contact:** No known effect on eye contact, rinse with water for a few minutes. **Skin Contact:** No known effect on skin contact, rinse with water for a few minutes. **Serious Skin Contact:** Not available.

**Inhalation:** Allow the victim to rest in a well ventilated area. Seek immediate medical attention. **Serious Inhalation:** Not available.

#### Ingestion:

Do not induce vomiting. Loosen tight clothing such as a collar, tie, belt or waistband. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek immediate medical attention. **Serious Ingestion:** Not available.

#### 3. Fire and Explosion Data

Flammability of the Product: May be combustible at high temperature. Auto-Ignition Temperature: 316°C (600.8°F) Flash Points: CLOSED CUP: 162°C (323.6°F). Flammable Limits: Not available. Products of Combustion: Not available. Fire Hazards in Presence of Various Substances: Not available. Explosion Hazards in Presence of Various Substances: Risks of explosion of the product in presence of mechanical impact: Not available. Risks of explosion of the product in presence of static discharge: Not available. Fire Fighting Media and Instructions: SMALL FIRE: Use DRY chemical powder. LARGE FIRE: Use water spray, fog or foam. Do not use water jet. Special Remarks on Fire Hazards: Not available.

## 4. Accidental Release Measures

#### Small Spill:

Use appropriate tools to put the spilled solid in a convenient waste disposal container. Finish cleaning by spreading water on the contaminated surface and dispose according to local and regional authority requirements.

#### Large Spill:

If the product is in its solid form: Use a shovel to put the material into a convenient waste disposal container. If the product is in its liquid form: Absorb with an inert material and put the spilled material in an appropriate waste disposal. Finish cleaning by spreading water on the contaminated surface and allow evacuating through the sanitary system.

## 5. Handling and Storage

#### Precautions:

Keep away from heat. Keep away from sources of ignition. Empty containers pose a fire risk, evaporate the residue under a fume hood. Ground all equipment containing material. Do not breathe dust.

#### Storage:

Keep container dry. Keep in a cool place. Ground all equipment containing material. Keep container tightly closed. Keep in a cool, well-ventilated place. Combustible materials should be stored away from extreme heat and away from strong oxidizing agents.

### 6. Exposure Controls/Personal Protection

#### **Engineering Controls:**

Use process enclosures, local exhaust ventilation, or other engineering controls to keep airborne levels below recommended exposure limits. If user operations generate dust, fume or mist, use ventilation to keep exposure to airborne contaminants below the exposure limit.

Personal Protection: Safety glasses. Lab coat. Personal Protection in Case of a Large Spill:

Splash goggles. Full suit. Boots. Gloves. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product. Exposure Limits: Not available.

#### 7. Physical and Chemical Properties

Physical state and appearance: Solid. Odor: Not available. Taste: Not available. Molecular Weight: Not available. Color: Not available. pH (1% soln/water): Not applicable. Boiling Point: Not available. Melting Point: 35°C (95°F) Critical Temperature: Not available. **Specific Gravity:** 0.952 (Water = 1) Vapor Pressure: Not applicable. Vapor Density: Not available. Volatility: Not available. Odor Threshold: Not available. Water/Oil Dist. Coeff.: Not available. Ionicity (in Water): Not available. Dispersion Properties: Not available. Solubility: Insoluble in cold water.

#### 8. Stability and Reactivity Data

Stability: The product is stable.
Instability Temperature: Not available.
Conditions of Instability: Not available.
Incompatibility with various substances: Not available.
Corrosivity: Non-corrosive in presence of glass.
Special Remarks on Reactivity: Not available.
Special Remarks on Corrosivity: Not available.
Polymerization: No.

#### 9. Toxicological Information

Routes of Entry: Ingestion. Toxicity to Animals: LD50: Not available. LC50: Not available. Chronic Effects on Humans: Not available. Other Toxic Effects on Humans: Hazardous in case of ingestion. Slightly hazardous in case of inhalation. Special Remarks on Toxicity to Animals: Not available. Special Remarks on Chronic Effects on Humans: Not available. Special Remarks on other Toxic Effects on Humans: Not available.

## **APPENDIX C**

## Safety Data for Crude Palm Oil

## 1. GENERAL

TRADE NAME: **CRUDE PALM OIL** CAS NO.: 8002-75-3 CHEMICAL NAME: TRIGLYCERIDE OF FATTY ACIDS CHEMICAL FAMILY: VEGETABLE OIL

## 2. HAZARDOUS COMPONENT

DOES NOT CONTAIN ANY KNOWN HAZARDOUS INGREDIENTS

# 3. PHYSICAL CHARACTERISTICS BOILING POINT N/D VAPOR PRESSURE N/A VAPOR DENSITY (AIR=1) EXCEEDS 1.0 SPECIFIC GRAVITY (H20=1) 0.9 MELTING POINT APPROX 30°C EVAPORATION RATE (BUTYL ACETATE = 1) N/A SOLUBILITY IN WATER INSOLUBLE APPEARANCE REDDISH-YELLOW FAT WEIGHT PER GALLON APPROX 7.6 LBS/GAL

#### 4. FIRE AND EXPLOSION HAZARD DATA

FLASH POINT >300°F (CC) FLAMMABLE LIMITS N/D EXTINGUISHING MEDIA FOAM, DRY CHEMICAL, CO2 FIRE FIGHTING: DO NOT USE WATER - may spread fire by dispersing oil. Water may be used to keep containers cool.

### **5. REACTIVITY DATA**

STABILITY: STABLE CONDITIONS TO AVOID: NONE INCOMPATIBILITY: CAN REACT WITH OXIDIZERS HAZARDOUS POLYMERIZATION: WILL NOT OCCUR HAZARDOUS DECOMPOSITION PRODUCTS: CO, CO2 ON BURNING

## 6. HEALTH HAZARD DATA

TRESHHOLD LIMIT VALUE: Liquid-none; oil mist 10 mg/m<sup>3</sup> total particulate.

EFFECTS OF OVEREXPOSURE: Excessive inhalation of oil mist may affect respiratory system. Oil mist is classified as a nuisance particulate by ACGIH. Sensitive individuals may experience dermatitis after long exposure of oil on skin.

EMERGENCY FIRST AID PROCEDURES: Wash skin with soap and water. Flush eyes with water and seek medical attention if irritation occurs. If ingested in large quantities, contact a physician if discomfort is encountered.

CARCINOGENICITY LISTING: None

#### 7. SAFE HANDLING AND USE

SPILL OR LEAK PROCEDURES: Spills of this material are very slippery. Cover spills with some inert absorbent material and scoop into a container. Wash floors with detergent or soap and hot water and rinse with hot water.

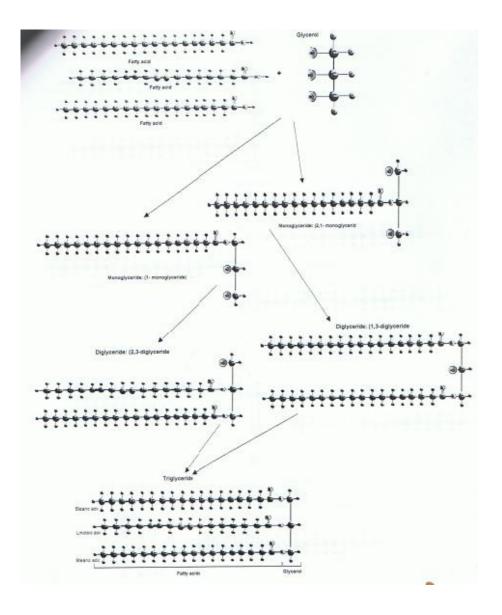
SPECIAL PRECAUTIONS: As with all unsaturated fats and oils, some porous materials such as rags, paper, insulation or clay when wetted with this product may undergo spontaneous combustion. Keep such wetted materials well ventilated to prevent possible heat build-up.

WASTE DISPOSAL: Disposal must be made in accordance with all local, state, and federal regulations.

#### **8. CONTROL MEASURES**

EXPOSURE CONTROL: Engineering controls are usually not necessary if good hygiene practices are strictly followed. Respiratory protection is generally not required during normal operations. Wear the following to prevent skin contact: work pants, long sleeve work shirt, and work gloves. Where there is the danger of eye contact, wear splash-proof goggles.

## APPENDIX D



Appendix D: Synthesis of Triglyceride