OIL RECOVERY FROM PALM OIL SOLID WASTES

DASHINY A/P GOPAL

UNIVERSITY MALAYSIA PAHANG

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of the degree Bachelor of Chemical Engineering

Signature : ________________________________

Name of Supervisor : ________________________________

Date : ________________________________

OIL RECOVERY FROM PALM OIL SOLID WASTES

DASHINY D/O GOPAL

Thesis submitted to the Faculty of Chemical and Natural Resources Engineering in
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I declare that this thesis entitled “Oil Recovery from Palm Oil Decanter Cake and Spent Bleaching Clay.” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : ……………………………………..

Name : ……………………………………..

Date : ……………………………………..

Special Dedication of This Grateful Feeling to My Beloved father and mother;

Mr. GOPAL S/O BALAKRISHNAN and Mrs. LALITHA D/O RAMASAMY

Loving siblings;

G. KUMARESWARAN AND G.SAMIHETA

Supportive families;

Uncles and Aunties

For Their Love, Support and Best Wishes.

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ABSTRAK

Kajian selama beberapa tahun telah dilakukan ke atas pengekstrakkan sisa minyak dari bahan buangan pepejal minyak kelapa sawit. Kek decanter ialah bahan buangan pepejal yang dihasilkan daripada kilang pengisar minyak kelapa sawit selepas air buangan industry minyak kelapa sawit dimampatkan, manakala tanah peluntur yang telah digunakan ialah bahan buangan pepejal hasil daripada penapisan minyak kelapa sawit. Biasanya, bahan buangan ini masih mengandungi 30-40% minyak dan bahan buangan pepejal sekarang ini dibuang terus di tempat buangan sampah tanpa dirawat di mana kerana perbuatan ini, pencemaran air dan udara yang serius berlaku. Pemulihan minyak dan penggunaan semula tanah liat peluntur yang telah digunakan serta kek decanter membuka peluang yang besar untuk menjimatkan kos dalam industry memproses minyak ini. Kajian ini menjelaskan tentang pengekstrakkan sisa minyak menggunakan tanah peluntur yang telah digunakan daripada penapisan minyak kelapa sawit. Pengekstrakkan sisa minyak dari kilang pengisar minyak kelapa sawit juga dijelaskan dalm kajian ini. Di sini, dua kaedah digunakan membandingkan pengekstrakkan minyak daripada kek decanter dan juga tanah liat yeng telah digunakan. Satu ialah kaedah pengekstrakkan soxlet dan satu lagi ialah pengekstrakkan pelarut kaedah lama. Perbandingan dua kaedah menunjukkan pengekstrakkan soxlet boleh member hasil yang banyak. Suhu optimum dan masa terbaik untuk mengeringkan kek decanter dan tanah liat yang telah digunakan juga ditentukan. Untuk kes ini, sampel-sampel dikeringkan dengan suhu 60^0C- 110^0C, dan suhu yang terbaik ialah 90^0C. Masa optimum telah ditentukan selama 12 jam. 4 pelarut yang berbeza telah digunakan untuk mengekstrak sisa minyak daripada sampel bahan buangan. Keputusan menunjukkan bahawa peratus pengekstrakkan minyak daripada metil etil ketone dan acetone lebih tinggi berbanding dengan hexane dan petroleum ether. Nilai iodin juga ditentukan untuk membandingkan kualiti miyak yang diekstrak. Nilai iodin yang diperoleh adalah dalam lingkungan 40-80. Mengikut analisis PORIM, nilai min minyak mentah ialah 51.3.

ABSTRACT

Many years of research have been done on extraction of residue oil from palm oil solid wastes. Decanter cake is the solid waste produced from palm oil milling company after decanting the palm oil mill effluent, while spent bleaching clay is the solid waste from palm oil refinery. Basically, this wastes still contains 30-40% of oil and this solid wastes are currently disposed directly in landfills without treatment, causing severe water and air pollution problems. Recovery of oil and the reuse of spent bleaching clay and decanter cake is the areas where great opportunity exists for cost saving in the oil processing industry. This study described the extraction of residual oils of spent bleaching earth (SBE) from palm oil refinery and also described the extraction of residue oil from palm oil milling industry. Here, two methods are used for comparison to extract the oil from decanter cake and also spent bleaching clay. There are soxhlet extraction method and also solvent decanting method. The comparison of two methods shows that soxhlet extraction can give higher yield. The optimum temperature and best duration of time to dry the decanter cake and spent bleaching clay also determined. In this case, the samples were dried at the temperatures 60^0C- 110^0C, and the best temperature is 90^0C. The optimum time was determined as 12 hours. Four different solvents were used to extract the residue oil from the waste samples. The results shows that the percentage of oil extracted from Methyl ethyl ketone and acetone was high compared to hexane and petroleum ether. The iodine value was also determined to compare the quality of the oil extracted. The range of iodine value obtained was 40-80. According to PORIM analysis mean value of crude oil is 51.3.

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LIST OF SYMBOLS

M - Molarity

m - Mass

N - Normality

V - Volume

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CHAPTER 1

INTRODUCTION

1.1 Overview of Research

The oil palm tree (Elais guineensis) originated from West Africa andwas introduced in Malaysia in early of 1870's. Today Malaysia is the largest producer and exporter of palm oil in the world. According to Malaysian Palm Oil Council currently we are producing 51% of world palm crude edible oil and accounts for 62% of world exports. Oil palm covers 67% of Malaysia's total agricultural land, and 500,000 people in Malaysia are engaged in the sector.

Palm oil is produced from palm fruit in mills through steaming and squeezing process. Figure 1.1 shows the palm oil tree, its fruit and also the cross sectional of the palm oil fruit. Each tree produces compact bunches weighing between 10 to 25 kilograms with 1000 to 3000 fruits per bunch. An individual palm fruit consists of a hard kernel (seed) inside a shell (endocarp) which is surrounded by thin fleshy mesocarp. Mesocarp is the part where we can extract crude palm oil.

Figure 1.1 Palm Oil fresh fruit bunch

The oil palm produces two types of oils; crude palm oil (CPO) from the fibrous mesocarp and crude palm kernel oil from the kernels. Besides that, the industry also produces large amounts of by-products such as palm kernel cake, palm oil sludge (decanter cake), empty fruit bunches and palm pressed fiber as residues.

The problem now in Malaysia is to manage the wastes generated during the palm fruit processes. From the figure 1.2 as presented below, we can see that on average one tone of crude palm oil is extracted from 5.8 ton of fresh fruit bunch. Fibre, shell, decanter cake and empty fruit bunch are 1.42 ton, 0.35 ton, 0.18 ton and 1.63 ton respectively. Empty fruit bunch is the bulk of solid residue and it isuse as a fuel for boiler and also used as a substrate for mushroom and as organic fertilizer. Palm fibres are mainly used as fuel for boilers (heating value of <5 MJ/kg dry fibres). Palm shells are used as feedstock of activated carbon due to undesirable emissions. Decanter cake is used as a fertilizer. Lastly, palm oil mill effluent which is the mixture of polluted effluent, is treated and digested anaerobically to yield biogas, which is used in modified diesel engine with a 90 kW induction motor. (Somporn et. al, 2004)

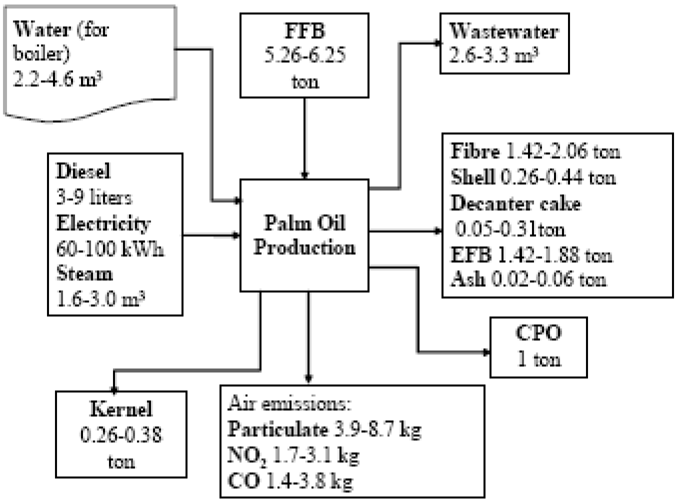

Figure 1.2 Unit process of palm oil production.

Palm oil sludge or decanter cake is the material that remains after decanting the palm oil effluent. Decanter cake produced after the oil mesh from digester passes through the centrifugal decanter to purify the oil before sending to a storage tank.

Decanter cake can be filter pressed before dried to produce dehydrated palm oil mill effluent. It can be centrifuged in the wet state after undergone anaerobic, thermophilic and acidophilic fermentation to produce fresh centrifuged sludge solid. Currently this sludge solid is used as fertilizers since it contains amino acid, crude protein and fibre.

The loss of oils in palm oil decanter cake is a concerned by the palm oil industry, as the oil can be used for industrial purposes. The residue oil in decanter cake should be recovered and re-used as raw material for industrial application. This somehow will save cost in the oil industry. One best approach is to convert the oil into fatty acid alkyl esters. Here , we need to treat decanter cake in organic (alcohol) phase at high temperature (110-270^{0}C) under moderate pressure to recover the oil and convert it into alkyl esters of C1 to C8 carbon atoms. The oil will be converted insitu to alkyl esters using methanol. The alkyl esters are very useful ingredients in non-food applications. (Loh et. al,2006)

1.2 Problem Statement

The decanter cake contains high percentage of oil. Disposal of decanter cake by incineration, inclusion in animal feeds, land filling method or concrete manufacturing is currently practiced by most of the palm oil milling companies. But, large quantity of decanter cake is disposed off in landfills, only causing fire and pollution hazards due to the residue oil content in the earth. So, the oil in the decanter cake would be just wasted if the oil was not recovered and this situation will cause loss of oil in the future.

In the refining of palm oil, bleaching clay dosages of 0.5-1% are generally used. From, Malaysia alone, with a production of 9 million tons of oil in 1999, a total of 70,000 tons of spent bleaching clay is estimated to be generated yearly. Spent bleaching clay contains about 30-40% of oil by weight of spent bleaching clay and this constitutes a major loss in oil as well a major cost from the clay since the spent clay is currently disposed untreated. In addition, the use and disposal of the spent bleaching clay is becoming a potential problem in the producing countries because of the rapid growth of the industry and currently, oil-laden spent bleaching clays are mainly disposed of in landfills or in waste dumps, as the spent clays are considered nontoxic.

Extracting oil from palm oil decanter cake has never been reported before. Now decanter cake is only used as fertilizers without extracting the residue oil. So, here we can extract the oil and can be used for so many purposes. As a suggestion the crude oil extracted can be converted to biodiesel because the oil extracted from palm oil solid waste has poorer quality compared to crude palm oil. After extracting oil, residue solid waste can be used as fertilizer or animal feed.

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. Other than that, land filling the sludge solid is expensive.

The highly increasing trend of oil price in this year has had a great impact on both agricultural and industrial sectors. This is because, increasing oil price also increasing cost of production. So in this situation Malaysian government needs to do researches on new sources of energy to substitute petroleum products. Using biodiesel is the best way to reduce foreign import of petroleum products. Furthermore there are a lot of advantages using biodiesel instead of conventional diesel fuel which are environmental benefits, less toxicity for humans because the raw materials used to produce it are natural and renewable making it biodegradable and non-toxic.

1.3 Objective of Study

The objectives of the studies are as follows;

- To determine the optimum temperature and optimum drying time to remove higher percentage of moisture from palm oil solid wastes.
- To extract crude oil from palm oil decanter cake by solvent decanting extraction and soxhlet extraction.
- To determine the higher percentage of oil yield using methyl ethyl ketone, acetone, hexane and petroleum ether as solvents.
- To determine the iodine value and acid value of the oil extracted from palm oil decanter cake and spent bleaching clay.

1.4 Scopes of study

Based on the objectives, the main scope of this project is an overview of the utilization of palm oil sludge solid in industry. The scope of study will involve collecting oil palm decanter cake sample from a nearby palm oil milling company and spent bleaching clay from palm oil refining company. Then, drying the sample with different temperature to remove moisture and preparation of the sample for extraction. This temperature parameter is to determine the optimum temperature to dry decanter cake to extract maximum oil. Then the solvent extraction method will be carried on using different type of solvents, metylethylketone, acetone, petroleum ether and hexane. There will also be a comparison between solvent decanting extraction method and soxhlet extraction method .This is to determine the effect of the solvent on the % of extracted oil. The effect of the solvent, extraction time, solvent/feed ratio, and mixing rate on the extraction efficiency was investigated through this work.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction and Historical Background

Chemical analyses play an important role in our life, from agricultural, environmental, clinical, pharmaceutical chemistry, oleo chemistry, toxicology, manufacturing, metallurgical, archaeology, to forensic. The value of a piece of land is determined by the content of carbon and nitrogen. The quality of air is determined by analyzing the percentage of the pollutants composition. The price of the coal will be determined by the percent of sulfur impurity present.

We look into our material world through the methods and the tools of analytical chemistry. Those analytical techniques existing now are always being sought to improve by analytical chemists to meet the arising requirements for better chemical measurement from our society. Usually one or more standard specific procedures are available for determination of an analyte in a provided sample. However, it is not exactly can be accepted by the other analyte. The analyst needs to depend on his experience and knowledge to carry out and analytical method for a sample. Since oil palm dominate most of the needs in our life, getting to know the process involve in oil extraction is important. The quality of oil is determined by the composition of saturated and unsaturated fatty-acids. Palm oil has higher saturated fatty acids and this make the oil more stable and less prone to oxidation at higher temperature.

2.2 The oil palm

Elaeis Guineensis Jacq is the scientific name of oil palm and this is the most important species in the genus Elaeis which belong to the family Palmae. Oil palm (*Elaeis guineensis*) is grown extensively in Southeast Asia and Equatorial Africa and it produces more oil per area than any other plant. (Poku et.al, 2002.) Oil palms start bearing bunches 2 ½ - 3 years after field planting. The usual frequency of a harvesting round is 10 to 15 days or 2-3 times a month. The fruit bunches are generally transported to palm oil mill on the day of harvesting.

The palm oil mil production capacity is around 45 tons of FFB per hour or around 1000 tons of FFB per day. The mill operates approximately 16-24 hours per day. (Mahlia et.al, 2000). Below are the technological process involve in extracting oil from the palm oil fresh fruit bunches and Figure 2.1 explains the palm oil milling process in a process flow diagram.

Loading ramp : After passing over the weighbridge the fruit has to be held for a time until it can enter the first stage of processing. For loading, ramp is the place where the FFB are transported and unloaded in the mill.

Sterilization : This process is done batchwise in an autoclave for 1 hour 40 minutes for the Fresh Fruit Bunch to be completely cooked. The temperature inside is about 120-130^{o}C. The steam condensate is the waste water generated at this step.

Stripping (threshing) : This process is to separate the sterilized fruits from bunch stalks. This process generates the empty fruit bunches (EFB).

Digestion : The separated fresh fruits are put into the place where they mashed under steam heated conditions. No residues occurred on this step.

Crude palm oil extraction: the homogenous oil mash from the digester is pushed through a screw press, and later passes through a vibrating screen, a hydrocyclone and decanters to remove fine solids and water. Decanter wastewater and decanter cake are the major wastes at this step. Centrifugal and vacuum driers are used to further purify the oil before sending it to a storage tank. The temperature of oil (60°C) in the storage is maintained with steam coil heating before the CPO is sold.

Nut/ Fibre Separation: the fibre and nuts from the screw press are separated in a cyclone. The fibre that passes out of the bottom of the cyclone is used as boiler fuel from which ash is produced after combustion.

Nut Cracking: the nuts are cracked in a centrifugal cracker. After the cracking process, the kernels and shells are separated by clay suspension (Kaolin). The separated shells from the kernels are sold to other mills as fuel. The kernels are sent to the kernel drying process in a silo dryer to sell (for extraction) to other mill.

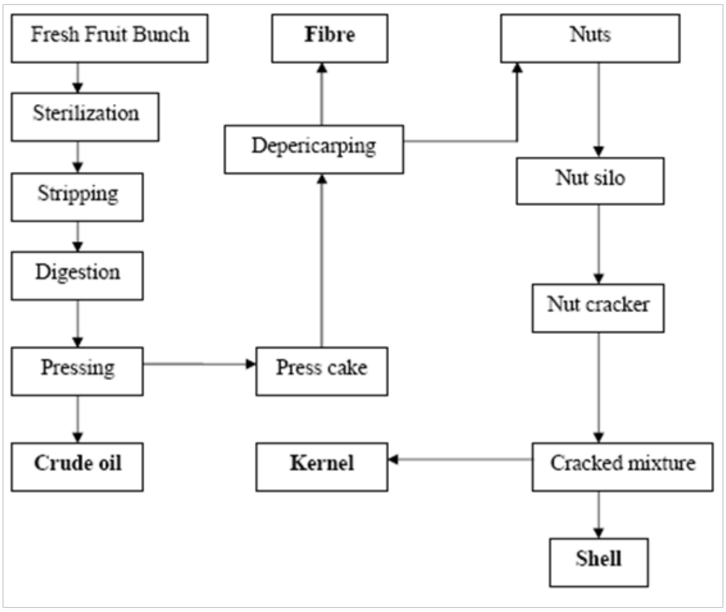

Figure 2.1 Process flow diagram of palm oil extraction

2.3 Characteristics of palm oil

The oil palm produces two types of oils, palm oil from the fibrous mesocarp and lauric oil from the palm kernel. In the conventional milling process, the fresh fruit bunches are sterilized and stripped of the fruitless which are then digested and pressed to extract the crude palm oil (CPO). The nuts are separated from fiber 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. Obtained 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.

2.3.1 Chemical properties of palm oil

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. (Marsin et. al, 2005). 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 color 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 artifacts 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 melting points of triglycerides are dependent on the structures and the position of the components acids present. They also effect the crystallization behavior of the oil. The semisolid nature of the palm oil at room temperature has been attributed to the presence of the oleo-disaturated fraction.

Palm oil contains palmitic acid (a fatty acid made by our body), the monounsaturated oleic acid, polyunsaturated linoleic acid (an essential fatty acid) and stearic acid. The typical blend in palm oil is 45% palmitic, 40% oleic, 10% linoleic and 5% stearic. Palm oil (and its products) has good resistance to oxidation. The palm oil is also rich in natural chemical compounds important for health and nutrition. Among others, it is a natural source of carotenoids and vitamin E as well as supplying fatty acids and other important fat-soluble micronutrients. The carotenoids, tocopherols, sterols, phosphatides, triterpenic, and aliphatic alcohols form the minor constituents of palm oil, and play significant role in the stability and refinability of the oil, in addition to increasing the nutritive value of the oil.

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. The combined effects of the properties of carotenoids, tocopherols, tocotrienols and the 50% unsaturation of the acids confer on palm oil a higher oxidative stability compared to other vegetable oils. (Fereidoon,2004). 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. In terms of sterols, palm oil contains far less cholesterol than other vegetable oil as shown in Table 2.1 below. The lower cholesterol levels in crude palm oil add nutritive value and advisable to be used in food industries.

Table 2.1: Cholesterol levels in crude oils and fats. Source: (Fereidoon,2004)

Oil type	**Average (ppm)**	**Range (ppm)**
Coconut oil	14	5-24
Cocoa butter	59	n.a
Palm kernel oil	17	9-40
Palm oil	18	13-19
Sunflower oil	17	8-44
Soybeen oil	28	20-35

Table 2.2 below discusses the summary of the inherent chemical properties of Malaysian Palm Oil. From the result, the iodine value is 52.9 and average saponification value is 195.7.

Table 2.2: Chemical Properties of Malaysian Palm Oil

Chemical properties	**Mean**	**Range**
Saponification value (mg KOH/g oil)	195.7	190.1-201.7
Unsaponification matter (%)	0.51	0.15-0.99
Iodine value (Wijs)	52.9	50.6-55.1
Slip melting point (^{o}C)	34.2	30.8-37.6

2.3.2 Physical properties of palm oil

The physical properties of the palm oil are shown in Table 2.3 below. The values shown are the average value of 215 samples that has done. The apparent density is an important parameter from the commercial point of view since it as used for volume to weight conversions. It can also be used as a purity indicator. The solid content of an oil is a measure (in %) of the amount of solid fat present in the oil at any one temperature. It is measured by means of wide-line nuclear magnetic resonance (NMR) spectrometry after a standard tempering procedure for the samples. The solid present in the oil at any one temperature is due to the process of crystallization occurring in the oil as a consequence of its chemical properties. The different molecular triglyceride structures with their differing chemical characteristics manifest their physical states at different temperatures, thus imparting certain crystallization and melting behavior to the oil. For the thermal characteristics, palm oil can be separated under controlled thermal conditions into two components, i.e., a solid (stearin) and a liquid (olein) fraction.

Table 2.3: Major physical properties of palm oil. Source: (Fereidoon,2004)

Property	**Mean (of 215 sample)**	**Range**
Apparent density at 50^{o}C (g/ml)	0.889	0.888-0.889
Refractive index at 50^{0}C	1.455	1.455-1.456
Solid fat content		
5^{0}C	60.5	50.7-68.0
10^{0}C	49.6	40.0-55.2
15^{0}C	34.7	27.2-37.9
20^{0}C	22.5	14.7-27.9
25^{0}C	13.5	6.5-18.5
30^{0}C	9.2	4.5-14.1
35^{0}C	6.6	1.8-11.7
Slip melting point (^{0}C)	34.2	31.1-37.6

Crude palm oil is the richest natural source of tocotrienols which is a fat-soluble vitamins related to the family of tocopherols. The latest technology to purify tocotrienols from the fruits of oil palm was developed to produce a superior quality and purity. Refined red palm oil is used for the treatment and prevention of vitamin A deficiency. Palm oil is indeed a nature's gift to Malaysia, and Malaysia's to the world. (Sumathi et.al, 2007).

2.4 Palm oil sludge solid or Decanter cake

Palm oil sludge is the material that remains after decanting the palm oil mill effluent. It can be either filter-pressed, before dried and ground to produce dehydrated palm oil mill effluent, or centrifuged in the wet state, after having undergone anaerobic, thermophilic and acidophilic fermentation. In the latter case, the product is known as fresh centrifuged sludge solids of 15 to 20% dry matter and may be dehydrated to form dry centrifuged sludge solids of between 94 and 97% dry matter. The composition of the essential amino acids in palm oil sludge and palm kernel meal is given in Table 2.4 below. From the table we could discuss that the composition of amino acid in palm kernel meal is higher compared to the palm oil sludge. So, from here we can discuss that palm oil kernel meal is more suitable as animal feed and fertilizer due to its high composition of amino acids.

Table 2.4: Composition of essential amino acid in palm oil sludge and palm kernel meal (% cp). Source; Ong (1982)

Amino acid	**Palm oil sludge**	**Palm kernel meal**	**Amino acid**	**Palm oil sludge**	**Palm kernel meal**
Arginine	0.19	2.20	Methionine + cystine	0.28	1.98
Histidine	0.14	0.27	Phenylalanine + tyrosine	0.77	1.28
Isoleucine	0.35	0.63	Threonine	0.34	0.54
Leucine	0.60	1.05	Tryptophan	0.12	0.17
Lysine	0.21	0.56	Valine	0.36	0.9

2.5 Properties 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/100 g) 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 (58.5 vs. 143.9 mg/kg) and Fe (1374.5 vs. 4086.0 mg/kg). Dry matter degradation characteristics and effective degradability varied significantly ($P < 0.05$) and were consistently highest in palm oil 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 a better quality of oil. Al-Zahrani et. al, (2000) have studied extraction of oil from spent bleaching clay using different type of solvents at optimum conditions.

2.6 Bleaching Clay

Bleaching earth is manufactured from the best quality selectively mined and scientifically blended montmorillonite clays under stringent process controls. The use of bleaching earth involves the removal of variety of impurities which include phosphatides, fatty acids, gums, metal trace, etc, followed by decolorization. Although there are 7 major clay groups and at least 33 different specific clay minerals (Fereidoon, 2004) , only 2 , calcium montmorillonite (bentonites) and a particular naturally occurring mixture of calcium montmorillonite and attapulgite called hormite, play a commercial role as bleaching clays. (Crossley, 2001). Both hormite and bentonite clays can be used in their natural or acid activated states. Acid activation is the process whereby the adsorptive powers of natural bleaching clays are enhanced. Figure 2.2 below shows the sample of spent bleaching clay from palm oil refinery.

Figure 2.2 Spent bleaching clay

Both natural and acid activated clays are used as adsorbents. Bleaching clay is composed mainly of smectite, an aluminosilicate mineral. It is well known that bentonites in natural state and have limited absorbing capacity. This ability is greatly enhanced by the treatment with strong acids. When bentonites are acid-activated , through treatment with hot mineral acid solutions, hydrogen ions attack the aluminosolicate layers via the interlayer region. (Taylor et al., 2007). This attacks alters the structure, chemical compositions and physical properties of the clay while increasing the adsorption capacity. This clay is widely used in palm oil refinery and after used this solid waste which is containing 25-35% retained oil disposed in landfills without treatment, causing severe water and air pollution problems. However, recently dumping of spent clay in landfills or public disposal sites has been prohibited in most countries. (Al-Zahrani et al., 2000).

Various methods of extracting the spent bleaching clay to recover the adsorbed oil have been studied. Table 2.5 below shows the characteristics of recovered oil form spent bleaching clay using solvent extraction method and supercritical carbon dioxide extraction method. We could discuss that, the oil recovery is almost same for both method. The good quality of oil could be recovered by supercritical carbon dioxide extraction method , because no content of Ferum and Copper in the recovered oil and also content very high vitamin E, compared to the oil recovered by solvent extraction method.

Table 2.5: Characteristics of residue oil from spent bleaching earth using two different type of extractions. Source : (Loh et .al,2006)

Characteristics of residue oil	**Solvent extraction**	**Supercritical carbon dioxide extraction**
FFA(%)	12.6	12.6
PV(meq/kg)	3.4	2.2
Phosporus (ppm)	18.7	15.8
Fe (ppm)	1.24	-
Cu (ppm)	0.38	-
B-carotene (ppm)	6	7
Total vitamin E (ppm)	0	38.8
Oil recovery (%)	21	20

2.7 Solvent Extraction

Many processes in chemical engineering require the separation of one or more of the components of a liquid mixture by treating the mixture with an immiscible solvent in which these components are preferentially soluble. In some cases purification of a liquid may be the function of the process, in others the extraction of a dissolved component for subsequent processes may be the important aspect. An example of the former is the preparation of the pure organic liquids from products of the oil industry. Liquid-liquid extractions may also be used as energy saving processes by, for example, eliminating distillation stages. It is possible, of course that the substance of interest may be heat-sensitive anyway and that distillation is accordingly an unacceptable process. There are two media involved in a typical extractor;

1. *Refined material*—a porous medium (palm oil decanter cake), usually characterized by an outer (bulk) (ε_b) porosity and an inner (pore) (ε_p) porosity. The refined material is initially composed of some fraction of the liquid species.

2. *Solvent*—a liquid (hexane, water, alcohol, etc.) to which the species *P* is transferred during extraction. (Veloso et. al, 2007).

Solvent extraction, in general, implies the removal of soluble material from an insoluble solid phase by dissolution in a liquid solvent. Absolute *n*-hexane, a petroleum-derived product, has been extensively used as a solvent for the oil extraction from soya beans and other oilseeds because of its low vapourisation temperature (boiling point 63–69 °C), high stability, low corrosiveness, low greasy residual effect, and better odour and flavour productivity for the milled products. (Seth et. al, 2007)

When separation by distillation is ineffective or very difficult, liquid extraction is one of the main alternatives to consider. Close-boiling mixtures or substances that cannot withstand the temperature of the distillation, even under a vacuum, may often be separated from impurities by extraction, which utilizes chemical differences instead of vapor pressure differences. For example, penicillin is recovered from fermentation broth by extraction with a solvent such as butyl acetate. Another example for liquid extraction is recovering acetic acid from dilute aqueous solutions; distillation would be possible in this case, but the extraction step considerably reduces the amount of water to be distilled.

Extraction equipment may be operated batchwise or continuous. The extract is the layer of solvent plus extracted solute and the raffinate is the layer from which solute has been removed. The extract may be lighter or heavier than the raffinate, and so the extract may be shown coming from top of the equipment in some cases and from the bottom in others. The operation may of course be repeated if more than one contact is required, but when the quantities involved are large and several contacts are needed, continuous flow becomes economical.

The rate at which a soluble component is transferred from one solvent to another will be dependent, among other things, on the area of the interface between the two immiscible liquids. Therefore it is very advantageous for this interface to be formed by droplets and films, the situation being analogous to that existing in packed distillation columns.

The following need to be carefully evaluated when optimizing the design and operation of the extraction processes.

- Solvent selection
- Operating Conditions
- Mode of Operation
- Extractor Type
- Design Criteria

2.7.1 Solvent selection

Solvents will be selected with its capabilities, depending on the solvent's and solute's chemical structure. Once the functional group is identified, possible solvents can be screened in the laboratory. The distribution coefficient and selectivity are the most important parameters that govern solvent selection. The distribution coefficient (m) or partition coefficient for a component (A) is defined as the ratio of concentration of a A in extract phase to that in raffinate phase. Selectivity can be defined as the ability of the solvent to pick up the desired component in the feed as compared to other components. The desired properties of solvents are a high distribution coefficient, good selectivity towards solute and little or no miscibility with feed solution. Also, the solvent should be easily recoverable for recycle. Designing an extractor is usually a fine balance between capital and operating costs. Consequently, while extracting larger quantities of solute, the solvent could also extract significant amount of feed solution. Other factors that affecting solvent selection are boiling point, density, interfacial tension, viscosity, corrosiveness, flammability, toxicity, stability, compatibility with product, availability and cost. (Christie John Geankoplis, 2005).

2.7.2 Selection of extraction conditions

According to most of the researches on extraction, the temperature, pH and residence time effect the yield and selectivity. Operating pressure has a negligible affect on extraction performance and therefore most extractions take place at atmospheric pressure unless governed by vapor pressure considerations. Temperature can also be used as a variable to alter selectivity. Elevated temperatures are sometimes used in order to keep viscosity low and thereby minimizing mass-transfer resistance. Other parameters to be considered are selectivity, mutual solubility, precipitation of solids and vapor pressure.

The pH becomes significant in metal and bio-extractions. In bio-extractions (e.g., Penicillin) and some agrochemicals (e.g. Orthene), pH is maintained to improve distribution coefficient and minimize degradation of product. In metal extractions, kinetic considerations govern the pH. In dissociation-based extraction of organic molecules, pH can play a significant role (e.g., cresols separation). Sometimes, the solvent itself may participate in undesirable reactions under certain pH conditions (e.g., ethyl acetate may undergo hydrolysis in the presence of mineral acids to acetic acid and ethanol). Residence time is an important parameter in reactive extraction processes (e.g., metals separations, formaldehyde extraction from aqueous streams) and in processes involving short-life components (e.g., antibiotics & vitamins). (Christie John Geankoplis, 2005).

Bassim et. al, (2003) has studied the removal of residue oil from palm oil mil effluent. In this study the effect of different solvents, extraction time, solvent/feed ratio and mixing rate on the extraction efficiency was investigated. The percentage of extraction will increase with the increasing of time. Experiments also conducted with solvent/feed ratio of 0.25, 0.5, 0.75 and 1.0. They found the best mixing rate is 200 rpm. The optimum solvent ratio obtained was found to be 1.0 for n-pentane and 0.5 for both n-heptane and n-hexane. The maximum percentage of extractions of 58%, 63% and 65% was achieved by using n-pentane, n-hexane and n-heptane.

Table 2.6 below shows the different type of solvent and its boiling point and its solubility in 100 parts of water. From the result we could discussed that cyclohexane is insoluble in water. These properties will be used to determine the performance of solvents in extraction process when rotary evaporator is used.

Table 2.6: Solubility in water and boiling point of some selected solvents. Source: (Bassim et.al, 2003).

Solvent	**Solubility in 100 Parts of Water**	**Boiling point (^{o}C)**
n-pentane	0.036^{15}	36.3
n-heptane	0.005^{15}	98.4
n-hexane	0.014^{15}	69.0
cyclohexane	Insoluble	80.1
chloroform	0.82^{20}	61.2
dichromethane	0.07^{20}	40.1
benzene	13	80.1
acetone	∞	56.5

2.7.3 Selection of mode operation

Extractors can be operated in crosscurrent or counter-current mode. The following briefly explain on how counter current mode and cross current mode operation.

2.7.3.1 Cross current operation

Crosscurrent mode is mostly used in batch operation. Batch extractors have traditionally been used in low capacity multi-product plants such as are typical in the pharmaceutical and agrochemical industries. For washing and neutralization operations that require very few stages, crosscurrent operation is particularly practical and economical and offers a great deal of flexibility. The extraction equipment is usually an agitated tank that may also be used for the reaction steps. In these tanks, solvent is first added to the feed, the contents are mixed, settled and then separated. Single stage extraction is used when the extraction is fairly simple and can be achieved without a high amount of solvent. A single extractor can be represent as Figure 2.3 below:

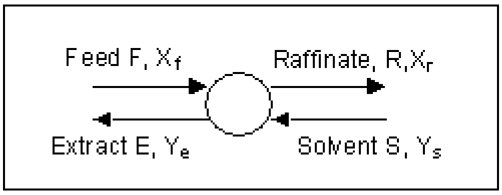

Figure 2.3 Cross current operation

F = Feed quantity / rate, mass

R = Raffinate quantity / rate, mass

S = Solvent quantity / rate, mass

E = Extract quantity / rate, mass

X_f, X_r, Y_s, and Y_e are the weight fractions of solute in the feed, raffinate, solvent and extract, respectively.

2.7.3.2 Countercurrent operation

As described above, the crosscurrent operation is mostly used in low capacity multi-product batch plants. For larger volume operation and more efficient use of solvent, countercurrent mixer-settlers or columns are employed. Countercurrent operation conserves the mass transfer driving force and hence gives optimal performance.

2.7.4 Selection of extractors’ type

The type of extractors which are widely used in industries now are mixer-settler, centrifugal devices, column contractors(static) and column contractors (agitated). Mixer settlers are usually a static or agitated mixed interspersed with settling stages. There are mostly used in metal industry where intense mixing and high residence time is required for the extraction processes. Centrifugal extractors are usually high-speed rotary machines with very low residence time. The number of stages in a centrifugal device is usually limited to one, but currently devices with multiple numbers of stages are common. Non polar or polar solvents can be used as extracting medium. Polar solvents such as acetone, methyl ethyl ketone, methylene chloride, perchloroethylene, isopropyl alcohol, chloroform can be used to extract oil. While the non polar solvents which applicable are petroleum benzene, petroleum ether, toluene, hexane, fluent extract. Anyway, the type of solvent used and the method of oil extraction will determine the quality of extracted oil. According to research, oil extracted by non-polar solvent like hexane is allowed to be used the extracted oil in foods. To obtain, better quality oil, we should carry out the extraction on fresh decanter cake and under nitrogen (Loh et al., 2006).

Marsin et. Al, (2005) have done a research in determining the quality of residue oil from palm pressed fiber using soxhlet extraction with n-hexane and chloroform as solvent. In this experiment the palm pressed fibre sample amount and total solvent usage were optimized based on the volume of the extraction and size of the Soxhlet apparatus. From the experiments, can be observed that the major forms of Vitamin E isomers present in residue oil from palm pressed fibre using n-hexane as extraction solvent are tocopherol(55.2 %) and tocotrienol (24.9%). On the other hand the forms of vitamin E isomers present in residue oil from palm pressed fibre using chloroform as extraction solvent were tocopherol (61.6%) and tocotrienol (19.6%). Based on the Table 2.4 below, we could notice that the highest value of β-carotene was obtained by using n-hexane as solvent. The table 2.4 describe the comparison result obtained on extraction of residue oil by using hexane and chloroform as solvent. We could also conclude that the residue oil extracted using hexane has less vitamin E contents, 3.3 mgml^{-1} compared to the residue oil using chloroform as solvent which is 3.45 mgmg^{-1}.

Table 2.7: Comparison of results obtained on the determination of residue oil contents. Source : (Marsin et. Al, 2005)

Parameter	**Soxhlet (n-hexane)**	**Soxhlet (chloroform)**
Total extraction time (h)	8	8
Total solvent usage (ml)	200	200
Total vitamin E contents (mgml^{-1})	3.1-3.5	3.3-3.6
Total β-carotene contents (mgml^{-1})	3.1-3.3	2.8-3.0
Method repeatability (R.S.D., %)	3.0-6.0	3.0-6.0

2.7.5 Soxhlet extraction

Soxhlet extraction involves leaching and is the solvent technique mostly used. In this extraction, when the liquid reaches the overflow level, a siphon well aspirate the solute of the thimble-holder and unloads it back into the distillation flask, carrying the extracted analytes into the bulk liquid. This operation will be repeated until complete extraction achieved. The advantage of Soxhlet extraction are the sample is repeatedly brought into contact with the fresh portions of solvent, helping to displace the transfer equilibrium and no filtration is required after the extraction step. Other than that the temperature of the system will remains high because heat is applied to the distillation flask. Samples are usually extracted at the boiling point of the solvent for a long time and the possibility of thermal decomposition of the target compounds cannot be ignored. Soxhlet device also provide agitation, which would accelerate the process. (Luque et. Al, 1998). Figure 2.4 shows a schematic diagram of soxhlet extactor.

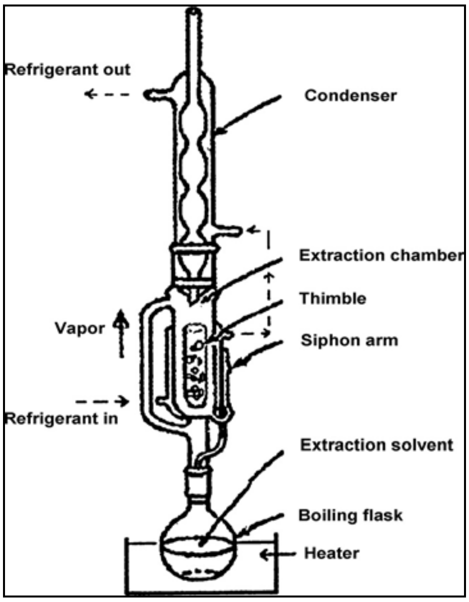

Figure 2.4 A schematic diagram of a modified soxhlet extractor. (Source :Setiyo et. al, 2008).

Soxhlet extraction requires large amount of organic solvents that are flammable and environmentally unfriendly, but the solvents can be recovered easily and it requires less sophisticated equipment than molecular distillation, operates under atmospheric pressure and lower temperature. (Setiyo et. al, 2008).

2.80 Oil analysis

Various methods were introduced to investigate the analysis of residue oil extracted from palm oil solid waste.

2.81 Acid Value

Acid value is the mass of potassium hydroxide in mg that is required to neutralize one gram of oil sample. Acid number is a measure of the amount of carboxylic acid groups in a chemical compound, such as fatty acid.

$$AN = (V_{eq} - b_{eq}) N \frac{56.1}{W_{oil}}$$

V_{eq} is the amount of titrant (ml) consumed by the crude oil sample and 1ml spiking solution at the equivalent point, b_{eq} is the amount of titrant (ml) consumed by 1 ml at the equivalent point, and 56.1 is the molecular weight of KOH. The molarity concentration of titrant (N) is calculated as such:

$$N = \frac{1000 W_{KHP}}{204.23 V_{eq}}$$

In which W_{KHP} is the amount (g) of KHP in 50 ml of KHP standard solution, V_{eq} is the amount of titrant (ml) consumed by 50 ml KHP standard solution at the equivalent point, and 204.23 is the molecular weight of .

There are standard methods for determining the acid number, such as ASTM D 974 and DIN 51558 (for mineral oils, biodiesel), or specifically for Biodiesel using the European Standard EN 14104 and ASTM D664 are both widely utilised worldwide. Acid number (mg KOH/g oil) for biodiesel should to be lower than 0.50 mgKOH/g in both EN 14214 and ASTM D6751 standard fuels. This is since the

FFA produced may corrode automotive parts and these limits protect vehicle engines and fuel tanks.

As oil-fats rancidify, triglycerides are converted into fatty acids and glycerol, causing an increase in acid number. A similar observation is observed with Biodiesel aging through analogus oxidation processes and when subjected to prolonged high temperatures (ester thermolysis) or through exposure to acids or bases (acid/base ester hydrolysis).

2.8.2 Iodine value

The iodine value is the mass of iodine in grams that is consumed by 100gm of oil sample. Chemically, vegetable and animal oils and fats are triglycerides, glycerol bound to three fatty acids. Animal fat such as tallow or lard is saturated, meaning that in the fatty acid portion, all the carbon atoms are bound to two hydrogen atoms, and there are no double bonds. This allows the chains of fatty acids to be straighter and more pliable so they harden at higher temperatures.

As we increase the number of double bonds in a fatty acid, you reduce that ability for oils to gain a confirmation that would make them solid, so they remain liquid. To picture it, imagine that you put a bunch of strings in a line. Now tie knots in various places on the strings and see how they don't fit together tightly.

To test a vegetable oil to see how many double bonds it has (how unsaturated it is) iodine is introduced to the oil. The iodine will attach itself over a double bond to make a single bond where an iodine atom is now attached to each carbon atom in that double bond. Higher iodine numbers do not refer to the amount of iodine in the oil, but rather the amount of iodine needed to "saturate" the oil, or break all the double bonds. Oils for the most part contain only trace amounts of iodine naturally. The figure 2.4 below shows how iodine attached itself to the fatty acid.

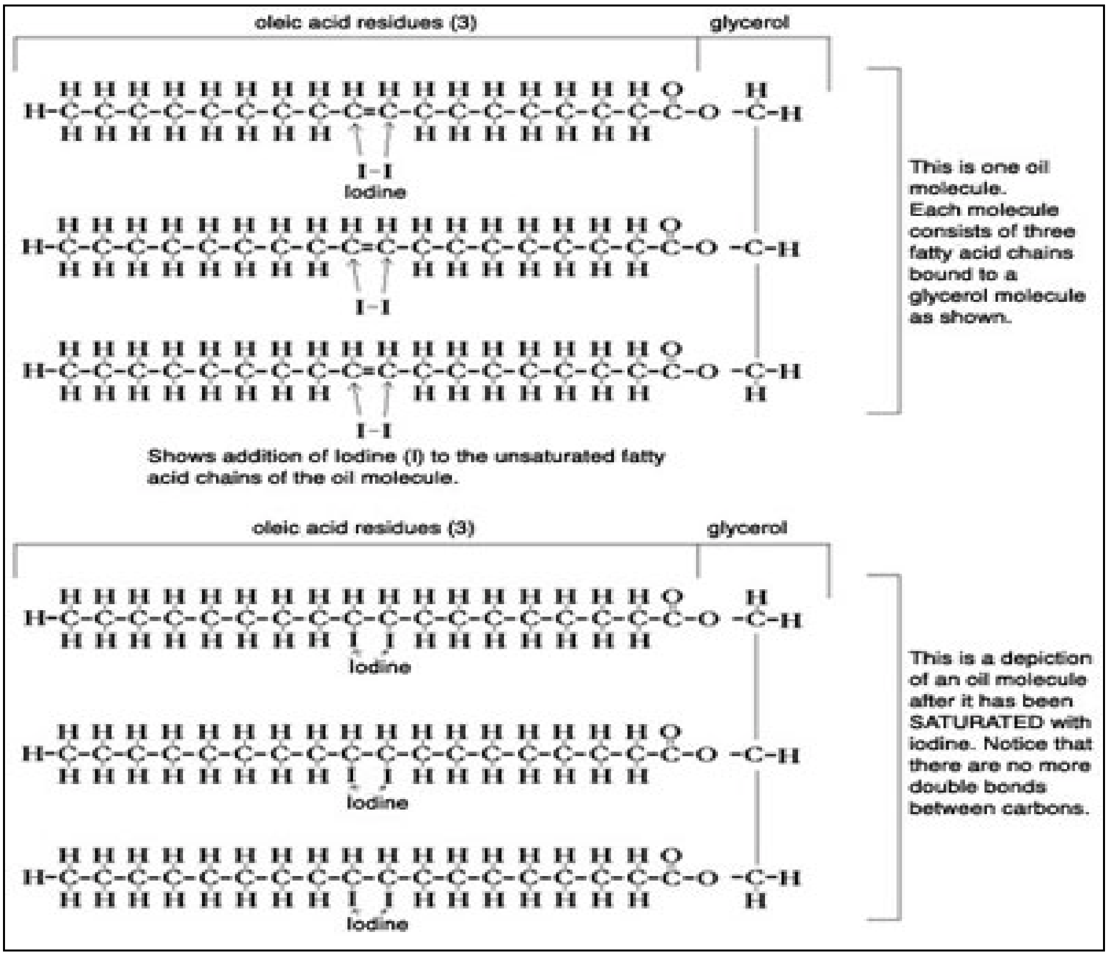

Figure 2.5 Attachment of iodine molecules to fatty acid.

The traditional measure of the degree of bonds available for this process is given by the 'Iodine Value' (IV) and can be determined by adding iodine to the fat or oil. The amount of iodine in grams absorbed per 100 ml of oil is then the IV. The higher the IV, the more unsaturated (the greater the number of double bonds) the oil and the higher is the potential for the oil to polymerize.

While some oils have a low IV and are suitable for use as fuel without any further processing other than extraction and filtering, the majority of vegetable and animal oils have an IV which may cause problems if used as a neat fuel. Generally speaking, an IV of less than about 25 is required if the neat oil is to be used for long term applications in unmodified diesel engines and this limits the types of oil that can be used as fuel.

The IV can be easily reduced by hydrogenation of the oil (reacting the oil with hydrogen), the hydrogen breaking the double bond and converting the fat or oil into a more saturated oil which reduces the tendency of the oil to polymerize. However this process also increases the melting point of the oil and turns the oil into margarine.

IV is a measure of unsaturation of oils and fats. Differential scanning calorimetry (DSC) is a thermoanalytical technique for monitoring changes in physical or chemical properties of materials by directly measuring the specific heat of a material. To form a double bond between two carbon atoms requires more energy (615 kJ/mol) than to form a single bond (350 kJ/mol). Therefore, the thermal behavior of oil will reflect the IV of the oil. (Haryati et. al 1997)

CHAPTER 3

METHODOLOGY

3.1 Introduction

In order to study the percentage of residue oil extraction from decanter cake and spent bleaching clay several important variables will be tested such as drying temperature, drying time, types of solvent and types of solvent extraction method. In this experiment, two major stages will be introduced in order to choose the best extraction method and best solvent to extract the maximum percentages of residue oil from palm oil decanter cake and spent bleaching clay. Basically the methodology of this research is consist of four major parts including drying the palm oil solid wastes, preparation the samples for extraction method, solvent extraction, separation of oil using rotary evaporator, and analysis of the residue oil extracted. Figure 3.1 shows the flow process for all method procedures. As a first part, literature review is done, to compare the methodology and the result that has done by other researcher. Then, the sample collection from factory is done, followed by the pre-treatment of the sample and solvent extraction method. The last part of this research is collect product and oil analysis.

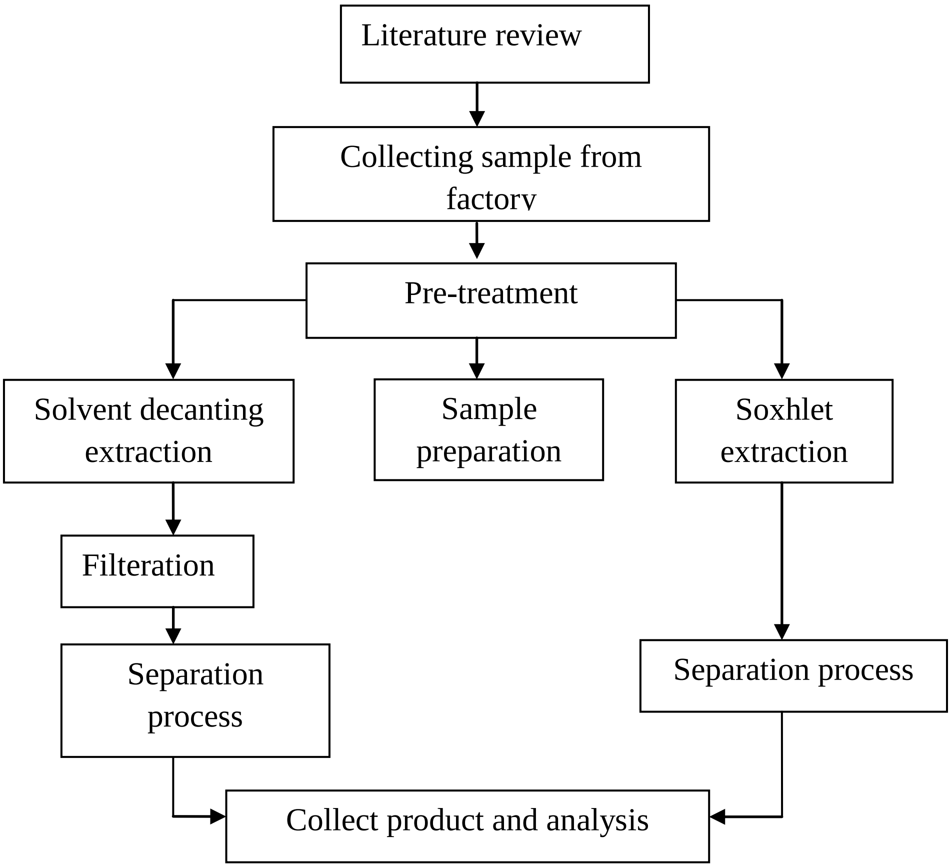

Figure 3.1 Process flow diagram

3.2 Sample and chemical preparation

The raw material that used in this experiment is spent bleaching clay from palm oil refinery and decanter cake from palm oil milling company. The decanter cake collected from LKPP palm oil milling company from Lepar Hilir, Pahang. The spent bleaching clay is collected from palm oil refining company in Gebeng, Pahang. These raw materials need to be dried at optimum temperature in the oven to remove maximum moisture in it before extracting the residue oil.

The solvents used in this experiment are methyl ethyl ketone, acetone, hexane, and petroleum ether (40^0C-60^0C). In this experiment, the amount palm oil solid waste used as raw material for each sample preparation is 20 g while the solvent used is 300ml. The properties of the solvents used in this experiment were shown in the table below. The table shows that the boiling point of methyl ethyl ketone is higher compared to other solvents. The boiling point is set when rotary evaporator is used to separate oil from solvent.

Table 3.1 Properties of solvents used in solvent extraction

Components	**Methylethylketone**	**Acetone**	**Hexane**	**Petroleum ether**
Formula	C_4H_8O	C_3H_6O	C_6H_{14}	-
Molecular weight	72.11g/mol	58.08g/mol	86.18g/mol	-
Boiling point	79.6^0C	56.5^0C	69^0C	$40\text{-}60^0C$
Density	$0.81g/cm^3$	$0.79g/cm^3$	$0.65g/cm^3$	$0.6\text{-}0.8g/cm^3$

3.3 Equipment / apparatus

Actually, the equipment used in this experiment can be divided into three parts which are equipments used for pre-treatment, equipment involved during experiment procedures and the others involved during analysis procedure. The equipment used during pre-treatment is oven. There are several equipments that will be used during experimental procedures such as orbital shaker, soxhlet extractor, stirring hotplate and rotary evaporator. The equipment that involved during analysis is set up according to the PORIM test method which used to determine the acid value and iodine value of the oil. Other apparatus such as beaker (500-1000ml), volumetric flask (1000ml), conical flask (250-500ml), filter paper (125mm diameter), pipette, and burette also involved on this experiment.

3.3.1 Experimental equipment/apparatus

Oven as shown in figure 3.2 is the main equipment used in pre-treatment experiment. The purpose of this equipment is to remove maximum moisture from the decanter cake and spent bleaching clay. Different temperature is set in order to find the optimum temperature to dry the solid waste.

Figure 3.2 Oven

Orbital shaker as shown in figure 3.3 is used in the solvent decanting extraction method. The mixture of sample and solvent is put to shake with 200rpm for 4 hours to produce homogenous mixture. This method is also done so that the oil could dissolved completely in the solvent.

Figure 3.3 Orbital shaker

The soxhlet extractor as shown in figure 3.4 is used in the solvent extraction method. The soxhlet extractor is place on to a flask containing solvent, and then equipped with condenser. The solvent is heated to reflux. The solvent vapour travels up a distillation arm, and floods into the chamber housing the thimble of solid waste. The condenser ensures that any solvent vapours cools, and drips back down into the chamber housing the solid material. The chamber containing the solid waste slowly fills with warm solvent. When the soxhlet chamber is almost full, the chamber is automatically emptied by a siphon side arm, with the solvent running back down to the distillation flask. This cycle will be allowed to repeat many times, over hours.

Figure 3.4 Soxhlet extractor

After the solvent extraction method, rotary evaporator as shown in figure 3.5 is used to separate the oil from the solvent. The temperature in rotary evaporator will be set up according to the boiling point of the solvents. Rotary evaporator is more often and conveniently applied to separate low boiling point solvents from oil sample.

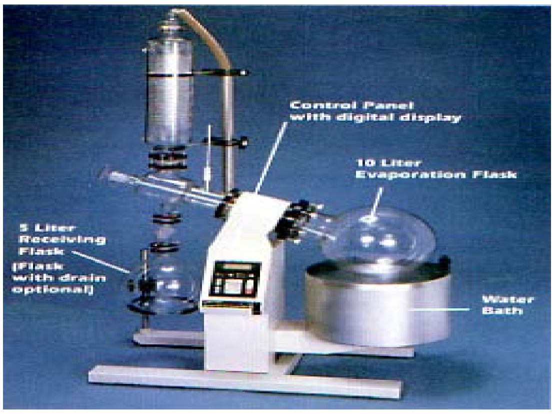

Figure 3.5 Rotary evaporator

3.4 Experimental procedure

3.4.1 Pre-treatment

Firstly, we need to collect the palm oil sludge waste from nearest palm oil mill and palm oil reinery. Then the 10g of four different sample will be put in oven at temperature of 60^{o}C, 70^{o}C, 80^{o}C, 90^{o}C, 100^{o}C and 110^{o}C each for 12 hours to remove water and to ensure the particles are free from any moisture before proceeding to the next step of the procedure. Temperature must be carefully maintained at this level so that not to burn the solid sludge and to avoid from the oil in decanter cake to be oxidized. The percentages of moisture lost for each sample is calculated with the formula below.

% of moisture loss = [(10g- A) / 10g] x 100

A = the mass of sample after drying

Then, 10g of sample is put in oven and the mass of sample after 5hr, 6hr, 7hr, 8hr, 9hr, 10hr, 11hr, and 12 hr is noted. The temperature was fixed as 90^{0}C. The percentage of moisture loss was also calculated. By this method we could determine the best temperature and time required remove the moisture from the palm oil decanter cake and also spent bleaching clay.

3.4.2 Sample preparation

The dried solid waste at the temperature of 90^0C and for 12 hours, is grind using grinder to produce powder. This is to increase the surface area of the waste sample, and increase the yield produced.

3.4.3 Solvent decanting extraction

The spent bleaching clay (20g) is weighed in four different 500ml conical flasks. For the first sample 300ml of MEK is poured and labeled as A. For the second flask 100 ml of MEK is poured and labeled as B, while for the third sample 150 ml of MEK is poured, and labeled as C and for the last sample also 150 ml of MEK is poured and labeled as D. Then all the flasks are put in orbital shaker at 200 rpm for 4 hours to produce homogenouse mixture. Then the mixture left for 12 hours, so that the oil will completely dissolve in the solvent. After that, the oil +solvent mixture was separated from the sample using filtration method. Then, 100 ml of MEK is added to the sample B and 150 ml of MEK is added to the sample C and sample D. The extract from sample A is left aside. After 12 hours, the sample D will be filtered to separate the residue oil and left aside. While for the sample B, 100 ml of MEK is added, and 150 ml of MEK is added to the sample C. Then, again the two samples will leave for 12 hours. Then all the extract from sample A, B, C and D was concentrated by rotary evaporation and then dried until complete removal of the solvent was reached. The same procedures were repeated again using hexane, acetone and petroleum ether as extracting medium. The procedures also repeated using 20g of decanter cake. The percentage of oil extracted from each sample was calculated using the formula below.

% of oil extracted = (oil extracted/ 20g) x100

3.4.4 Soxhlet extraction

The spent bleaching clay (20g) was placed in a thimber inside a soxhlet extractor. 300ml of Methyl ethyl ketone was poured into a 500ml round bottom flask as extracting medium. The extraction was continued for 10-12 hours until the extracted solvent was colourless. After extraction, the extract was concentrated by rotary evaporation and then dried until complete removal of the solvent was reached. The same procedures were repeated again using hexane, acetone and petroleum ether as extracting medium. The procedures also repeated using 20g of decanter cake. Then, three trials were done in order to get approximate results. The percentage of oil extracted from each sample was calculated using the formula below.

% of oil extracted = (oil extracted/ 20g) x100

3.4.5 Oil Analysis

After solvent recovery, all the oil samples are ready to be analyzed. In this method there were two analysis (iodine value and acid value) were carried out in order to determine the quality of the oil. All these methods are discussed below.

3.4.5.1 Iodine value analysis

Reagents

Potassium iodide (10% w/v), Starch solution, Standardized sodium thiosulphate (0.1N), Equal volumes of Cyclohexane and glacial acetic acid (1:1), Wijs reagent.

Procedures

Take approximately 0.15g to 0.30g of the test sample, weighed to the nearest 0.001g in a glass stoppered conical flask. Dissolve the test portion using 20ml of solvent (actetic acid : cyclohexane). Add 25ml of Wijs reagent using a precission pipette. Insert the stopper, swirl carefully to place the flask in the dark. Prepare a blank solution using the same procedure but omitting the test sample. Leave the flask in the dark for one hour. At the end, of the reaction time add 200ml of KI using pipette and followed by the addition of 100ml water. Titrate with standardized sodium thiosulphate solution until the yellow colour due to iodine has almost disappeared. Add few drops of starch solution and continue the titration until the blue colour just disappears after very vigorous shaking. Carry out a blank test using the blank solution concurrently.

Iodine value= $(12.69 \times N \times (V_b - V_s)) / m$

V_b = volume of sodium thiosulphate used for blank solution, V_s = volume of sodium thiosulphate solution for sample titration, m = mass of oil sample, N= normality of sodium thiosulphate.

3.4.5.2 Acid value analysis

Reagents

Standard sodium hydroxide (0.1M), Phenolphtalein indicator solution, (1.0% in 95% ethanol), Neutralized iso-propanol.

Procedures

Add 5-6 drops of phenolphthalein solution into the industrial grade iso-propanol preparation conical flask. Neutralize the iso-propanol using standardized sodium hydroxide until light pink colored with stirring. Determine the size of sample. Weigh specified amount of sample into a 250 ml of conical flask. Add in 50ml of neutralized iso-propanol into sample beaker. Add 3-5 drop of phenolphthalein solution. Put in magnetic stirrer. Put on hotplate stirrer and stir.

Read burette before titration. Titrate with standardized sodium hydroxide until light pink colored. The color must be persist for 30 seconds. Read burette after titration.

$$\text{Acid value} = (56.1 \times N \times (A-B)) / M$$

N = normality of sodium hydroxide, A = Burette reading after titration, B= Burette reading before titration, M = mass of oil sample.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

Solvent extraction is the convenient way in extracting residue oil from palm oil solid wastes. In this experiment, the parameters such as temperature and time were studied in order to obtain the optimum conditions to remove maximum moisture from the fresh palm oil decanter cake and spent bleaching clay. The optimum conditions to dry the solid wastes are at 90^{o}C and for 12 hours. In this experiment, two types of solvent extraction methods were used as a comparison and four types of solvents were used to study their performance towards oil extraction. As previously mentioned, the solvents used are methyl ethyl ketone, acetone, hexane and petroleum ether. The residue oil obtained from the two types of solvent extraction and four types of solvent was characterized. The percentages of oil extracted were higher for the experiments using acetone and methyl ethyl ketone as extracting medium. The color of the residue oil extracted is dark for the sample using MEK and acetone as solvent. Each experiment is repeated for three times in order to get closer and accurate results. After all experiments was done, result was obtained from the analysis data and the effect of time and temperature to wards drying process, effect of solvent towards residue oil yield, acid value and iodine value of the residue oil were studied. After completing 5 months of research, the scheduled experiment was accomplished. Figure 4.1 shows the residue oil obtained from spent bleaching clay and decanter cake through soxhlet extraction method and figure 4.2 shows the obtained residue oil by solvent decanting method. In the figures, the first four samples are the residue oil from spent bleaching clay and the last four samples is the samples from decanter cake. The samples are arranged by the solvent used, methyl ethyl ketone, acetone, hexane and petroleum ether. In the figure we can observe that the oil extracted using acetone and methyl ethyl ketone is darker while the oil extracted using hexane and petroleum ether is lighter.

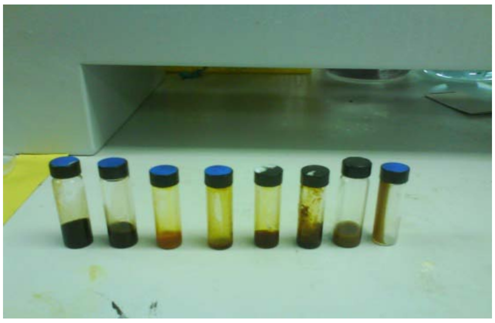

Figure 4.1 Extracted oil sample by soxhlet extraction method

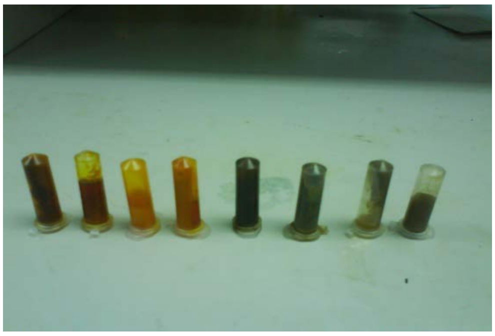

Figure 4.2 Extracted oil sample by solvent decanting method

4.2 Pre-treatment

4.2.1 Effect of temperature and time towards the moisture removal

The first stage in this research is drying the fresh solid waste in oven. The temperatures used were in the range of 60^0C to 90^oC and time was fixed as 12 hours. This drying process is necessary because palm oil wastes contain a lot of moisture and removal of moisture may maximize the oil yield. Table 4.1 shows the results obtained for the first stage experiment. The other parameter, drying time was varied from 5hr-12hr with the temperature fixed as 90°C. Table 4.2 shows the effect of drying time on percentage of moisture removed.

Table 4.1 Effect of temperature on the weight loss

Temperature (^{0}C)	Percentage of weight loss (%)	
	Decanter cake	Spent bleaching clay
60	63.5	0.9
70	65.3	1.2
80	68.1	1.4
90	73.2	2.2
100	73.5	2.8
110	73.8	3.3

Table 4.2 Effect of drying time on the weight loss

Time (hr)	Percentage of weight loss (%)	
	Decanter cake	Spent bleaching clay
5	56.0	1.50
6	58.5	1.60
7	60.0	1.75
8	61.8	1.80
9	63.5	2.00
10	66.0	2.18
11	73.0	2.20
12	73.3	2.23

Result for the effect of drying period and drying temperature on the moisture removal from spent bleaching clay and decanter cake. The data are plotted on graph in Figure 4.3 and Figure 4.4.

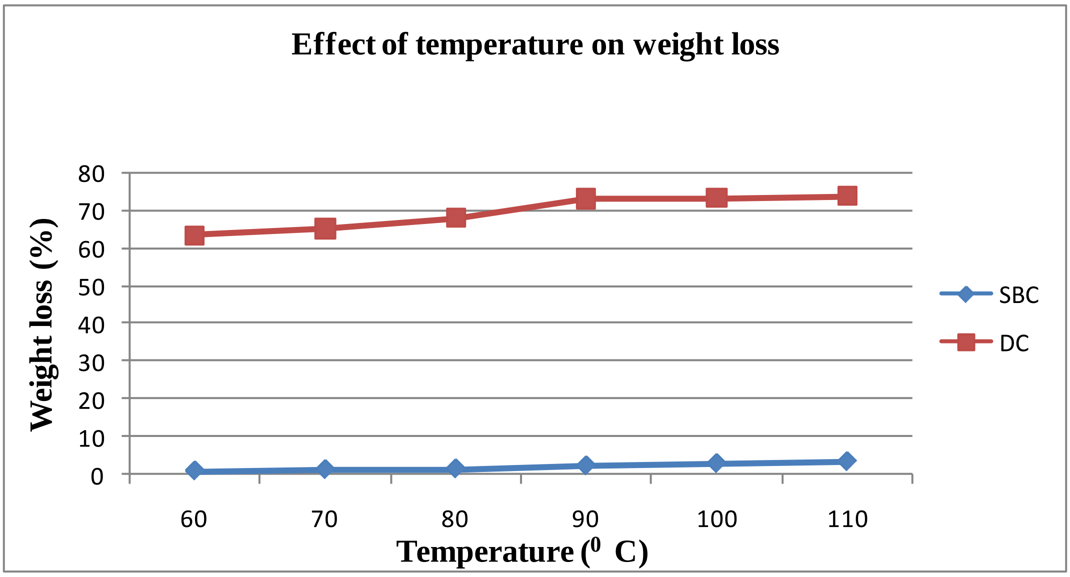

Figure 4.3 Effect of Temperature on weight loss due to moisture content

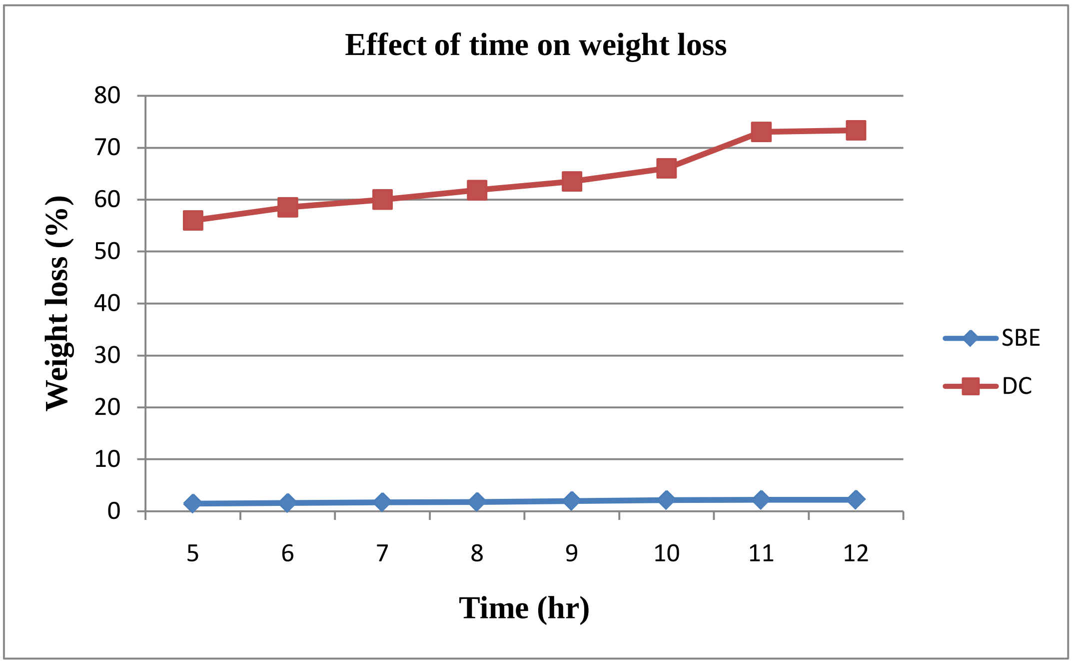

Figure 4.4 Effect of Time on weight loss due to moisture content

In the first stage of experiment, the effect of temperature was studied towards the moisture content in decanter cake and spent bleaching clay. Experiment results (figure 4.1) showed that, when the temperature is increases, the moisture content remove from palm oil wastes also increases and become constant after 90^{0}C. The percentage of weight loss due to moisture content from decanter cake is close to

73%, while percentage of weight loss due to moisture content from spent bleaching clay is only 3% because spent bleaching clay content very less moisture. The maximum moisture removal was achieved at temperature of 90^0C for decanter cake and spent bleaching clay. In figure 4.2 the weight loss due to moisture content increases when the time increases and maximum moisture removal achieved at 11 hours for both decanter cake and spent bleaching clay. From the graphs the optimum condition to dry the palm oil solid wastes is at 90^0C for 12 hours.

4.3 Oil Yield

4.3.1 Soxhlet extraction method

After drying, the decanter cake was ground into small particles; and then undergoes solvent extraction method using different type of solvents. Each experiment was repeated three times in order to get the reproducible result. The amount of oil recovered from samples using various solvents using soxhlet extraction method is shown in Table 4.3 and Table 4.4.

Table 4.3 Percentage of oil extracted from spent bleaching clay by soxhlet extraction

	Percentage of oil extracted (%)		
Solvent	**Trial 1**	**Trial 2**	**Trial 3**
Methylethylketone	29.30	24.40	26.80
Acetone	27.15	27.80	28.20
Hexane	19.05	17.40	19.05
Petroleum ether	17.30	18.41	18.70

Table 4.4 Percentage of oil extracted from decanter cake by soxhlet extraction

	Percentage of oil extracted (%)		
Solvent	**Trial 1**	**Trial 2**	**Trial 3**
Methylethylketone	26.38	28.80	27.10
Acetone	29.00	26.15	29.90
Hexane	23.35	19.05	19.80
P.Ether	15.15	14.15	17.00

From the result, it clearly shows that percentages of oil extracted from decanter cake and spent bleaching clay is dependent on the type of solvent used. The result is shown graphically in the Figure 4.3 and Figure 4.4. It is clear that the total oil yields in terms of percentage of grams of oil extracted per gram of sample obtained by the polar solvents were higher than non polar solvents. In general the color of the polar solvent-extracted oils was darker than those extracted with non polar solvents. The increased quantity of polar components extracted using the polar solvents undoubtedly includes minor components present in the crude oils, such as phosphatides, fatty acids, and sterols as well as other oxidized, hydrolyzed and polymerized products from reactions catalyzed by the solid wastes. From the figure 4.5 below the average of oil extracted from spent bleaching clay by MEK is 26.83%, acetone is 28.38%, hexane 18.50% and Petroleum ether 17.14% respectively. The average oil extracted from decanter cake for MEK is 27.43%, acetone is 28.35%, hexane is 20.73% and petroleum ether is 15.43% respectively. From this, we know that the oil yield from MEK and acetone is high compared to hexane and petroleum ether. From the observation the color of oil extracted from palm oil solid waste using MEK and acetone is darker compared to hexane and petroleum ether.

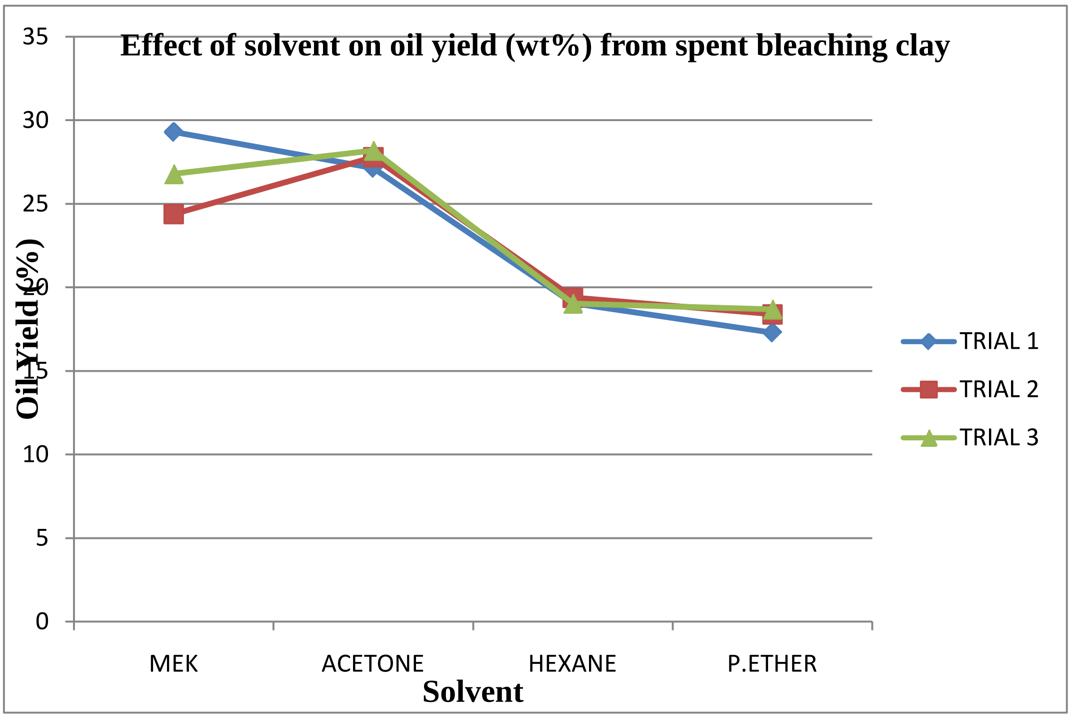

Figure 4.5 Effect of solvent on oil yield from spent bleaching clay using soxhlet extraction

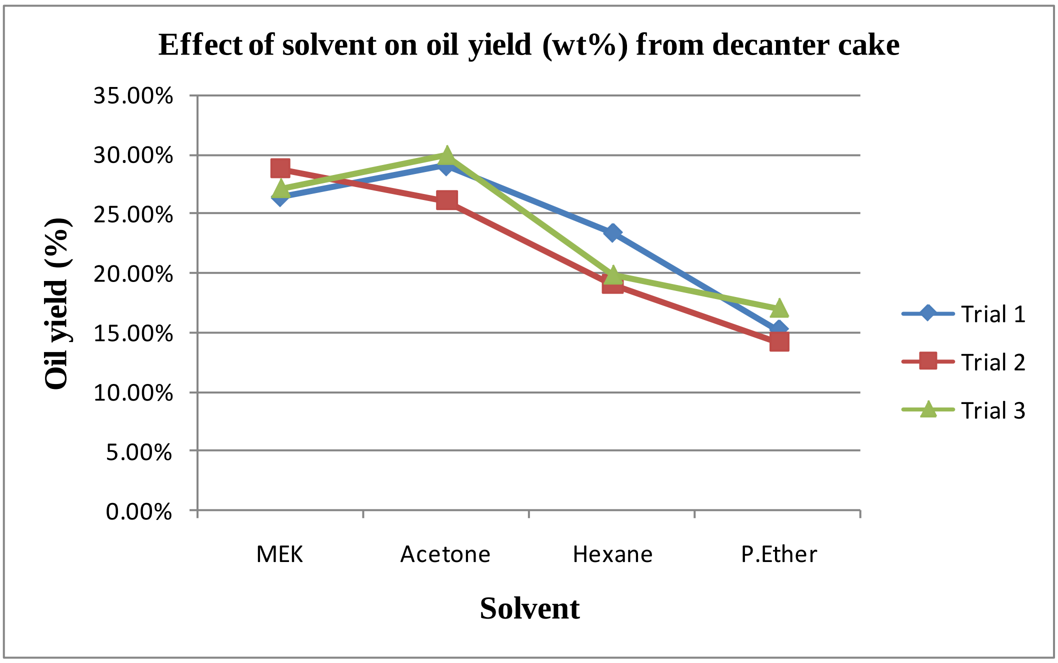

Figure 4.6 Effect of solvent on oil yield from decanter cake using soxhlet method

4.3.2 Solvent decanting method

After extracting the residue oil using soxhlet extraction solvent decanting extraction was done as a comparison to compare which extraction method can extract more yield. This method is done four times with different amount of solvent to get reproducible result. Table 4.5 and table 4.6 below shows the result obtained for the four runs.

Table 4.5 Percentage of oil extracted from spent bleaching clay using solvent decanting method

	Percentage of Oil Extracted (%)			
Solvent	**Run 1**	**Run 2**	**Run 3**	**Run 4**
Methylethylketone	21.5	25.5	23.5	22.5
Acetone	21.0	24.0	22.5	21.5
Hexane	15.5	17.5	17.0	16.0
Petroleum ether	12.0	15.5	14.0	13.0

Table 4.6 Percentage of oil extracted from decanter cake using solvent decanting method

	Percentage of Oil Extracted (%)			
Solvent	**Run 1**	**Run 2**	**Run 3**	**Run 4**
Methylethylketone	21.5	24.5	22.5	21.0
Acetone	19.0	23.0	21.5	20.0
Hexane	11.0	14.0	12.5	11.5
Petroleum ether	10.5	13.0	11.5	12.0

From the result it is so clear that, the percentage of oil extracted is maximum in Run 2. In run 2 the total amount of solvent used is 300 ml (100 ml added three times). Then the run 3, with the total amount of 450 ml (150 ml added three times) has the second higher percentage of oil extracted. In the figure 4.7 and figure 4.8 we could clearly see that the run 4 with the amount of 300 ml solvent give the least percentage of oil extracted. From here, we can discussed that the oil yield is maximum at run 2, because in run 2 the solvent is added three times, each time 100ml of solvent is added. While in run 3 the same procedure repeated but the amount of solvent added is 150 ml each time. So, from here we could conclude that the best amount of solvent to be used in decanting method is 100ml. In run 4 the amount of solvent used is 300ml, with each time 150ml of solvent is added. The oil yield in this run is slightly higher than the run 1. So we could discussed that, adding the amount of solvent could increase the oil yield. The Figure 4.7 and Figure 4.8 below is the graphical result for the solvent decanting method.

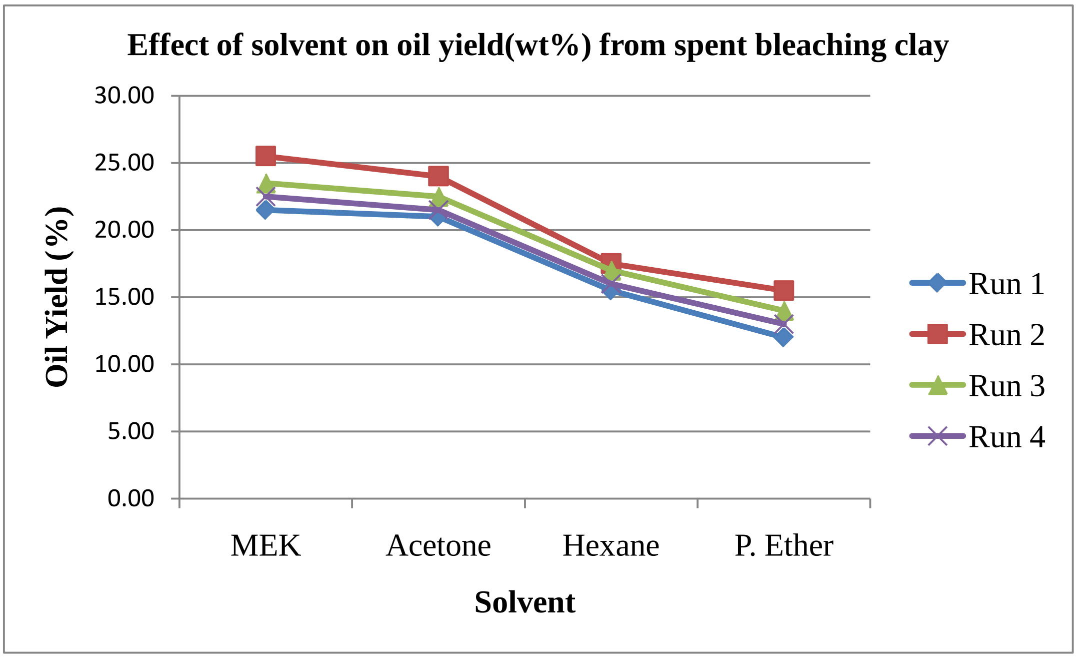

Figure 4.7 Effect of solvent on oil yield from spent bleaching clay using solvent decanting method

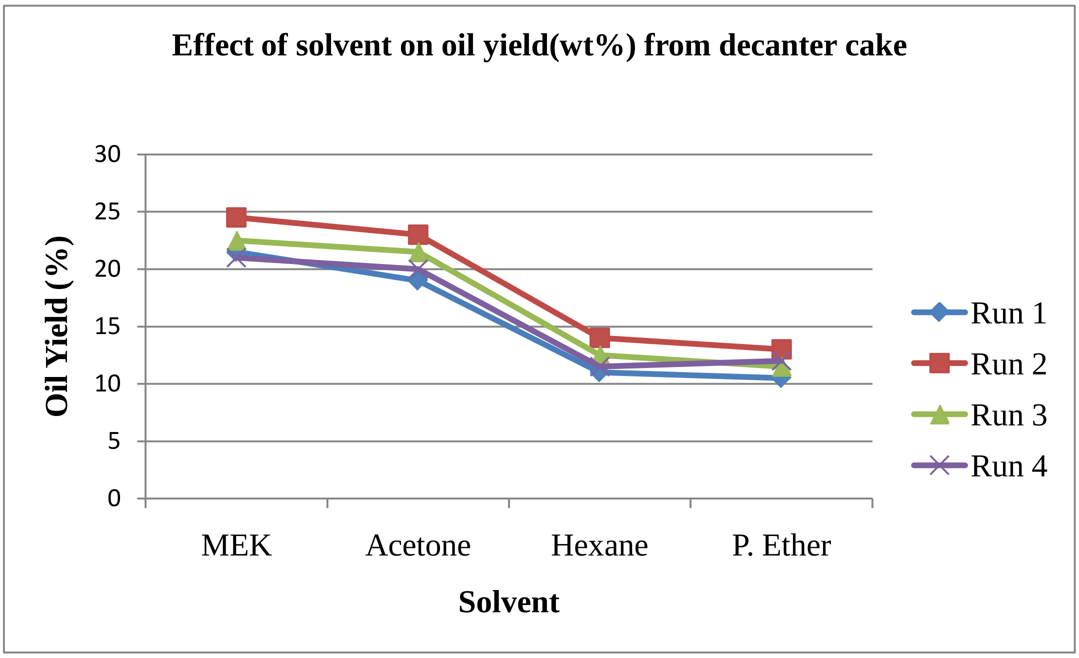

Figure 4.8 Effect of solvent on oil yield from decanter cake using solvent decanting method.

4.3.2 Comparison of Solvent decanting method and soxhlet extraction method

From the figure 4.9 and 4.10, we can see that the residue oil yield is high for the soxhlet method compared to solvent decanting method. In figure 4.9 the oil extracted using MEK is 26.83% by soxhlet extraction and only 24.5% by solvent decanting method. So, it is so clear that percentage oil yield is maximum by using soxhlet extraction method, because in soxhlet extraction the fresh solvent is continuously in contact with the sample without having to introduce more solvent, which will dilute the extract and there experiment take place unattended and can conveniently occur overnight if desired. Other than that, the advantage of soxhlet extraction is , instead of many portion of warm solvent being passed through the sample, just one batch of solvent is recycled. While in solvent decanting extraction the solvent can be vaporized, and loss of oil happen when the oil sample is filtered.

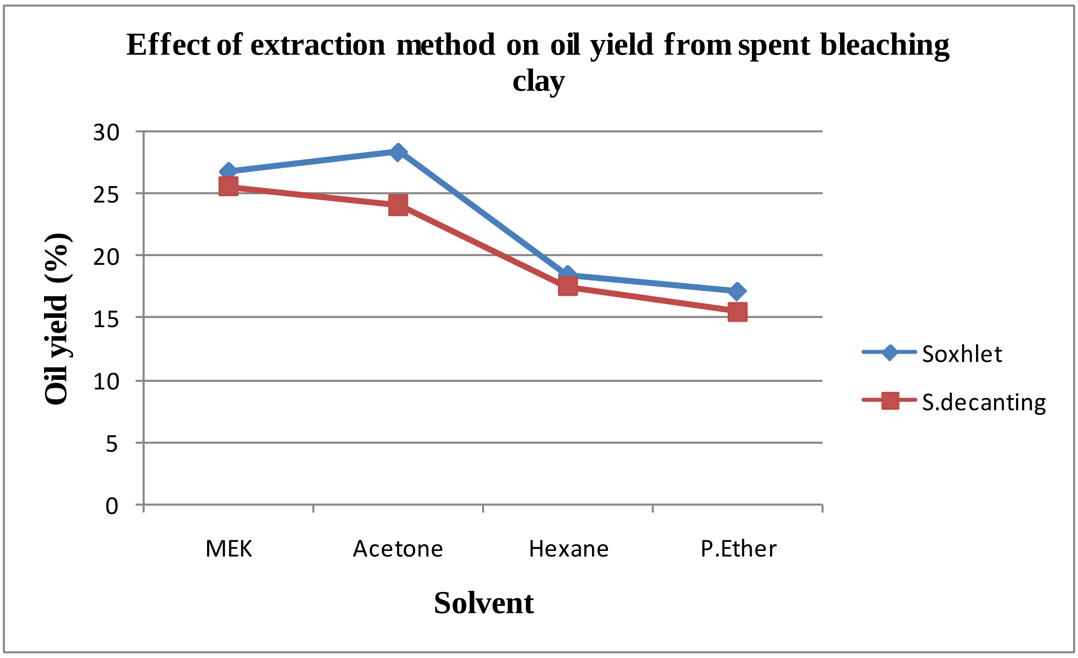

Figure 4.9 Effect of solvent decanting method and soxhlet method towards oil yield for spent bleaching clay.

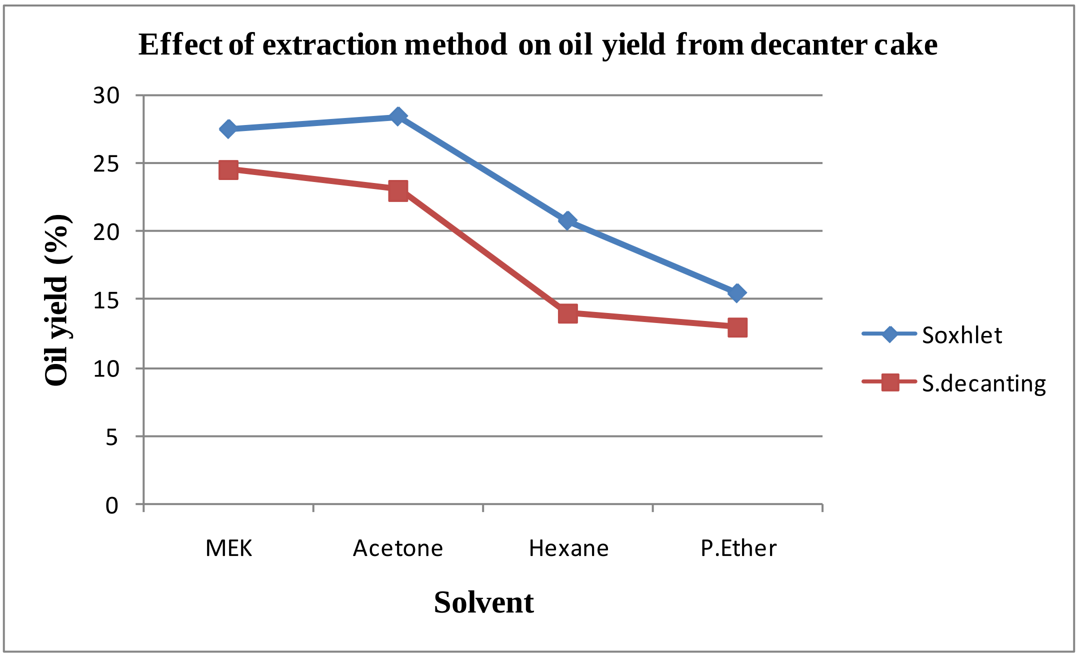

Figure 4.10 Effect of solvent decanting method and soxhlet method towards oil yield for decanter cake.

4.4 Determination of acid value and iodine value

After extracting process, the residue oil extracted was characterized by chemical analysis as shown in the Table 4.7.

Table 4.7 Acid value and Iodine value of residue oil

Solvent	**Acid value**		**Iodine value**	
	SBC	**DC**	**SBC**	**DC**
MEK	214.83	243.72	54.34	50.16
Acetone	239.45	223.22	54.21	46.13
Hexane	237.95	216.77	60.40	61.79
P.Ether	242.71	243.09	66.68	59.50

The acid value is the measurement of free fatty acid present in the oil. Some deterioration that took place during storage of either raw materials from which oil is obtained (storage of oil) or oil isolation results from the hydrolysis of triglycedic to yield free fatty acid. (Lee et.al, 2000). Iodine value gives total degree of unsaturated substance expressed in the percentage of iodine absorbed by the oil. From the table 4.5 the range of acid value of residue oil from spent bleaching clay and decanter cake is between200-250. The acid value for the four different crude oils were high thus, the oil extracts would need some form of purification (refining) to enhance stability and storage. Low acid values suggest stability of oil. Pocklington (2002) reports that acid value in crude oil estimate the amount of oil that will be lost during refining. Thus, the higher the acid value the higher the amount of oil that could be lost during processing. (Nwabueze et. al, 2008).While the iodine value is range between 50 -70. The whole analysis was carried out and the values were calculated by comparing them with standard value from literature review. From literature review, acid value for crude palm oil is 217-230 and iodine value is 51.3. (PORIM). This implies that all the values lie within the experimental values, therefore these values could be accepted.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The experimental studies show that through soxhlet extraction method almost 25% of oil can be extracted from palm oil decanter cake and spent bleaching clay. In this research, the first stage is to remove moisture from the samples, because the palm oil solid wastes contain lot of moisture in it, and if the moisture is not removed this will effect the oil yield. In order to obtain the optimum conditions to remove the moisture the waste sample first was dried at different temperature and for different duration. The best condition to remove the maximum moisture from palm oil decanter cake and spent bleaching clay is at 90^0C and for 12 hours because maximum percentage moisture is removed as this condition.

It can be seen that, the types of solvent used as extracting medium and the extraction method used were the main factors affecting the oil extraction process. Although two different samples were studied, spent bleaching clay and decanter cake used, the oil yield from both samples are almost same because both wastes contains the same amount of oil around 25-35%. In this research, the best method to recover oil from palm oil decanter cake and spent bleaching clay is soxhlet extraction method. The main reason is in soxhlet extraction the contact between solvent and waste sample is longer compared to solvent decanting method.

From the research done, we can conclude that methylethyl ketone and acetone give higher oil yield compared to hexane and petroleum ether for both decanter cake and spent bleaching clay. MEK and acetone are characterized as polar solvents and hexane and petroleum ether are non polar solvents. In general, the color of oil extracted from polar solvent is darker than the oil extracted from non polar solvent. This is because polar solvent also extract the other minor polar component from the sample wastes.

The last stage in this research, is determining the iodine value and the acid value of the oil. The amount of iodine in grams absorbed per 100 ml of oil is the Iodine value of oil. The range iodine value for the residue oil extracted from decanter cake and spent bleaching clay is 40-60, while the acid value is at 200-250. According to the PORIM test, the iodine value for the crude palm oil is 51.3 and the acid value is 217-230. The quality of the oil extracted from the decanter cake and spent bleaching clay using different type of solvent are almost the same, because the sources are same. The iodine value and acid value are also closer for all the oil extracted.

5.2 Recommendation

Based on the result and discussion, a few measurements can be considered to upgrade future studies in extracting residue oil from decanter cake and spent bleaching clay. There are still many other methods that have not been explored for the extraction of oil from palm oil wastes. For further study, other methods like microwave extraction, supercritical extraction, and hydro distillation can be use to extract residue oil.

After extracting oil, the residue waste samples can be used as activated carbon, membrane and useful materials. This is because, other than oil the decanter cake and spent bleaching clay also contain more components. So, further studies is required to maximize the usage of spent bleaching clay and decanter cake rather than throw it to landfills.

The iodine value and acid value for the residue oil is high compared to pure crude oil. So, the quality of oil is poorer compared to pure crude palm oil. Instead of using high grade or edible oil in synthesizing of biodiesel waste, the residue oil extracted from decanter cake and spent bleaching clay can be used as raw material. The higher the iodine value, the more unsaturated (the greater the number of double bonds) the oil and the higher is the potential for the oil to polymerize. The iodine value can be easily reduced by hydrogenation of the oil (reacting the oil with hydrogen), the hydrogen breaking the double bond and converting the fat or oil into a more saturated oil which reduces the tendency of the oil to polymerize. Due, to high FFA content of residue oils recovered from both decanter cake and spent bleaching clay, their application in foods are limited. However these residue oils can be used in non-food applications such as converting them into methyl esters as bio-fuels. Other possible applications include bio-lubricants, grease, plasticizers, detergents, agricultural chemicals, emulsifiers, soap, candle and other oleo chemicals.

Other than that, the further analysis like peroxide value , p-anisidine value and totox value can be done in order to determine the quality of the oil. Analysis equipment like HPLC and GC also can be used to analyze the content of the oil. The equipment used in this research need to be supervised by it owner to make sure that it is safe to use and also to prevent any damage of the equipment.

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APPENDIX A

PRE- TREATMENT OF THE SOLID WASTE

A.1 Effect of temperature on percentage of moisture removal.

A.1.1 Calculation for moisture removal (%) from spent bleaching clay.

The value of moisture removed for each sample prepared for the effect of temperature is calculated below.

i) Sample 1 (60^0C)

Mass of raw material = 10g

Mass of sample after drying = 9.914g

Mass of moisture = 0.086g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

$$= \frac{0.086g}{10g} \text{ x } 100\%$$

$$= 0.86\%$$

ii) Sample 2 (70^oC)

Mass of raw material = 10g

Mass of sample after drying = 9.88g

Mass of moisture = 0.12g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

$$= \frac{0.12g}{10g} \text{ x } 100\%$$

$$= 1.20\%$$

iii) Sample 3 (80^0C)

Mass of raw material = 10g

Mass of sample after drying = 9.86g

Mass of moisture = 0.14g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

= $\frac{0.14g}{10g}$ x 100%

= 1.40%

iv) Sample 4 (90^0C)

Mass of raw material = 10g

Mass of sample after drying = 9.78g

Mass of moisture = 0.22g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

= $\frac{0.22g}{10g}$ x 100%

= 2.2%

v) Sample 5 (100^0C)

Mass of raw material = 10g

Mass of sample after drying = 9.72g

Mass of moisture = 0.28g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

= $\frac{0.28g}{10g}$ x 100%

= 2.8%

vi) Sample 6 (110^0C)

Mass of raw material = 10g

Mass of sample after drying = 9.67g

Mass of moisture = 0.33g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

$$= \frac{0.33g}{10g} \times 100\%$$

$$= 3.3\%$$

A.1.2 Calculation for moisture removal (%) from decanter cake.

i) Sample 1 (60^0C)

Mass of raw material = 10g

Mass of sample after drying = 3.65g

Mass of moisture = 6.35g

$$\text{\% of moisture removal} = \frac{\text{Mass of moisture}}{\text{Mass of raw material}} \times 100\%$$

$$= \frac{6.35g}{10g} \times 100\%$$

$$= 63.50\%$$

ii) Sample 2 (70^0C)

Mass of raw material = 10g

Mass of sample after drying = 3.47g

Mass of moisture = 6.53g

$$\text{\% of moisture removal} = \frac{\text{Mass of moisture}}{\text{Mass of raw material}} \times 100\%$$

$$= \frac{6.53g}{10g} \times 100\%$$

$$= 65.30\%$$

iii) Sample 3 (80^0C)

Mass of raw material = 10g

Mass of sample after drying = 3.20g

Mass of moisture = 6.80g

$$\text{\% of moisture removal} = \frac{\text{Mass of moisture}}{\text{Mass of raw material}} \times 100\%$$

$$= \frac{6.80g}{10g} \times 100\%$$

$$= 68.0\%$$

iv) Sample 4 (90^0C)

Mass of raw material = 10g

Mass of sample after drying = 2.68g

Mass of moisture = 7.32g

$$\% \text{ of moisture removal} = \frac{\text{Mass of moisture}}{\text{Mass of raw material}} \times 100\%$$

$$= \frac{7.32g}{10g} \times 100\%$$

$$= 73.2\%$$

v) Sample 5 (100^0C)

Mass of raw material = 10g

Mass of sample after drying = 2.65g

Mass of moisture = 7.35g

$$\% \text{ of moisture removal} = \frac{\text{Mass of moisture}}{\text{Mass of raw material}} \times 100\%$$

$$= \frac{7.35g}{10g} \times 100\%$$

$$= 73.5\%$$

vi) Sample 6 (110^0C)

Mass of raw material = 10g

Mass of sample after drying = 2.62g

Mass of moisture = 7.38g

$$\% \text{ of moisture removal} = \frac{\text{Mass of moisture}}{\text{Mass of raw material}} \times 100\%$$

$$= \frac{7.38g}{10g} \times 100\%$$

$$= 73.8\%$$

A.2 Effect of drying time on percentage of moisture removal

A.2.1 Calculation for moisture removal (%) from spent bleaching clay.

i) Sample 1 (5 hr)

Mass of raw material = 10g

Mass of sample after drying = 9.85g

Mass of moisture = 0.15g

$$\% \text{ of moisture removal} = \frac{\text{Mass of moisture}}{\text{Mass of raw material}} \times 100\%$$

$$= \frac{0.15g}{10g} \times 100\%$$

$$= 1.50\%$$

ii) Sample 2 (6 hr)

Mass of raw material = 10g

Mass of sample after drying = 9.84g

Mass of moisture = 0.16g

$$\% \text{ of moisture removal} = \frac{\text{Mass of moisture}}{\text{Mass of raw material}} \times 100\%$$

$$= \frac{0.16g}{10g} \times 100\%$$

$$= 1.60\%$$

iii) Sample 3 (7 hr)

Mass of raw material = 10g

Mass of sample after drying = 9.825g

Mass of moisture = 0.175g

$$\% \text{ of moisture removal} = \frac{\text{Mass of moisture}}{\text{Mass of raw material}} \times 100\%$$

$$= \frac{0.175g}{10g} \times 100\%$$

$$= 1.75\%$$

iv) Sample 4 (8 hr)

Mass of raw material = 10g

Mass of sample after drying = 9.82g

Mass of moisture = 0.18g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

= $\frac{0.18g}{10g}$ x 100%

= 1.80%

v) Sample 5 (9 hr)

Mass of raw material = 10g

Mass of sample after drying = 9.80g

Mass of moisture = 0.20g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

= $\frac{0.20g}{10g}$ x 100%

= 2.00%

vi) Sample 6 (10 hr)

Mass of raw material = 10g

Mass of sample after drying = 9.782g

Mass of moisture = 0.218g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

= $\frac{0.218g}{10g}$ x 100%

= 2.18%

vii) Sample 7 (11 hr)

Mass of raw material = 10g

Mass of sample after drying = 9.78g

Mass of moisture = 0.22g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

$= \frac{0.22g}{10g} \times 100\%$

$= 2.20\%$

viii) Sample 8 (12 hr)

Mass of raw material = 10g

Mass of sample after drying = 9.777g

Mass of moisture = 0.223g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}} \times 100\%$

$= \frac{0.223g}{10g} \times 100\%$

$= 2.23\%$

A.2.2 Calculation for moisture removal (%) from decanter cake.

i) Sample 1 (5 hr)

Mass of raw material = 10g

Mass of sample after drying = 4.4g

Mass of moisture = 5.6g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}} \times 100\%$

$= \frac{5.6g}{10g} \times 100\%$

$= 56.0\%$

ii) Sample 2 (6 hr)

Mass of raw material = 10g

Mass of sample after drying = 4.15g

Mass of moisture = 5.85g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}} \times 100\%$

$= \frac{5.85g}{10g} \times 100\%$

= 58.5%

iii) Sample 3 (7 hr)

Mass of raw material = 10g

Mass of sample after drying = 4.00g

Mass of moisture = 6.00g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

= $\frac{6.0g}{10g}$ x 100%

= 60%

iv) Sample 4 (8 hr)

Mass of raw material = 10g

Mass of sample after drying = 6.18g

Mass of moisture = 3.82g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

= $\frac{6.18g}{10g}$ x 100%

= 61.80%

v) Sample 5 (9 hr)

Mass of raw material = 10g

Mass of sample after drying = 3.65g

Mass of moisture = 6.35g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

= $\frac{6.35g}{10g}$ x 100%

= 63.50%

vi) Sample 6 (10 hr)

Mass of raw material = 10g

Mass of sample after drying = 3.40g

Mass of moisture = 6.60g

% of moisture removal = $\frac{\text{Mass of moisture}}{\text{Mass of raw material}}$ x 100%

$$= \frac{6.6g}{10g} \times 100\%$$

$$= 66.0\%$$

vii) Sample 7 (11 hr)

Mass of raw material = 10g

Mass of sample after drying = 2.70g

Mass of moisture = 7.30g

% of moisture removal = $\frac{\text{Mass of moisture}}{Mass\ of\ raw\ material}$ x 100%

$$= \frac{7.30g}{10g} \times 100\%$$

$$= 73.0\%$$

viii) Sample 8 (12 hr)

Mass of raw material = 10g

Mass of sample after drying = 2.67g

Mass of moisture = 7.33g

% of moisture removal = $\frac{\text{Mass of moisture}}{Mass\ of\ raw\ material}$ x 100%

$$= \frac{7.33g}{10g} \times 100\%$$

$$= 73.3\%$$

APPENDIX B

OIL YIELD

B.1 Oil yield from spent bleaching clay by soxhlet extraction method

Trial 1

i) Sample 1 (Methylethylketone)

Mass of raw material = 20g

Mass of oil extracted = 5.86g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ \ raw\ material}$ x 100%

$= \frac{5.86g}{20g}$ x 100%

= 29.30%

ii) Sample 2 (Acetone)

Mass of raw material = 20g

Mass of oil extracted = 5.43g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ \ raw\ material}$ x 100%

$= \frac{5.43g}{20g}$ x 100%

= 27.15%

iii) Sample 3 (Hexane)

Mass of raw material = 20g

Mass of oil extracted = 3.81g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ \ raw\ material}$ x 100%

$= \frac{3.81g}{20g}$ x 100%

= 19.05%

iv) Sample 4 (Petroleum ether)

Mass of raw material = 20g

Mass of oil extracted = 2.86g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{2.86g}{20g}$ x 100%

= 14.30%

Trial 2

i) Sample 1 (Methyl ethyl ketone)

Mass of raw material = 20g

Mass of oil extracted = 4.88g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{4.88g}{20g}$ x 100%

= 24.40%

ii) Sample 2 (Acetone)

Mass of raw material = 20g

Mass of oil extracted = 5.56g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{5.56g}{20g}$ x 100%

= 27.80%

iii) Sample 3 (Hexane)

Mass of raw material = 20g

Mass of oil extracted = 3.48g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{3.48g}{20g}$ x 100%

= 17.40%

iv) Sample 4 (Petroleum ether)

Mass of raw material = 20g

Mass of oil extracted = 3.68g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{3.68g}{20g}$ x 100%

= 18.40%

Trial 3

i) Sample 1 (Methyl ethyl ketone)

Mass of raw material = 20g

Mass of oil extracted = 5.36g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{5.36g}{20g}$ x 100%

= 26.80%

ii) Sample 2 (Acetone)

Mass of raw material = 20g

Mass of oil extracted = 5.64g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{5.64g}{20g}$ x 100%

= 28.20%

iii) Sample 3 (Hexane)

Mass of raw material = 20g

Mass of oil extracted = 3.81g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{3.81g}{20g}$ x 100%

= 19.05%

iv) Sample 4 (Petroleum ether)

Mass of raw material = 20g

Mass of oil extracted = 3.74g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{3.74g}{20g}$ x 100%

= 18.70%

B.2 Oil yield from decanter cake by soxhlet extraction method

Trial 1

i) Sample 1 (Methylethylketone)

Mass of raw material = 20g

Mass of oil extracted = 5.28g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{5.28g}{20g}$ x 100%

= 26.38%

ii) Sample 2 (Acetone)

Mass of raw material = 20g

Mass of oil extracted = 5.80g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{5.80g}{20g}$ x 100%

= 29.00%

iii) Sample 3 (Hexane)

Mass of raw material = 20g

Mass of oil extracted = 4.67g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{4.67g}{20g}$ x 100%

= 23.35%

iv) Sample 4 (Petroleum ether)

Mass of raw material = 20g

Mass of oil extracted = 2.86g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{3.03g}{20g}$ x 100%

= 15.15%

Trial 2

i) Sample 1 (Methylethylketone)

Mass of raw material = 20g

Mass of oil extracted = 5.42g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{5.42g}{20g}$ x 100%

= 27.10%

ii) Sample 2 (Acetone)

Mass of raw material = 20g

Mass of oil extracted = 5.98g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{5.98g}{20g}$ x 100%

= 29.90%

iii) Sample 3 (Hexane)

Mass of raw material = 20g

Mass of oil extracted = 3.96g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{3.96g}{20g}$ x 100%

= 19.80%

iv) Sample 4 (Petroleum ether)

Mass of raw material = 20g

Mass of oil extracted = 3.40g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{3.40g}{20g}$ x 100%

= 17.00%

Trial 3

i) Sample 1 (Methylethylketone)

Mass of raw material = 20g

Mass of oil extracted = 5.75g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{5.75g}{20g}$ x 100%

= 28.80%

ii) Sample 2 (Acetone)

Mass of raw material = 20g

Mass of oil extracted = 5.23g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{5.23g}{20g}$ x 100%

= 26.15%

iii) Sample 3 (Hexane)

Mass of raw material = 20g

Mass of oil extracted = 4.67g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{3.81g}{20g}$ x 100%

= 19.05%

iv) Sample 4 (Petroleum ether)

Mass of raw material = 20g

Mass of oil extracted = 2.83g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material}$ x 100%

$= \frac{2.83g}{20g}$ x 100%

= 14.15%

B.3 Oil yield from spent bleaching clay by solvent decanting method.

Run 1

i) Sample 1 (Methylethylketone)

Mass of raw material = 20g

Mass of oil extracted = 4.30g

$$\% \text{ of oil yield } = \frac{\text{Mass of oil extracted}}{\text{Mass of raw material}} \text{ x } 100\%$$

$$= \frac{4.30g}{20g} \text{ x } 100\%$$

$$= 21.5\%$$

ii) Sample 2 (Acetone)

Mass of raw material = 20g

Mass of oil extracted = 4.20g

$$\% \text{ of oil yield } = \frac{\text{Mass of oil extracted}}{\text{Mass of raw material}} \text{ x } 100\%$$

$$= \frac{4.20g}{20g} \text{ x } 100\%$$

$$= 21.0\%$$

iii) Sample 3 (Hexane)

Mass of raw material = 20g

Mass of oil extracted = 3.1g

$$\% \text{ of oil yield } = \frac{\text{Mass of oil extracted}}{\text{Mass of raw material}} \text{ x } 100\%$$

$$= \frac{3.1g}{20g} \text{ x } 100\%$$

$$= 15.5\%$$

iv) Sample 4 (Petroleum ether)

Mass of raw material = 20g

Mass of oil extracted = 2.4g

$$\% \text{ of oil yield } = \frac{\text{Mass of oil extracted}}{\text{Mass of raw material}} \text{ x } 100\%$$

$$= \frac{2.4g}{20g} \text{ x } 100\%$$

$$= 12.00\%$$

	Run 2		**Run 3**		**Run 4**	
Solvent	**Oil extracted (g)**	**% oil extracted**	**Oil extracted (g)**	**% oil extracted**	**Oil extracted (g)**	**% oil extracted**
MEK	5.1	25.5	4.7	23.5	4.5	22.5
Acetone	4.8	24	4.5	22.5	4.3	21.5
Hexane	3.5	17.5	3.4	17	3.2	16
P. Ether	3.1	15.5	2.8	14	2.6	13

B.4 Oil yield from decanter cake by decanting solvent extraction method

Run 1

i) Sample 1 (Methylethylketone)

Mass of raw material = 20g

Mass of oil extracted = 4.3g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material} \times 100\%$

$= \frac{4.3g}{20g} \times 100\%$

= 21.5%

ii) Sample 2 (Acetone)

Mass of raw material = 20g

Mass of oil extracted = 3.8g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ raw\ material} \times 100\%$

$= \frac{3.8g}{20g} \times 100\%$

= 19%

iii) Sample 3 (Hexane)

Mass of raw material = 20g

Mass of oil extracted = 2.2g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ \ raw\ material}$ x 100%

$= \frac{2.2g}{20g}$ x 100%

= 11%

iv) Sample 4 (Petroleum ether)

Mass of raw material = 20g

Mass of oil extracted = 2.1g

% of oil yield $= \frac{Mass\ of\ oil\ extracted}{Mass\ of\ \ raw\ material}$ x 100%

$= \frac{2.1g}{20g}$ x 100%

= 10.5%

	Run 2		**Run 3**		**Run 4**	
Solvent	**Oil extracted (g)**	**% oil extracted**	**Oil extracted (g)**	**% oil extracted**	**Oil extracted (g)**	**% oil extracted**
MEK	4.9	24.5	4.5	22.5	4.2	21
Acetone	4.6	23	4.3	21.5	4.0	20
Hexane	3.8	14	2.5	12.5	2.3	11.5
P. Ether	2.6	13	2.2	11.5	2.4	12

APPENDIX C

IODINE VALUE ANALYSIS

In order to determine the iodine value of the residue oil, each sample analysis will be repeated three times to obtain the average value. Calculation for the iodine value is shown below.

C.1 Iodine value for the oil from spent bleaching clay using soxhlet extraction method.

Trial 1

i) Sample 1 (Methylethylketone)

Mass of sample = 0.321g

Titration of blank solution, V_b = 50ml

Titration of sample, V_s = 40.5 ml

Normality of natrium thiosulphate = 0.1M

$$IV = \frac{12.69 \ x \ N \ x \ (Vb - Vs)}{Mass \ of \ oil \ sample}$$

$$= \frac{12.69 \ x \ 0.1 \ x \ (50 - 40.5)}{0.321}$$

$$= 37.67$$

ii) Sample 2 (Acetone)

Mass of sample = 0.295g

Titration of blank solution, V_b = 50ml

Titration of sample, V_s = 38.6 ml

Normality of natrium thiosulphate = 0.1M

$$IV = \frac{12.69 \ x \ N \ x \ (Vb - Vs)}{Mass \ of \ oil \ sample}$$

$$= \frac{12.69 \ x \ 0.1 \ x \ (50 - 38.6)}{0.295}$$

= 49.04

iii) Sample 3 (Hexane)

Mass of sample = 0.312g

Titration of blank solution, V_b = 50ml

Titration of sample, V_s = 35.5 ml

Normality of natrium thiosulphate = 0.1M

IV $= \frac{12.69\ x\ N\ x\ (Vb-Vs)}{Mass\ of\ oil\ sample}$

$= \frac{12.69\ x\ 0.1\ x\ (50-35.5)}{0.312}$

= 58.97

iv) Sample 4 (Petroleum ether)

Mass of sample = 0.325g

Titration of blank solution, V_b = 50ml

Titration of sample, V_s = 34.2 ml

Normality of natrium thiosulphate = 0.1M

IV $= \frac{12.69\ x\ N\ x\ (Vb-Vs)}{Mass\ of\ oil\ sample}$

$= \frac{12.69\ x\ 0.1\ x\ (50-34.2)}{0.325}$

= 61.69

Trial 2

i) Sample 1 (Methylethylketone)

Mass of sample = 0.294g

Titration of blank solution, V_b = 50ml

Titration of sample, V_s = 35.8 ml

Normality of natrium thiosulphate = 0.1M

IV $= \frac{12.69\ x\ N\ x\ (Vb-Vs)}{Mass\ of\ oil\ sample}$

$= \frac{12.69\ x\ 0.1\ x\ (50-35.8)}{0.294}$

= 61.29

ii) Sample 2 (Acetone)

Mass of sample = 0.303g

Titration of blank solution, V_b = 50ml

Titration of sample, V_s = 37.2ml

Normality of natrium thiosulphate = 0.1M

$$IV = \frac{12.69 \ x\ N\ x\ (Vb - Vs)}{Mass\ of\ oil\ sample}$$

$$= \frac{12.69\ x\ 0.1\ x\ (50 - 37.2)}{0.303}$$

= 53.61

iii) Sample 3 (Hexane)

Mass of sample = 0.320g

Titration of blank solution, V_b = 50ml

Titration of sample, V_s = 33.6 ml

Normality of natrium thiosulphate = 0.1M

$$IV = \frac{12.69 \ x\ N\ x\ (Vb - Vs)}{Mass\ of\ oil\ sample}$$

$$= \frac{12.69\ x\ 0.1\ x\ (50 - 33.6)}{0.320}$$

= 65.04

iv) Sample 4 (Petroleum ether)

Mass of sample = 0.285g

Titration of blank solution, V_b = 50ml

Titration of sample, V_s = 36.3 ml

Normality of natrium thiosulphate = 0.1M

$$IV = \frac{12.69 \ x\ N\ x\ (Vb - Vs)}{Mass\ of\ oil\ sample}$$

$$= \frac{12.69\ x\ 0.1\ x\ (50 - 36.3)}{0.285}$$

= 61.00

Trial 3

i) Sample 1 (Methylethylketone)

Mass of sample = 0.313g

Titration of blank solution, V_b = 50ml

Titration of sample, V_s = 34.2ml

Normality of natrium thiosulphate = 0.1M

$$IV = \frac{12.69 \ x\ N\ x\ (Vb - Vs)}{Mass\ of\ oil\ sample}$$

$$= \frac{12.69 \ x\ 0.1\ x\ (50-34.2)}{0.313}$$

= 64.06

ii) Sample 2 (Acetone)

Mass of sample = 0.311 g

Titration of blank solution, V_b = 50ml

Titration of sample, V_s = 35.3ml

Normality of natrium thiosulphate = 0.1M

IV $$= \frac{12.69 \ x\ N\ x\ (Vb-Vs)}{Mass\ of\ oil\ sample}$$

$$= \frac{12.69 \ x\ 0.1\ x\ (50-35.3)}{0.311}$$

= 59.98

iii) Sample 3 (Hexane)

Mass of sample = 0.315

Titration of blank solution, V_b = 50ml

Titration of sample, V_s = 35.8 ml

Normality of natrium thiosulphate = 0.1M

IV $$= \frac{12.69 \ x\ N\ x\ (Vb-Vs)}{Mass\ of\ oil\ sample}$$

$$= \frac{12.69 \ x\ 0.1\ x\ (50-35.8)}{0.315}$$

= 57.20

iv) Sample 4 (Petroleum ether)

Mass of sample = 0.315g

Titration of blank solution, V_b = 50ml

Titration of sample, V_s = 30.8 ml

Normality of natrium thiosulphate = 0.1M

IV $$= \frac{12.69 \ x\ N\ x\ (Vb-Vs)}{Mass\ of\ oil\ sample}$$

$$= \frac{12.69 \ x\ 0.1\ x\ (50-30.8)}{0.315}$$

= 77.35

C.2 Iodine value for the oil from decanter cake using soxhlet extraction method.

Since the calculation method for determining iodine value can be determine by repeating three times of valued. Each trial of sample will be calculated using the same equation below as calculated in part A.

$$IV = \frac{12.69 \ x \ N \ x \ (Vb-Vs)}{Mass \ of \ oil \ sample}$$

Table C.1 Iodine value for the oil from decanter cake

Sample	**Trial 1**	**Trial 2**	**Trial 3**	**Average value**
MEK	30.35	66.39	53.75	50.16
Acetone	36.48	50.04	51.86	46.13
Hexane	44.78	73.70	66.89	61.79
P.Ether	58.57	68.14	51.81	59.50

C.2 Iodine value for the oil from spent bleaching clay using solvent decanting method.

Since the calculation method for determining iodine value can be determine by repeating three times of valued. Each trial of sample will be calculated using the same equation below as calculated in part A.

Table C.2 Iodine value for the oil from spent bleaching clay

Sample	**Trial 1**	**Trial 2**	**Trial 3**	**Average value**
MEK	39.01	55.87	49.78	48.22
Acetone	67.57	41.28	51.22	53.35
Hexane	72.38	68.73	65.94	69.02
P.Ether	79.20	57.82	58.54	65.18

C.3 Iodine value for the oil from decanter cake using solvent decanting method.

Since the calculation method for determining iodine value can be determine by repeating three times of valued. Each trial of sample will be calculated using the same equation below as calculated in part A.

Table C.3 Iodine value for the oil from decanter cake

Sample	**Trial 1**	**Trial 2**	**Trial 3**	**Average value**
MEK	33.20	50.14	56.14	46.49
Acetone	68.77	47.20	61.76	59.24
Hexane	51.17	40.82	58.82	50.27
P.Ether	60.66	71.21	61.21	64.36

APPENDIX D

ACID VALUE ANALYSIS

D.1 Iodine value for the oil from spent bleaching clay using soxhlet extraction method.

In order to determine the iodine value of the residue oil, each sample analysis will be repeated two times to obtain the average value. Calculation for the iodine value is shown below.

Trial 1

i) Sample 1 (MEK)

Mass of sample = 1.035 g

Burette reading before titration, B = 0 ml

Burette reading after titration, A = 40.5 ml

Normality of KOH = 0.1 M

Acid value $= \frac{56.1 \ x \ N \ x \ (A-B)}{Mass \ of \ oil \ sample}$

$= \frac{56.1 \ x \ 0.1 \ x \ (40.5-0)}{1.035 \ g}$

= 219.38 mg KOH/g

ii) Sample 2 (Acetone)

Mass of sample = 1.172 g

Burette reading before titration, B = 0 ml

Burette reading after titration, A = 44.8 ml

Normality of KOH = 0.1 M

Acid value $= \frac{56.1 \ x \ N \ x \ (A-B)}{Mass \ of \ oil \ sample}$

$= \frac{56.1 \ x \ 0.1 \ x \ (44.8-0)}{1.172 \ g}$

= 214.56 mg KOH/g

iii) Sample 3 (Hexane)

Mass of sample = 0.971 g

Burette reading before titration, B = 0 ml

Burette reading after titration, A = 43.7 ml

Normality of KOH = 0.1 M

Acid value $= \frac{56.1 \; x \; N \; x \; (A-B)}{Mass \; of \; oil \; sample}$

$= \frac{56.1 \; x \; 0.1 \; x \; (43.7-0)}{0.971 \; g}$

= 252.93 mg KOH/g

iv) Sample 4 (P. Ether)

Mass of sample = 0.982 g

Burette reading before titration, B = 0 ml

Burette reading after titration, A = 41.3 ml

Normality of KOH = 0.1 M

Acid value $= \frac{56.1 \; x \; N \; x \; (A-B)}{Mass \; of \; oil \; sample}$

$= \frac{56.1 \; x \; 0.1 \; x \; (41.3-0)}{0.982 \; g}$

= 236.23 mg KOH/g

Trial 2

i) Sample 1 (MEK)

Mass of sample = 1.371 g

Burette reading before titration, B = 0 ml

Burette reading after titration, A = 54.6 ml

Normality of KOH = 0.1 M

Acid value $= \frac{56.1 \; x \; N \; x \; (A-B)}{Mass \; of \; oil \; sample}$

$= \frac{56.1 \; x \; 0.1 \; x \; (54.6-0)}{1.371 \; g}$

= 223.45 mg KOH/g

ii) Sample 2 (Acetone)

Mass of sample = 1.238 g

Burette reading before titration, B = 0 ml

Burette reading after titration, A = 48.2 ml

Normality of KOH = 0.1 M

Acid value $= \frac{56.1 \, x \, N \, x \, (A-B)}{Mass \; of \; oil \; sample}$

$= \frac{56.1 \, x \, 0.1 \, x \, (48.2-0)}{1.238 \; g}$

= 218.50 mg KOH/g

iii) Sample 3 (Hexane)

Mass of sample = 1.031 g

Burette reading before titration, B = 0 ml

Burette reading after titration, A = 45.3 ml

Normality of KOH = 0.1 M

Acid value $= \frac{56.1 \, x \, N \, x \, (A-B)}{Mass \; of \; oil \; sample}$

$= \frac{56.1 \, x \, 0.1 \, x \, (45.3-0)}{1.031 \; g}$

= 246.43 mg KOH/g

iv) Sample 4 (P. Ether)

Mass of sample = 1.211 g

Burette reading before titration, B = 0 ml

Burette reading after titration, A = 50.9 ml

Normality of KOH = 0.1 M

Acid value $= \frac{56.1 \, x \, N \, x \, (A-B)}{Mass \; of \; oil \; sample}$

$= \frac{56.1 \, x \, 0.1 \, x \, (50.9-0)}{1.211 \; g}$

= 251.32 mg KOH/g

D.2) Iodine value for the oil from decanter cake using soxhlet extraction method.

Since the calculation method for determining iodine value can be determine by repeating two times of valued. Each trial of sample will be calculated using the same equation below as calculated in part A.

Acid value $= \frac{56.1 \, x \, N \, x \, (A-B)}{Mass \; of \; oil \; sample}$

Table D.1: Acid value for the oil from decanter cake

Sample	Trial 1 (mg KOH/g)	Trial 2 (mg KOH/g)	Average value (mg KOH/g)
MEK	251.79	258.42	255.11
Acetone	237.30	243.65	240.48
Hexane	250.58	250.89	250.74
P. Ether	250.38	252.12	251.25

D.2) Iodine value for the oil from spent bleaching clay using solvent decanting extraction method.

Since the calculation method for determining iodine value can be determine by repeating two times of valued. Each trial of sample will be calculated using the same equation below as calculated in part A.

$$\text{Acid value} = \frac{56.1\ x\ N\ x\ (A-B)}{Mass\ of\ oil\ sample}$$

Table D.2 Acid value for the oil from spent bleaching clay

Sample	Trial 1 (mg KOH/g)	Trial 2 (mg KOH/g)	Average value (mg KOH/g)
MEK	218.34	211.32	214.83
Acetone	247.46	231.45	239.46
Hexane	224.56	251.35	237.95
P. Ether	254.43	230.98	242.71

D.3) Iodine value for the oil from decanter cake using solvent decanting extraction method.

Since the calculation method for determining iodine value can be determine by repeating two times of valued. Each trial of sample will be calculated using the same equation below as calculated in part A.

$$\text{Acid value} \quad = \frac{56.1\ x\ N\ x\ (A-B)}{Mass\ of\ oil\ sample}$$

Table D.3 Acid value for the oil from decanter cake

Sample	**Trial 1 (mg KOH/g)**	**Trial 2 (mg KOH/g)**	**Average value (mg KOH/g)**
MEK	245.78	241.65	243.72
Acetone	223.76	222.67	223.22
Hexane	213.77	219.76	216.77
P. Ether	238.09	248.09	243.09