Optimization of Bleaching Earth and Extraction of Free Fatty Acid (FFA) in Palm Oil Refinery Process

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Optimization of Bleaching Earth and Extraction of Free Fatty Acid (FFA) in Palm Oil Refinery Process

SITI HAIZAN BT MOHD MUKHTAR AFFANDI

Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering and Natural Resources

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SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering and Natural Resources.

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Position   : SENIOR LECTURER
Date   : JANUARY 2015

STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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Date   : JANUARY 2015

Dedication

      Dedicated to my beloved father, Mohd Mukhtar Affandi Bin Nasir, for his constant encouragement and motivation.

      To my beloved mother, Zaleha Bt Abu Bakar , for her inspiration and to my forever supportive family members Mohd Afiza , Hartini, Mohd Azim and Siti Arbani.

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ABSTRACT

Physical refining has become the major processing route for crude palm oil in Malaysia. Degumming, bleaching and deodorization are essential processes that involves in palm oil refining. The purpose is the removal of gums, trace metals, pigments, peroxides, oxidation products and other breakdown products in the crude oil by adsorption on the active surface of the bleaching earth to improve colour and stability of the final oil (Siew et al., 1992). This research aims to study the Optimization of Bleaching Earth and Extraction of Free Fatty Acid (FFA) in Palm Oil Refinery Process which involves process of degumming and bleaching using neutral and acid-activated clays. Degumming is the pre-treatment stage of physical refining crude palm oil and these stages are identified as the major contributors to the total operating cost of the plant due to the cost of chemicals (phosphoric acid and bleaching earth) that are being used in these processes .Bleaching absorbs the undesirable impurities and other pigments. It also reduces the oxidation products, absorbs the phospholipids precipitated by phosphorus acid, and removes any excess present in the oil. Last stage for physical refining is deodorization. The pre-treated oil is deoerated and then heated to deodorization temperature and pressure. Under these condition , which the free fatty acids, which are still present in the oil are distilled together with the more volatile odoriferous and oxidation products such as aldehydes and ketone, which otherwise will affect the odor and the taste of the oil. In this research, a process model for degumming and bleaching operation will be designed in order to help the refiner's to predict the exact ratio of phosphoric acid and bleaching earth to the crude palm oil. By doing so, we could reduce the operating costs and time of the overall palm oil refining process.

ABSTRACT

Penapisan fizikal telah menjadi laluan pemprosesan utama bagi minyak sawit mentah di Malaysia. Degumming , pelunturan dan penyahbauan adalah proses penting yang melibatkan penapisan minyak sawit. Tujuannya adalah penyingkiran gusi , logam surih, pigmen , peroksida , produk pengoksidaan dan produk kerosakan lain dalam minyak mentah oleh penjerapan di permukaan aktif pelunturan bumi untuk meningkatkan warna dan kestabilan minyak akhir (Siew et al. , 1992). Kajian ini bertujuan untuk mengkaji Pengoptimuman Pelunturan Bumi dan Pengeluaran Asid Lemak Bebas (FFA) dalam Proses Penapisan Minyak Sawit yang melibatkan proses degumming dan pelunturan menggunakan tanah liat neutral dan asid- diaktifkan. Degumming adalah peringkat pra- rawatan penapisan fizikal minyak sawit mentah dan peringkat ini dikenal pasti sebagai penyumbang utama kepada jumlah kos operasi kilang kerana kos bahan kimia (asid fosforik dan pelunturan bumi) yang digunakan dalam proses ini. Pelunturan menyerap kekotoran yang tidak diingini dan pigmen lain. Ia juga mengurangkan produk pengoksidaan, menyerap phospholipid yang dihasilkan oleh asid fosforus, dan memindahkan lebihan bendasing di dalam minyak. Peringkat akhir untuk penapisan fizikal adalah penyahbauan. Minyak pra- dirawat deoerated dan kemudian dipanaskan kepada suhu dan tekanan penyahbauan. Di bawah keadaan ini, asid lemak bebas yang masih ada dalam minyak akan disuling bersama-sama dengan bendasing yang berbau busuk dan pengoksidaan produk lebih tidak menentu seperti aldehid dan keton , yang boleh memberi kesan bau dan rasa pada minyak sekali gus meningkatkan kualiti minyak yang terhasil. Dalam kajian ini, satu model proses degumming dan pelunturan operasi akan direka bentuk untuk membantu penapis untuk meramalkan nisbah yang tepat asid fosforik dan pelunturan bumi untuk minyak sawit mentah. Dengan berbuat demikian, kita boleh mengurangkan kos operasi dan masa proses penapisan minyak sawit secara keseluruhan.

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CHAPTER 1

INTRODUCTION

1.1 Introduction

The background of the research which are include raw materials, process and the product that will be produce in this research. In this chapter, we will include the identification of problems, research objectives, research scopes and rational and significant of the study.

1.2 Background of Study

Palm oil is one of the various types of vegetable oils, belonging to the group called liquids, because of its fatty acids content. Oil palm tree (*Elaeis guineensis Jacq)* is one of the two most important vegetable oils in the world's oil and fats market with one hectare of oil palm producing between 10 and 35 tones of fresh fruit bunch (FFB) per year ([Hartley, C.N.S., 1988;Ma, A.N., Y. Tajima, M. Asahi and J. Hannif, 1996]).The extraction and purification processes generate different kinds of waste generally known as palm oil mill effluent (POME) (Mohd Izwan,2010) and typically,1 t of crude palm oil production requires 5–7.5 t of water; over 50% of which ends up as POME(J.C. Igwe and C.C. Onyegbado,2007) . POME has a high nutrient content (Zakaria et al. 1994), and large oil palm plantations prefer to use it directly as fertilizer. The POME is first treated to reduce the organic load (Ma et al. 1993). In crude palm oil, the amounts of such impurities vary, depending on its quality. As say by siew,1987, phosphorus varies from 10 to 40 ppm of which a portion is inorganic, and iron from 2-10 pm (Tan et al 1999).the quality of CPO is determined by its free fatty-acid content ,and the oxidative parameter(Swodoba,1982).
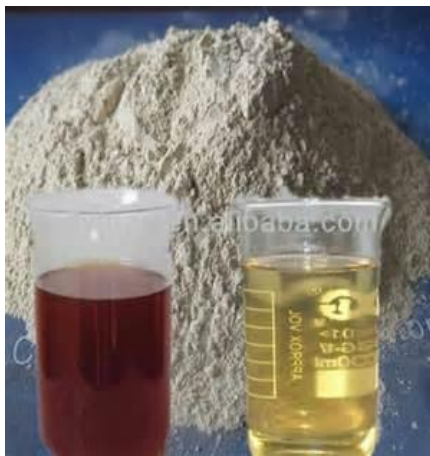
Figure 1 : Palm Oil before (left) and
after (right) Undergo Bleaching Process

Proceeded generally by degumming and refining(neutralization) processes, bleaching is required to removed specific detrimental contaminants that are not effectively removed by these processes before the oil progresses through deodorization.

Bleaching originally described as a process of mixing oil and clay adsorbent to remove color, reduces the content of chlorophyll, residual soap and gums, oxidative products, trace metals and indirectly impacts on deodorized oil color

Bleaching earth is also known as fuller's earth and is essentially a physical adsorption process using activated carbon or bleaching earth to removed undesired matters from oils. Bleaching earth is a type of clay mined in Asia, England, India and United States. Once you obtain bleaching earth you get a substance that is rich in minerals and it is used for various purposes including for bleaching, absorbing and filtering.

If bleaching earth is used for industrial purposes, the earth then has to be put through processing which involves recycling. After this recycling, the earth will be ready to be used again and it will also be in a state in which it can safely be disposed of but also beware that these substances are quite flammable and they have harmful characteristics as well. Attapulgite ,bentonite and montmorillonite clays are the most commonly found in bleaching earth.. Once obtained from the earth, this mineral-rich substance is processed and used for its absorbing, bleaching, and filtering properties.

Figure 2 : Reactor in Refining Process

1.3   Motivation and statement of problem

In the palm oil refinery, the critical part is degumming and bleaching process stage where at these stages separation of minor components must be carefully monitored (Goh *et al.*, 1985). Any imperfection during these processes will tremendously affect refining processes or the later stages and finally affect the finished product byproduct. These stages are considered as major contributors to the total operating cost of the plant due to the cost of chemicals which is phosphoric acid and bleaching earth clay that are being used in these processes.

Bleaching is one of the most cost-intensive processes for refining vegetable oils caused mainly by the consumption of bleaching agents like bleaching earth and activated carbon, oil losses in the spent bleaching and in certain cases for disposal of the spent agents. Because of these reason, all refineries to trying to find a way to reduce the cash flow out for this process as much as they

can. The price of bleaching earth is RM 700-800 per MT and RM 3000 per MT for phosphoric acid and it is about 20% of total operating cost are due to bleaching process. In current situation the amounts of chemicals added in are usually fixed within typical ranges of doses usually used throughout certain period. It means that, even though the incoming CPO has lower impurities or minor components content, the amount of chemicals added would not be changed. Thus, it important to have a process model which can suggest a suitable ratio of phosphoric acid and bleaching earth for the varying CPO quality.

1.4   Objectives of research

The objectives of this research are:

1.3.1   To predict the most optimal ratio of phosphoric acid and bleaching earth for the bleaching process of crude palm oil.

1.3.2   To decrease the percentage of FFA by optimizes physical refining bleaching process condition.

1.3.3   Identification of the most optimal operating parameters and conditions for bleaching processes.

1.5   Scope of study

In order to achieve the objective, the following scopes have been identified and to be applied:

1.4.1   The effect of time contact (20, 40, 60 minutes) of bleaching clay in bleaching process

1.4.2   The effect of temperature range 60℃-90℃ in physical bleaching process

1.4.3   The effect of ratio of acid (0.05-0.1 %w) to earth clay(0.2-2.0%w) in physical refinery process and the number of experiments need to be run are determined through the design of experiment (DOE) method.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In this chapter, the finding of related articles from goggle scholar, MPOB website and I-portal is needed in order to do literature review. The literature review is research done in the past by other people and it is needed to support our research objectives.

2.2 Oil Palm

2.2.1 History, habitat, tree and industrial development

The oil palm tree is a tropical palm tree and has two species which better known originally come from Guinea, Africa is originally illustrate by Nicholaas Jacquin in year of 1763 and hence, oil palm is known as *Elaeis guineensis Jacq* as after his name. Elaia is come from Greek word that means olive which indicates its fruits rich in oil. Besides that, *Elaeis guineensis* is also a member of the family *Palmae* with their subfamily, *Cocoidae* which is includes the coconut.

The most suitable area for oil palm is soil that free from draining with low pH and does not thrive at very high pH which is greater than 7.5. Its culture is well done in low altitude less than 500 m above sea level with 15˚ from the equator in the humid tropics. The soil is properly drained with distributed of rainfall of 1,800 to 2,000 mm/year but will tolerate rainfall up 5,000 mm/year. If there are three months in row, with less than 100 mm rainfall per month, productive yield will be reduced as oil palm is sensitive toward poor drainage and drought. This is one of the reason why it is agreed that oil palm *Elaeis guineensis Jacq,* is originated from equatorial tropical rain forest Africa.

Figure 3 : Fresh Fruit Bunch

The most suitable area for oil palm is soil that free from draining with low pH and does not thrive at very high pH which is greater than 7.5. Its culture is well done in low altitude less than 500 m above sea level with 15° from the equator in the humid tropics. The soil is properly drained with distributed of rainfall of 1,800 to 2,000 mm/year but will tolerate rainfall up 5,000 mm/year. If there are three months in row, with less than 100 mm rainfall per month, productive yield will be reduced as oil palm is sensitive toward poor drainage and drought. This is one of the reason why it is agreed that oil palm *Elaeis guineensis Jacq,* is originated from equatorial tropical rain forest Africa.

Oil palm is native to Africa, but Malaysia was the first country to embark on large-scale planting and processing. In order to boost production, Malaysia had to create and develop its own technology and adopt innovative policies. In 1917, the first commercial oil palm estate in Malaysia was set up at Tennamaran Estate, Selangor. However, in order to get through over dependence on natural rubber which are major commodity during previous years ,it was only in the 1960s, oil palms were commercially cultivated in bigger scale.(Kifli,1981) Currently, there are more than three million hectares of oil palm plantations. In total, about 90 million mt of renewable biomass (trunks, fronds, shells, palm press fiber and the empty fruit bunches) are produced each year.(M. Suhaimi and H.K. Ong,2011)

Since then, palm oil industry has expended rapidly and has emerged as the most remunerative agricultural commodity, overtaking the natural rubber (Arrifin and Fairus 2002).The present and increasing of the industry has been phenomenal and accounting for 52 percent of world production and 64 percent of world exports in 1999, Malaysia is now the largest producer and exporter of palm oil in the world, and Figure 6 below, shows world annual production and annual exports of palm oil according to the respective countries. This was complemented by the government allocating land to the poor and landless to plant more oil palm, in great part causing the area to increase from 62 000 ha in 1975 to 1.02 million hectares in 1980 and 2.03 million hectares in 1990. By 2007, there were 4.3 million hectares of oil palm, constituting nearly two-thirds of the national agricultural area. With a production of 16.20 million tonnes in 2006, Malaysia continues to be the world's largest palm oil producer. The success of the crop is largely market driven with good long term price prospects for palm oil making oil palm more attractive than most other crops. Palm oil contributes more than one-third of the national agricultural GDP, generating RM 31.81 billion in export earnings in 2006, making it one of the pillars of Malaysia's economy. At present, the industry employs more than 1.5 million people in the core and related sectors.
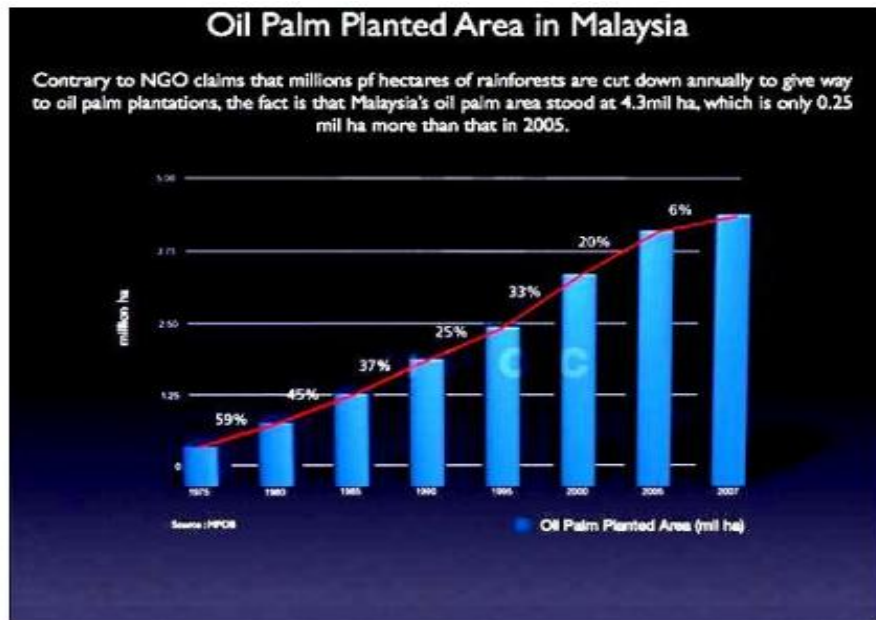
Figure 4 : Oil Palm Planted Area in Malaysia 1975-2007

Country	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003
Malaysia	7,403	7,221	8,386	9,069	8,319	10,554	10,842	11,804	11,909	13,354
Indonesia	3,421	4,008	4,540	5,380	5,361	6,250	7,050	8,030	9,200	9,750
Nigeria	645	640	670	680	690	720	740	770	775	785
Colombia	323	353	410	441	424	501	524	548	528	543
Cote d'Ivoire	310	300	280	259	269	264	278	220	240	251
Thailand	297	316	375	390	475	560	525	620	600	630
Papua New Guinea	223	225	272	275	210	264	336	329	316	325
Ecuador	162	178	188	203	200	263	222	201	217	247
Costa Rica	84	90	109	119	105	122	138	138	140	144
Honduras	80	76	76	77	92	90	97	108	110	112
Brazil	54	71	80	80	89	92	108	110	118	132
Venezuela	21	34	45	54	44	60	73	80	80	79
Guatemala	16	22	36	50	47	53	65	70	81	91
Others	1,265	1,676	815	869	844	832	879	919	922	940
TOTAL	14,304	15,210	16,282	17,946	17,169	20,625	21,877	23,947	25,236	27,383

Figure 5 : World Major Producers of Palm Oil '000 tonnes (MPOB, 2003)

Country	1994	1995	1996	1997	1998	1999	2000	2001	2002	2003
Malaysia	6,750	6,513	7,212	7,490	7,465	8,911	9,081	10,618	10,886	12,248
Indonesia	2,173	1,856	1,851	2,982	2,260	3,319	4,140	4,940	6,379	6,830
Papua New Guinea	231	220	267	275	213	254	336	328	324	325
Cote d'Ivoire	148	120	99	73	102	101	72	75	65	63
Colombia	20	21	29	61	70	90	97	90	85	105
Singapore*	328	399	289	298	241	292	240	224	220	256
Hong Kong*	234	275	305	173	103	94	158	192	318	206
Others	876	791	711	860	680	787	884	1,107	956	1,083
TOTAL	10,760	10,195	10,763	12,212	11,134	13,848	15,008	17,574	19,233	21,116

Figure 6 : World Major Exporters of Palm Oil '000 tonnes (MPOB, 2003)

Natural pigments that present in vegetable oils are mainly the caused by carotenoid, giving yellow and red colors, and the chlorophylls which give green colors. Color deterioration of crude palm oil can take place during the refining process, which removes contaminants that adversely impact the appearance and performance of this oil. Many of these impurities have to be removed from the oil to achieve the high quality oil standards necessary for edible applications.

2.2.2 What is Crude Palm Oil?

There are two types of oil produced from oil palm. The first is crude palm oil (CPO) and the second is crude palm kernel oil (CPKO) from the palm kernel. The two types of oil differ in terms of chemical composition and nutritional content. Palm oil has a balanced ratio of saturated and unsaturated fatty acids while palm kernel oil has mainly saturated fatty acids (Teoh, C.H. (2002). Crude palm oil (CPO) is the oil obtained from the mesocarp part of palm oil fruit. Figure 8 shows the processes undergone by fresh fruit bunches (FFB) to produce CPO. The crude palm oil (CPO) produced, is further processed to yield either red or bleached cooking oil or detergents.

Figure 7 : Crude Palm Oil

Fresh fruit bunch (FFB)

FFB enters the plant mills for processing

Sterilization in large pressure vessels / cages

Extraction in a homogenous oil

Purifications in a continuous clarification tank

Stripping in rotating drum "Stripper"

Crude palm oil (CPO)

Refining process
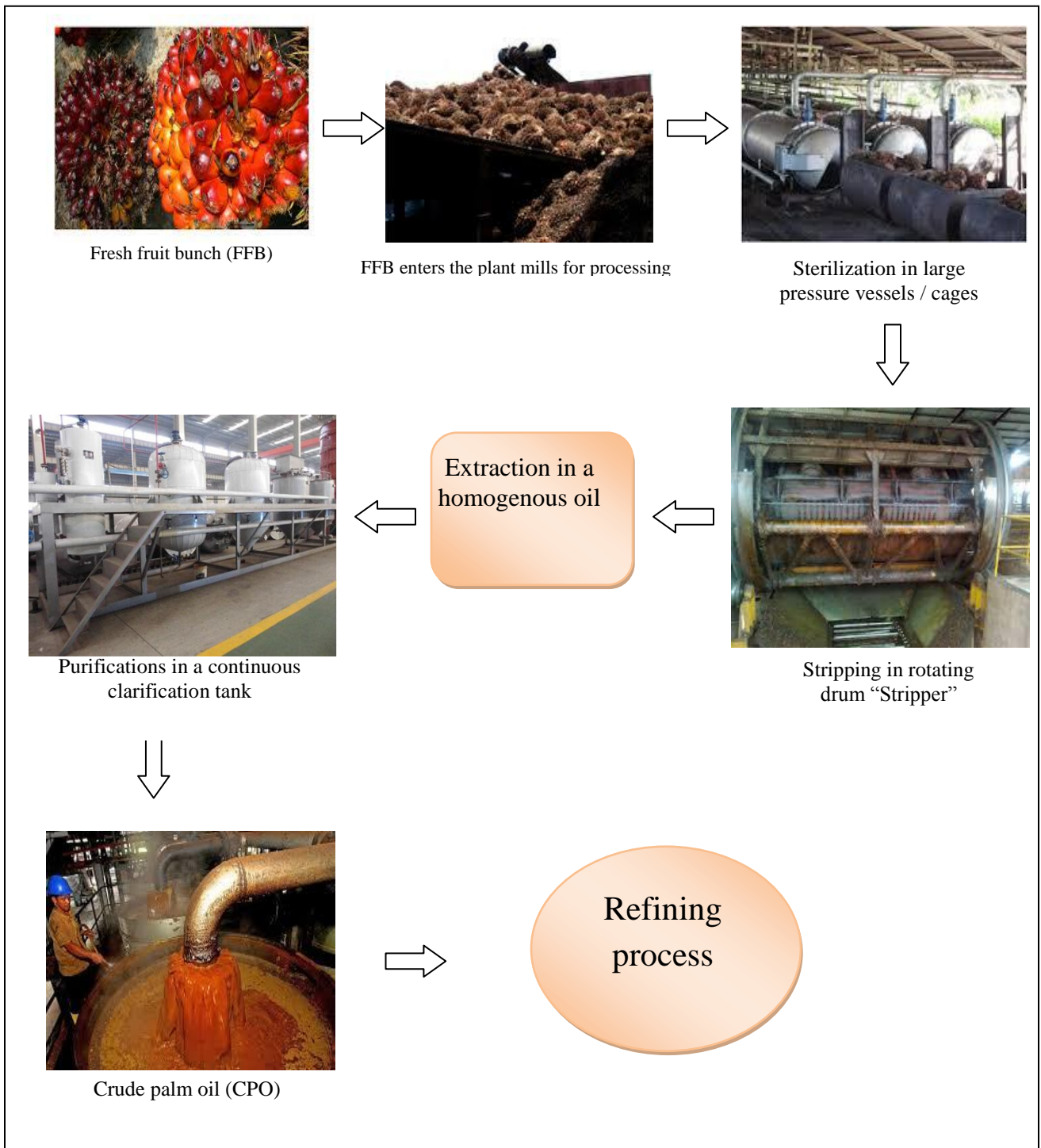
Figure 8 : Flowchart of Crude Palm Oil (CPO) Production

2.2.3　Chemistry of Palm Oil

Higuchi (1983), The empty bunch is a solid waste product of the oil palm milling process and has a high moisture content of approximately 55-65% and high silica content, from 25% of the total palm fruit stated that crude vegetables oil commonly consists of desirable triglycerides, unsaponifiable matter together with small amount of impurities (Keu, S.T., 2005). Like all oils, TGs are the major constituents of palm oil. Over 95% of palm oil consists of mixtures of TGs, that is, glycerol molecules, each esterified with three fatty acids. During oil extraction from the mesocarp, the hydrophobic TGs attract other fat- or oil-soluble cellular components. These are the minor components of palm oil such as phosphatides, sterols, pigments, tocopherols, tocotrienols and trace metals. Other components in palm oil are the metabolites in the biosynthesis of TGs and products from lipolytic activity. These include the monoglycerols (MGs), diglycerols (DGs) and free fatty acids (FFAs).

The fatty acids are any of a class of aliphatic acids, such as palmitic (16:0), stearic (18:0) and oleic (18:1) in animal and vegetable fats and oils. The major fatty acids in palm oil are myristic (14:0), palmitic, stearic, oleic and linoleic (18:2).5

The typical fatty acid composition of palm oil from Malaysia is presented in Table 2.1. Palm oil has saturated and unsaturated fatty acids in approximately equal amounts.

Most of the fatty acids are present as TGs. The different placement of fatty acids and fatty acid types on the glycerol molecule produces a number of different TGs. There are 7 to 10% of saturated TGs, predominantly tripalmitin.

The fully unsaturated TGs constitute 6 to 12%. The Sn-2 position has specificity for unsaturated fatty acids. Therefore, more than 85% of the unsaturated fatty acids are located in the Sn-2 position of the glycerol molecule. The triacylglycerols in palm oil partially define most of the physical characteristics of the palm oil such as melting point and crystallisation behaviour The crude palm oil compositions can also be classified as a mixture of 5 main chemical groups as per shown in Figure 9 below.

Group	Components in the group
Oil	- Triglyceride, Diglyceride , Monoglyceride - Phospholipids, Glycolipid and Lipoprotein - Free fatty acids
Oxidized Products	- Peroxides, Aldehydes, Ketones, Furfurals (from sugars)
Non-oil (but oil solubles)	- Carotene - Tocopherols - Squalene - Sterols
Impurities	- Metal particles - Metal ions - Metal complexes
Water Solubles	- Water (moisture) - Glycerol - Chlorophyll pigments - Phenols - Sugars (soluble carbohydrates)

Figure 9 : General Composition of Crude Palm Oil (Abdul Azis, 2000)

Table 2.1: Typical Fatty Acid Composition (%) of Palm Oil

Fatty acid chain length	Mean	Range observed	Standard deviation
12 : 0	0.3	0-1	0.12
14 : 0	1.1	0.9-1.5	0.08
16 : 0	43.5	39.2-45.8	0.95
16 : 1	0.2	0-0.4	0.05
18 : 0	4.3	3.7-5.1	0.18
18 : 1	39.8	37.4-44.1	0.94
18 : 2	10.2	8.7-12.5	0.56
18 : 3	0.3	0-0.6	0.07
20 : 0	0.2	0-0.4	0.16

2.2.4 Finished Products (RBDPO) Quality

In Malaysia, the generally accepted trading specifications for crude palm oil are; 5 % maximum FFA; 2.5 % maximum moisture and impurities (Goh, 1991),while the Palm Oil Refiners Association of Malaysia (PORAM) standard specifications for refined palm oils are given in Table 2.2. Refined, bleached and deodorised palm oil is obtained from crude or semi-refined palm oil which has been bleached, deodorised and deacidified by physical means (PORIM, 2000). The melting and crystallisation characteristic of the oil can be followed using the DSC technique (Gunstone, 2011).

Table 2.2 : Standard Specification For Refined Palm Oils (Victoria et al., 2011)

Parameter	RBD Palm Oil
Free Fatty Acid,FFA (% as Palmitic)	0.1 max
Moisture and Impurities (%)	0.1 max
Iodine Value (Wijs)	50-55
Melting Point(C-AOCS Cc 3-25)	33-39
Color(5.25"Lovibond Cell)	3 or 6 red max

2.2.5 Uses of Palm Oil

As mentioned, the oil palm produces two types of oils, palm oil from the fibrous mesocarp and palm kernel oil from the palm kernel. Palm oil and palm kernel oil have a wide range of applications; about 80% are used of food applications while the rest is feedstock for a number of non-food applications (Salmiah. 2000).

Among the food uses, refined, bleached and deodorised (RBD) olein is used mainly as cooking and frying oils, while RBD stearin is used for the production of shortenings and margarine. RBD palm oil, which is the unfractionated palm oil, is used for producing margarine, shortenings, vanaspati(vegetable ghee), frying fats and ice cream (Salmiah, 2000).

2.3   Refinery Method in Industry

Refining process is an important step for the production of edible oils and fats products. The objective is to remove the impurities and other components that will affect the quality of finished product. Crude palm oil of poor quality was degummed, bleached and deodorized to evaluate the effectiveness of various bleaching clays using different dosages (Ng, Sook Kuen, 2006).

The flavor, shelf-life stability and color of the finish products need to be monitored to maintain its quality. (Leong, 1992).In industry perspective, to convert the crude oil to a quality edible oil is the main aim of refining by removing objectionable impurities in the most efficient manner to the desired levels. This also means that, where possible, losses in the desirable components are kept minimal and cost effective.

The objectionable substance or impurities in palm oil maybe biogenic synthesized by plant themselves but they can be impurities taken up by the plants from their environment (Borner et al., 1999). The impurities maybe acquired during upstream of bleaching process which are extraction, storage or transportation of the crude palm oil from mill to the refinery. It is important to have proper refining process in order to produce high quality of finished products with specified quality range and meet users' requirements.

Parameter that used to assess the efficiency of various stages of a refining process is called Refining factor (RF). It is dependent upon the yield of the product and the quality of the input and it is calculated as:

$$RF = \frac{oil\ loss\%}{FFA\%}$$

The RF is usually quantified for various stages of refining process individually and monitoring of the RF in the refinery is usually by means of weight calculated from volumetric measurements adjusted for temperature or by using accurate cross-checked flow meters.

There are 2 basic types of refining technology available for palm oil:

(i) Chemical (alkaline) refining

(ii) Physical refining

The differences between these 2 types are basically based on the type of chemicals used and mode of removing the FFA.

Table 2.3: Basic Step of Refining Process (Ng, Sook Kuen, 2006).

Alkali or chemical refining	Main groups of compound removed	Physical refining
Degumming	Phospholipids	Degumming
Neutralization	Free fatty acids	
Bleaching	Pigments / metals / soaps	Bleaching
Winterization	Waxes / saturated triacylglycerols	Winterization
Deodorization	Volatiles / free fatty acids	Deodorization / deacidification

2.3.1 Physical Refinery

Physical refining of vegetables oils is a distillation process. It appears to practically replace the use of chemical (alkali) refining in palm oil as the consequence of high acidity content (FFA) in chemically refined oil. The deacidification (deodorisation) process stage in the physical refining is able to overcome such situation.

Apart from that, according to the literature, this method is preferred because it is acknowledged to be suitable for low-content phosphatides vegetable oil such as palm oil. Thus, physical refining is proven to have a higher efficiency, less losses (refining factor (RF) < 1.3), less operating cost, less capital input and less influent to handle .

2.3.1.1 Dry Degumming

Degumming process is mainly to remove phospholipids or gums from the crude oil. There are two types of phospholipids present in crude oils according to their level of hydration which is hydratable and non-hydratable ones, the latter mainly present as calcium and/or magnesium salts of phosphatidic acid and phosphatidylethanolamine. Most of the phospholipids are hydrated and are insoluble in the oil if treat with water. Further filtration or centrifugations are needed to separate the hydrated compounds efficiently. The oil is usually treated with phosphoric acid for the elimination of the non-hydratable fraction, (0.05 to 1%), which chelates the Ca and Mg converting the phosphatides into the hydratable forms. Analysis of phosphorous prior to acid treatment is necessary to ensure that the acid dosage is correct due to the variable content of phospholipids in crude oils, especially when the content of Ca and Mg salts is high.

The degumming step can be eliminated depending on the oil composition and during the next step of neutralization; the phosphatides are also removed along with the soaps. Furthermore, degumming is mandatory for physical refining and the content of phosphorous after degumming should be lower than 10 mg/kg.

2.3.1.2 Bleaching Process

Bleaching is a treatment that removes the colour substances and other impurities such as fat in oil. The usual method of this process is by adsorption of the impurities on an adsorbent material or bleaching agent.

This step is applied to both physical and alkali refining, where the hot oil is slurried with acid-activated bleaching earth .Then, adsorption of colour bodies, trace metals and oxidation products such as residual soaps and phospholipids remaining will takes place.

The reaction time has to exceed 15 minutes and cannot more than 30 minutes at optimal bleaching temperatures for the great adsorption of oxidation products to be produce. The removal of chlorophyllic pigments is very important since they are not eliminated in any other stage of refining, as carotenoid compounds are in deodorization. Moreover, because of their iron content in the activated earths filtration must eliminate completely the activated earths as the presence of traces act as prooxidants during oil storage.

Although synthetic silicas and active carbons are applied in industrially, acid-activated clays are the major adsorbent used nowadays. Active carbons are used to eliminate polycyclic aromatic hydrocarbons (PAH) from some oils, especially fish oils and pomace oils, while synthetic silicas are good in adsorbing secondary oxidation products, phospholipids and soaps (Leon et all,2003).

Two types of adsorption occur between the compounds to be adsorbed and the absorbent and this is the critical part in order to obtain good quality oils. Because of the need to control the presence of refined oils in virgin oils, chemical changes taking place at this stage have been well studied in olive oil. The two main reactions found in all the vegetable oils are the following:

Figure 10 : Sunflower

o Hydroperoxides decomposition. Previous steps do not modify the peroxide value and it may even increase if air is available in the earlier stages. But, during bleaching, hydroperoxides decompose to form volatiles and oxidized triacylglycerols containing keto and hydroxy functions. After bleaching, there should be no peroxide value, but the significant increase in the anisidine value is detected where there are presence of aldehydes and ketones .

o Alcohols dehydration. Partial dehydration of hydroxyl is perform by earth catalysis.A rapid increase in UV absorption at 232 nm is observed as the function is at an allylic position because of the formation of conjugated dienes from oleic acid hydroperoxides and in UV absorption at 268 nm due to formation of conjugated trienes from linoleic acid hydroperoxides. Besides, sterols undergo significant dehydration and the formation of the

hydrocarbon 3, 5-stigmastadiene from the major sterol (β-sitosterol) is considered a proof of the presence of refined oil in virgin olive oil (Cert et al, 1994).

2.3.1.3 Deodorization Process

Last stage for physical refining is deodorization. At deodorization temperature and pressure the pre-treated oil is deoerated. Under these condition , which the free fatty acids, which are still present in the oil are distilled together with the more volatile odoriferous and oxidation products such as aldehydes and ketone, which otherwise will affect the odor and the taste of the oil.
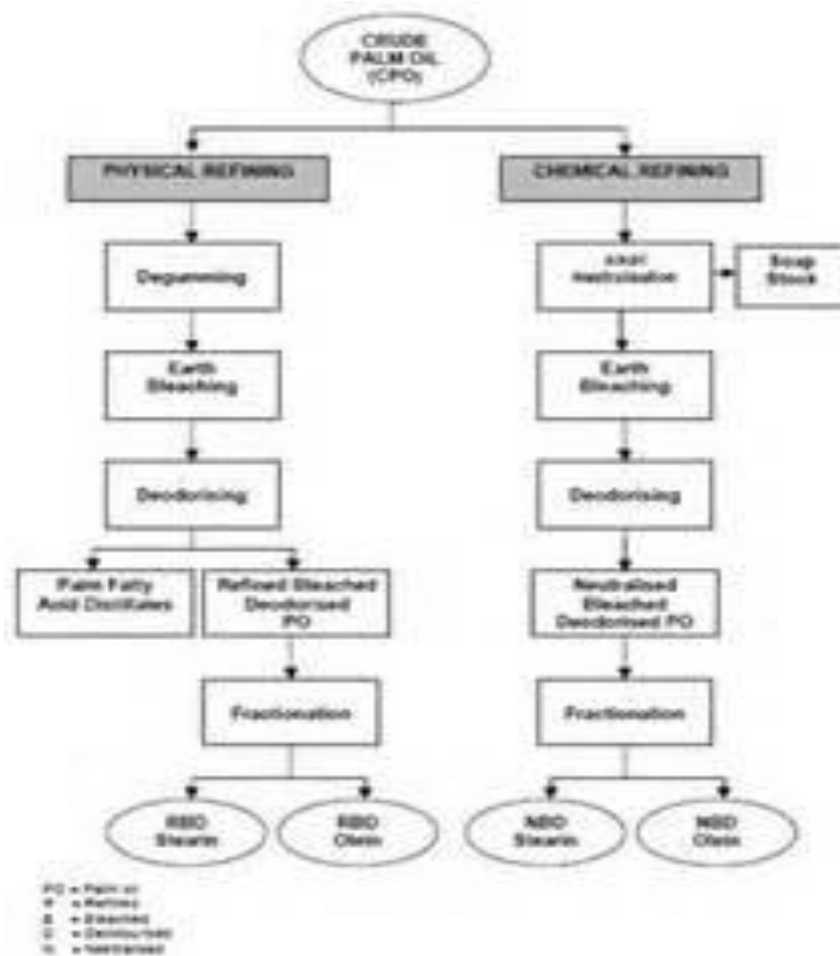
Figure 11 : Chemical and Physical Refining Routes

In general, chemical refining requires more processing stage, more equipment and more chemicals compared to the physical refining. The processing routes for chemical and physical refining are as per illustrated inFigure 11.
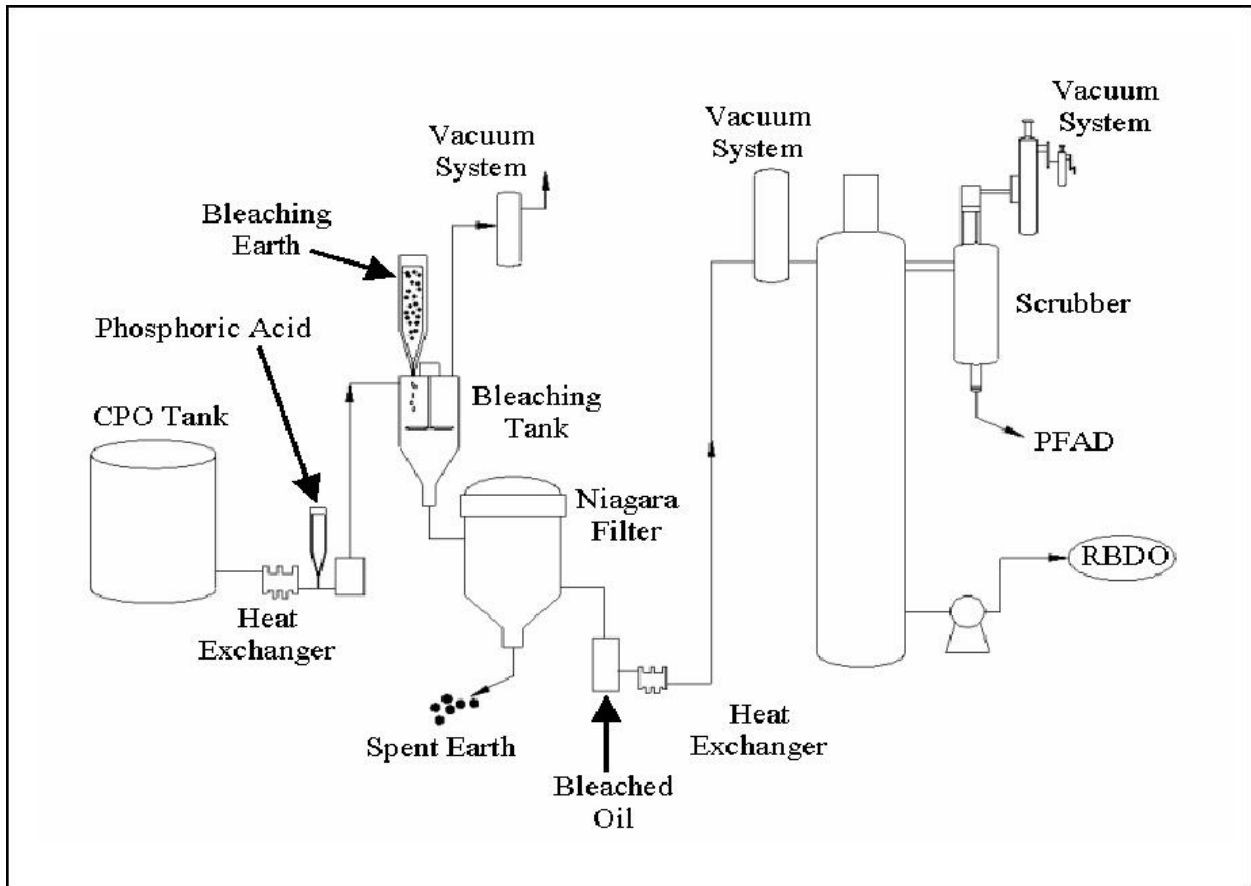
Figure 12 : Simplified Flow Sheet of Physical Refining

2.4   Types Bleaching Methods

There are 3 types of bleaching methods can be used in edible oil industry told by Gunstone and Norris, 1983, namely:

2.4.1   Heat bleaching

Pigments such as the carotenes may become colorless if heated sufficiently. But, these pigment molecules will leaves in the oil and may have adverse effect on oil quality. Also, when the oil became contact with air colored degradation product such as chroman-5, 6-quinones from γ-tocopherol may form and these type of pigment are difficult to remove. (Taylor, 1992)

2.4.2   Chemical Oxidation

Some pigments for example carotenoids are made colorless or less colored by oxidation. But such oxidation invariably affects the glycerides and destroys natural antioxidants. Consequently, it is never used for edible oil but restricted to oils for technical purposes, such as soap-making.

2.4.3   Adsorption

Adsorption is the common method usually used for bleaching the edible oil by using bleaching agents. Examples of bleaching agents are bleaching earths, activated carbon and silica gel. Bleaching agents normally posses a large surface that has a more or less specific affinity for pigment-type molecules, thus removing them from oil without damaging the oil itself.

2.5 Mechanisms of Degumming and Bleaching Process

Oils and fats are bleached in order to remove undesired colorants because these colorants can negatively affect the taste of the oil and in part because the color would disturb the consumers, therefore on the whole, these colorants limit use and market ability. In addition to that, some particles or pigments that promote deterioration to oil quality is also being removed during bleaching process mainly due to their pro-oxidative properties that promotes oxidation (Bockish, 1998).

During bleaching or purification, the oil is brought into contact with a surface–active adsorbent, and then the undesired particles or other components are selectively retained on the pore surface and triglycerides escapes. Gradually, the concentration of undesired particles on the available surface-active of the adsorbent and the concentration remaining in the oil come into balance, so further exchange is negligible.

Best temperature for oil must be chosen, as well as duration of contact because an excess of either factor will impart undesirable side effects to the process. In order to have efficient use of adsorbent, any material like gum or soap should be removed at earlier degumming stage as the presence of these particles will compete for room on the adsorbent surface. That explains why an effective degumming process is required in refining process of vegetable oils.

Theoretically, colored particles that should be removed during bleaching are present in the oil either in dissolved or in a colloidal form. For both types, the process reaction happens at the surface of bleaching agent.

2.6 Bleaching Agents

There are few types of bleaching agents that are being used in vegetable oil industry such as acid activated bleaching earth ; bentonite, $H_2Al_2(SiO_3)_4-nH_2O$,natural bleaching earth, activated carbon, synthetic silicates and synthetic resins.

Bleaching earth is characterized by high bleaching efficiency, fast filtration rate, low oil retention, ability minimize the increase of free fatty acids , removing impurities like soap and trace metals without affecting appearance, flavor and nutritional properties of oil.

Acid activated bleaching earth or clay, which is also called bentonite, is the adsorbent material that has been used most extensively. This substance consists primarily of hydrated aluminum silicate. Usually, bleaching earth does not remove all the color materials, much of which are actually removed by thermal destruction during the deodorization process. Activated carbon is also used as a bleaching adsorbent to a limited extent.

Activated carbon has very higher surface area, often in excess of 1000 m2/g. Much of that surface area is, however, associated with microspores; pores <20 A ° (<2 nm) in diameter. The surface area associated with mesopores ; pores 20 to 500 A ° (2 to 50 nm) in diameter ; is considerably lower (typically in the range 10—100 m2/g). Most liquid-based applications involve the adsorption of high-molecular-weight contaminants whose molecular dimensions prevent penetration into microspores; therefore, activated carbon containing significant mesoporosity is most desirable in these applications (Kaghazchi and Soleimani., 2011)

2.7 Factors Affecting Degumming and Bleaching Processes of Crude Palm Oil

Refining process can be affect by many factors. The factors that are mentioned here are particularly focused on the upstream operation of the refining process, which are degumming and bleaching. The factors are, bleaching time, quality of CPO, operating pressure, operating temperature, bleaching earth dosage and phosphoric acid dosage (Rohani, 2006).

2.7.1 Quality of CPO

As to the refiners, the most importance as it relates directly to the product's shelf life and the processing cost is the quality of the crude palm oil (Wong, 1983). This is because refiners are requested to meet stringent specifications. A crude palm oil (CPO), which can be easily processed to bland and light colored oil with good oxidative and color stability is the objective of all refiners. All requirements should preferably be obtained at the minimum refining cost, in other words, low oil losses with minimal use of bleaching aids. Therefore, an ideal CPO should posses the quality as shown in Table 2.4 below;

Table 2.4 : Ideal Quality Target of Crude Palm OIl (Ai. 1990)

Parameters	Crude Palm Oil (maximum)
FFA, %	3.5
M & I,%	0.25
Peroxide value, meq/kg	1.0
Anisidine value	5
β-carotene content,ppm	500-800
DOBI	2.5
Phosphorus,ppm	15
Iron (Fe),ppm	5
Copper (Cu),ppm	0.1
Colour (5.25" Lovibond Cell)	-

2.7.2   Bleaching Temperature

Bleaching temperature is one of the factors that affecting the performance of bleaching and degumming process of crude palm oil. Bleaching temperature was found that it can be affecting the color, Figure 13, of oil and the FFA content, Figure 14, of the oil.
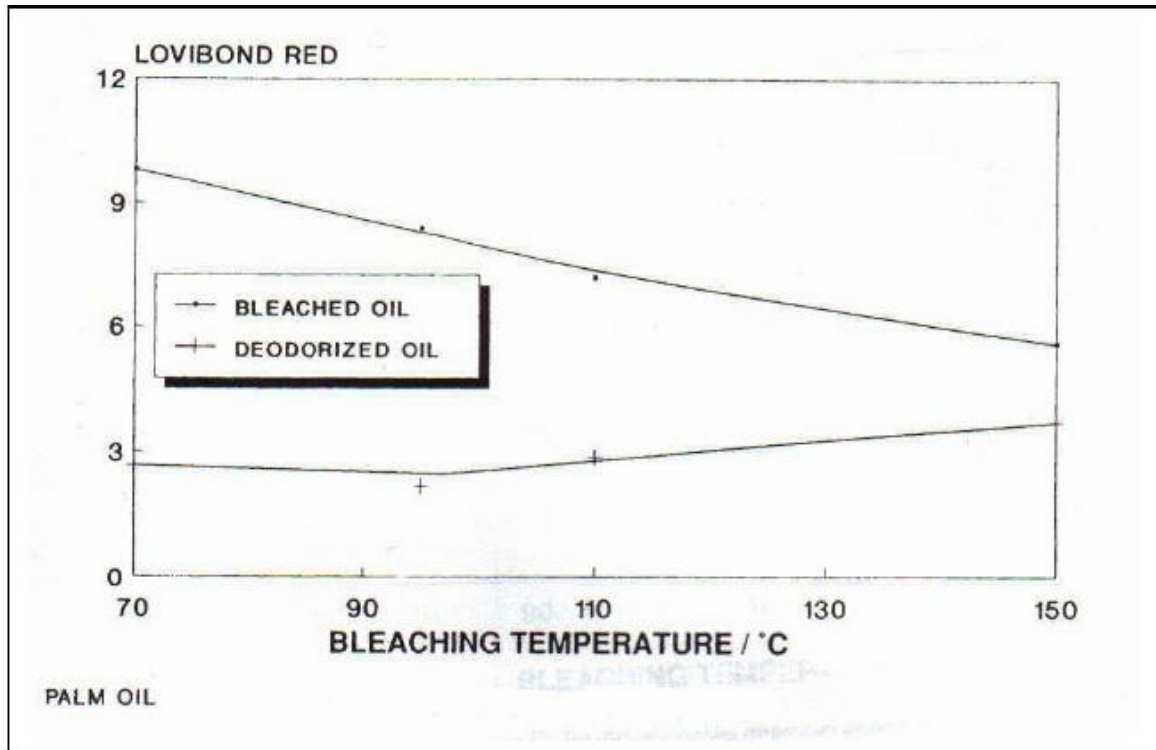
Figure 13 : Effect of Bleaching Temperature on Oil Colour
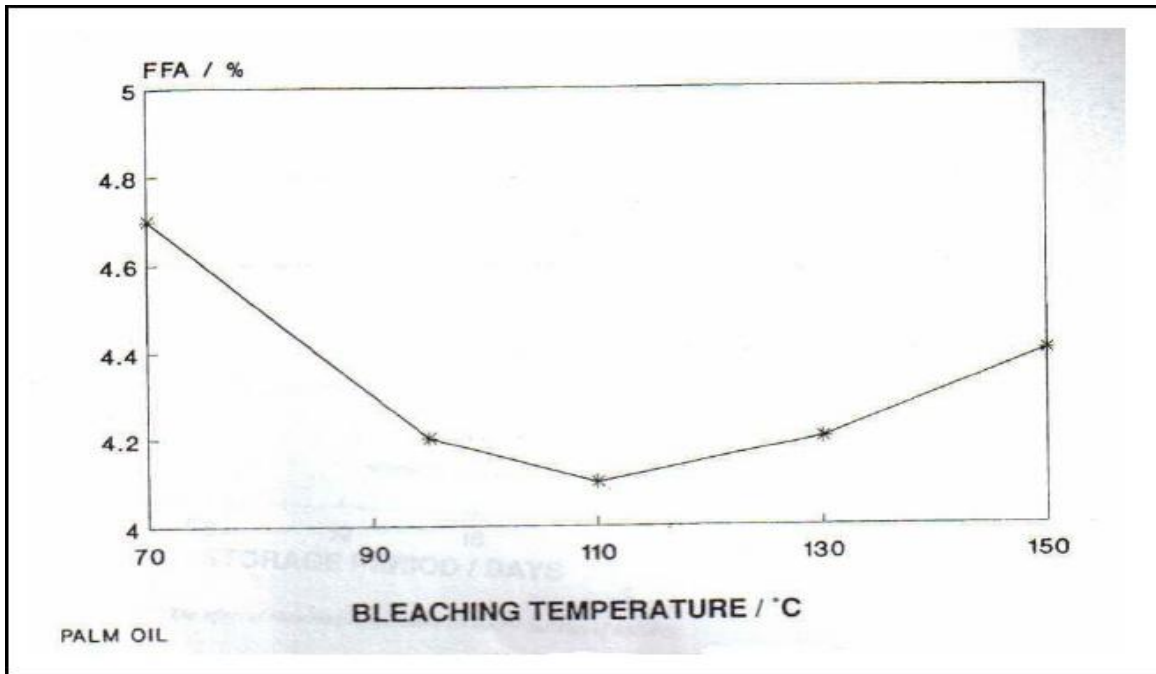
Figure 14 : Effect of Bleaching Temperature on Free Fatty Acid (FFA)

2.7.3   Bleaching Earth Dosage

Bleaching earth dosage is also one of the factors that can influence the efficiencies of degumming and bleaching processes. This factor is one of the easiest variables to change. Amount of dosage added will affect the value of FFA extraction. Higher dosage will increase the adsorption but also not good for oil quality if too much of bleaching agent is added. It eventually also affect the colour of the RDBPO.
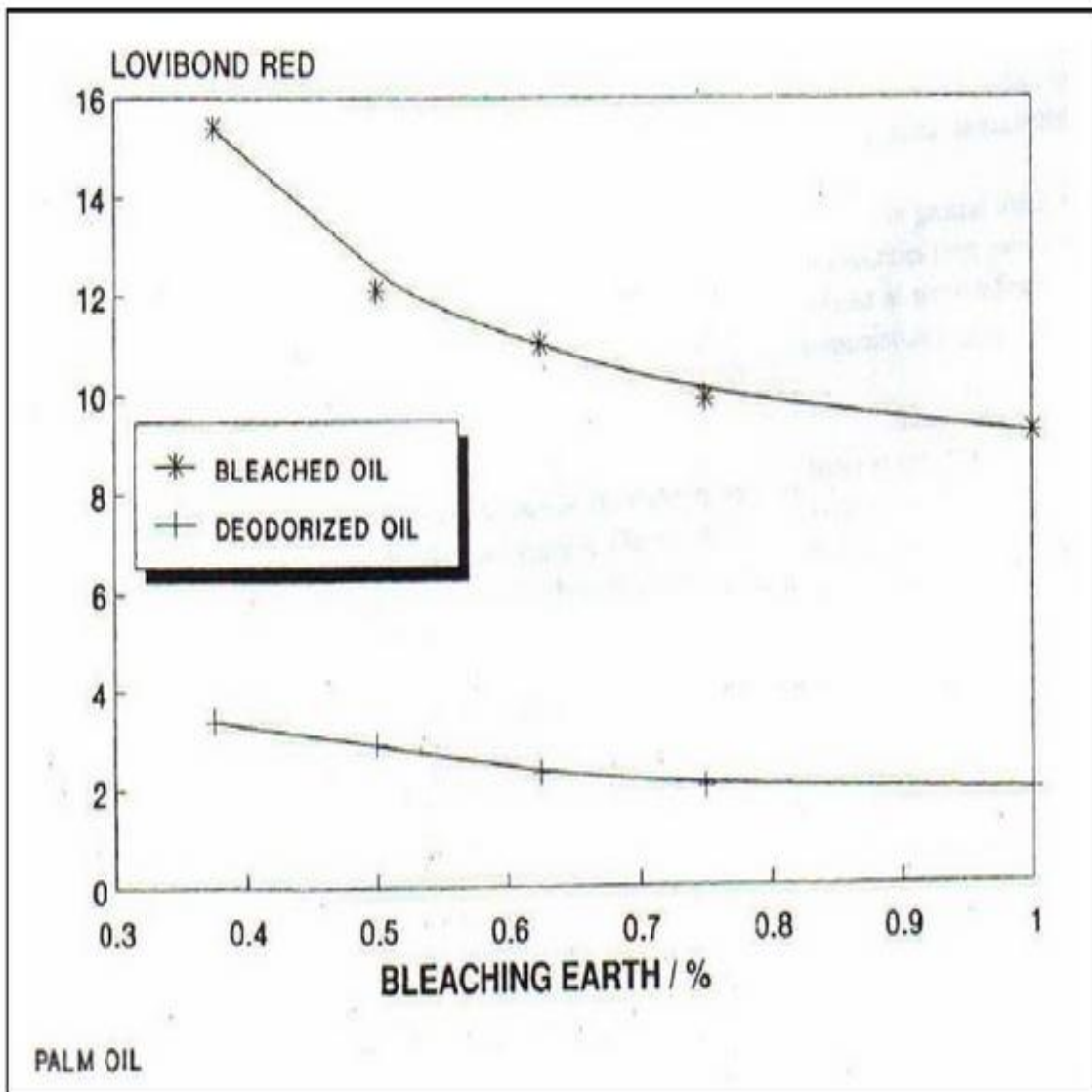
Figure 15 : Effect of Bleaching Dosage on Color of Palm Oil)

2.7.4 Phosphoric Acid Dosage
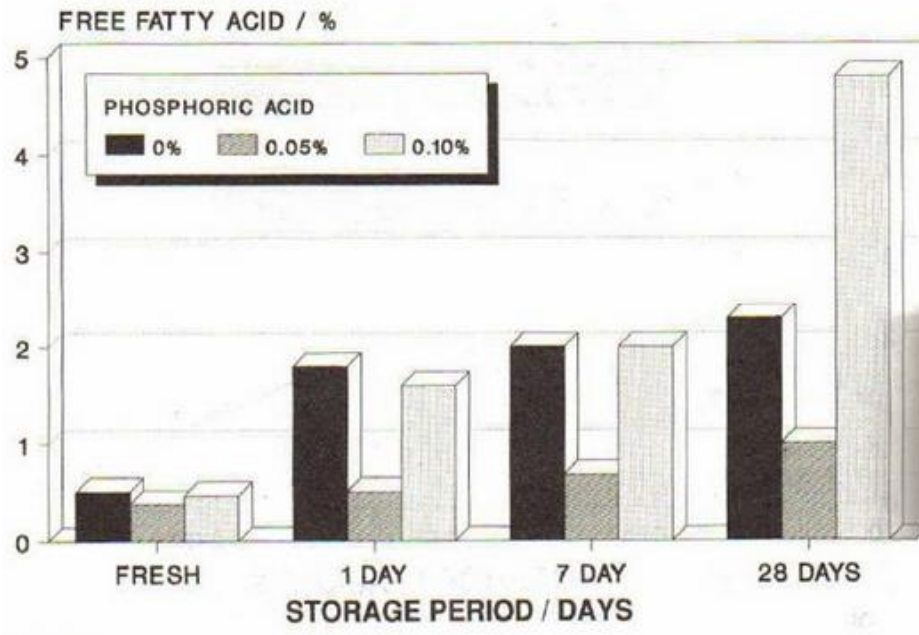
Figure 16 : Effect of Phosphoric Acid Dosage on FFA

2.8 Conclusion

In an industry, past researchers shows similar statement about the optimization of bleaching earth in refinery process. There are many methods to be used to produce a good quality of vegetable oil and by using different raw material and different parameter.

Therefore, this research will be find out either the chosen changes of parameter will yielding the best quality product or not.

CHAPTER 3

METHODOLOGY

3.1 Introduction

More detail about the method to conduct in this research on optimization of bleaching earth and

extraction of Free Fatty Acid(FFA) in palm oil refinery process.

3.2 Operational Conditions

Based on preliminary studies through surveys and literature it is found that the optimal operating conditions for a typical configuration of palm oil refinery are identified as per shown in Table 3.1 below;

Table 3.1 : Operational Condition

Parameter	Operating condition
Temperature	100°C
Pressure	Vacuum (50 torr)
Contact time	30 minutes

3.3 Quality Checking Parameters

Parameters that are usually being checked either it is on daily basis, weekly basis or monthly basis. The parameters are (Siew et al., 1995);

(i)Free Fatty Acid (FFA) content

(ii)Moisture Content

(iii)Deterioration of Bleach ability Index (DOBI) value

The ranges are identified based on the maximum acceptable and allowable values used by the refiners as Table 3.2 ;

<div align="center">Table 3.2: Ranges of Parameter</div>

Parameters		Range
Dosage of phosphoric acid		Maximum : 1.00 % (per 1MT of CPO)
Dosage of bleaching earth		Maximum : 2.00% (per 1MT of CPO)
Quality of CPO	FFA	Maximum: 5 %
	Moisture	Maximum : 2 %
	DOBI	2-3.5

But, for this research, we only focus on FFA content varying with different dosage of acid and bleaching earth, contact time and temperature.

3.4   Materials and methods

3.4.1 Raw material

Crude palm oil (CPO) can be found from any source. For this experiment, CPO sample has been collected from Kilang Swit LCSB Lepar.

3.4.2 Chemical and equipment

Chemicals   : Phosphoric acid (H_3O_4)

: Sodium hydroxide (NaOH)

: Bentonite (bleaching earth) ($Al_2H_2o_{12}Si_4$)

: Crude palm oil (CPO)

: Ethanol

: Phenolphthalein

Equipment   : Vacuum pressure indicator

: Rotavapor unit

: Filter paper

: Thermometer

: 250 ml conical flask

: Magnetic stirrer with heater

: Tripod stand

: Volumetric flask

Physical refinery equipment in plant

: Continuous bleacher

: Column

: Crude distillation unit tank

: Vacuum pump

: Mixer

: Heat exchanger

: Cooling unit

: Earth tank

: Barometric condenser

: Earth-oil pump

: Earth dosing unit
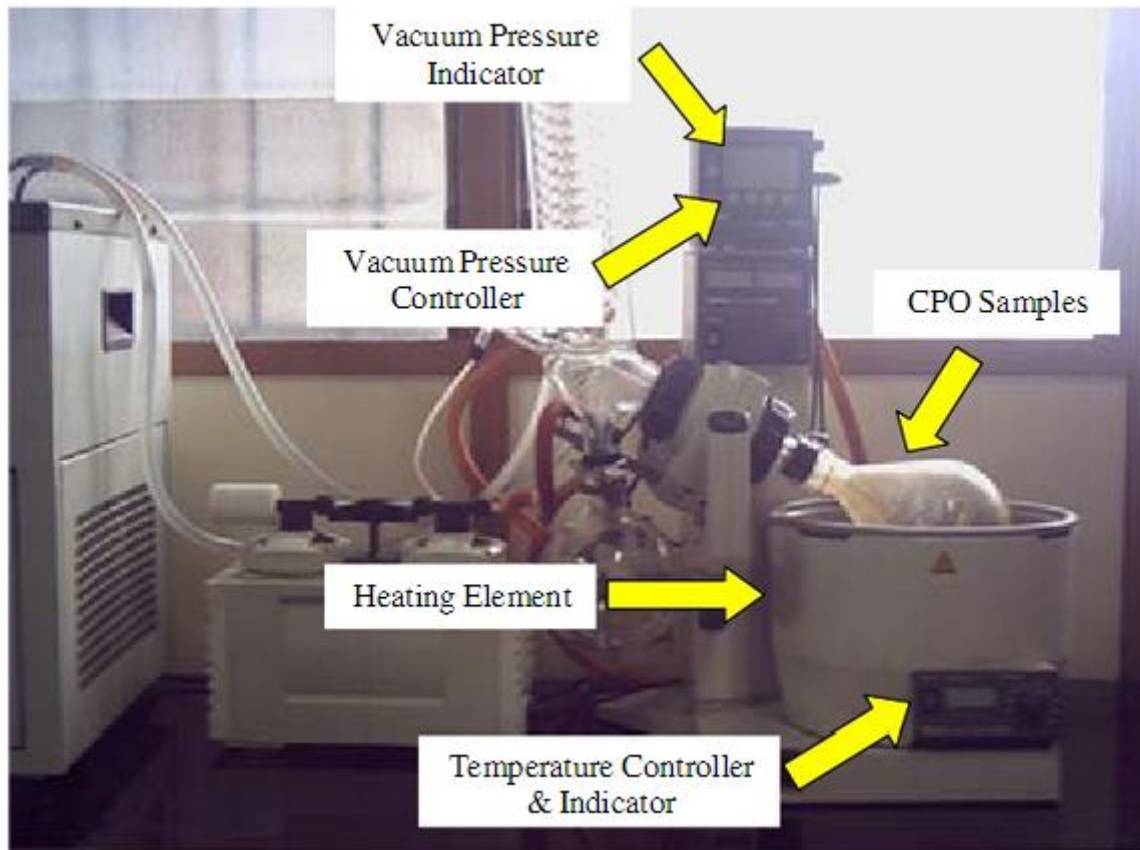
Figure 17 : Rota-Vapor Unit Used for Degumming and Bleaching Process Vacuum

3.5 Experimental steps for bleaching and degumming process

This experiment is to extract free fatty-acid in crude palm oil (CPO) by doing physical refining process. This process is chosen because it offers the advantages of higher efficiency; improve yields, less capital cost, lower operating costs, reduced processing time, immediate recovery of high purity distilled fatty acids, and simple effluent.

100 g of CPO sample is weighed.

The mixture of phosphoric acid and CPO will be heated to 90℃. Bleaching earth are added.

*The bleaching earth and phosphoric acid are added into the mixture based on the ratio in table 3.1

After the temperature is achieved,the process is carried out for 20 minutes.

The mixture is filtered.

The percentage of FFA is determined. The experiment are repeated at different temperature and time contact..

The data is analyzed.

Figure 18 : Overall General Methodology

3.5.1 Determination of Free Fatty-Acid (FFA)

10g of sample DBPO is weighed in the conical flask.

50 ml of ethanol have been neutralized by adding little drops of 0.1 N NaOH and phenolphthalein indicator.

The mixture is heated up on the hot plate for about 40C.

Then, the mixture is titrated with 0.1N NaOH until the solution change its color.

The reading in the burrete is taken.
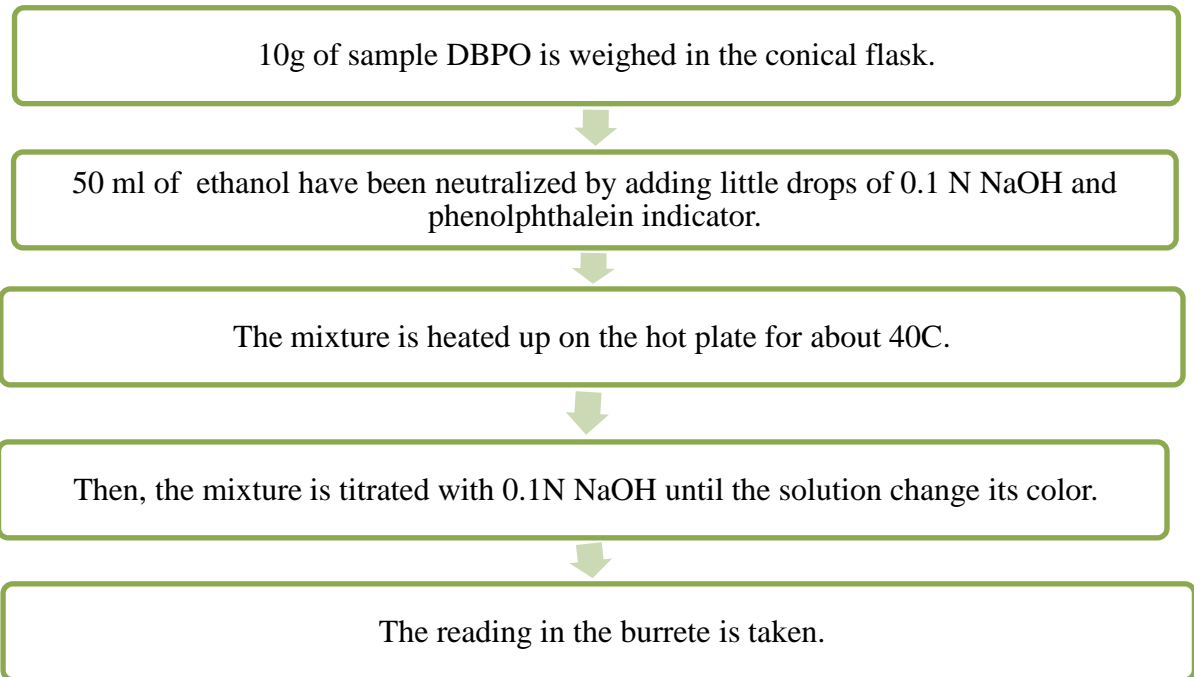
Free fatty-acid;

$$FFA = \frac{2.5 \; x \; V}{W}$$

Where W   =Weight of sample used in grams

V   = volume of NaOH use in mL
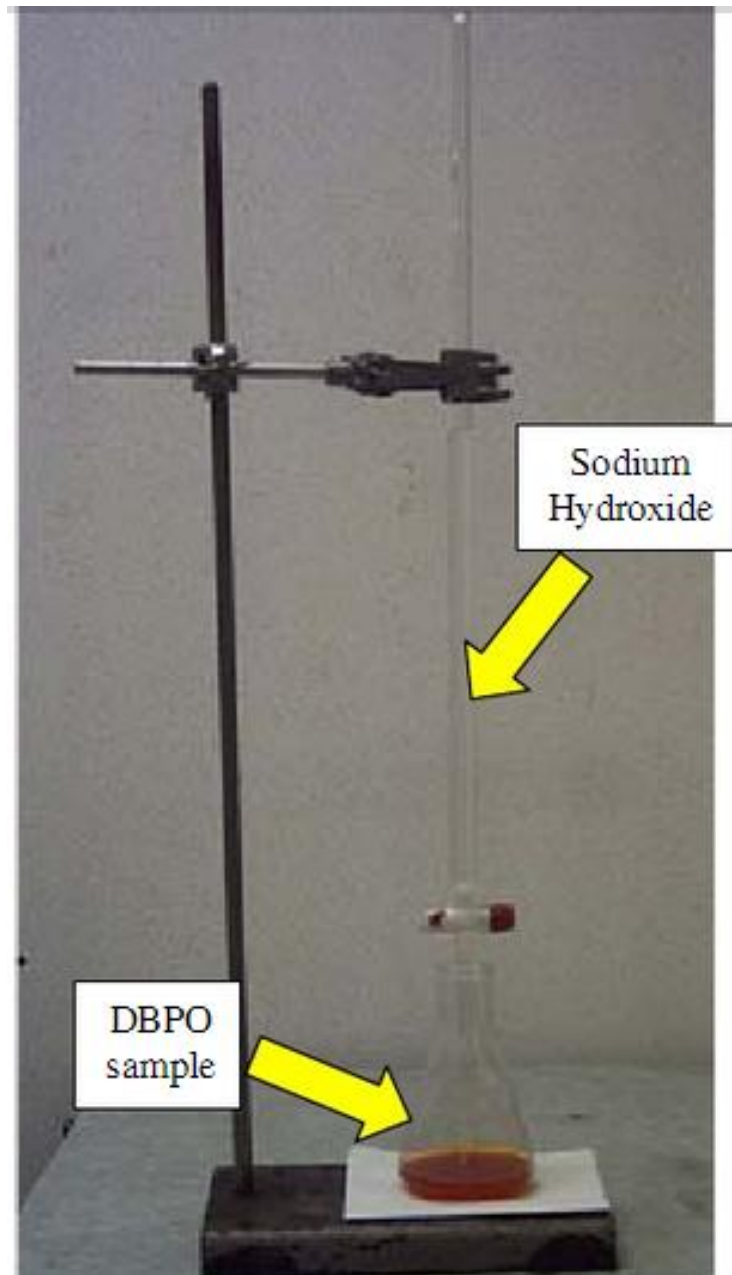
Figure 19 : Titration method for FFA experiment

CHAPTER 4

RESULT AND DISCCUSSION

4.1 Introduction

In this chapter, the results of the experiment and the discussion on the result obtained will be shown. The experiment had been done by varying the three parameter ; ratio of acid to bleaching earth, time contact during degumming and bleaching process and temperature of the process to achieve the objective of the research.

4.2 The effect of Free Fatty Acid (FFA)

One of the major effects in FFA content in RDBPO is volume of phosphoric acid added. After these impurities are absorbed by certain ratio of acid to bleaching agent, at certain temperature and different contact time, Table 4.1, the FFA content will started to decrease. The FFA content was determined by using titration method (PORIM, 2000).

Table 4.1 : Data Collection of FFA with Different Parameter

Dosage ,%wt		characterisation											
		FFA%											
Dosage ,%wt	temp	80-90℃	time contact			70-80℃	time contact			60-70℃	time contact		
acid	bleaching earth	initial	60mnt	40mnt	20mnt	initial	60mnt	40mnt	20mnt	initial	60mnt	40mnt	20mnt
0.50	0.0	3.740	4.225	4.234	4.227	3.740	4.378	4.311	4.432	3.740	4.402	4.325	4.442
0.50	1.0	3.740	4.125	3.972	4.208	3.740	4.353	4.297	4.401	3.740	4.357	4.307	4.423
0.50	1.5	3.740	4.000	3.333	4.221	3.740	4.321	4.242	4.342	3.740	4.331	4.243	4.352
0.50	2.0	3.740	3.970	3.432	4.010	3.740	4.272	4.227	4.298	3.740	4.277	4.232	4.302
1.00	0.0	3.740	5.070	4.953	5.083	3.740	5.070	5.019	5.088	3.740	5.084	5.024	5.089
1.00	0.5	3.740	4.130	4.014	4.976	3.740	4.983	4.911	5.024	3.740	5.023	4.921	5.056
1.00	1.0	3.740	3.825	3.632	4.999	3.740	4.535	4.446	5.001	3.740	4.576	4.452	5.013
1.00	1.5	3.740	3.790	3.486	4.446	3.740	4.698	4.573	4.878	3.740	4.672	4.582	4.999
1.00	2.0	3.740	3.924	3.401	4.442	3.740	4.319	4.211	4.772	3.740	4.330	4.220	4.791

From the data collected, the most optimum condition that gives lowest value of FFA was detected. Free fatty acids (FFA) are released naturally in crude palm oil (CPO) and can be increased by the action of enzymes in the palm fruit and by microbial lipases. During storage, FFA was produced by the reaction of oil with water. (M.H. Moha, and F.R. van de Voortb., 1999). Table 4.1 shows the value of FFA content after undergo process at different parameter condition. Different dosage of acid and bleaching earth added at different time contact and temperature give different value of FFA content at the end. Adsorption occurred mostly at the first minutes (20 min) of the process. The FFA losses were directly related to the bleaching earth dosage: 1-2wt% (Luis, A.M. and Nohemí, G. 2011). The degummed and bleached oil was filtered twice with Whatman filter paper #1 under vacuum as soon as possible to prevent any undesirable oxidation.
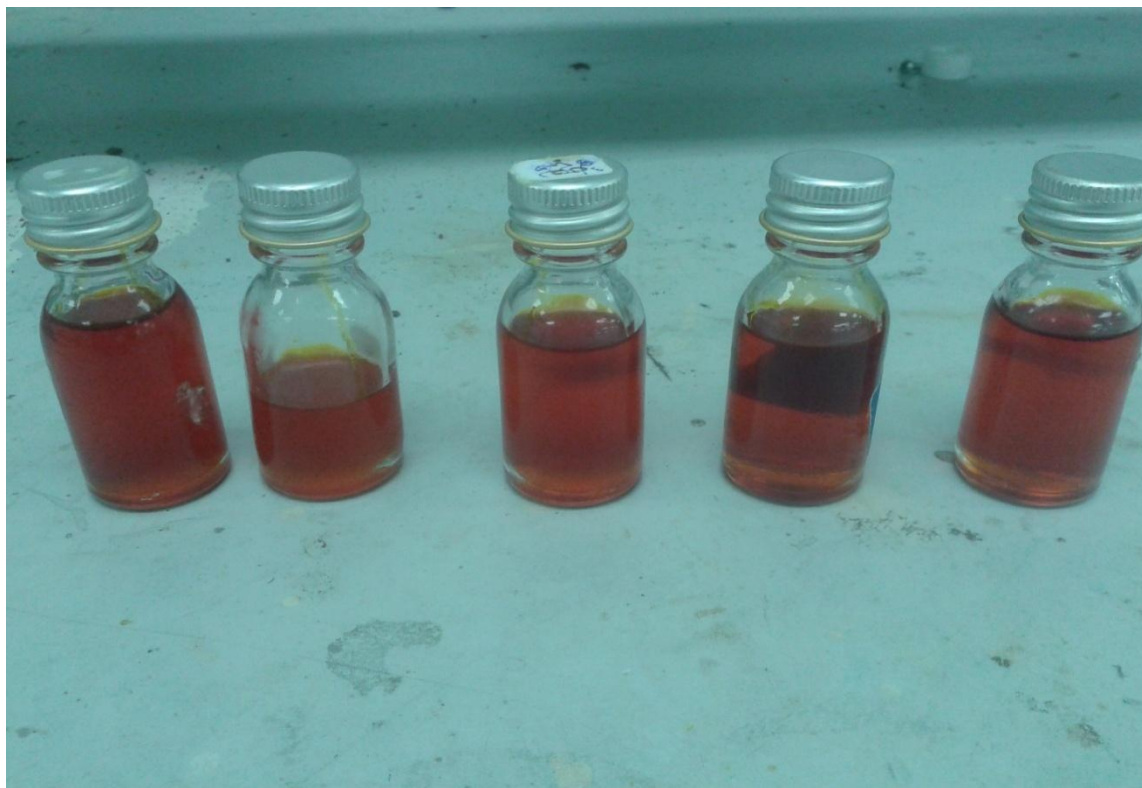
Figure 20 : Sample After Degumming and Bleaching process

Figure 20 shows the sample of CPO after degumming and bleaching process at different parameter. The colour of the sample is much lighter than CPO before the process. Most of the impurities present such as ketones and aldehydes that affect the color had been absorbed by bleaching agent.

1. *Reaction process at 80℃-90℃*
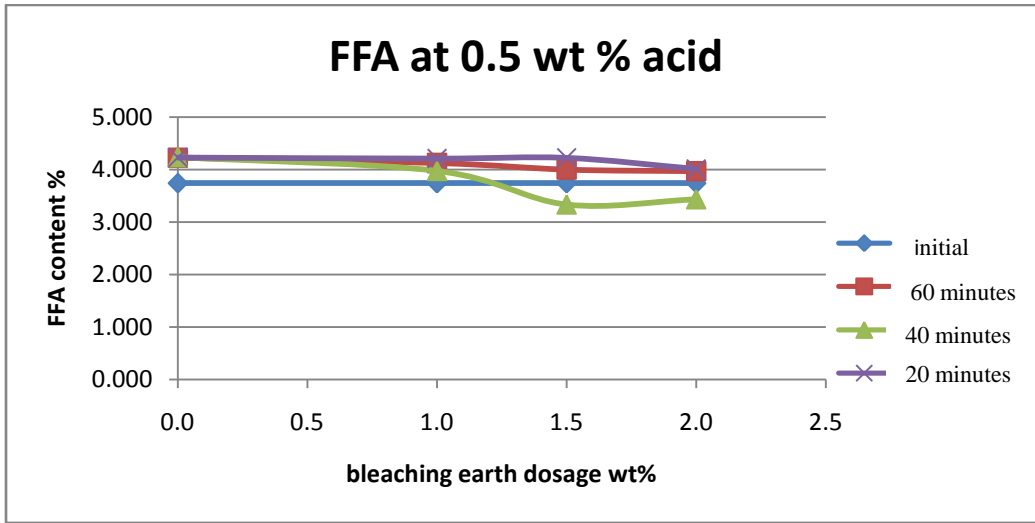
Figure 21 : FFA content at 0.5 wt % acid and 80℃-90℃
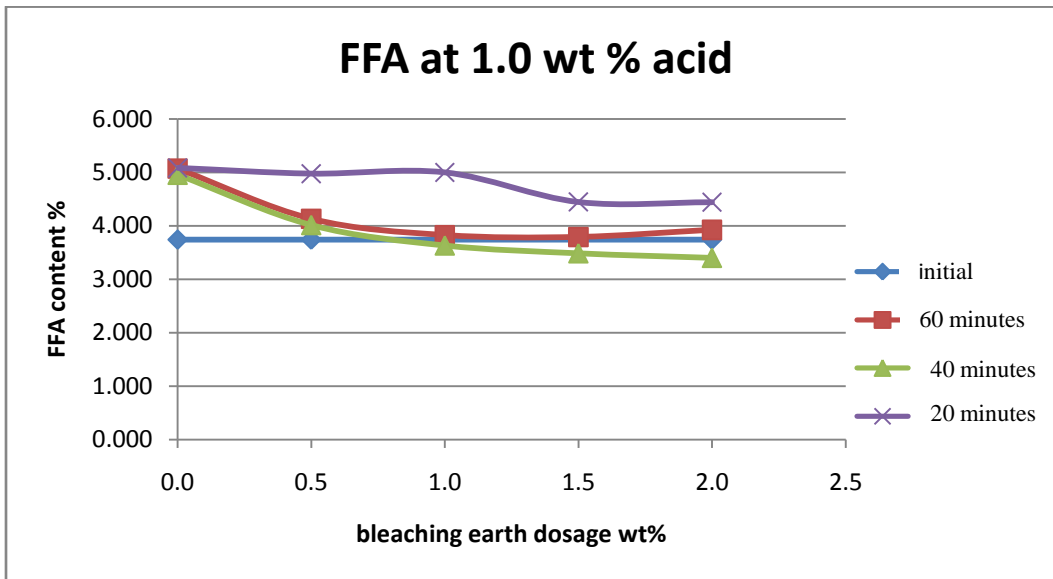
Figure 22 : FFA content at 1.0 wt % acid and 80℃-90℃

The present of FFA shows the quality of the oil present. Figure 21 and Figure 22 shows the effect on FFA content by different bleaching earth dosage and phosphoric acid percentage at temperature 80℃-90℃. Blue line indicates initial value of FFA content in the sample, red is for FFA value after 60 minutes time contact, green is 40 minute and purple is 20 minute time

contact. Initially , the process was run with the absence of bleaching earth clay, so, no adsorption process occurs. But, the FFA value seem increased from its initial value. This happened due to the acidic nature of phosphoric acid and due to hydrolysis of triglyceride occurs when heating.

Oil bleaching temperatures typically range from 90–125°C (195–257°F). Temperature affects oil viscosity and adsorption kinetics. Oil viscosity decreases with increasing temperature resulting in better dispersion of bleaching earth particles, improved clay/oil interactions, and less resistance to flow. The ability to maintain a particle in slurry suspension is inversely related to the viscosity of the oil, implying that clays with coarser particle size distributions (PSD) will require a more powerful agitation to stay in suspension and that clay with finer PSDs may more rapidly disperse into the oil at higher temperatures.

A higher temperature may give you benefits on chlorophyll removal, colour, and filtration rates, however, depending on oil type and oil quality, may in turn worsen the colour of the deodorized oil, and its oxidative stability.FFA levels in crude oils can be adversely affected by dosage of bleaching as well dosage of the phosphoric acid used.

For the next run, from both figure we can see the content of FFA is still found higher, however, after a mean time, the content of FFA started to decrease as the bleaching earth dosage and time contact increase. The reaction started to take place when 0.5 bleaching agent was added. The lowest content of FFA were detected at 1.5 wt% of bleaching earth, 0.5 wt % acid and 40 minutes times contact.

2. Reaction process at 70℃-80℃
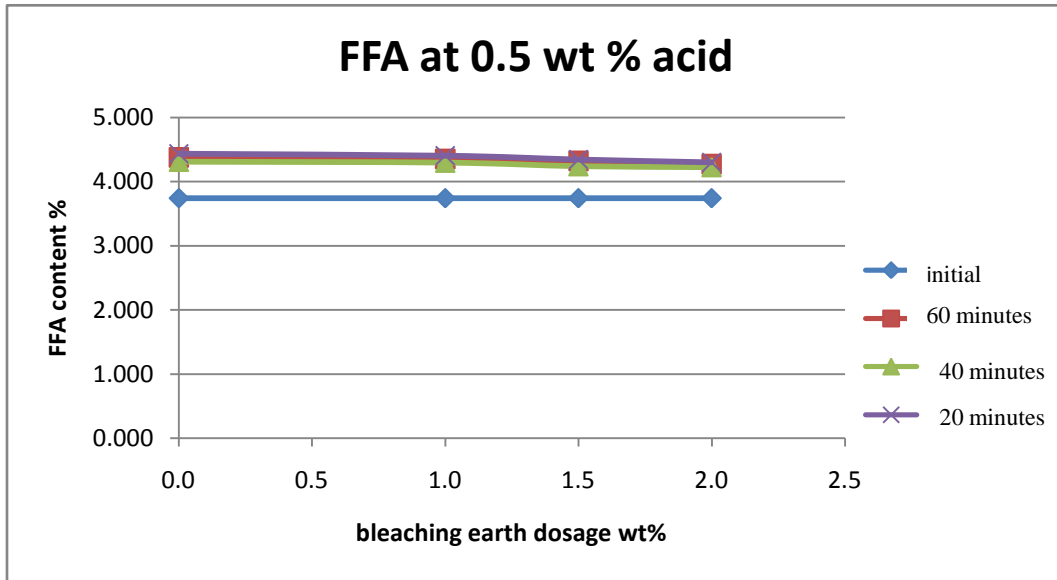
Figure 23 : FFA content at 0.5 wt % acid and 70℃-80℃
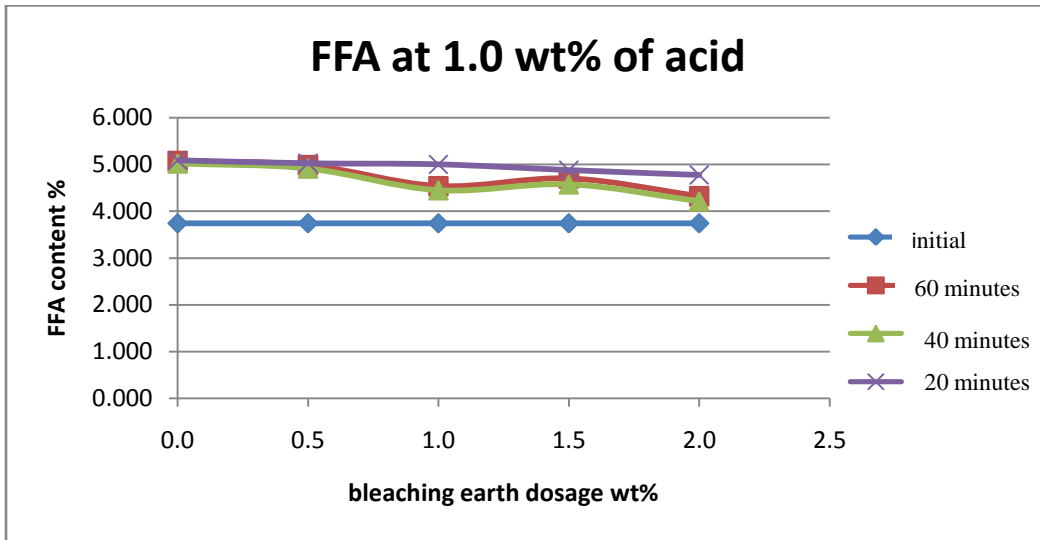
Figure 24 : FFA content at 1.0 wt % acid and 70℃-80℃

Figure 23 and Figure 24 show the processes undergo at temperature 70℃-80℃. For Figure 23, the acid use was 0.5 wt % varying with different dosage of bleaching earth at different time contact. At this condition, the obtained removal of FFA was not very high because the present of low acid. From the graph, it also show increment initially where when there is no bleaching earth added to the degumming and bleaching treatment (means adsorption process); the rise in FFA

content is quite high. This rise of FFA is also attributed to the higher acidity of phosphoric used during the treatment which is 1.0 wt%. From the results it is obvious that the degumming result was better when the phosphoric is at 0.5wt% and at 40 minutes.

3. *Reaction process at 60℃-70℃*
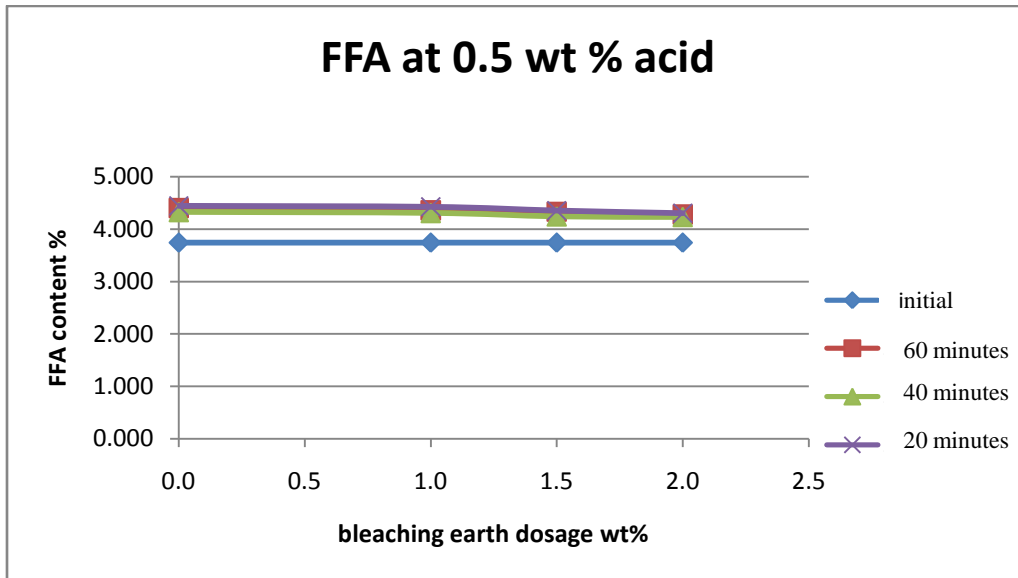
Figure 25 : FFA content at 0.5 wt % acid and 60℃-70℃
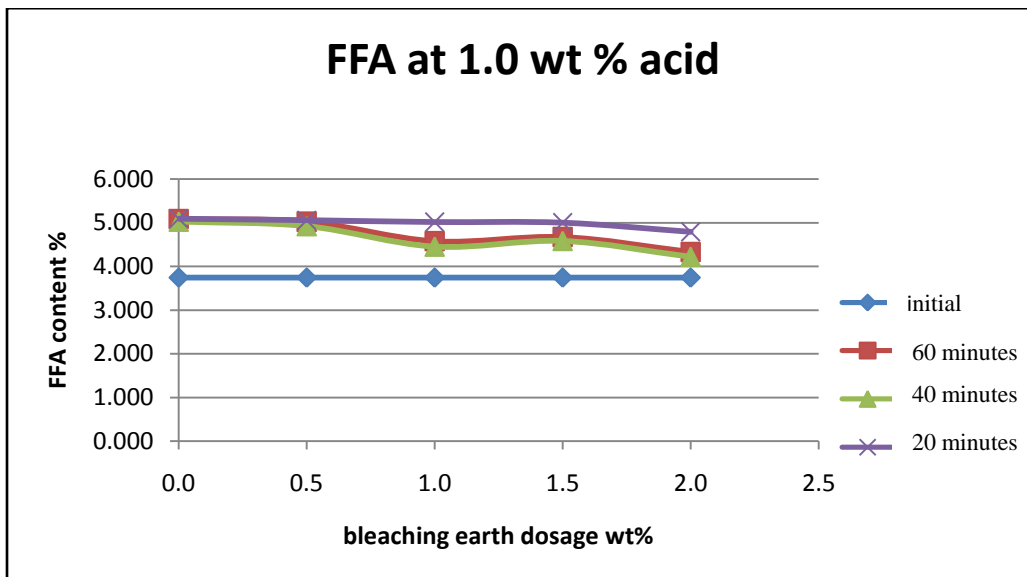
Figure 26 : FFA content at 1.0 wt % acid and 60℃-70℃

As shown in Figure 25 and Figure 26 , they have similar trend with the previous graph. The degumming result was influenced mainly by the acid and bleaching earth dosage and less by the temperature or the contact time. Acid dosage together with the present of bleaching agent has high effect on FFA content. Varying the temperature from 20 to 60 minutes removed 10-15% of the FFA at 2.0 bleaching earth dosage.

For Figure 25, the graph was plot at 0.5wt% of phosphoric present at different dosage of bleaching earth in CPO. At different time contact it's nearly seem as no decreasing of FFA content after the process take place. Meanwhile for Figure 26, there is a gap between 20 minute and 40 minute time contact. The acid dosages added seem sufficient enough to react with bleaching agent to remove the FFA. Treatment of palm oil with bleaching with phosphoric acid followed by bleaching earth gave residual iron levels much lower than bleaching earth alone or phosphoric alone(Rohani , 2006).

From the observation, the degumming result was improved with longer contact time (40 min). The degumming for a 20 min contact time was examined, but it did not contribute to a significantly improved purification of the oil. While at 60 minute, the reaction started to slow down back. The reduction of FFA slow even when the present of bleaching agent because the limited off active site on bleaching agent. It is generally accepted and understood that bleaching agent have a limited number of active sites or "parking places" for contaminants. The filling of these sites progresses well provided the amount of contaminant is matched by adequate pore space or volume and surface area. . But, excessive contact times may result oxidation and reduction reactions to occur on the active surface of the bleaching clay and no adsorption occurs.

As the proverbial parking garage nears capacity, the level of residual contaminants is so low that it is not worth spending additional production time to find a place to park the remaining residual contaminants.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In this research, the Optimization of Bleaching Earth and Extraction of Free Fatty Acid (FFA) in Palm Oil Refinery Process have been completed. The analysis is to determine the percentage of FFA content after degumming and bleaching process at the most optimize condition with good. These results gives some valuable information that would suggest some improvements to the degumming and bleaching practices in Malaysia's palm oil refineries. Thus, there are several conclusions that can be making**:**

1. The treatment of palm oil with bleaching with phosphoric acid followed by bleaching earth gave residual iron levels much lower than bleaching earth alone or phosphoric alone.

2. The optimal operating conditions for this process operation of degumming and bleaching processes is about 80℃-90℃ and 40 minutes of contact time for a palm oil refinery that operating using physical refining method.

3. The removal or reduction level FFA are mainly due to bleaching treatment process, whereas degumming treatment is responsible for influencing the increment in FFA level in CPO. For bleaching processes, there are essential to ensure the removal process run effectively.

4. Based on the experimental analysis, the range of suitable dosage of phosphoric acid to be used is 0.5 - 1.0wt% and bleaching earth dosage is between 1.0 - 2.0 wt %.

5.2   Recommendation for Future Study

Based on the result and discussion of the experiment, several important recommendation should be carried out in operating condition during the degumming and bleaching process such as;

1. The FFA reduction can be increase with the suitable OSN membrane to separate FFA from palm oil in palm oil industry. The concept is rather simple and yet cost effective as it can be operated at ambient temperature. It also has no damage to minor compounds in palm oil.
2. From the observation, the type of bleaching agent use also must suitable with the crude palm oil feed in order to improve the quality of the CPO.
3. After degumming and bleaching process, the filtration must take place directly as soon as possible to avoid more oxidation in the palm oil.

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APPENDIX A

Calculation of Free Fatty Acid (FFA)

At temperature 80-90℃

    I.   60 minutes

1st sample:	weight of CPO sample	: 10.2 g
	Volume NaOH	: 16.83 mL
	FFA %	$: \dfrac{2.56 \times 16.83}{10.2} = 4.225\%$

2nd sample:	weight of CPO sample	: 10.187 g
	V NaOH	: 16.41 mL
	FFA %	$: \dfrac{2.56 \times 16.41}{10.187} = 4.125\%$

3rd sample:	weight of CPO sample	: 10.2g
	V NaOH	: 15.94 mL
	FFA %	$: \dfrac{2.56 \times 15.94}{10.2} = 4.000\%$

4th sample:	weight of CPO sample	: 10.18g
	V NaOH	: 15.78 mL
	FFA %	$: \dfrac{2.56 \times 15.78}{10.18} = 3.970\%$

5th sample:	weight of CPO sample	: 10.187 g
	V NaOH	: 20.17 mL
	FFA %	$: \dfrac{2.56 \times 20.17}{10.187} = 5.070\%$

6th sample:	weight of CPO sample	: 10.187g
	V NaOH	: 16.43 mL
	FFA %	$: \dfrac{2.56 \times 16.43}{10.187} = 4.130\%$

7th sample: weight of CPO sample   : 10.154 g

                V NaOH   : 15.17 mL

                FFA %   : $\dfrac{2.56 \times 15.17}{10.154} = 3.825\ \%$

8th sample: weight of CPO sample   : 10.141 g

                V NaOH   : 15.01 mL

                FFA %   : $\dfrac{2.56 \times 15.01}{10.141} = 3.790\%$

9th sample: weight of CPO sample   : 10.221 g

                V NaOH   : 15.66 mL

                FFA %   : $\dfrac{2.56 \times 15.66}{10.221} = 3.924\ \%$

  II.   <u>40 minutes</u>

1st sample: weight of CPO sample   : 10.3 g

                Volume NaOH   : 17.03 mL

                FFA %   : $\dfrac{2.56 \times 17.03}{10.3} = 4.234\%$

2nd sample: weight of CPO sample   : 10.112 g

                V NaOH   : 15.70 mL

                FFA %   : $\dfrac{2.56 \times 15.70}{10.112} = 3.972\%$

3rd sample: weight of CPO sample   : 10.125g

                V NaOH   : 13.18 mL

                FFA %   : $\dfrac{2.56 \times 13.18}{10.125} = 3.333\ \%$

4th sample: weight of CPO sample   : 10.052 g

                V NaOH   : 13.48 mL

                FFA %   : $\dfrac{2.56 \times 13.48}{10.052} = 3.432\ \%$

5th sample: weight of CPO sample   : 10.23 g

V NaOH   : 19.79 mL

FFA % $: \dfrac{2.56 \times 19.79}{10.23} = 4.953\ \%$

6th sample: weight of CPO sample   : 10.17g

V NaOH   : 15.95 mL

FFA % $: \dfrac{2.56 \times 15.95}{10.17} = 4.014\%$

7th sample: weight of CPO sample   : 10.1 g

V NaOH   : 14.33 mL

FFA % $: \dfrac{2.56 \times 14.33}{10.1} = 3.632\ \%$

8th sample: weight of CPO sample   : 10.087 g

V NaOH   : 13.74 mL

FFA % $: \dfrac{2.56 \times 13.74}{10.141} = 3.790\%$

9th sample: weight of CPO sample   : 10.222 g

V NaOH   : 13.58 mL

FFA % $: \dfrac{2.56 \times 13.58}{10.222} = 3.486\ \%$

III.   <u>20 minutes</u>

1st sample: weight of CPO sample   : 10.06 g

Volume NaOH   : 16.61 mL

FFA % $: \dfrac{2.56 \times 16.61}{10.06} = 4.227\%$

2nd sample: weight of CPO sample   : 10.023 g

V NaOH   : 16.47 mL

FFA % $: \dfrac{2.56 \times 16.47}{10.023} = 4.208\%$

3rd sample: weight of CPO sample   : 10.321g

                  V NaOH   : 17.02 mL

                  FFA % $: \dfrac{2.56 \times 17.02}{10.321} = 4.221\ \%$

4th sample: weight of CPO sample   : 10.098 g

                  V NaOH   : 15.82 mL

                  FFA % $: \dfrac{2.56 \times 15.82}{10.098} = 4.010\ \%$

5th sample: weight of CPO sample   : 10.091 g

                  V NaOH   : 20.05 mL

                  FFA % $: \dfrac{2.56 \times 20.05}{10.091} = 5.083\ \%$

6th sample: weight of CPO sample   : 10.08 g

                  V NaOH   : 19.60 mL

                  FFA % $: \dfrac{2.56 \times 19.60}{10.08} = 4.976\ \%$

7th sample: weight of CPO sample   : 10.21 g

                  V NaOH   : 19.94 mL

                  FFA % $: \dfrac{2.56 \times 19.94}{10.21} = 4.999\ \%$

8th sample: weight of CPO sample   : 10.197 g

                  V NaOH   : 17.71 mL

                  FFA % $: \dfrac{2.56 \times 17.71}{10.197} = 4.446\ \%$

9th sample: weight of CPO sample   : 10.14 g

                  V NaOH   : 17.6 mL

                  FFA % $: \dfrac{2.56 \times 17.6}{10.14} = 4.442\ \%$

At temperature 70-80℃

I.   60 minutes

1st sample: weight of CPO sample   : 10.06 g

              V NaOH   : 17.12 mL

              FFA %   : $\frac{2.56 \times 17.12}{10.06}$ =4.378 %

2nd sample: weight of CPO sample   : 10.023 g

              V NaOH   : 17.14 mL

              FFA %   : $\frac{2.56 \times 17.14}{10.023}$ =4.353%

3rd sample: weight of CPO sample   : 10.321 g

              V NaOH   : 17.23 mL

              FFA %   : $\frac{2.56 \times 17.23}{10.321}$ = 4.321%

4th sample: weight of CPO sample   : 10.098 g

              V NaOH   : 17.52 mL

              FFA %   : $\frac{2.56 \times 17.52}{10.098}$ = 4.272 %

5th sample: weight of CPO sample   : 10.091 g

              V NaOH   : 19.98 mL

              FFA %   : $\frac{2.56 \times 19.98}{10.091}$ = 5.070 %

6th sample: weight of CPO sample   : 10.08 g

              V NaOH   : 19.62 mL

              FFA %   : $\frac{2.56 \times 19.62}{10.08}$ =4.983 %

7th sample: weight of CPO sample   : 10.21 g

              V NaOH   : 18.1 mL

              FFA %   : $\frac{2.56 \times 18.1}{10.21}$ = 4.535 %

8th sample: weight of CPO sample   : 10.197 g

            V NaOH   : 18.71 mL

            FFA %   : $\frac{2.56 \times 18.71}{10.197} = 4.698\ \%$

9th sample: weight of CPO sample   : 10.14 g

            V NaOH   : 17.11 mL

            FFA %   : $\frac{2.56 \times 17.11}{10.14} = 4.319\ \%$

II.   <u>40 minutes</u>

1st sample: weight of CPO sample   : 10.06 g

            V NaOH   : 16.94 mL

            FFA %   : $\frac{2.56 \times 16.94}{10.06} = 4.311\%$

2nd sample: weight of CPO sample   : 10.023 g

            V NaOH   : 16.83 mL

            FFA %   : $\frac{2.56 \times 16.83}{10.023} = 4.297\%$

3rd sample: weight of CPO sample   : 10.321 g

            V NaOH   : 17.11 mL

            FFA %   : $\frac{2.56 \times 17.11}{10.321} = 4.24\%$

4th sample: weight of CPO sample   : 10.098 g

            V NaOH   : 16.67 mL

            FFA %   : $\frac{2.56 \times 16.67}{10.098} = 4.227\%$

5th sample: weight of CPO sample   : 10.091 g

            V NaOH   : 19.78 mL

            FFA %   : $\frac{2.56 \times 19.78}{10.091} = 5.019\ \%$

6th sample: weight of CPO sample   : 10.08 g

V NaOH   : 19.34 mL

FFA % $: \dfrac{2.56 \times 19.34}{10.08} = 4.911\ \%$

7th sample: weight of CPO sample   : 10.21 g

V NaOH   : 17.73 mL

FFA % $: \dfrac{2.56 \times 17.73}{10.21} = 4.446\%$

8th sample: weight of CPO sample   : 10.197 g

V NaOH   : 18.22 mL

FFA % $: \dfrac{2.56 \times 18.22}{10.197} = 4.573\ \%$

9th sample: weight of CPO sample   : 10.14 g

V NaOH   : 16.68 mL

FFA % $: \dfrac{2.56 \times 16.68}{10.14} = 4.211\ \%$

III.   <u>20 minutes</u>

1st sample: weight of CPO sample   : 10.06 g

V NaOH   : 17.34 mL

FFA % $: \dfrac{2.56 \times 17.34}{10.06} = 4.432\ \%$

2nd sample: weight of CPO sample   : 10.023 g

V NaOH   : 17.33 mL

FFA % $: \dfrac{2.56 \times 17.33}{10.023} = 4.401\%$

3rd sample: weight of CPO sample   : 10.321 g

V NaOH   : 17.32 mL

FFA % $: \dfrac{2.56 \times 17.32}{10.321} = 4.342\ \%$

4th sample: weight of CPO sample   : 10.098 g

V NaOH   : 17.63mL

FFA %   : $\dfrac{2.56 \times 17.63}{10.098} = 4.298\ \%$

5th sample: weight of CPO sample   : 10.091 g

V NaOH   : 20.05 mL

FFA %   : $\dfrac{2.56 \times\ 20.05}{10.091} = 5.088\ \%$

6th sample: weight of CPO sample   : 10.08 g

V NaOH   : 19.78 mL

FFA %   : $\dfrac{2.56 \times 19.78}{10.08} = 5.024\ \%$

7th sample: weight of CPO sample   : 10.21 g

V NaOH   : 19.95 mL

FFA %   : $\dfrac{2.56 \times 19.95}{10.21} = 5.001\ \%$

8th sample: weight of CPO sample   : 10.197 g

V NaOH   : 19.43 %

FFA %   : $\dfrac{2.56 \times 19.43}{10.197} = 4.878\ \%$

9th sample: weight of CPO sample   : 10.14 g

V NaOH   : 18.91 %

FFA %   : $\dfrac{2.56 \times 18.91}{10.14} = 4.772\ \%$

At temperature 60-70°C

I.   60 minutes

1st sample: weight of CPO sample   : 10.2 g

V NaOH   : 17.54 mL

FFA %   : $\dfrac{2.56 \times\ 17.54}{10.2} = 4.402\ \%$

2nd sample: weight of CPO sample   : 10.22 g

V NaOH   : 17.39 mL

FFA %   : $\dfrac{2.56 \times 17.39}{10.22}$ = 4.357 %

3rd sample: weight of CPO sample   : 10.51 g

V NaOH   : 17.78 mL

FFA %   : $\dfrac{2.56 \times 17.78}{10.51}$ = 4.331 %

4th sample: weight of CPO sample   : 10.078 g

V NaOH   : 16.84 mL

FFA %   : $\dfrac{2.56 \times 16.84}{10.078}$ = 4.277 %

5th sample: weight of CPO sample   : 10.075 g

V NaOH   : 20.01 mL

FFA %   : $\dfrac{2.56 \times 20.01}{10.075}$ = 5.084 %

6th sample: weight of CPO sample   : 10.18 g

V NaOH   : 19.97 mL

FFA %   : $\dfrac{2.56 \times 19.97}{10.18}$ = 5.023 %

7th sample: weight of CPO sample   : 10.15 g

V NaOH   : 18.14 mL

FFA %   : $\dfrac{2.56 \times 18.14}{10.15}$ = 4.576 %

8th sample: weight of CPO sample   : 10.23 g

V NaOH   : 18.67 mL

FFA %   : $\dfrac{2.56 \times 18.67}{10.23}$ = 4.672 %

9th sample: weight of CPO sample   : 10.31 g

                V NaOH   : 17.44 mL

                FFA %   : $\dfrac{2.56 \times 17.44}{10.31} = 4.330\%$

II.   40 minutes

1st sample: weight of CPO sample   : 10.2 g

                V NaOH   : 16.92 mL

                FFA %   : $\dfrac{2.56 \times 16.92}{10.2} = 4.325\%$

2nd sample: weight of CPO sample   : 10.22 g

                V NaOH   : 16.96 mL

                FFA %   : $\dfrac{2.56 \times 16.96}{10.22} = 4.307\%$

3rd sample: weight of CPO sample   : 10.51 g

                V NaOH   : 16.92 mL

                FFA %   : $\dfrac{2.56 \times 16.92}{10.51} = 4.243\%$

4th sample: weight of CPO sample   : 10.078 g

                V NaOH   : 17.36 mL

                FFA %   : $\dfrac{2.56 \times 17.36}{10.078} = 4.232\%$

5th sample: weight of CPO sample   : 10.075 g

                V NaOH   : 19.80 mL

                FFA %   : $\dfrac{2.56 \times 19.80}{10.075} = 5.024\ \%$

6th sample: weight of CPO sample   : 10.18 g

                V NaOH   : 19.38 mL

                FFA %   : $\dfrac{2.56 \times 19.38}{10.18} = 4.921\ \%$

7th sample: weight of CPO sample   : 10.15 g

V NaOH   : 17.76 mL

FFA %   : $\dfrac{2.56 \times 17.76}{10.15} = 4.452\ \%$

8th sample: weight of CPO sample   : 10.23 g

V NaOH   : 18.25 mL

FFA %   : $\dfrac{2.56 \times 18.25}{10.23} = 4.582\ \%$

9th sample: weight of CPO sample   : 10.31 g

V NaOH   : 16.72 mL

FFA %   : $\dfrac{2.56 \times 16.72}{10.31} = 4.220\ \%$

III.   <u>20 minutes</u>

1st sample: weight of CPO sample   : 10.2 g

V NaOH   : 17.46 mL

FFA %   : $\dfrac{2.56 \times 17.46}{10.2} = 4.442\ \%$

2nd sample: weight of CPO sample   : 10.22 g

V NaOH   : 17.32 mL

FFA %   : $\dfrac{2.56 \times\ 17.32}{10.22} = 4.423\%$

3rd sample: weight of CPO sample   : 10.51 g

V NaOH   : 17.55 mL

FFA %   : $\dfrac{2.56 \times 17.55}{10.51} = 4.352\ \%$

4th sample: weight of CPO sample   : 10.078 g

V NaOH   : 16.97 mL

FFA %   : $\dfrac{2.56 \times 16.97}{10.078} = 4.302\ \%$

5th sample: weight of CPO sample   : 10.075 g

V NaOH   : 20.06 mL

FFA % $: \dfrac{2.56 \times 20.06}{10.075} = 5.089\ \%$

6th sample: weight of CPO sample   : 10.18 g

V NaOH   : 19.91 mL

FFA % $: \dfrac{2.56 \times 19.791}{10.18} = 5.056\ \%$

7th sample: weight of CPO sample   : 10.15 g

V NaOH   : 19.99 mL

FFA % $: \dfrac{2.56 \times 19.99}{10.15} = 5.013\ \%$

8th sample: weight of CPO sample   : 10.23 g

V NaOH   : 19.91 %

FFA % $: \dfrac{2.56 \times 19.91}{10.23} = 4.999\ \%$

9th sample: weight of CPO sample   : 10.31 g

V NaOH   : 18.98 %

FFA % $: \dfrac{2.56 \times 18.98}{10.31} = 4.791\ \%$

Project Activities	w1	w2	w3	w4	w5	w6	w7	w8	w9	w10	w11	w12	w13	w14	w15	w16	w17
Visit Factory		■			■					■							
literature value	■	■	■	■	■	■	■	■	■								
Detail Research on Methodology	■	■	■	■	■	■	■	■	■	■							
1)Determine Percentage of FFA Content					■	■	■	■	■	■	■	■					
Preparation of Experiment					■	■	■	■	■	■	■	■					
Experimental Work					■	■	■	■	■	■	■	■	■				
Data Analysis And Report Writing								■	■	■	■	■	■	■			
Technical Paper Submission														■			
Draft Thesis Submission														■			
Correction Of Report Writing														■	■	■	
Final Presentation																	■
Compile And Submit The Report																	■

GANTT CHART /PROJECT SCHEDULE

APPENDIX B