

Recovery of Oil from Palm Oil Mill Effluent (POME) by Solvent Extraction

by

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Dissertation submitted in partial fulfilment of
the requirements for the
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(Chemical Engineering)

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CERTIFICATION OF APPROVAL

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Chemical Engineering Programme
Universiti Teknologi PETRONAS
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BACHELOR OF ENGINEERING (Hons)
(CHEMICAL ENGINEERING)

Approved by,

(A.P. Dr. M. Azmuddin Abdullah)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
May 2011

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MAISARAH BINTI SALLEH

ABSTRACT

Palm oil mill effluent (POME) will be a major source of pollution if it not treated properly because oil and grease present in POME. Accumulation of residual oil in POME will prevent effective wastewater treatment subsequently can cause environmental problem. Residual oil recovered from POME (sludge palm oil) is used for non-edible applications such as in the producing of laundry soap and biodiesel. Focus of this research is to recover oil from POME by solvent extraction method. N-hexane, n-pentane, diethyl ether and ethanol were used as solvents. The quality of oil extraction and best solvent in single solvent extraction and combination of solvents extraction was determined at different solvent ratios (1:15 and 1:1.5). Result showed that ethanol is the best single solvent with 20.61% oil recovery at 1:15 ratio and 32.85% oil recovery at 1:1.5 ratio, meanwhile combination of ethanol and n-hexane is the best solvent combination with 2.14% oil recovery at 1:15 ratio and 10.41% oil recovery at 1:1.5 ratio. Extraction at smaller POME to solvent ratio (1:1.5) gave higher percentage of oil recovery.

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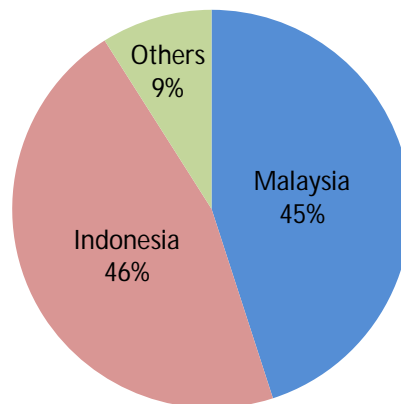
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CHAPTER 1

INTRODUCTION

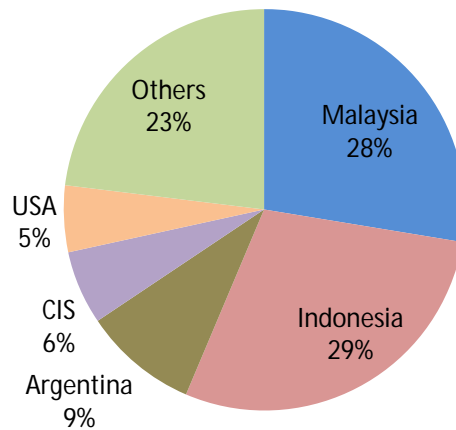
1.1 Background of Study

Palm oil is extracted from oil palm tree (*Elaeis guineensis*), which originated from West Africa. In early 1870's, oil palm tree was introduced in Malaysia as ornamental plant by the British. Nowadays oil palm tree is one of the most important commercial crops in Malaysia. Malaysia is known as one of the world's largest palm oil producer, which contributes to 11% of the world's oil and fat production and 27% of export trade of oils and fats. According to Malaysia Palm Oil Council (MPOC), about 4.49 million hectares of land in Malaysia is under oil palm cultivation; producing 17.73 million tons of palm oil and 2.13 tons of palm kernel oil.



(Source: GOFB)

Figure 1.1: World palm oil exporters, 2009: 35.36 million tonnes



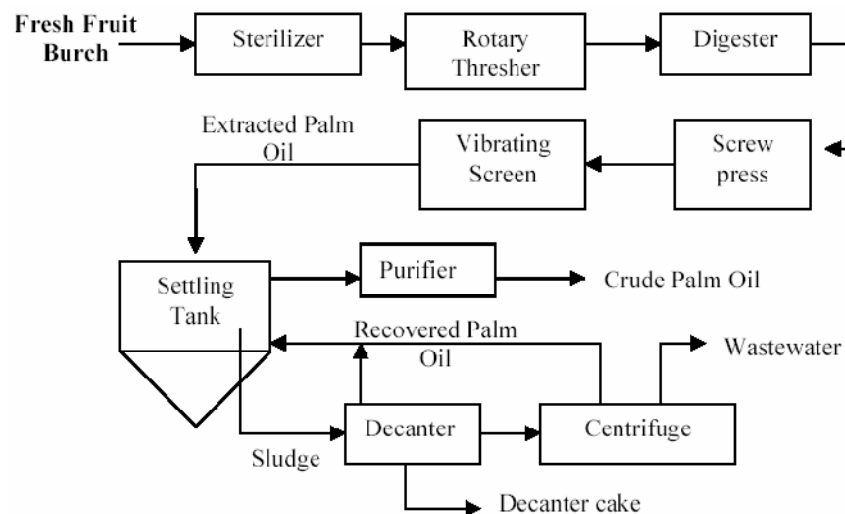
(Source: MPOC)

Figure 1.2: World major exporters of oil and fat, 2009: 63.09 million tonnes

The most common type of oil palm species grown in Malaysia is *Tenera* species, which is the cross-breed of *Dura* and *Pisifera* species. There are two types of oils that can be produced from oil palm fruit, namely: crude palm oil (CPO) and crude palm kernel oil (CPKO). Crude palm oil is obtained from the fibrous mesocarp and crude palm kernel oil from the kernels. The *Tenera* species can produce about 4 to 5 tonnes of crude palm oil per hectare per year and about 1 tonne of palm kernels.

In order to produce crude palm oil, palm oil mills will generate large amount of solid and liquid by-products such as empty fruit bunch (EFB), fibre, shell and palm oil mill effluent (POME). The production of one tonne of crude palm oil requires five to seven tones of water and about half of the water ends up as liquid waste known as POME. POME is non-toxic, organic in nature but has an unpleasant odour and is highly polluting which originated from the mixture of a sterilizer condensate, separator sludge and hydrocyclone wastewater (Ahmad *et al.*, 2009).

Ahmad *et al.* (2009) reported that the oil droplets of POME can be found in two phases; suspended in solids or floating in the supernatant. POME is considered highly pollutant if the oil content exceeding 6000 mg/l according to Environmental Quality Regulations 1977 for crude palm oil. Generally in palm oil mills, oil losses can occur at various stages of milling, particularly at the sterilizer station and oil recovery station. POME must be treated to an acceptable level set by the Government before discharge due to its potential hazard to the environment.



Source: (Chungsiriporn *et al.*, 2005)

Figure 1.3: Palm oil milling process

There are numerous methods available for treatments of POME especially to remove residual oil in POME. Wahab *et al.* (2010) mentioned that there are two common methods in order to remove residual oil in POME. The first one is the conventional method that is done by skimming the oil from the surface of the cooling pond and the second method is by solvent extraction with hexane or petroleum ether as the solvent. Meanwhile, Ibrahim *et al.* (2003) stated that solvent extraction and adsorption are used for removal of residual oil in POME on batch basis.

Solvent extraction usually used in extraction of vegetable oil. Solvent extraction is a process of separating compounds based on their relative solubilities in two different immiscible liquids, usually water and an organic solvent. Majority used n-hexane and petroleum ether as the solvent for solvent extraction. However, most of them conducted their study by using single solvent extraction only where it is rare to find research on different solvent combinations for palm oil recovery.

1.2 Problem Statement

POME will be a major threat to environment if it not treated properly. The production of one tonne of crude palm oil requires five to seven tones of water and about half of the water ends up as liquid waste known as POME (Ahmad *et al.*, 2009). With 416 mills operating in Malaysia, huge amount of POME will be generated. Oil and grease present in POME and has poor solubility in water. Oil and grease must be removed in order to prevent interfaces in water treatment units, reduce fouling in process equipment, avoid problems in biological treatment stages and comply with water discharge requirements (Ahmad *et al.*, 2005). The accumulation of oil in treatment pond will lead to ineffective wastewater treatment and increase the organic loading rate in anaerobic pond. With the increasing of the organic loading rate in anaerobic pond, the conversion of organic matter to methane gas will increase and may cause odor problems to the resident down-wind (Pittaway, 2001). Oil recovery can reduce the accumulation of oil in pond system and one of the options is to use solvent extraction method. Further study can be conducted in order to determine the best solvent with high efficiency rate of extraction.

1.3 Objective

The objectives of this research are;

- 1) To determine the best combination of solvents for high yield of oil recovery.
- 2) To establish the best POME to solvent ratio for high oil recovery.
- 3) To characterize oil quality recovered for solvent extraction.

1.4 Scope of Work

In order to achieve the main objective of this research to recover oil from POME, solvent extraction method is used. Solvent extraction method is used because it is simple, suitable for liquid-liquid extraction and gives high extraction rate. N-hexane, n-pentane, diethyl ether and ethanol are solvents that were used in this study. In this research there are two type of solvent extraction method is applied which are single solvent and combination of solvents. Both of the extraction methods are done at two different solvent to POME ratios. The percentage of oil recovered from the POME can be determined by the given equation. Afterward, the recovered oil from the POME is also analyzed by using analysis equipment such as Fourier Transform Infrared (FTIR), UV-Spectrometry, X-Ray Diffraction (XRD) and CHNS elemental analyzer.

CHAPTER 2

LITERATURE REVIEW

2.1 Oil Palm Industries

According to Malaysian Palm Oil Council (2011), Malaysia currently accounts for 39 % of world palm oil production and 44% of world exports. Being one of the biggest producers and exporters of palm oil and palm oil products, Malaysia has gained lots of positive returns particularly in economy sector.

Fresh fruit bunch (EFB) is the ripe bunch which contains 1000 to 3000 fruits. The oil palm tree requires only 0.26 hectares of land in order to produce 1 tonne of oil whereas soybean, sunflower and rapeseed require 2.22, 2 and 1.52 hectares respectively to produce the same. This makes the oil palm tree as the most efficient oil-bearing crop in the world (MPOC, 2011).

However, only 10% of oil palm fruit is economically valuable and is used for the production of crude palm oil (palm oil and palm kernel oil), whereas the remaining 90% is constituted of oil palm trunks (OPT), oil palm fronds (OPF), empty fruit bunches (EFB), and palm pressed fibers (PPF) (Ratnasingam *et al.*, 2008). The generation of by-products which is greater than the production of crude palm oil can contribute to environmental problem. Obot *et al.* (2007) reported that many agricultural by-products from agricultural activities and agro-based processing litter the environments and constitute waste problems.

It is also estimated that for each ton of crude palm oil that is produced, 5 – 7.5 ton of water are required, and more than 50% of this water ends up as POME (Ahmad *et al.*, 2003). POME is the liquid waste that is generated in order to produce the crude palm oil and it is highly polluting. With 4.49 million hectares of land in Malaysia is under oil palm cultivation which producing 17.73 million tons of palm oil and 2.13 tons of palm kernel oil, the amount of POME generated can cause major source of pollution if it is not treated properly. In order to avoid such problem to occur, solid and liquid palm oil waste must be manipulated in order to transform into more profitable sources.

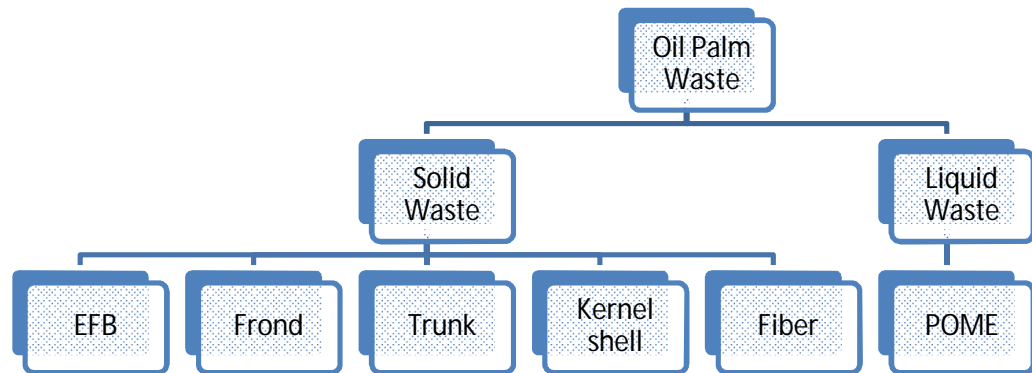


Figure 2.1: Oil Palm Waste

2.2 Palm Oil Mill Effluent (POME)



Figure 2.2: Palm Oil Mill Effluent (POME)

Palm oil mill effluent (POME) is the waste water produced by the palm oil industry. It consists of colloidal suspension of 95-96% water, 0.6-0.7% oil and 4-5% total solid including 2-4% suspended solids originating in the mixing of sterilizer condensate, separator sludge and hydrocyclone waste water that are mostly debris from palm fruit mesocarp (Ahmad *et al.*, 2005). The characteristics of POME for each process are shown in Table 2.1. It is estimated that the oil content in the sterilizer condensate and sludge or decanter is about 0.16% and 0.46% of the fresh fruit bunches (FFB) respectively (Othman *et al.*, 2003).

Table 2.1: Characteristics of POME

Parameters	Sterilizer effluent (g/L)	Hydrocyclone effluent (g/L)	Centrifuge effluent (g/L)	Mixed effluent (g/L)
BOD	10 – 25	-	17 – 35	11 – 30
COD	30 – 60	-	40 – 75	30 – 70
TS	40 – 50	5 – 15	35 – 70	30 – 65
SS	3 – 5	5 – 12	12 – 18	9 – 25
Oil	2 – 3	1 – 5	5 – 15	5 – 13
A – N	0.02 – 0.08	-	0.02 – 0.08	0.02 – 0.08
TN	0.35 – 0.60	0.07 – 0.15	0.5 – 0.9	0.5 – 0.9
pH	4.5 – 5.5	-	3.5 – 4.5	3.5 – 4.5

(Source: Borja *et al.*, 1995)

There will be considerable environmental issues that can happen if the effluent is left untreated and discharged directly into waterway. This is because of the high biochemical oxygen demand (19020 mg/l), chemical oxygen demand (53630 mg/l), oil and grease (8370 mg/l), total solids (43635 mg/l) and suspended solids (19020 mg/l) in the effluent (Ma, 1995). The raw or untreated POME has an extremely high content of degradable organic matter, which is due in part to the presence of unrecovered oil (Lin, 2005).

POME also contains soluble materials for example gases like CH₄, SO₂, NH₃, halogens or soluble liquids or solids which contain ions which originated either from organic or non-organic with concentration above threshold value and very harmful to the environment as mentioned by Igwe *et al.* in 2007.

Igberaharha (1998) and Igwe (2007) also reported that in industrial effluents have the characteristics as follows:

- 1) Soluble organic resulting in dissolve oxygen depleting
- 2) Organic suspended solid resulting in dissolve oxygen depleting
- 3) Inert suspended solid causing turbidity and resulting in bottom sediments
- 4) Toxic substances and heavy metals
- 5) Oil floating materials
- 6) Dissolved salts particularly phosphates, chlorides and nitrates.

Okwute & Isu (2007) and Ahmad *et al.* (2003) also stated that POME is also rich in mineral content, particularly phosphorus, potassium, magnesium and calcium, aside from organic composition. Therefore, the government has introduced the POME discharge limit in order to help to protect and sustain the environment. The standard discharge limit according to Malaysian Department of the Environment can be shown in Table 2.2.

Table 2.2: Characteristics of POME and its respective standard discharge limit

Parameter	Concentration (mg/L)	Standard Limit (mg/L)
pH	4.7	5 – 9
Oil and Grease	4,000	50
BOD	25,000	100
COD	50,000	50
TS	40,500	-
SS	18,000	400

TN	750	150
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(Source: Abdul Latif *et al.*, 2003)

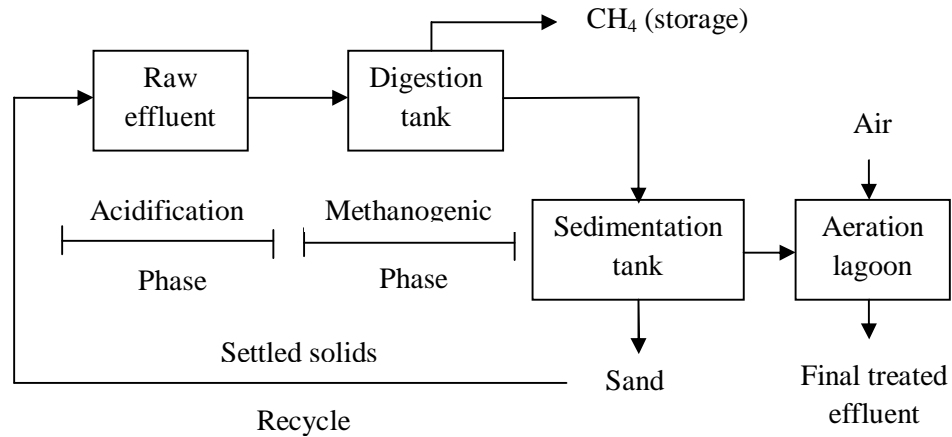
2.3 Palm Oil Mill Effluent (POME) Treatment

POME is known as one of major contributor to the industrial pollution in palm oil industry. This is because it contains compound that can offer potential hazard to the environment if it left untreated. Thus, proper effluent management is needed in order to protect the environment.

There are lots of methods or techniques have been employed in order to treat the effluent. One of techniques used is mechanical technique which involves sedimentation, filtration, and decolorization of effluent. Mechanical technique is used at the first stage of purification process in order to remove the suspended solids which devices such as sieve, sedimentation bed and filter is used. This technique is often called as primary treatment (Igwe *et al.*, 2007).

Another technique is physic-chemical technique which consists of coagulation of finely dispersed and suspended solid particles, adsorption of the dissolved impurities such as heavy metal (Igwe *et al.*, 2003 & 2005; Namasivayam *et al.*, 1998 and Ngah *et al.*, 1999), selective crystallization, reverse osmosis and ion-exchange process (Chow *et al.*, 1981). According to Igwe *et al.* (2007), reverse osmosis is commonly used at the final stage of effluent treatment.

The final treatment technique is biological technique, which commonly known as secondary treatment. This secondary treatment is widely used for effluent treatment in palm oil mill industries which includes process such as activated sludge, tricking filters, contact stabilization, etc as reported by Chow *et al.* (1981).



(Source: *Igwe et al.*, 2007)

Figure 2.3: Flow chart for process treatment of the effluent water

2.4 Recovery of Oil

Recovery of oil from POME is one of the alternatives to minimize oil losses during the milling processes (*Wahab et al.*, 2010). Recovery of oil also is done in order to abide the standard discharge limit for oil and grease, according to Environmental Quality (prescribed premises) (crude palm oil) Regulations 1977 the standard discharge limit for oil and grease is 50 mg/L while the concentration of oil and grease in POME is about 6000 mg/L.

Sludge palm oil (SPO) is the recovered oil from POME. SPO is the third grade oil because of the low quality compared to the typical crude palm oil. It contains high fatty acid (FFA), high moisture and impurity contents (*Ainie et al.*, 1995). The SPO can be sold and the price is about 40% to 60% of the normal price of CPO. The application of SPO is in non-edible applications such as in producing laundry soap, fatty acids, candles and biodiesel. Therefore recovering oil in POME not only can minimize oil losses and reduce environmental problem, it also can generate profit because there is market demand for SPO.

There are several methods that have been developed for treatment of POME. The most conventional method is biological treatment of aerobic and anaerobic. This treatment method relies on microorganisms by breaking down the pollutant in POME. Evaporation process also one of the POME treatment method as mentioned by Ma (1998) and Ibrahim *et al.* (2003).

Oil can be recovered by removing and extracting residual oil in POME during the treatment process. According to Andrew *et al.* (2000) and Ahmad *et al.* (2005), adsorption, flocculation, electro-coagulation and flotation are been used to remove residual oil from wastewater. As mentioned earlier in the report, Wahab *et al.* (2010) stated that removal of residual oil in POME is done by skimming the oil from the surface of the cooling pond and by solvent extraction with hexane or petroleum ether as the solvent. Meanwhile, Ibrahim *et al.* (2003) reported that solvent extraction and adsorption are used in order to remove residual oil in POME on batch basis.

2.4 Solvent Extraction

POME consists of organic component and inorganic component. In order to separate these two components, solvent extraction method is used. Solvent extraction or liquid-liquid extraction method is the separation of constituents solution from a liquid solution by contact with another liquid in which the constituents are more soluble (Freeman, 1989). In order to remove the organic components in the POME, organic solvent will be used. The organic component is more soluble in the organic solvent, thus it will combine in the organic solvent and simultaneously, separating the organic component from the inorganic component. It is an ex situ separation and concentration process in which a non-aqueous liquid is used to remove organic contaminants (Silva *et al.*, 2005).

Solvent extraction method is used commercially in hydrometallurgy and widely within the chemical industry including organic chemical, petrochemical and pharmaceuticals (Ahmad *et al.*, 2003). Almost all of vegetable oil recovery plants are using solvent extraction method for oil recovery and purification.

According to Belhateche (1995), here are four basic components for the solvent extraction that can be listed as follows;

- 1) Contact between wastewater and solvent;
- 2) Separation of extracted wastewater and solvent;
- 3) Treatment of solvent to remove extracted constituents;
- 4) Treatment of wastewater to remove residual solvent

Table 2.3: Oil yield, FFA and peroxide value of solvent-extracted oil

Solvent	Oil yield (%)	FFA (%)	Peroxide value
Methanol	24.4	17.25	-
Ethanol	42.4	10.90	-
Isopropanol	44.2	8.70	-
Petroleum ether	36.6	8.03	2.12
Pentane	38.0	8.46	0.59
Hexane	37.7	8.11	0.32
Heptane	37.6	7.96	0.26
Crude palm oil	-	3.76	0.82

(Source: Lee *et al.*, 2000)

CHAPTER 3

METHODOLOGY

3.1 Materials Preparation

3.1.1 Fresh POME Sample

The fresh POME sample was collected from a palm oil mill, Felcra Nasaruddin Palm Oil Mill, Bota, Perak.

3.1.2 Reagents

Solvents that were used in this experiment are n-hexane, n-pentane, diethyl ether and ethanol.

3.1.3 Apparatus

Main apparatus that were needed in this for this experimental work are Soxhlet extractor for extraction process, heating mantle for heating purposes, drying oven for drying process and analytical balance for weight measuring.

3.2 Experimental Methodology

3.2.1 Single Solvent Extraction

Single solvent extraction only requires one type of solvent for the extraction process. 10 ml of fresh POME sample was weighted and mixed with 150 ml of solvent in solvent vessel. The mixture was left for extraction process for four hours. Then, the mixture was filtered and transferred into separation funnel for complete layer separation. The extracted oil was transferred into rotary evaporator in order to distill off all the solvent. Then the volume and weight of extracted oil was measured. The drying process was done in drying oven at 30°C for 48 hours or until constant weight was obtained. The volume and weight of the extracted POME after the drying process was measured. Finally, all the samples were sent for analysis.

Table 3.1: Single solvent extraction

Sample	Solvent
1	n-hexane
2	n-pentane
3	diethyl ether
4	ethanol

3.2.2 Combination of Solvents Extraction

Combination of solvents solvent require more than one type of solvent for the extraction process. However, solvent were used in this experiment are combination of two type of solvents. 10 ml of fresh POME sample was weighted and mixed with 75 ml of solvent A and 75 ml of solvent B in solvent vessel. The mixture was left for extraction process for four hours. Then, the mixture was filtered and transferred into separation funnel for

complete layer separation. The extracted POME was transferred into rotary evaporator in order to distill off all the solvent. Then the volume and weight of extracted POME was measured. The drying process was done in drying oven at 30°C for 48 hours or until constant weight was obtained. The volume and weight of the extracted POME after the drying process is measured. Finally, all the samples were sent for analysis.

Table 3.2: Combination of solvents extraction

Sample	Solvent A	Solvent B
5	n-hexane	n-pentane
6	n-hexane	diethyl ether
7	n-pentane	diethyl ether
8	ethanol	n-hexane
9	ethanol	n-pentane
10	ethanol	diethyl ether

3.2.3 Different POME - Solvent Ratio Extraction

For different POME - solvent ratio extraction, the same experimental methodology for both single and combination of solvents was repeated, however the volume of fresh POME sample used was 100 ml instead of 10 ml. In this experiment, the ratio was reduced from 1:15 to 1:1.5. the percentage of oil extracted is shown as follows:

$$\% \text{ Oil recovery} = \frac{\text{Weight of oil extracted}}{\text{Weight of POME}} \times 100$$

3.2.4 General Experimental Steps

Figure 3.1 shows the general sequence of experimental steps for each experiment in this research.

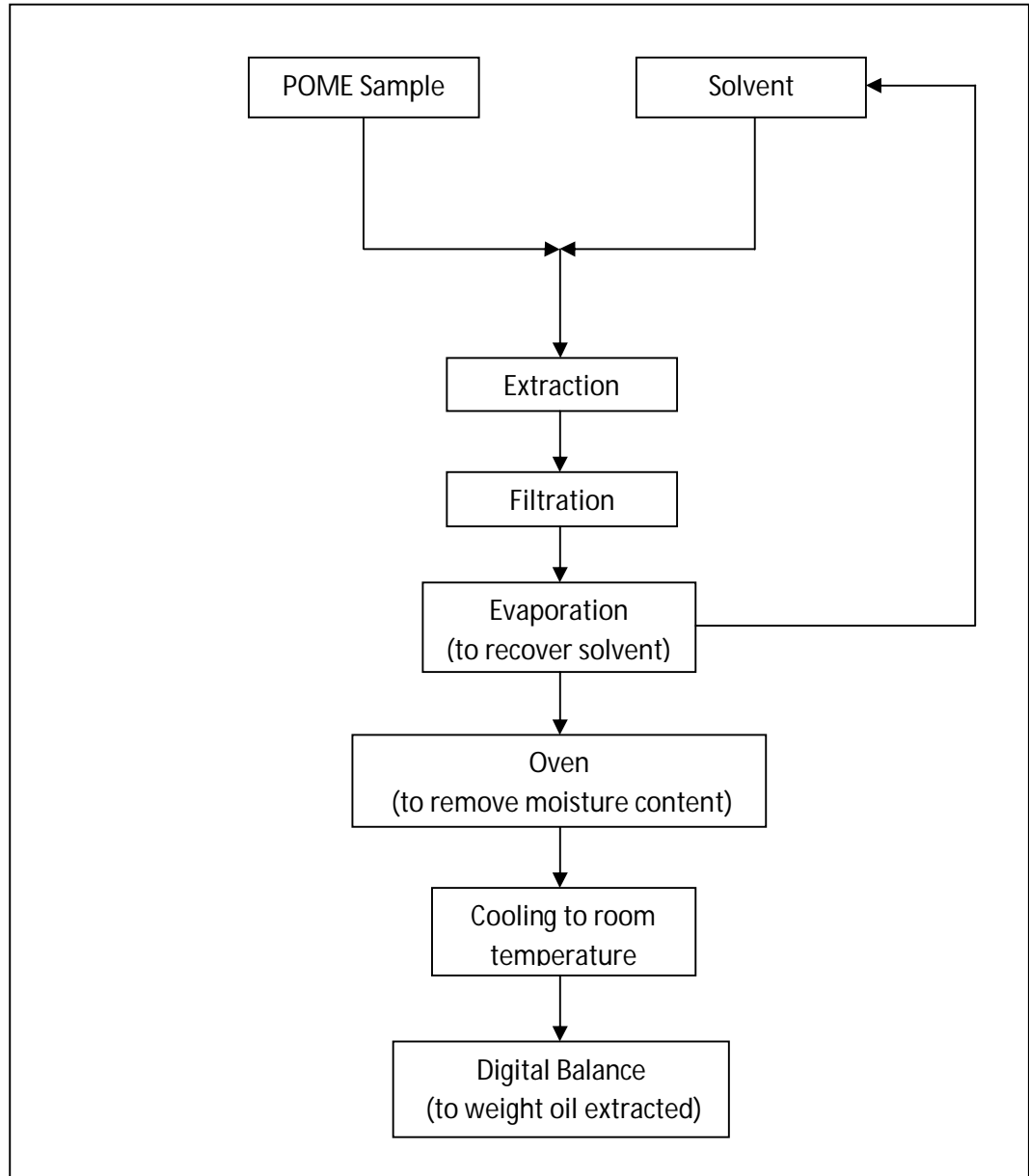


Figure 3.1: General sequence for experimental steps

3.3 Characterization

3.3.1 UV-Visible

UV-Visible analyzer used will provide quantitative determination of different analytes, such as transition metal ions, highly conjugated organic compounds, and biological macromolecules.

3.3.2 FTIR

Fourier Transform Infra-Red (FTIR) analysis test will identify chemicals compound that are present in the sample. The compound may either organic or inorganic.

3.3.3 CHNS

CHNS elemental analyzers will determine the carbon, hydrogen, nitrogen and sulphur in organic matrices and other types of materials.

3.4 Gantt Chart

The Gantt chart (Figure 3.2) shows the overall progress of this research throughout this semester.

ACTIVITIES / WEEK	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	
Project Work	█	█	█	█	█	█	MID SEM BREAK	█	█	█	█	█				
Progress Report								█								
Pre-EDX											█					
EDX												█				
Draft Report													█			
Dissertation (soft bound)														█		
Technical Paper														█		
Oral Presentation															█	
Dissertation (hard bound)																█

Figure 3.2: Gantt Chart

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Percentage Oil Recovery

Table 4.1: Percentage of oil recovery at different solvent ratio.

Sample	Solvent	% Oil Recovery	
		Solvent Ratio 1:15	Solvent Ratio 1:1.5
1	n-hexane	10.38	29.30
2	n-pentane	0.83	1.42
3	diethyl ether	1.69	2.63
4	ethanol	20.61	32.85
5	n-hexane + n-pentane	1.77	7.53
6	n-hexane + diethyl ether	1.13	1.77
7	n-pentane +diethyl ether	0.17	1.09
8	ethanol + n-hexane	2.14	10.41
9	ethanol + n-pentane	1.73	3.79
10	ethanol + diethyl ether	1.60	5.24

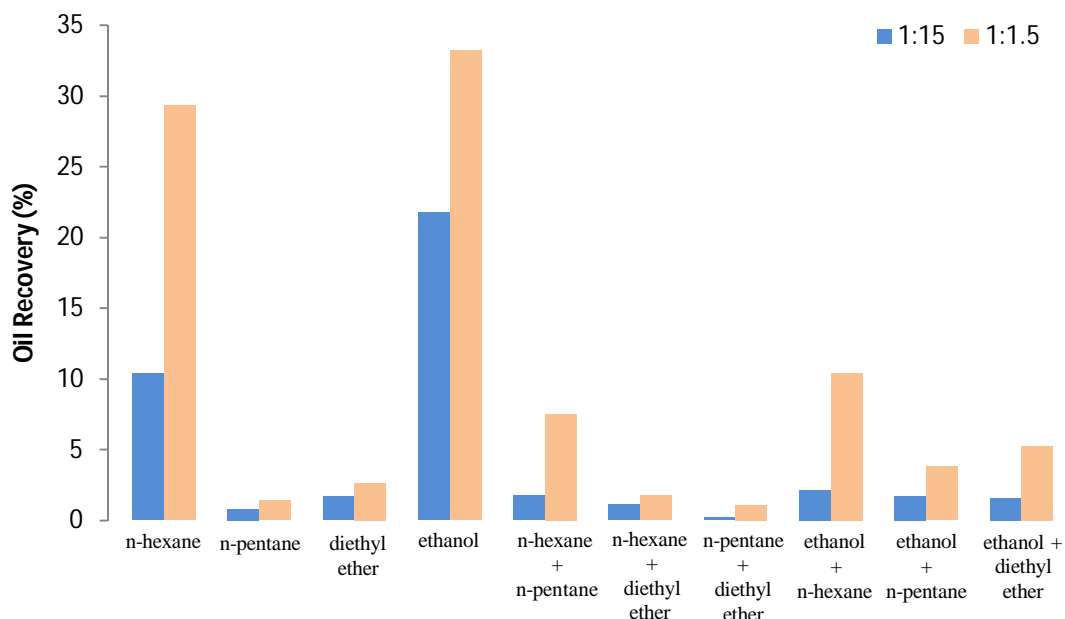


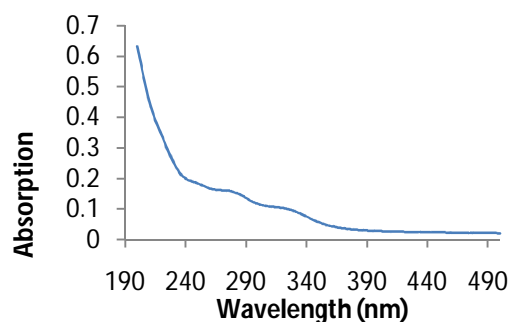
Figure 4.1: Oil Recovery

Table 4.1 shows the percentage of oil recovery in both single solvent extraction and combination of solvent extraction at two different solvent to POME ratios; 1:15 and 1:1.5. For single solvent extraction at 1:15 solvent to POME ratio, the highest yield of oil recovery is in ethanol with 20.61% of oil recovery. Then, n-hexane, diethyl ether and followed by n-pentane, 0.83%. The similar trend is obtained for single solvent extraction at 1:1.5 solvent to POME ratio, which ethanol is the highest oil recovery with 32.85%. This is proven by the previous study conducted by Ahmad *et al.* in 2009.

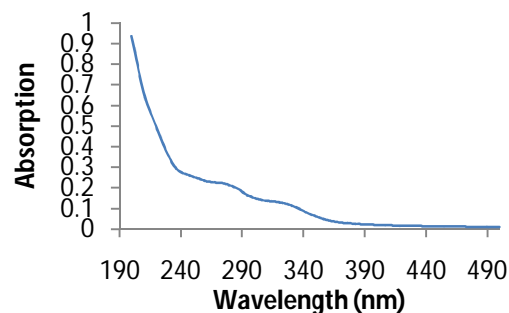
For combination of solvents extraction at both 1:15 and 1:1.5 ratios, combination of n-hexane and ethanol give the highest yield of oil recovery which is 2.14% and 10.41% respectively. The percentage of oil recovery in combination of n-pentane and diethyl ether is the lowest with 0.17% at 1:15 ratio and 1.09% at 1:1.5 ratio. Combination of solvent extraction shows low oil recovery than single solvent extraction. From table 4.1 also, it is observed that the percentage of oil recovery at 1:1.5 ratio is slightly higher than 1:15 ratio. Thus, minimizing the volume of solvent can increase the percentage of oil recovery.

For single solvent extraction, ethanol ($\text{CH}_3\text{CH}_2\text{OH}$) has the highest yield of oil extracted because ethanol is one type of polar solvent. Polar solvent tend to dissolve polar compound and polarity of solvent is generated from bond dipole of O-H bond. In this study, polar solvent can extract more oil than non-polar solvents (n-hexane, diethyl ether and n-pentane). Percentage of oil recovery in combination of solvents extraction is lower than single solvent extraction because solvents in this type of extraction might have reacted with one another and slightly altered their original solvent properties. Thus, the rate of oil extracted is reduced.

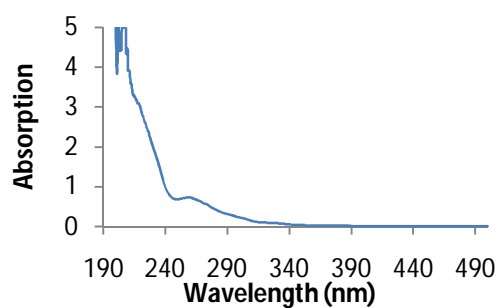
4.2 UV-VIS



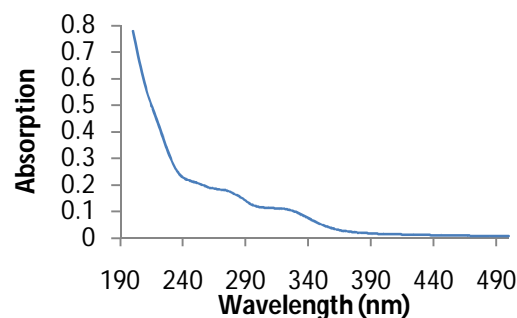
(a)



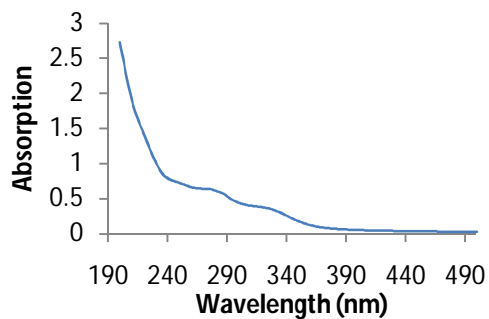
(b)



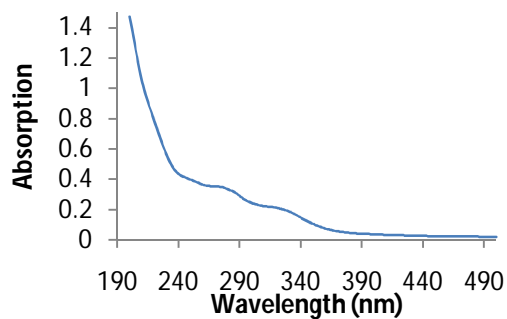
(c)



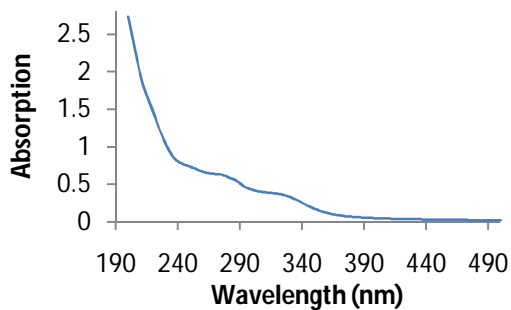
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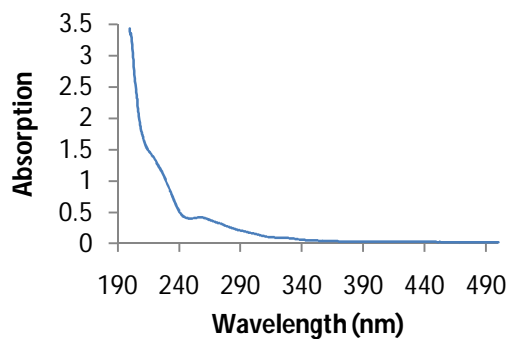
(e)



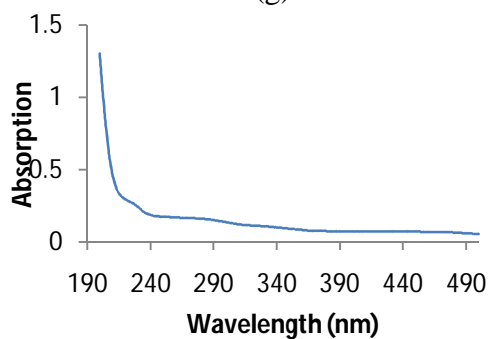
(f)



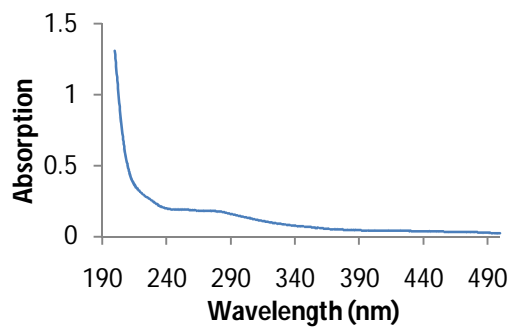
(g)



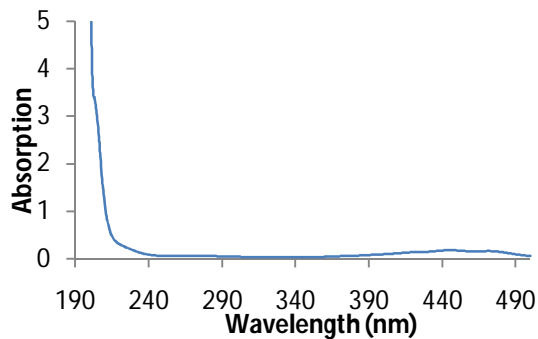
(h)



(i)

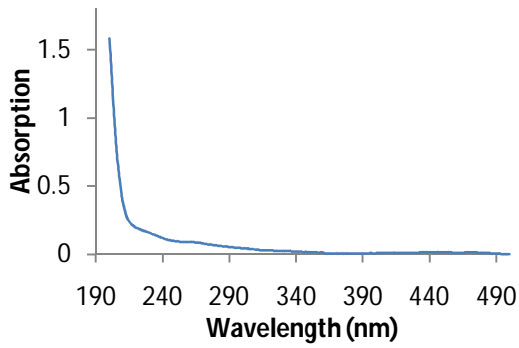


(j)

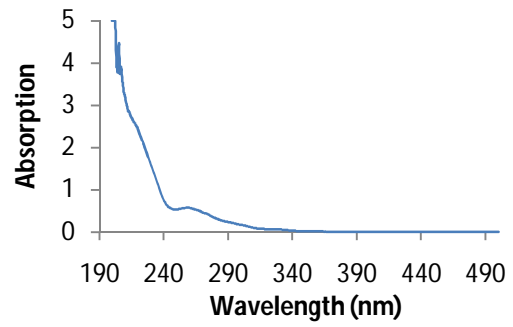


(k)

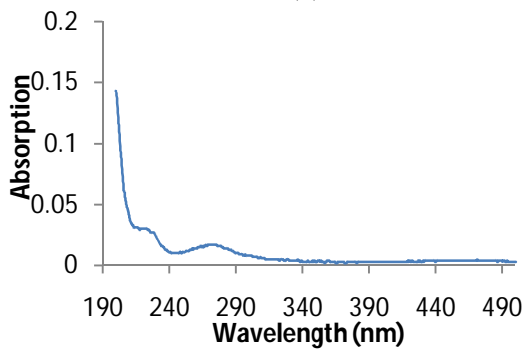
Figure 4.2: UV- Spectrometry of (a) n-hexane, (b) n-pentane, (c) diethyl ether, (d) ethanol, (e) n-hexane and n-pentane, (f) n-hexane and diethyl ether, (g) n-pentane and diethyl ether, (h) ethanol and n-hexane, (i) ethanol an n-pentane and (j) ethanol and diethyl ether at 1:15 ratio and (k) crude palm oil.



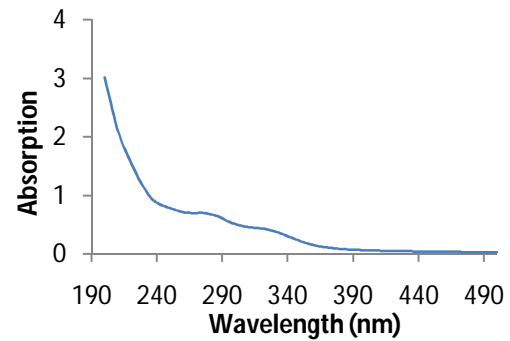
(a)



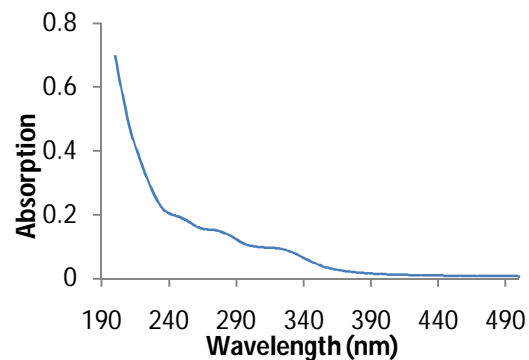
(b)



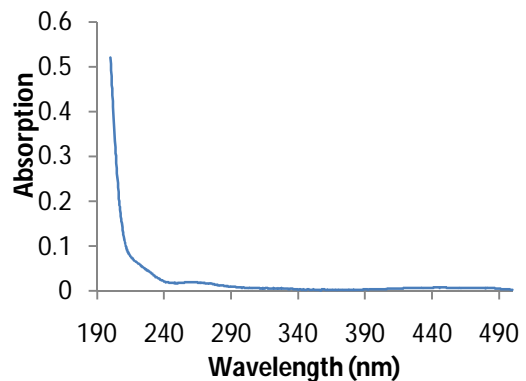
(c)



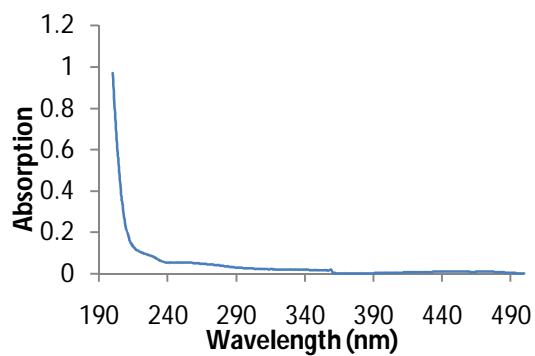
(d)



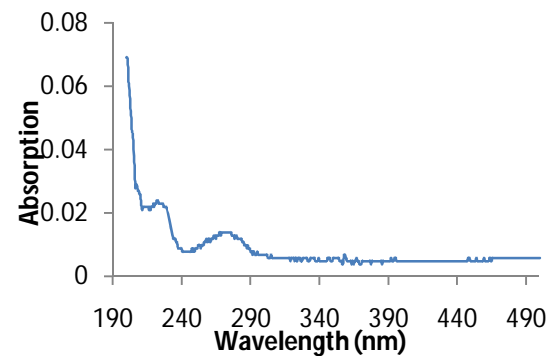
(e)



(f)



(g)



(h)

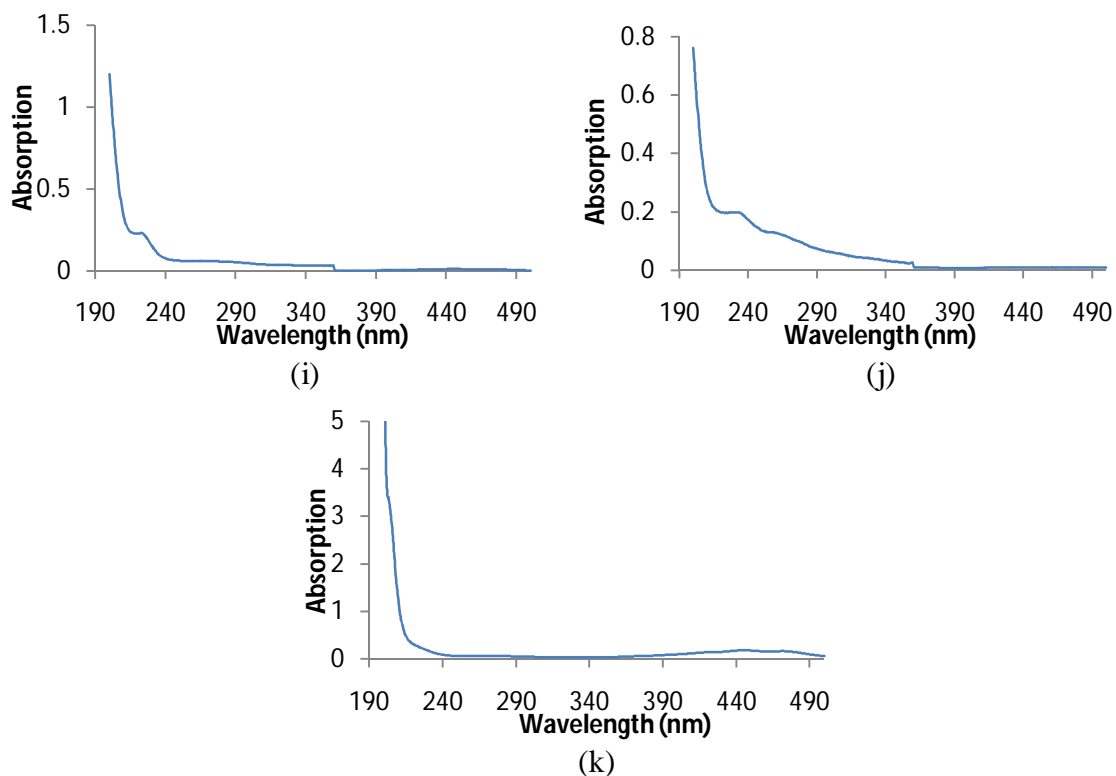
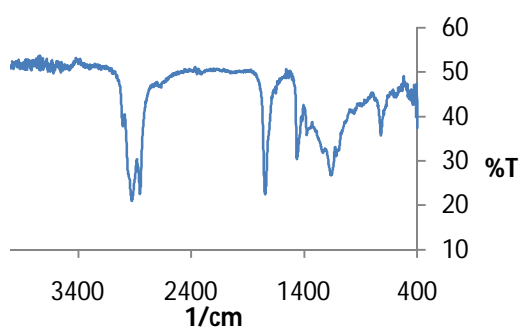


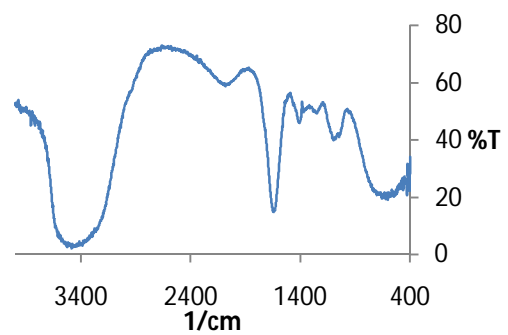
Figure 4.3: UV- Spectrometry of (a) n-hexane, (b) n-pentane, (c) diethyl ether, (d) ethanol, (e) n-hexane and n-pentane, (f) n-hexane and diethyl ether, (g) n-pentane and diethyl ether, (h) ethanol and n-hexane, (i) ethanol an n-pentane, (j) ethanol and diethyl ether at 1:1.5 ratio and (k) crude palm oil.

Based on figure 4.2, it is observed that the all samples have similar trend with standard crude palm oil sample. In addition, most of the samples also have peaks within wavelength range of 200 to 280, which indicate the presence carbonyl group in the sample. Hence, the oil presence in the sample is confirmed.

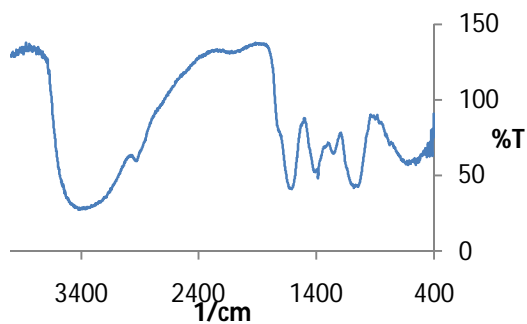
4.3 FTIR



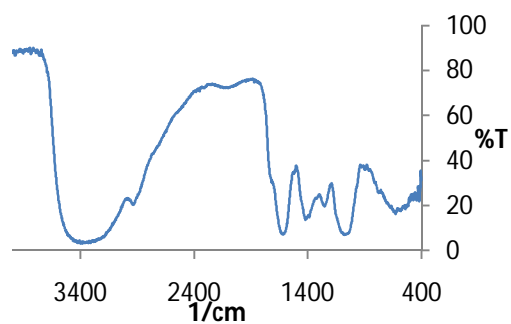
(a)



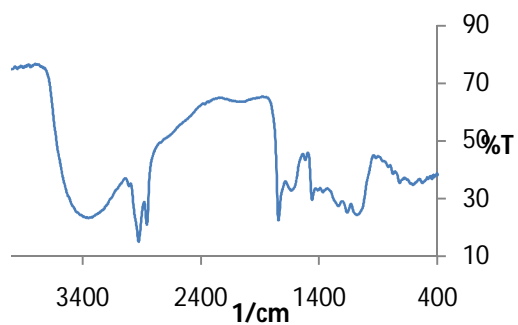
(b)



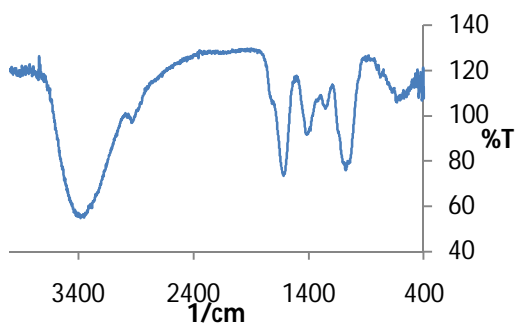
(c)



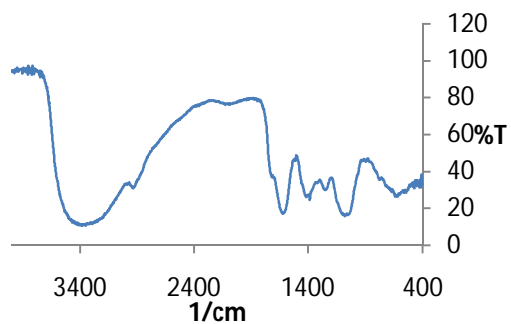
(d)



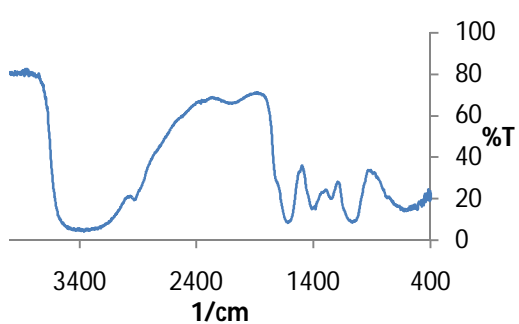
(e)



(f)



(g)



(h)

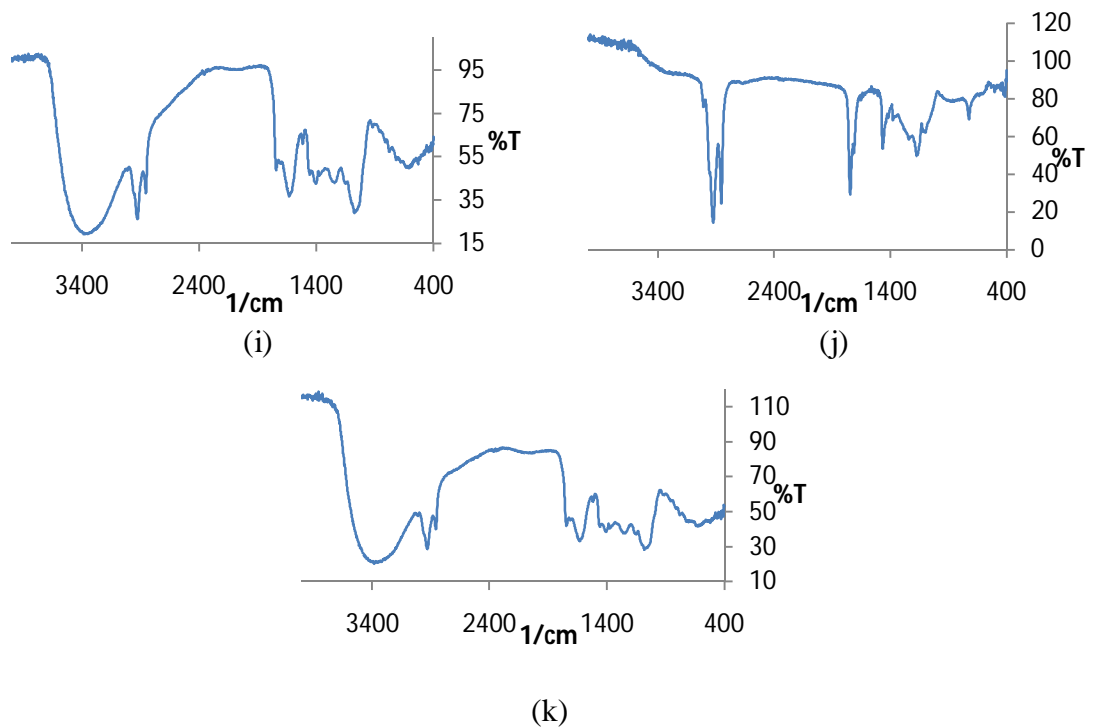


Figure 4.4: Fourier transform infrared (FTIR) of (a) crude palm oil, (b) n-hexane(c) n-pentane, (d) diethyl ether, (e) ethanol, (f) n-hexane and n-pentane, (g) n-hexane and diethyl ether, (h) n-pentane and diethyl ether, (i) ethanol and n-hexane, (j) ethanol and n-pentane and (k) ethanol and diethyl ether at 1:15 ratio.

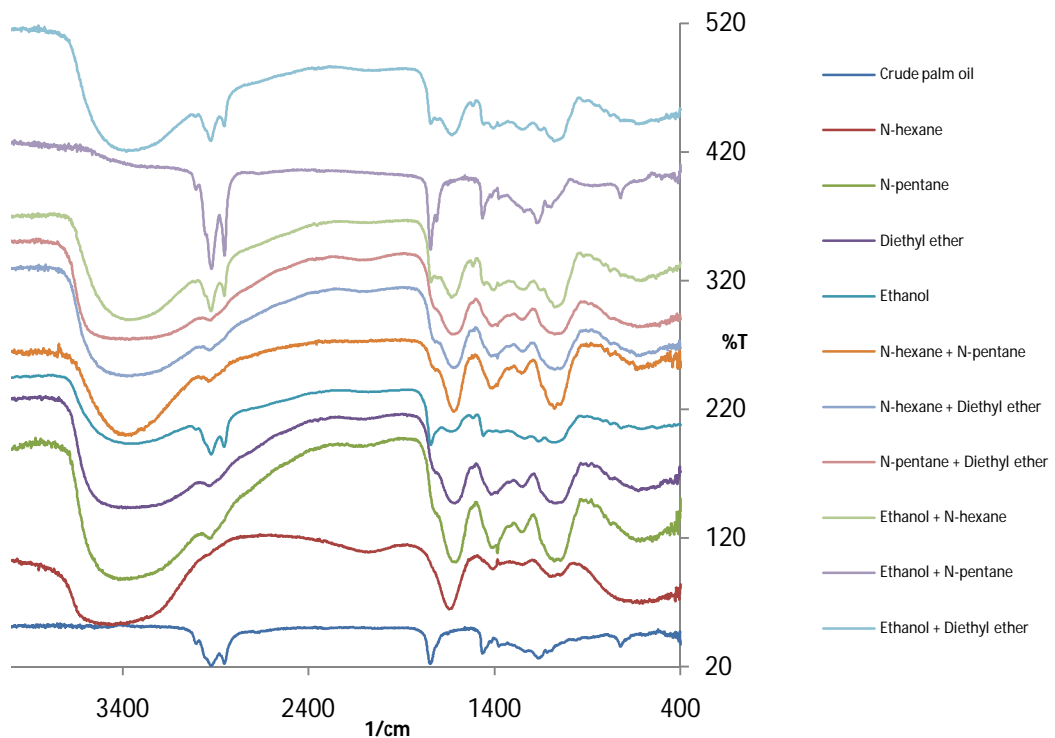
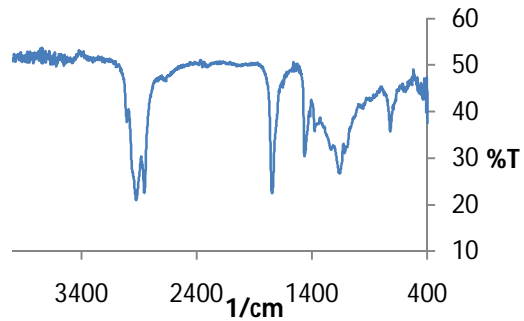
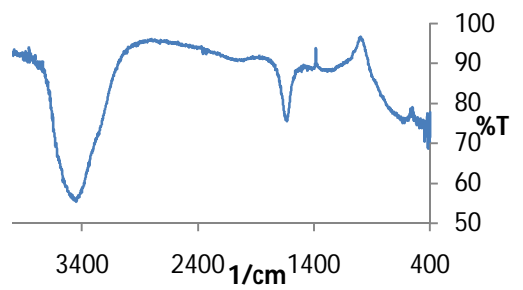


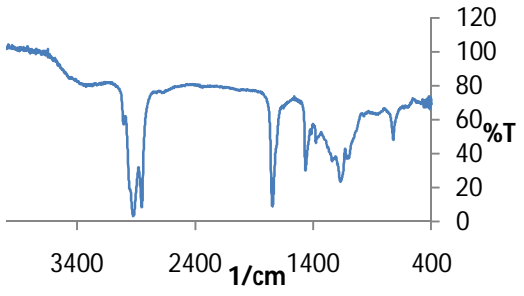
Figure 4.5: Fourier transform infrared (FTIR) at 1:15 ratio.



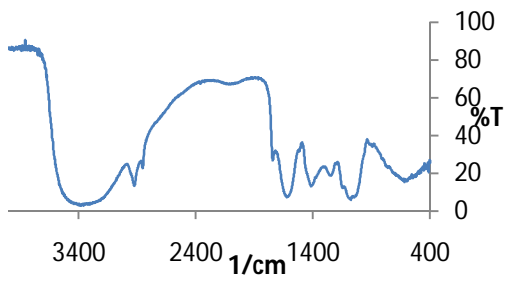
(a)



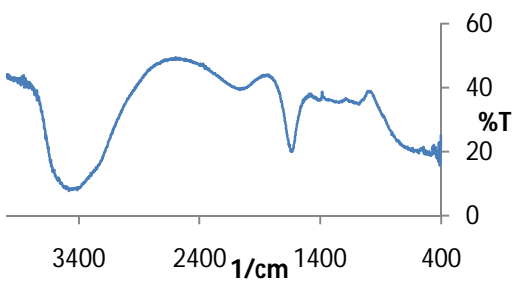
(b)



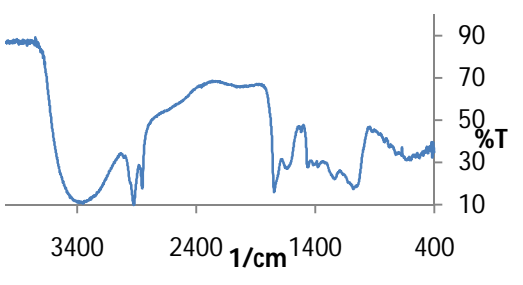
(c)



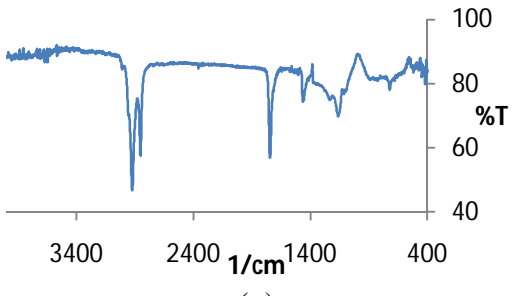
(d)



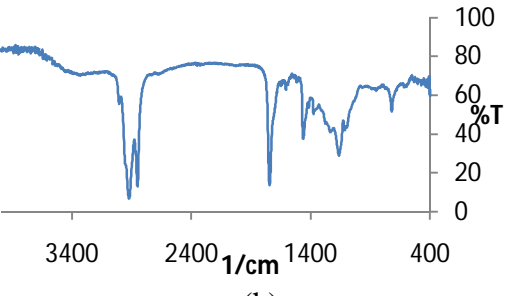
(e)



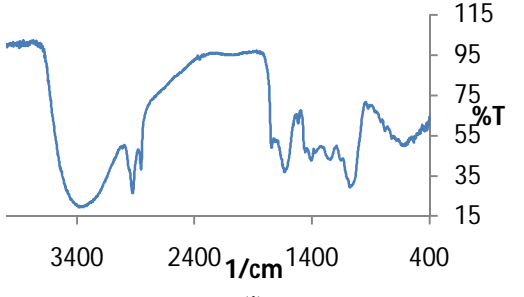
(f)



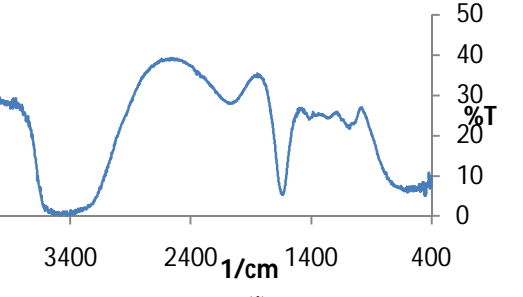
(g)



(h)



(i)



(j)

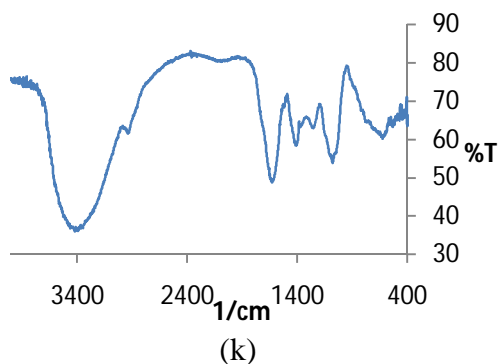


Figure 4.6: Fourier transform infrared (FTIR) of (a) crude palm oil, (b) n-hexane(c) n-pentane, (d) diethyl ether, (e) ethanol, (f) n-hexane and n-pentane, (g) n-hexane and diethyl ether, (h) n-pentane and diethyl ether, (i) ethanol and n-hexane, (j) ethanol and n-pentane and (k) ethanol and diethyl ether at 1:1.5 ratio.

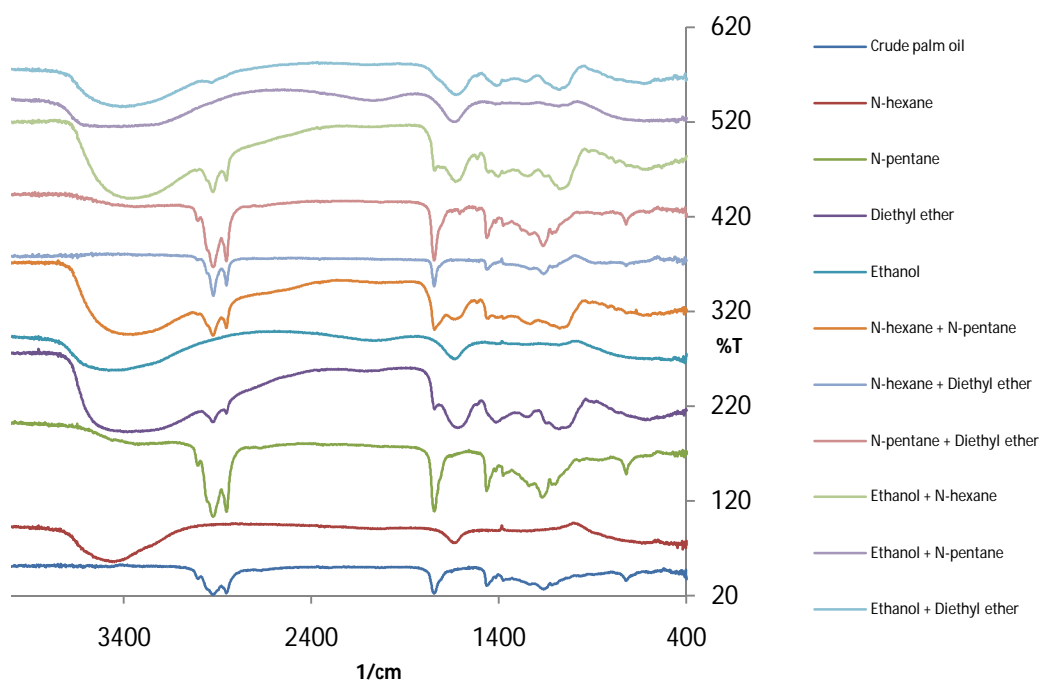


Figure 4.7: Fourier transform infrared (FTIR) at 1:1.5 ratio.

For FTIR analysis, crude palm oil sample is used as a standard sample in order to compare results obtained from the samples. The fingerprint of oil region is observed at 1000 to 1500 cm^{-1} (Che Man et al., 1999). According to Higson (2004), the group frequency absorptions of carboxyl region is at 1690 to 1760 cm^{-1} . Figure 4.3 show that most of the samples have peaks in the oil fingerprint region. Therefore, it is proven that oil is extracted from the POME.

4.4 CHNS

Table 4.2: CHNS analysis

Sample	Ratio	C	H	N	S
1	1:15	34.52	4.382	2.816	0.440
	1:1.5	49.83	6.120	2.670	0.180
2	1:15	35.00	5.026	2.766	0.688
	1:1.5	37.90	4.793	2.213	0.218
3	1:15	46.97	7.346	3.231	0.357
	1:1.5	44.28	5.294	2.860	0.063
4	1:15	35.02	4.439	2.514	-0.136
	1:1.5	56.81	8.321	2.668	0.588
5	1:15	52.26	8.633	2.737	0.414
	1:1.5	40.85	5.249	2.460	0.355
6	1:15	51.69	7.220	2.995	0.383
	1:1.5	34.82	4.787	2.304	0.110
7	1:15	51.31	6.687	2.822	0.287
	1:1.5	43.57	5.253	2.537	-0.005
8	1:15	77.29	11.49	4.010	2.004
	1:1.5	93.41	13.41	4.271	1.375
9	1:15	39.55	5.181	2.597	0.056
	1:1.5	45.73	5.638	2.499	0.143
10	1:15	39.66	4.907	2.588	0.057
	1:1.5	43.09	5.640	2.272	0.195

Based on table 4.2, most of the samples have high percentage of carbon and hydrogen element. This is because the compounds that present in the samples consist of mainly carbon and hydrogen element. For example, carboxylic acid and carotene is made up mostly from carbon and hydrogen element. As mentioned earlier in the report, POME also contains soluble materials for example gases like SO and NH₃ (Igwe *et al.*, 2007), so this is the reason why small percentage of nitrogen and sulphur present in the sample.

CHAPTER 5

CONCLUSION

5.1 Conclusion

- Solvent to POME ratio at 1:1.5 gives better oil recovery than 1:15 ratio.
- Ethanol is the best solvent in single solvent extraction with 20.61% oil recovery at 1:15 ratio and 32.85% oil recovery at 1:1.5 ratio.
- Combination of ethanol and n-hexane gives the highest oil extraction in combination of solvents extraction with 2.14% oil recovery at 1:15 ratio and 10.41% oil recovery at 1:1.5 ratio.

5.2 Recommendation

For future work, more analysis on the oil quality of oil can be conducted. There is only a few studies is conducted on the suspended solid of the POME, in this research there is no further analysis is done on the suspended solid from the POME due to time constraint. Thus, further analysis on the suspended solid from POME is recommended in order to find out another potential use of suspended solid for palm oil mill waste utilization.

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J. Chungsiriporn, S. Prasertsan and C. Bunyakan (2006). *Minimization of water consumption and process optimization of palm oil mills.* *Clean Techn Environ Policy.* 8:151-158

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J.C. Igwe, A.A. Abia (2005). *Sorption kinetics and intraparticle diffusivities of Cd, Pb and Zn on maize cob.* *Afr. J. Biotechnol.* 4: 509-512.

J.C. Igwe and C.C. Onyegbado (2007). *A review of palm oil mill effluent (Pome) water treatment.* *Global Journal of Environmental Research.* 1(2):54-62.

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APPENDICES

APPENDIX 1: Raw Data

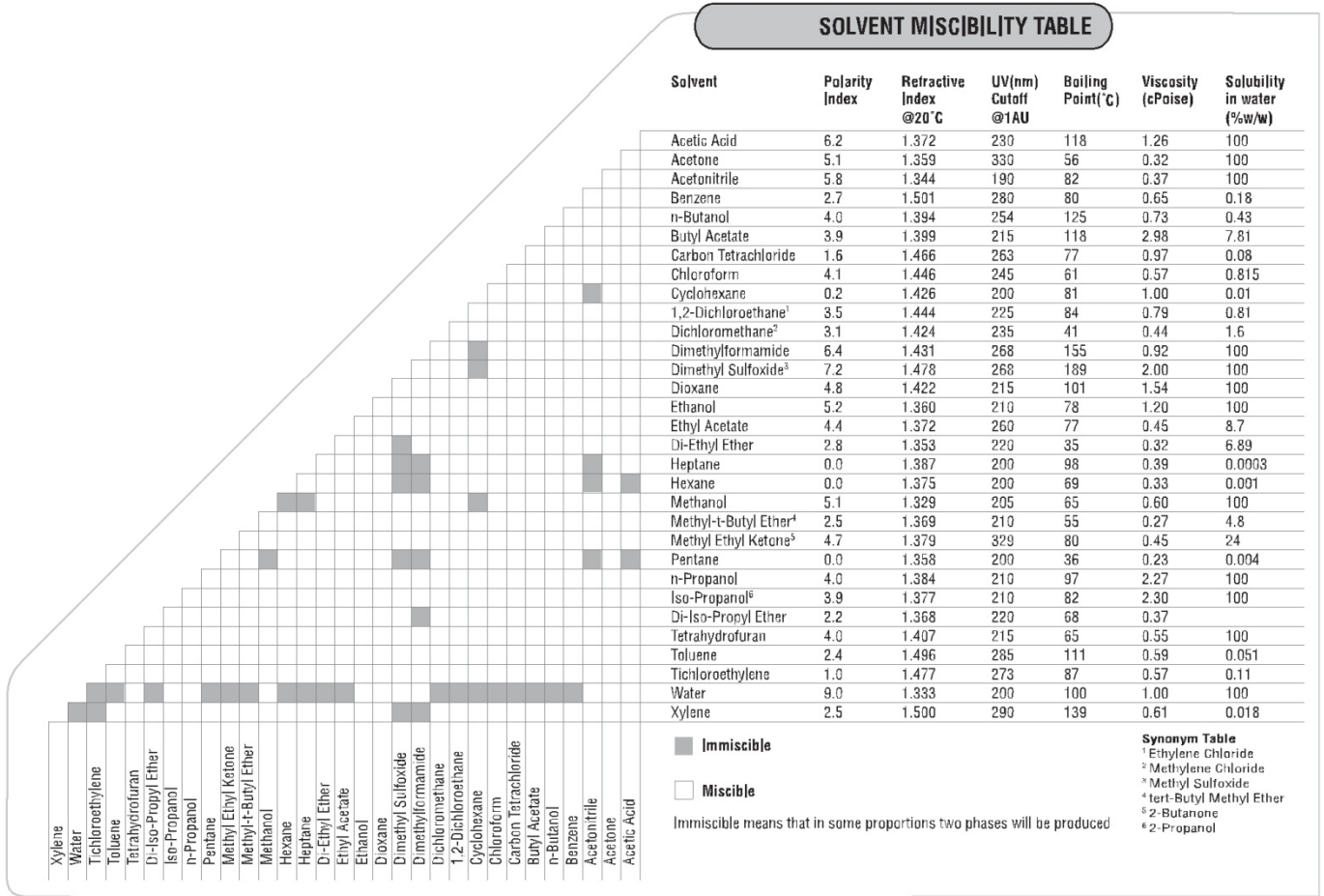
Solvent extraction at POME to solvent ratio 1:15

Sample	Solvents	Weight of 10 ml POME (g)	Weight before drying (g)	Weight after drying (g)
1	n-hexane	10.1555	5.2965	1.054
2	n-pentane	10.0100	2.2795	0.0830
3	diethyl ether	10.9555	3.2220	0.1853
4	ethanol	10.744	39.0922	2.2140
5	n-hexane + n-pentane	10.3550	2.3020	0.2218
6	n-hexane + diethyl ether	10.7800	1.6068	0.1214
7	n-pentane + diethyl ether	10.1330	3.8994	0.1832
8	ethanol + n-hexane	10.4262	21.8282	0.2231
9	ethanol + n-pentane	10.2366	36.8766	0.1772
10	ethanol + diethyl ether	10.0682	39.9386	0.1608

Solvent extraction at POME to solvent ratio 1:1.5

Sample	Solvents	Weight of 10 ml POME (g)	Weight before drying (g)	Weight after drying (g)
1	n-hexane	99.72	63.9872	29.2
2	n-pentane	97.3922	40.4062	1.3864
3	diethyl ether	97.3718	54.5574	2.562
4	ethanol	100.5648	89.8094	33.0350
5	n-hexane + n-pentane	94.8388	48.336	7.14
6	n-hexane + diethyl ether	96.6354	7.314	1.709
7	n-pentane + diethyl ether	99.5904	28.5646	1.855
8	ethanol + n-hexane	100.0328	88.1872	10.4134
9	ethanol + n-pentane	96.7218	28.3032	3.6658
10	ethanol + diethyl ether	99.3088	106.742	5.2038

APPENDIX 2: SOLVENT MISCIBILITY



Solvent Polarity Chart

Relative Polarity	Compound Formula	Group	Representative Solvent Compounds
Nonpolar	R - H	Alkanes	Petroleum ethers, ligroin, hexanes
	Ar - H	Aromatics	Toluene, benzene
	R - O - R	Ethers	Diethyl ether
	R - X	Alkyl halides	Tetrachloromethane, chloroform
	R - COOR	Esters	Ethyl acetate
Increasing	R - CO - R	Aldehydes and ketones	Acetone, methyl ethyl ketone
	R - NH ₂	Amines	Pyridine, triethylamine
	R - OH	Alcohols	Methanol, ethanol, isopropanol, butanol
	R - COHN ₂	Amides	Dimethylformamide
	R - COOH	Carboxylic acids	Ethanoic acid
Polar	H - OH	Water	Water