ULTRASONIC EXTRACTION OF PALM PRESSED FIBRE FOR OIL RECOVERY

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ABSTRACT

Palm pressed fiber (PPF) is the solid waste produced from palm oil milling company after the pressing process. Recovery of oil and reuse of the palm pressed fiber is a great opportunity for cost saving in the oil processing industry. The objective of the research is to recover residual oil which still left in the PPF by using ultrasonic-assisted extraction method (UAE). The process is carried out by priory drying process of the PPF at 60 °C for 1 hour. Then the dried sample will undergo the extraction process in the ultrasonic bath by using methanol as a solvent. The effect of solid: liquid ratio and extraction time are varies in the range of (1:3-1:8) and (0 minutes-60 minutes) respectively. Yield of the extracted oil are measured for all conditions. Results from studies shows that the highest yield is 12.90% which obtained at the condition 1:5 (g/ml) of the solid: liquid ratio and 30 minutes of the extraction time. Hence, the result from Gas chromatography–mass spectrometry (GC-MS) has confirmed that the properties of the extracted oil are almost similar to the crude palm oil (CPO).

Keywords: Palm pressed fiber (PPF), ultrasonic, extraction.

ABSTRAK

Sabut kelapa sawit hempit (PPF) adalah sisa pepejal yang dihasilkan daripada syarikat pengilangan kelapa sawit selepas proses menekan. Pemulihan minyak dan penggunaan semula serat kelapa ditekan adalah satu peluang besar wujud untuk penjimatan kos dalam industri pemprosesan minyak. Objektif kajian ini adalah untuk mendapatkan semula sisa minyak yang masih tinggal dalam PPF dengan menggunakan kaedah pengekstrakan ultrasonik dibantu (UAE). Proses ini dilakukan dengan pengeringan biara daripada PPF pada 60 ° C selama 1 jam. Kemudian sampel kering akan menjalani proses pengekstrakan di mandi ultrasonik dengan menggunakan metanol sebagai pelarut. Kesan daripada pepejal: Nisbah cecair dan masa pengekstrakan adalah berbeza dalam julat (1: 3-1: 8) dan masing-masing (0 minit-60 minit). Hasil minyak yang diekstrak diukur untuk semua keadaan. Hasil daripada kajian menunjukkan bahawa hasil yang tinggi adalah 12,90% diperolehi pada keadaan optimum pada 1: 5 (g / ml) pepejal: Nisbah cecair dan 30 minit masa pengekstrakan. Oleh itu, hasil daripada (GC-MS) telah mengesahkan bahawa sifat-sifat minyak yang diekstrak hampir serupa dengan minyak sawit mentah.

Kata Kunci: Sabut kelapa sawit hempit (PPF), ultrasonik, pengekstrakan.

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CHAPTER 1

INTRODUCTION

1.1 Motivation and Statement of Problem

Palm oil (Elaeis guineensis) is the most important product and one of the key economic of the agricultural sector in developing countries such as Malaysia. Today Malaysia is the largest producer and exporter of palm oil in the world (Basiron & Weng, 2004). In the process of extraction of palm oil from oil palm fruit, biomass materials such as palm empty fruit bunch (EFB) and palm pressed fibre (PPF) are generated as waste products (Ohimain & Chibuezeizah, 2014)

Based on the Malaysian Palm Oil statistic, a total of 79.3 million tonnes of fresh fruit bunch is processed in 2006 to produce 15.9 million tonnes of crude palm oil (CPO) and

2.0 million tonnes of palm kernel oil (PKO). In the process of extracting these oils, 4.3 million tonnes of palm kernel shell, 10.7 million tonnes of palm pressed fibres (PPF) and 17.4 million tonnes of empty fruit bunch (EFB) are generated (Nafissa *et al.*, 2008).

In general, a palm oil mill extracts 20% of oil from the fresh fruit bunch and produces 23% empty bunch, 15% fibre, and 12% nut. Oil losses occur in various by-products, including the fibre, which remains after the mesocarp oil is extracted by a screw press (Choo *et al.*, 1996). The oil retained in pressed mesocarp fibre in Malaysia ranges from 5.0% to 11.0% oil as a ratio to dry matter, but the fibre is normally burned as fuel to provide energy for the mill. (Subramaniam *et al.*, 2013)

These practices create environmental pollution problems as incineration and boiler emit gases with particulates such as tar and soot droplets of 20-100 microns and a dust load of about 3000 to 4000 mg/nm and indiscriminate dumping of EFB and PPF causes additional methane emission into the atmosphere (Nafissa *et al.*, 2008). Furthermore the disposed of oil palm waste without any treatment causing the loss of much valuable oil. So, to minimize pollution and to avoid the loss of much valuable oil, the recovery oil from the solid wastes is the best method in the palm oil mill. From table 1-1, it shows palm pressed fibre constitutes about 15% by weight of fresh fruit bunches.

Table 1.1 Products/Wastes From Each Bunch Of FFB
(Abdullah & Sulaiman, 2013).

Products/wastes	Percentages by weight to FFB (dry basis)
Palm oil	21
Palm kernel	7
Fibre	15
Shell	6
Empty fruit bunches	23
POME	28
Total	100

In terms of extraction treatment, it can be defined that, as separation process consisting in the separation of a substance from a matrix. Basically, the extractions are referring to liquid-liquid extraction, and solid phase extraction. The example of technique in extraction process includes ultrasonic solvent extraction, heat reflux extraction, supercritical carbon dioxide extraction, enzymatic extraction and microwave-assisted extraction. The choice of extraction technique is basically decided upon based on initial cost, operating cost, simplicity of operation, amount of organic solvent required and sample throughout. Among of this extraction treatment, ultrasonic solvent extraction is the best method for extraction process due to its advantages of other extraction process. It is expected the use of ultrasonic extraction for sample preparation purposes in environmental analytical chemistry will become more widespread, owing to its simplicity, speed, and enhanced safety when compared with others (Petigny et al., 2013)

1.2 **Problem statement**

Palm pressed fibre contain a rich source of residual oil such as carotenoids, vitamin E, sterols and phospholipids. However, the solid wastes PPF are currently disposed directly without any treatment causing the loss much valuable oil. So, to increase the oil recovery in palm oil mill, the extraction of residual oil from PPF is important.

Ultrasound-assisted extraction (UAE) is a new simple technique for the recovery of oil and bioactive compounds from different sources. Consequently, ultrasonic treatment has been proven as an effective treatment of PPF and able to get high yield of oil recovery from PPF which can save disposal cost, produce new valuable product and at the same time protect the environment.

Extraction is the first key step to isolate natural bioactive compounds from plants and materials. Different extraction methods such as solvent extraction, expelling extraction process, supercritical fluid extraction (SFE) and microwave assisted extraction (MAE) have been developed for the recovery of bioactive compounds and essential oils. However, several disadvantages like extra solvent amount in solvent extraction, low yield in the process, massive investment in supercritical fluid extraction (SFE) and the requirement for the aqueous phase in microwave assisted extraction indicates the demand of comprehensive extraction method to recover different target compounds in economic condition.

1.3 **Objective**

The objective of the research is to recover residual oil left in the PPF by using ultrasound-assisted extraction

1.4 **Scopes of this research**

i. The scopes of the study are to investigate the effect of the key parameters for recovery oil in palm pressed fibre. These parameters include:

- To study the effect of sample (PPF) to solvent ratio (1:3-1:8)

- To study the extraction time (0-60 min)

ii. To obtain the high yield of residual oil by using ultrasound-assisted extraction.

CHAPTER 2

LITERATURE REVIEW

4.1 **Introduction of palm oil**

Palm oil or Elaeis guineesis traditionally was grown in tropica Africa and it was introduces to Malaysia for planting in the Botanical Gardens in Singapore in 1870. Now, the oil palm (Elaeis guineensis) is the most important product from Malaysia that has helped to change the scenario of it is agriculture and economy. Two types of oil can be obtained from the fruits, namely palm oil from the oil cell in the mesocarp, and the kernel oil from the seed of the nut (kernel) (Choo *et al.*, 1996). Palm oil is used mainly for production of margarine and compounds in cooking fat and oil. Besides that, the palm oil also are used for production of candles, detergents, soap and cosmetics products. Production of palm kernel oil is about 12% of the production of its palm oil (Abdullah & Sulaiman, 2013).

Figure 4-1 Fresh Fruit Bunch

The total oil palm planted area in Malaysia increased by 2.8% to 4.17 million hectares in 2006. The area expansion occurred mainly in Sabah and Sarawak with a combined growth of 4.5% compared to 1.6% in Peninsular Malaysia. Sabah remained the largest oil palm planted state with 1.24 million hectares or 30% of the total planted area. Table 2-1 shows the oil palm planted areas by state in Malaysia for 2005 until 2008 (in hectares).

Table 4.1 Oil Palm Planted Area 2005-2008 (Hectares) (Abdullah & Sulaiman.,2013).

State	2005	2006	2007	2008
Johor	667,872	671,425	670,641	na
Kedah	75,472	76,329	75,096	na
Kelantan	89,886	94,542	99,763	na
Melaka	52,015	52,232	49,113	na
N. Sembilan	155,164	161,072	170,843	na
Pahang	606,821	623,290	641,452	na
Perak	340,959	348,000	350,983	na
Perlis	278	258	260	na
P. Pinang	14,074	14,119	13,304	na
Selangor	132,100	128,9115	129,315	na
Terengganu	163,967	164,065	161,287	na
Peninsular Malaysia	2,298,608	2,334,247	2,362,057	-
Sabah	1,209,368	1,239,497	1,278,244	na
Sarawak	543,398	591,471	664,612	na
Sabah & Sarawak	1,752,766	1,830,968	1,942,856	-
Malaysia	4,051,374	4,165,215	4,304,913	4,487,957

Figure 2-1 below shows that the crude palm oil (CPO) was extracted from the digested palm fruit by pressing process.

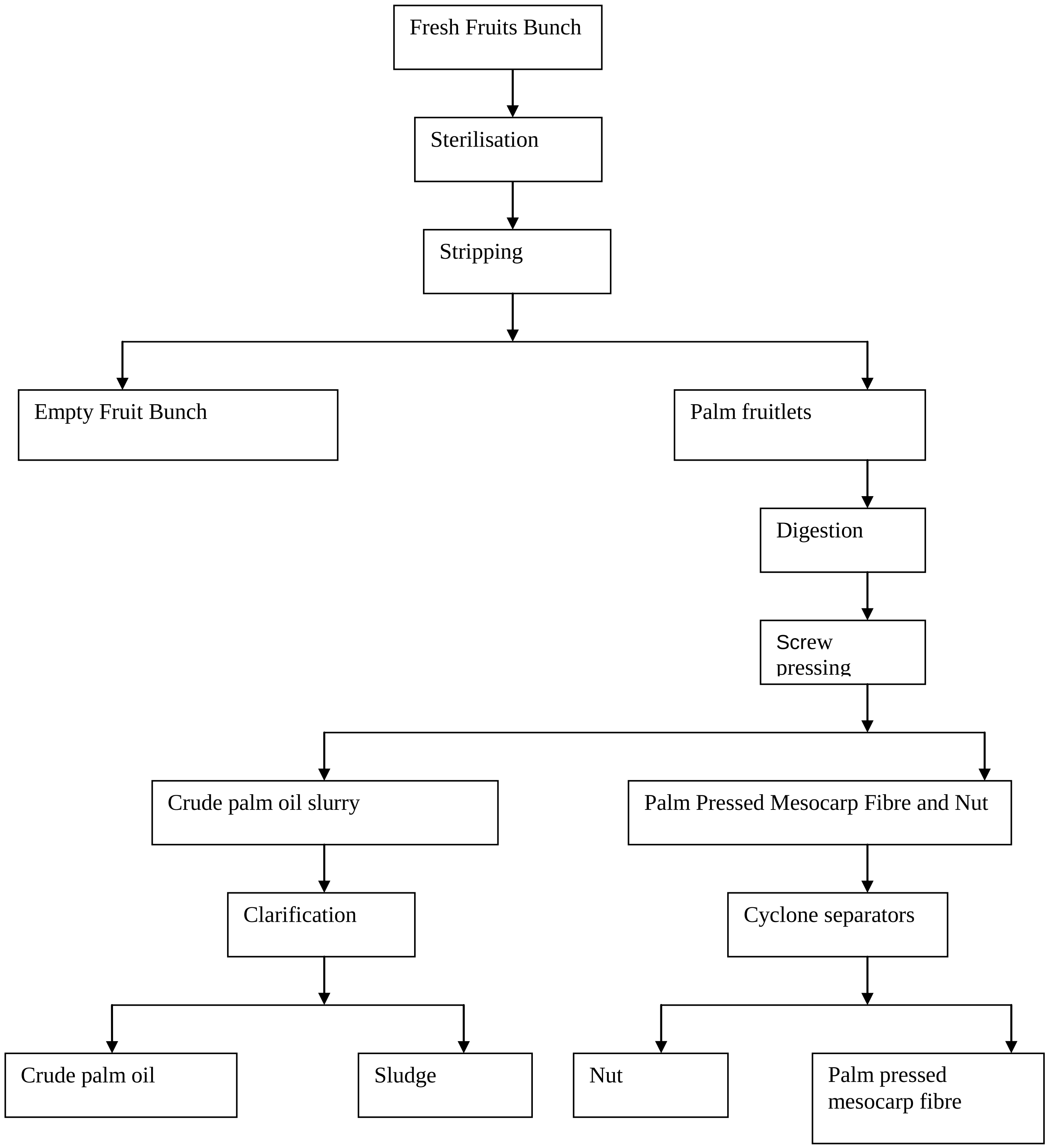

Figure 4-2 Process Flow Diagram of Palm Oil Extraction

(Lik Nang Lau *et al.*, 2008)

4.1.1 Properties of palm oil

Palm oil is edible oil that has been used widely in food industry. It is produced from crude palm oil (CPO) which has gone through several treatment or refining processes. The CPO consisted of triglyceride (TG) which is the major component in palm oil, digylceride, monoglyceride, free fatty acid (FFA) and minor components such as carotenoids, tocopherols, sterols, phospholipids and squalene (Akanda *et al.*, 2012). Crude palm oil (CPO) is produced in the palm oil mills by mechanically extracting it from the digested mesocarp of the palm fruits. Currently, most palm oil mills uses screw presses for the oil extraction process.

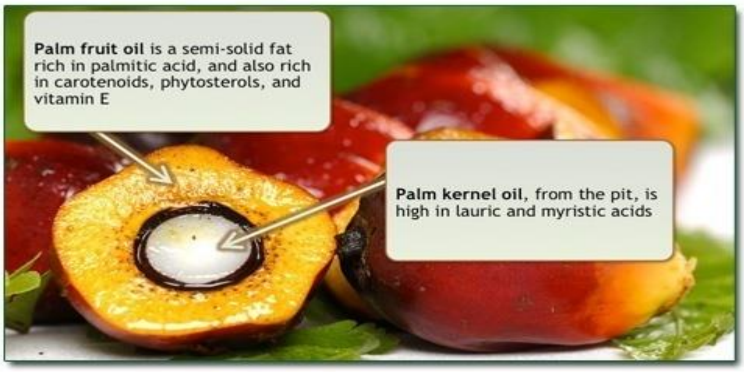

Figure 4-3 Palm Oil Fresh Fruit Bunch

Crude palm oil is one of the major sources of vitamin E and contains high quantities of tocopherols and tocotrienols in the range of 600-1000 ppm (Choo *et al.*, 1996). Palm oil has a balanced ratio of saturated and unsaturated fatty acids while palm kernel oil has mainly saturated fatty acid which is broadly similar to the composition of coconut oil.

Palm oil is semi-solid at room temperature. In its virgin form, the oil is bright orange-red in colour due to its high content of carotene. Triglycerides forms the major component and bulk of the glyceridic material present in palm oil with small amounts of monoglycerides and diglcerides, which are minor composition of the extraction process. Knowledge about the detailed structures of the triglycerides present in palm oil is important, because they define the physical characteristics of the oil. The palm oil are melts over the wide range of temperatures namely 25-50 ^{o}C, while it is 20-30 ^{o}C for the kernel oil. Table 2-2 shows the characteristics of crude palm and palm kernel oil.

Table 4.2 Characteristics of crude palm and palm kernel oils (Nagaraj, 2009)

Characteristic	Palm oil	Palm kernel oil
Density	0.891	0.908
Refractive index, n_D 40 ^{o}C	1.453-1.459	1.4495-1.4515
Melting range, ^{o}C	27-50	25-30
Titer, ^{o}C	40-46	20-28
Acid value, mg KOH/g oil	2-15	3-17
Saponification value, mg KOH/ g oil	196-209	244-264
Iodine value	46-60	14-20
Thiocyanogen value	44-48	13-18
Hydroxyl value	7-24	-
Unsaponifiable matter, %	0.3-1.0	0.2-0.8

4.1.2 Fatty acid composition

Palm oil contains equal proportions of saturated fatty acids and unsaturated fatty acids. The saturated fatty acids are made up of 44% palmitic acid and 5% stearic acids. The unsaturated fatty acids consist of 39% oleic acid and 10% linoleic acid.

Table 4.3 Fatty acid composition of palm oil and palm kernel oil (Nagaraj, 2009)

Fatty acid	Typical Range	Immature fruit	Elaeis oleifera	Palm kernel oil
Carprylic acid	-	-	-	2.4-6.2
Capric acid	-	-	-	3.0-6.3
Lauric acid	-	-	0.05	44.5-52.0
Myristic acid	0.5-5.9	trace	0.3	14.1-18.6
Palmitic acid	32-47	57.5	25.0	6.5-10.4
Stearic acid	2-8	7.0	1.2	1.3-3.5
Arachidic acid	-	trace	0.1	0-1.9
Palmitoleic acid	-	-	1.4	0-0.6
Oleic acid	40-52	0.9	68.6	10.5-18.5
Linoleic acid	5-11	29	2.1	0.7-2.5
Linolenic acid	-	2	0.9	-

4.1.3 Minor components of crude palm oil (CPO)

Palm oil has some very important minor constituents which are carotenoids, tocopherols, sterols, phosphatides, triterpenic, and aliphatic alcohols. Mainly they work as antioxidants and help in free radical quenching, anti-aging and anti-cancer agents. Vitamin A and E have their normal physiological roles as vitamins. Crude palm oil contains between 500-700 ppm of carotenoids mainly in the forms of α- and β-carotenes In crude palm oil , the presence of these carotenoids appears to offer some oxidative protection to the oil through a mechanism where they are oxidized to the triglcerides (Choo *et al.*, 1996).

Table 4.4 Unsaponifiable matter of palm oil

(Nagaraj, 2009)

Constituents	Content
Carotenoids	500-700 mg/kg
α-Carotene	36.2%
β-Carotene	54.4%
γ-Carotene	3.3%
Lycopene	3.8%
Xanthophylls	2.2%
Tocopherols	500-800 mg/kg
α-tocopherol	35%
γ-tocopherol	35%
δ-tocopherol	10%
Sterols	About-300 mg/kg
Cholesterol	4%
Campesterol	21%
Stigmasterol	21%
β-Sitosterol	63%
Phosphatides	500-1000 mg/kg
Total alcohol	about-800mg/kg
Triterpenic alcohols	80%
Aliphatic alcohols	20%

4.1.4 Application of palm oil

Palm oil has a wide range of application in food uses and oleo chemical products for non food uses. About 80% of the world production is used for food applications and the rest is feedstock for a non-food applications. Refined, bleached and de-odorized (RBD) palm oil, is obtained by removing the free fatty acids, colour and unwanted flavour through refining (Nagaraj, 2009). The four main traditional uses of palm oil in food products are for cooking or frying oil, shortenings, margarine and confectionary fats. Palm oil is popularly used in both solid fat products as well as in the liquid cooking oil sector especially in industrial frying applications. It offers several technical characteristics desirable in food applications, such as resistance to oxidation, which contributes towards longer shelf life of end products.

Palm oil products also find wide applications in the non-food sector, especially in the production of soaps, detergents, and cleansing agents such as surfactant. Palm oil also is an excellent raw material for the production of oleo chemical products like fatty acids, fatty alcohol, glycerol and other derivatives. These are widely used in the manufacture of cosmetics pharmaceuticals, household and industrial products. Oleo chemicals manufactured from palm oil and palm kernel oil are now popular ingredients in the manufacture of environment friendly detergents as they are biodegradable (Edem, 2002)

Palm oil and kernel oil play an important role as feedstock for the production of oleo chemicals. These oleo chemicals can be used for the production of candles, cosmetics products, soaps, pharmaceuticals, textiles and in rubber or plastic processing (Salmiah., 2000) Table 2-5 shows the example of application of palm oil.

Table 4.5 Example of basic application of palm oil and palm kernel oils

Food Applications	Oleochemical	Energy, Biomass & Others
Cooking oil	Surfactants	Biodiesel
Industrial Frying Fats	Personal care	Furniture
Margarine	Cosmetics	Charcoal
Vegetable Ghee	Agrochemical	Pulp & paper
Confectionary Fats	Lubricant/grease	Animal Feeds
Ice Cream	Toilet Soap	Bio-composite
Non-dairy creamer	Industrial cleaning	Fertilizer
Salad Dressing	Printing Ink	Briquettes
Cheese analog	Polyols	
Supplements/vitamins	Polyurethane	

4.2 **Palm pressed fibre (PPF)**

Palm pressed fibre (PPF) is the fibrous residues separated from the mesocarp and kernel during palm oil extraction (Nordin *et al.*, 2013). The oil losses usually occur in various by-products, including the fibre, which remains after the mesocarp oil is extracted by a screw press. Because the screw press process for the extraction of oil from mesocarp is conventionally done by single stage pressing, so, the oil loss is considerable (Sanagi, See, Ibrahim, & Naim, 2005). PPF is a combination of palm mesocarp fibre, kernel shell, crushed kernel and debris. Hence, the oil recovered from this fibre has combined characteristics of all these products (Neoh et al., 2011). Recent studies (Subramaniam *et al.*, 2013) show that the oil retained in pressed mesocarp fibre in Malaysia ranges from 5.0% to 11.0% oil.

Figure 4-4 Palm Pressed Fibre

Based on the Malaysian Oil Palm Statistics 2011, a total of 92.9 million tonnes of fresh fruit bunch (FFB) were processed to produce about 18.9 million tonnes of crude palm oil (CPO). In the process of the oil extraction, 12.5 million tonnes of palm pressed fibres (PPF) were generated (Wahab, Aziz, Berahim, & Ali, 2005). Usually, most palm oil mills burn the fresh fibre for electricity generation, resulting in the loss of much valuable oil and nutrients (Lik Nang Lau *et al.,* 2008). Now PPF also has been expected to contain high yield of oil (Choo *et al.,* 2004). However, the presence of these oil palm wastes has created a major disposal problem. The fundamental principles of waste management are to minimise and recycle the waste, recover the energy and finally dispose the waste. Palm pressed fibre contain a rich source of residual oil such as carotenoids, vitamin E, sterols and phospholipids. So the recovery oil from PPF is a great opportunity to increase the production in palm oil mill.

Table 4.6 The type of residual oil in palm pressed fiber (PPF)

Type of extraction	solvent used	Type of residual oil recovery	journals
Supercritical fluid extraction	Carbon dioxide	SFE extract highest carotenoid	(Cardenas *et al.*, 2014).
High- pressure soxhlet extraction	Hexane	Carotenoids, vitamin E and sterols	(Choo *et al.*, 1996).
Ultrasonic extraction	Methanol	Phospholipid	(Chua *et al.*, 2009)
Cold extraction	Hexane	Diaclyglycerol (DAG), Triacylglycerol (TAG),and monoaclyglycerol (MAG)	(Neoh *et al.*, 2011)
Supercritical fluid extraction	Carbon dioxide	Carotene and lipids	(Franc & Meireles, 2000)
Ethanolic extract (FOES)	Ethanol	Phospholipids	(Choo *et al.*, 2004)
Supercritical fluid extraction	Carbon dioxide	Caratone, vitamin E, squalene & sterols	(Lik Nang Lau *et al.*, 2008)
Pressurized liquid extraction	n-hexane	Carotene, tocopherols and tocotrienols	(Sanagi, *et al.*, 2005)