



THE EFFECT OF DRYING TEMPERATURE TOWARDS QUANTITY AND QUALITY
OF PALM OIL EXTRACTED FROM DRIED DECANTER CAKE

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ABSTRACT

The production of palm oil by the palm oil refinery produces a large quantity of waste. One of the wastes produced is palm oil decanter cake. Decanter cake is generated by the separation process of the suspended solids from crude palm oil in a decanter. Usually, the generated decanter cake still contains about 8% of oil in it. So, this research described the extraction of the residual oil from the decanter cake and its quality resulting from the pretreatment applied on the decanter cake before the extraction. There are three stages involved which are the pretreatment of the decanter cake, the extraction of oil from the pretreated decanter cake, and the analysis of the extracted oil. The pretreatment method is the drying of the decanter cake to remove the moisture content of the decanter cake for optimal extraction performance. The decanter cake samples were dried at different temperature in the range of 60°C to 120°C. All dried decanter cake samples were then analyzed using Thermal Gravimetric Analysis to determine the water, volatile compound and the fixed carbon content of each dried decanter cake. Base on the weight loss profile, the samples showed only slight differences in water, volatile compound and fixed carbon content. The extraction of the oil from the decanter cake was done with Soxhlet extraction method using hexane as solvent. The decanter cake dried at 100°C sample gives the highest oil yield. The quality of the extracted oil was analyzed using acid and iodine value analysis. The oil extracted from dried decanter cake of 60°C has the highest acid value. The acid value decreases with increase in drying temperature and reach a constant value for oil from decanter cake dried at 100°C. The iodine value of extracted oil initially decreases with drying temperature and reach a minimum for sample dried at 100°C and increases at higher drying temperature. These result suggested the optimum temperature for drying under the condition of this study is 100°C. The range of the iodine value obtained was 40-50. The iodine value for the crude palm oil and cooking oil are 13.8 and 55.0.

ABSTRAK

Dalam pengeluaran minyak sawit di kilang penapisan minyak sawit, terdapat kuantiti sisa buangan yang banyak dihasilkan. Salah satu sisa buangan yang dihasilkan ialah kek *decanter* minyak sawit. Kek *decanter* dihasilkan oleh proses pemisahan pepejal terampai daripada minyak sawit mentah oleh *decanter*. Biasanya, kek *decanter* yang dihasilkan masih mengandungi kira-kira 8% minyak di dalamnya. Jadi, kajian ini menjelaskan tentang pengekstrakan baki minyak dari kek *decanter* dan kesan prarawatan yg dikenakan terhadap kek *decanter* sebelum pengekstrakan terhadap kualiti minyak yang diekstrak daripadanya. Terdapat tiga peringkat yang terlibat iaitu prarawatan kek *decanter*, pengekstrakan kek *decanter* yang telah dirawat, dan analisis minyak yang telah diekstrak. Kaedah prarawatan yang digunakan ialah pengeringan kek *decanter* untuk membuang kandungan lembapan di dalam kek *decanter* untuk mengoptimalkan prestasi pengekstrakan. Sampel kek *decanter* dikeringkan pada suhu berbeza dalam julat 60 °C hingga 120 °C. Semua sampel kek *decanter* kering dianalisis menggunakan *Thermal Gravimetric Analysis (TGA)* untuk menentukan kandungan air, bahan meruap dan karbon tetap dalam setiap kek *decanter* kering. Berdasarkan profil pengurangan berat, semua sampel menunjukkan hanya sedikit perbezaan dalam kandungan air, bahan meruap dan karbon tetap yang tinggal selepas dikeringkan. Pengeluaran minyak dari kek *decanter* dilakukan menggunakan kaedah pengekstrakan Soxhlet dengan heksana sebagai pelarut. Sampel kek *decanter* yang dikeringkan pada 100 °C memberikan hasil minyak yang paling tinggi. Kualiti setiap minyak yang diekstrak daripada setiap sampel kek *decanter* kering dianalisis menggunakan analisis nilai asid dan nilai iodine. Minyak yang diekstrak daripada kek *decanter* yang dikeringkan pada 60 °C mempunyai nilai asid tertinggi. Nilai asid menurun dengan kenaikan suhu pengeringan dan mencapai nilai sekata untuk minyak yang diekstrak daripada cake *decanter* yang dikeringkan pada suhu 100°C. Nilai iodine minyak yang diekstrak menurun dengan suhu pengeringan pada awalnya dan mencapai nilai minimum untuk sampel yang dikeringkan pada suhu 100°C dan meningkat pada suhu pengeringan lebih tinggi. Hasil ini mencadangkan suhu optima untuk pengeringan dibawah keadaan kajian ialah 100°C. Julat nilai iodine yang diperolehi adalah diantara 40-50. Nilai iodine untuk minyak sawit mentah dan minyak masak adalah 13.8 dan 55.0.

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LIST OF SYMBOLS

b_{eq}	Amount of titrant (ml) consumed by 1 ml spiking solution at the equivalent point
m	Mass of sample
m_b	Mass of sample before drying
m_a	Mass of sample after drying
N	Normality
V_a	Burette reading after titration
V_b	Burette reading before titration
V_b	Volume of titrant for blank
V_s	Volume of titrant for sample
V_{eq}	Amount of titrant
W_{KHP}	Amount (g) of KHP in 50 ml of KHP standard solution

LIST OF ABBREVIATIONS

BOD	Biochemical oxygen demand
CP	Crude protein
CPKO	Crude palm kernel oil
CPO	Crude palm oil
DM	Dry matter
EFB	Empty fruit bunch
FAs	Fatty acids
FFA	Free fatty acid
FFB	Fresh fruit bunch
KHP	Potassium Hydrogen Phtalate
KOH	Potassium hydroxide
ME	Metabolisable energy
PKC	Palm kernel cake
POME	Palm oil mill effluent
POS	Palm oil sludge
PPF	Palm press fiber

CHAPTER 1

INTRODUCTION

1.1 Research overview

In 1800's, the oil palm tree or *Elaeis guineensis* which originated from West Africa was introduced in Malaysia by the British as an ornamental plant. Now, palm oil is one of the key economic growth drivers in Malaysia. Malaysia is one of world's largest producers of palm oil. Malaysia currently accounts for 39 % of world palm oil production and 44% of world exports. Our palm oil is mostly exported to China, the European Union, Pakistan, United States and India.

Oil palm produces palm oil from extraction process of palm fruit in the mills and this process is called milling. Milling process includes various processes such as summarized in Figure 1.1. The oil palm produces two types of oils which are crude palm oil (CPO) from the fibrous mesocarp and crude palm kernel oil from the kernels as shown in Figure 1.2. Besides producing these two oils, oil palm industry produces a wide variety of wastes in large quantities. There are liquid wastes and the solid wastes. The liquid waste is known as the palm oil mill effluent (POME) which is generated mainly from oil extraction, washing and cleaning processes in the mill. It contains cellulosic material, fat, oil, and grease (Agamuthu, 1995). While the solid waste are the leaves, trunk, decanter cake, empty fruit bunches, seed shells and fibre from the mesocarp. Fiber, shell, decanter cake and empty fruit bunch (EFB) accounts for 30, 6, 3 and 28.5% respectively of the fresh fruit bunch (FFB) processing (Pleanjai et al., 2004).

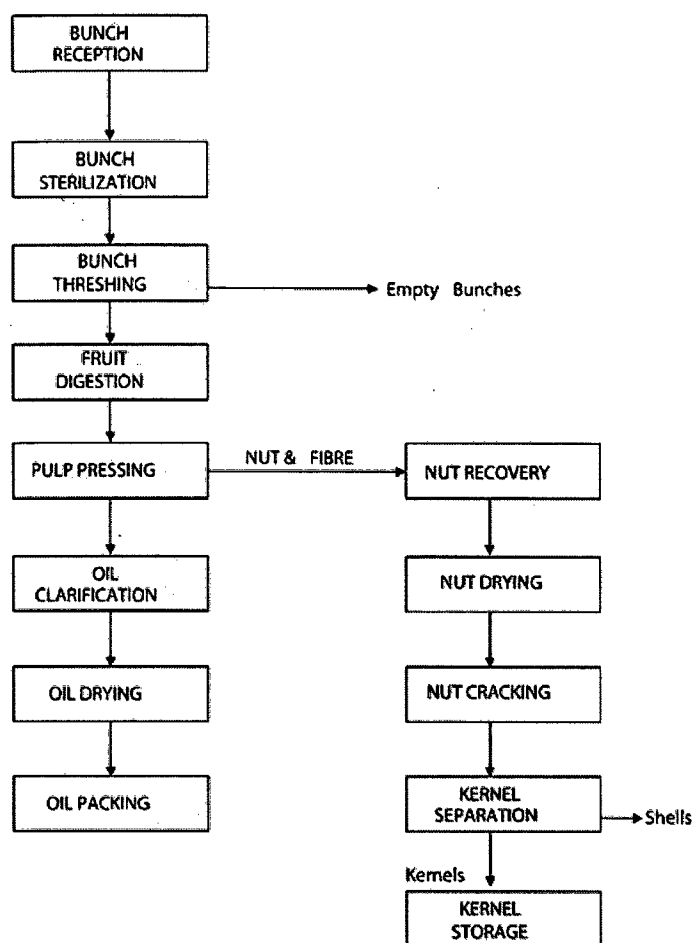


Figure 1.1: Palm oil milling process

Source: Poku, 2002

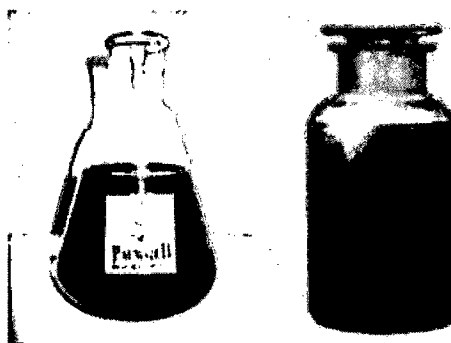


Figure 1.2: Crude palm oil (CPO) and crude palm kernel oil (CPKO)

Problems arising from the production of the palm oil is the wastes generated during the production process of the palm oil. Discharging the effluents may results in pollution and might deteriote the surrounding environment. Treatment of POME is necessary to avoid environmental pollution. There is a growing interest in composting as well as vermicomposting process (Rupani et. al, 2010). Palm kernel cake can be processed into animal feed and chicken feed (Ismail, 2004). The fibre produced is mostly used as solid fuel for boilers in the palm oil mills, while shells are sold as solid fuel to other industries. (Paepatung et. al, 2006). EFB, with a high moisture content of 60–70%, are difficult to use as fuel for power boilers. Partial EFB and decanter cake are currently utilized as fertilizers and soil cover materials in palm oil plantation areas, whilst the rest of EFB is dumped in areas adjacent to the mill because of the high generation rate along with its limitations for current utilization (Paepatung et. al, 2009). Empty fruit bunch can also be incinerated to produce potash, which is applied in the plantation as fertilizer by mulching. The fibre and shell materials are used as boiler fuel. The palm kernel is usually sold to palm kernel oil producers for the extraction of palm kernel oil (Thani et. al, 1999).

1.2 Problem Statement

The solid waste from palm oil mill, decanter cake which is produced by the decanter still contains oils in it. Most of the palm oil mill plant companies disposed the decanter cake by incineration, inclusion in animal feeds, land filling method or concrete manufacturing. But then, there is also huge quantity of decanter cake being deposited in landfills which would cause fire and pollution hazards due to the residue oil content in the earth. The oil in decanter cake would just be wasted if it is not being recovered. This would cause the loss of the oil which actually can be used for other purpose.

There is little research on extracting oil from palm oil decanter. Currently, some palm oil mill companies turn decanter into fertilizers and animal feed without extracting the residue oil. The residual oil can be extracted and used for so many uses. As the oil extracted from palm oil solid waste has poorer quality compared to crude palm oil, the

oil can be used as a biodiesel. Just then, after the oil was extracted from the decanter cake the remaining solid can be use as fertilizer. Utilization of palm oil decanter cake also will improve the environment. This is because the disposal of sludge solid waste will increase the Biochemical Oxygen Demand (BOD) of the land (G.Dashiny, 2009)

1.3 Objectives of Study

The objectives of this research are:

- 1) To extract residual oil from palm oil decanter cake.
- 2) To determine the quality of extracted oil from Soxhlet extraction method of decanter cake.
- 3) To study the relationship of pretreatment of POME with the extraction performance and the quality of oil extracted.

1.4 Scope of study

Based on the objectives, the main scope of this project is likely to elucidate the approach to effectively recover valuable constituents from decanter cake. The scope of this study includes collecting decanter cake sample from a nearby palm oil mill. Then, the sample is exposed to pretreatment which is drying methods at different temperature in order to prepare the sample for extraction. The remaining water, volatile compound and fixed carbon contents of each dried decanter cake were analyzed using Thermal Gravimetric Analysis. Then, the effect of the different drying temperature of the decanter cake sample on the extraction process is studied after the extraction process has been done. The extraction of oil from decanter cake is done using Soxhlet extraction unit. In the extraction process, hexane is used as the solvent. After the extraction process is done, the quality of extracted oil from each dried decanter cake sample is determined using acid value and iodine value titration. The effects of the drying temperature of each dried decanter cake on both acid and iodine value were studied.

CHAPTER 2

LITERATURE REVIEW

2.1 Palm Oil

Oil palm or by its scientific name, *Elaeis Guineensis* Jacq is the most important species in the genus *Elaeis* which belong to the family *Palmae*. *Elaeis guineensis* Jacq is the most productive oil palm variety in the world, with one hectare of oil palm producing 10–35 tonnes of fresh fruit bunch (FFB) per year (Singh et al., 2010). Industrial processing of palm oil from palm fruits is carried out in palm oil mills and the process of oil extraction is called milling (Corley and Tinker, 2003). The process involved in oil palm milling after the fresh fruit bunches (FFB) are transported to the palm oil mills consist of sterilization, threshing, digestion, crude palm oil extraction, nut/fibre separation and nut cracking. The process flow diagram of palm oil extraction is as illustrated in Figure 2.1. The process flow diagram shows the process involved in the palm oil extraction, the products and wastes produced from each process.

Sterilization

This process is done batch wise in an autoclave for about 2 hour with the temperature inside the autoclave about 120–130°C. Sterilization is done to check further formation of free fatty acids due to enzyme action and to facilitate stripping and prepare the mesocarp for subsequent processing. The steam condensate coming out of the sterilizer constitutes one of the major sources of wastewater (Thani et al., 1999).

Threshing

Also known as stripping process where the FFB are fed to a rotary drum-stripper after sterilized. In this process the fruits are stripped from the fruit bunches and then the detached fruits passed through the bar screen of the stripper and are collected below by a bucket conveyor and discharged into a digester. This process generates empty fruit bunches (EFB).

Digestion

Separated fresh fruits are put into a digester, where in this process they are mashed under steam heated conditions by the rotating arms. The mashing of the fruits under heating breaks the mesocarp oil-bearing cells. Twin screw presses are generally used to press out the oil from digested mash of fruit under high pressure. To enhance the flow of the oils, hot water is added. In this step, no residue occurs.

Crude palm oil extraction

Homogenous oil mash from the digester is passed through a screw press then going through the vibrating screen, a hydrocyclone and decanters in order to remove fine solids and water. For further purification of the oil, the centrifugal and vacuum driers are used before the oil is sent to a storage tank. The temperature of oil in the storage is maintained at around 60 °C with steam coil heating before the crude palm oil (CPO) is sold (Singh et al., 2010).

Nut/fibre separation

The press cake discharged from the screw press contains moisture, oily fibre and nuts and the cakes. It is then conveyed to a depericarper where the fibre and nuts are separated by strong air current induced by a suction fan. The fibre is usually used as boiler fuel in the boiler house. While, the remaining fibre on the nuts is removed using the rotating drum before they are sent to a nut cracker.

2.1.1 Characteristic of Palm Oil

The oil palm produces two types of oils which are palm oil from the fibrous mesocarp and lauric oil from palm kernel. In the conventional milling process, the fresh fruit bunches are sterilized and stripped of the fruitlets which are then digested and pressed to extract the crude palm oil (CPO). The nuts are separated from fibre in the press cake and cracked to obtain palm kernels which are crushed in another plant to obtain crude palm kernel oil (CPKO) and a by-product, palm kernel cake which is used as an animal feed. Fractionation of CPO and CPKO in the refinery produces the liquid olein fraction and a solid stearin component.

The fatty acid compositions the palm oil products, compared with coconut oil and soy oil are presented in Table 2.1. Palm oil has a balanced ratio of saturated and unsaturated fatty acids while palm kernel oil has mainly saturated fatty acids which are broadly similar to the composition of coconut oil. Compared to soy oil, palm oil has a higher amount of saturated fatty acids but this makes it more stable and less prone to oxidation at high temperatures (Teoh, 2002). Table 2.1 shows the fatty Acid Compositions of Palm Oil Products, Soy Oil and Coconut Oil

Table 2.1: Fatty Acid Compositions of Palm Oil Products, Soy Oil and Coconut Oil

Fatty Acids	Weight Percentage						
	Palm Oil	Palm Olein	Palm Stearin	Palm Kernel Oil	Palm Kernel Olein	Coconut Oil	Soy Oil
C6:0				0.3	0.4	0.2	
C8:0				4.4	5.4	8.0	
C10:0				3.7	3.9	7.0	
C12:0	0.2	0.2	0.3	48.3	41.5	48.2	
C14:0	1.1	1.0	1.3	15.6	11.8	18.0	
C16:0	44.0	39.8	55.0	7.8	8.4	8.5	6.5
C18:0	4.5	4.4	5.1	2.0	2.4	2.3	4.2
C18:1	39.2	42.5	29.5	15.1	22.8	5.7	28.0
C18:2	10.1	11.2	7.4	2.7	3.3	2.1	52.6
Others	0.8	0.9	0.7	0.1	0.1		8.0
Iodine Value	53.3	58.4	35.5	17.8	25.5	9.5	133.0

Source: Salmiah (2000)

2.1.2 Chemical properties of palm oil

Palm oil contains several saturated and unsaturated fats in the forms of glyceryl laurate (0.1%, saturated), myristate (1%, saturated), palmitate(44%,saturated), stearate (5%, saturated), oleate (39%, monounsaturated), linoleate (10%, polyunsaturated), and linolenate (0.3%, polyunsaturated). It is also composed of fatty acids, esterified with glycerol just like any ordinary fat. It is high in saturated fatty acids. Palm oil gives its name to the 16-carbon saturated fatty acid palmitic acid. Monounsaturated oleic acid is also a constituent of palm oil. Unrefined palm oil is a large natural source of tocotrienol, part of the vitamin E family. The approximate concentration of fatty acids (FAs) in palm oil is 44.3% of palmitic acid, 38.7% of Oleic acid, 10.5% of Linoleic acid, 4.6% of Stearic acid, 1.0% of Myristic acid and 0.9% of other and unknown fatty acids.

2.2 Waste production of palm oil mills

The oil palm mill generates a wide variety of wastes in large quantities which can be categorized into two, liquid wastes and solid wastes such as illustrated in Figure 2.2. Liquid wastes came from extraction and processing of the oil while the solid wastes are the the leaves, trunk, decanter cake, empty fruit bunches, seed shells and fibre from the mesocarp.

Liquid effluent

In the production of palm oil, there are large quantities of polluted wastewater being generated which is commonly referred as palm oil mill effluent (POME) (Lorestani, 2006). It contains various suspended components including cell walls, organelles, short fibres, a variety of carbohydrates ranging from hemicelluloses to simple sugars, a range of nitrogenous compounds from proteins to amino acids, free organic acids and an assembly of minor organic and mineral constituents (Ugoji, 1997).

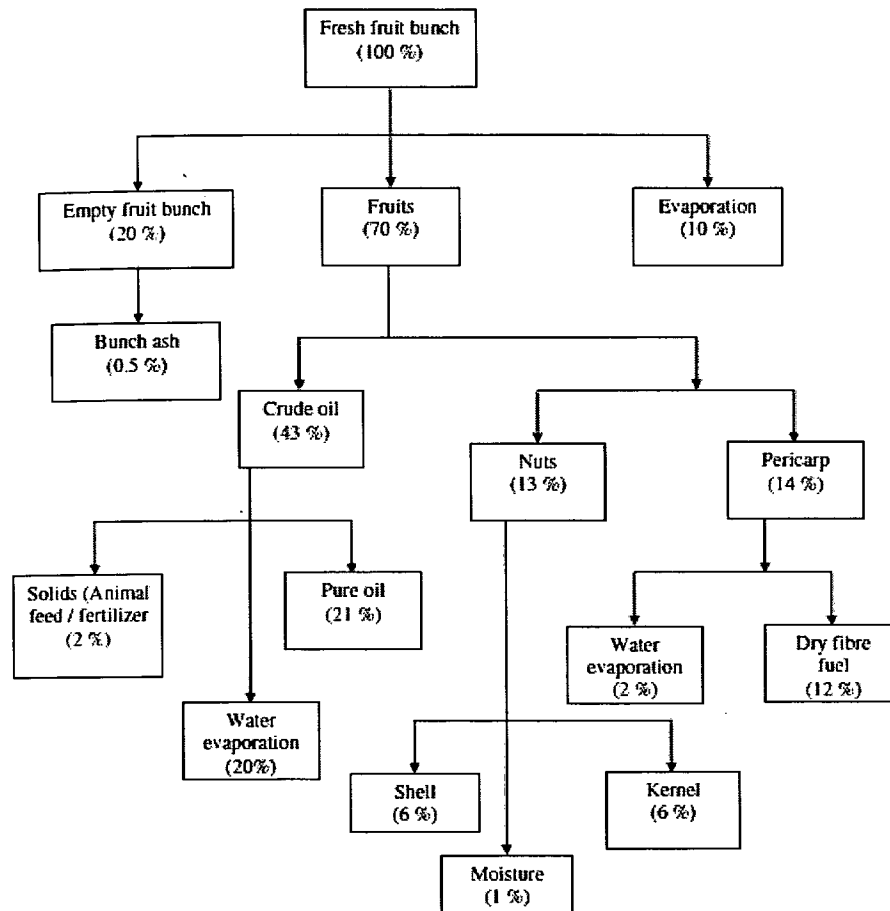


Figure 2.2: Products from palm oil mill process (Lorestani, 2006)

Solid wastes

Palm oil extraction also generates the solid waste materials and other by-products as illustrated in Figure 2.3. The empty fruit bunch, palm oil mill sludge (POMS), palm kernel cake (PKC) and decanter cake are the most common among these by-products. Palm oil mill sludge (POMS) produced from the treatment of the POME. Therefore due to large quantity of POME production each year, the amount of POMS increases respectively. These sludges results in bad odors and is considered as a source of surface and ground pollution (Rupani et al., 2010). Palm kernel oil (white palm oil) is obtained from the seed known as kernel or endosperm. When oil has been extracted from the kernel, what remains is known as ‘palm kernel cake’ (PKC) (Singh et al., 2010).

In Thailand about 60 crude palm oil mills produced approximately 1.24 million tonnes of crude palm oil from 6.4 million tonnes of fresh fruit bunches (FFB) in 2007 (Paepatung et al. 2006). The average values of waste generation rate per ton FFB from palm oil mills in Thailand were 140 kg of fibre, 60 kg of shells, 240 kg of empty fruit bunch (EFB) and 42 kg of decanter cake. The productions of fibre, shells, EFB and decanter cake were estimated to be respectively 0.894, 0.13, 1.53, and 0.27 million tonnes per year (Chavalparit et al. 2006).

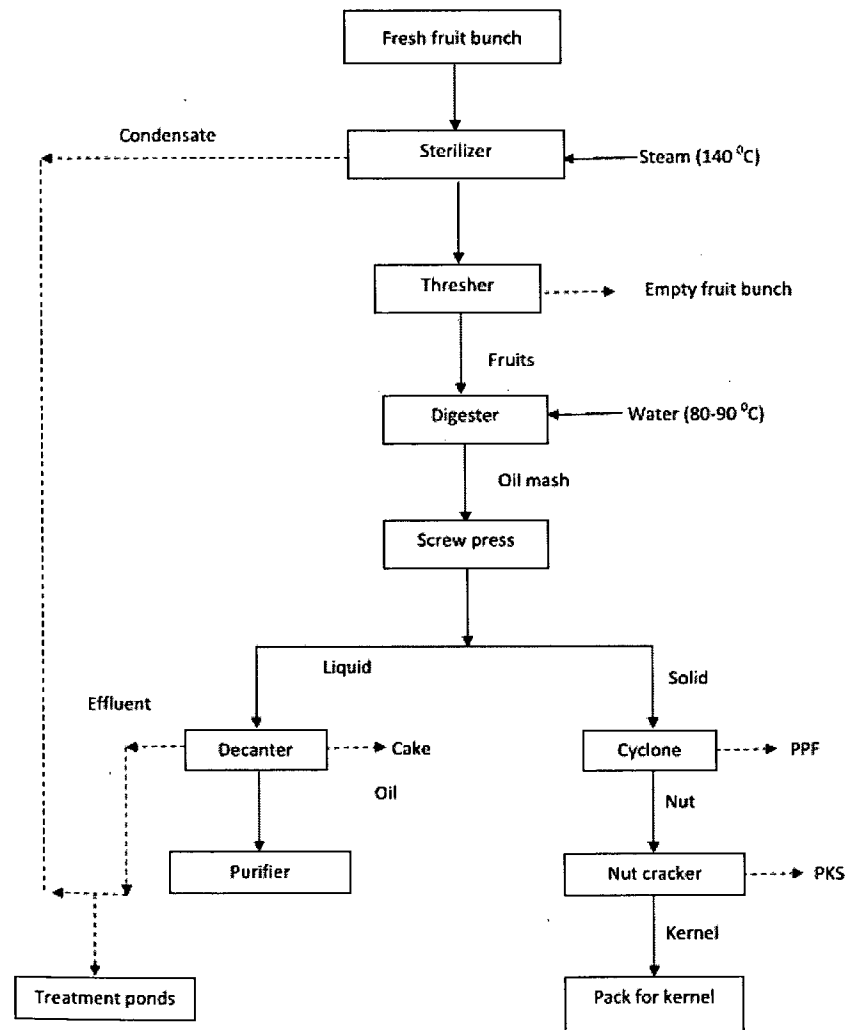


Figure 2.3: Oil extraction and waste generation process in palm oil mill

Source: Prasertsan and Prasertsan (1996)

2.3 Decanter Cake

Decanter cake is a waste from palm oil mills and a mill with 90 t/hr FFB processing capacity will produce about 160 to 200 tone of decanter cake (K.Haron et al., 2008). Decanter cake is produced by the decanter in the decanting process of the palm oil mill sludge. This results in a reduced BOD load generated by the standard decanter process despite its higher BOD and oil concentrations (W.Roge, 1981; L. C. Lok, 1987; Stuttgart, 1980). Decanter cake was reported to have plant nutrients in it. Table 2.2 shows the plant nutrient contents and pH of decanter cake.

The nutritive value of by-products (palm press fiber (PPF) and palm oil sludge (POS)) of palm fruit processing were studied through analysis of their chemical components and degradability of their dry matter (DM) in the rumen of steers. Chemical analysis showed that the materials have similar organic matter components (mean = 95.2%), while crude protein (CP, g/100g) and metabolisable energy (ME, MJ/kg DM) were highest in palm oil sludge (10.02 and 9.43). Mineral contents showed that POS had the lowest and highest concentration, respectively, of Mg (0.07 vs. 0.11%), Cu (958.5 vs. 143.9 mg/kg) and Fe (1374.5 vs. 4086.0 mg/kg). Dry matter degradation characteristic and effective degradability varied significantly ($P < 0.05$) and were consistently highest in palm oil mill sludge (Ong et al., 1982). It is concluded that palm oil sludge cake and palm pressed fiber can be harnessed directly as feed resources for ruminant animals. Solvent extraction process for recovering oil from decanter cake is chosen because they give higher yield and better quality of oil (G. Dashiny, 2009).

Table 2.2: Plant Nutrient Contents (%) and Ph of Decanter Cake

N	P2O5	K2O	CaO	MgO	pH
2.42	0.51	1.24	1.68	0.54	4.8

Source: K.Haron et al. (2008)

2.4 Thermal Gravimetric Analysis

Thermo gravimetric analysis or thermal gravimetric analysis (TGA) determine changes in weight in relation to change in temperature. Its principal uses include measurement of a material's thermal stability and composition. The applications of TGA are for thermal stability, compositional analysis, filler content, sample preparation, comparative analysis specification testing.

Thermogravimetric techniques have a very wide field of application. The technique can be used in the examination of absorptive surfaces, together with the nature and processes involved in thermal decomposition and oxidation processes. The technique has also been applied to the examination of water of crystallization and in forensic work involving the identification and comparison of varnishes and other surface coating. Thermogravimetric analysis has also been involved in determining the age of art treasures, particularly paintings and in determining the stability of explosives. The technique is commonly used to control the dehydration procedures for crops particularly in the control of the drying processes used for tobacco. Thermogravimetric analysis is also used extensively in the pharmaceutical industry in the examination of drug stability and the rate of degradation of certain drugs when expose to air.

The results from thermogravimetric analyses are usually reported in the form of curves relating the mass lost from the sample against temperature. In this form the temperature at which certain processes begin and are completed are graphically demonstrated. (Scott). Figure 2.4 shows the example of the TGA Curve for Silica Gel. Thermogravimetric analysis curve obtained is from heating a sample of silica gel from 30 °C to 1000 °C. The curve shows the loss in weight that occurred at different temperatures, as different types of water are lost from the surface.

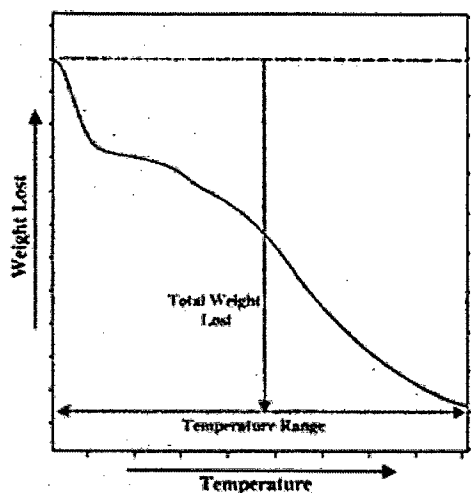


Figure 2.4: The TGA Curve for Silica Gel

Source: Scott (n.d.)

2.5 Solvent Extraction

Solvent extraction method is one of most powerful separation and purification tool in the process industries. It is used commercially within the chemical industry including organic chemical, petrochemical and pharmaceuticals (A.L. Ahmad et al., 2003). Solvent extraction or liquid/liquid extraction method is a method to separate compounds based on their relative solubilities in two different immiscible liquids, usually water and an organic solvent. It is an extraction of a substance from one liquid phase into another liquid phase. The solvent extraction process consists of four basic components (Belhateche, 1995).

- Contact between wastewater and solvent;
- Separation of extracted wastewater and solvent;
- Treatment of solvent to remove extracted constituents; and
- Treatment of wastewater to remove residual solvent.