MICROEMULSION-BASED PEANUT OIL EXTRACTION USING SURFACTANT SOLUTION

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This project is submitted in partial fulfillment of the requirements for the degree of Bachelor of Science with Honours (Resource Chemistry)

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DECLARATION

I hereby declare that no portion of the work referred to in this thesis has been submitted in support of an application for another degree or qualification to this or any other university or institution of higher learning.

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Nur Syafiqah Amira Binti Mohd Zulkefli

24630

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<td>APES</td>
<td>alkyl-propoxylate-ethoxylate-surfactant</td>
</tr>
<tr>
<td>APS</td>
<td>alkyl-propoxylate-surfactant</td>
</tr>
<tr>
<td>Brij-30</td>
<td>polyoxylene (4) lauryl ether</td>
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<tr>
<td>CMC</td>
<td>Critical Micelle Concentration</td>
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<tr>
<td>cm⁻¹</td>
<td>per centimeter</td>
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<tr>
<td>FTIR</td>
<td>Fourier Transform Infrared Spectroscopy</td>
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<td>g</td>
<td>gram</td>
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<td>GE-460</td>
<td>glycolic acid ethoxylate lauryl ethers</td>
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<tr>
<td>HLB</td>
<td>Hydrophile-Lipophile Balance</td>
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<tr>
<td>IFT</td>
<td>Interfacial Tension</td>
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<tr>
<td>mg/L</td>
<td>Milligram per liter</td>
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<td>Milliliter</td>
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<td>mm</td>
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<td>nm</td>
<td>Nanometer</td>
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<tr>
<td>Rpm</td>
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<td>Surfactant</td>
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Microemulsion-Based Peanut Oil Extraction Using Surfactant Solution
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Resource Chemistry Programme
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ABSTRACT
Nowadays, vegetable oil is an alternative for the substitution of biodiesel. Vegetable oil extraction is more favourable by using surfactant compared to hexane which is hazardous to human. In this study, extraction efficiency and physical appearance of oil extracted by Brij-30, GE-460 and their mixture were evaluated at optimum conditions. Microemulsion-based extraction was conducted by mixing these surfactants with ground peanut samples (0.2-0.5mm) and agitated at 150 rpm. This enables the oil to be collected because it’s liberated from the samples as a separate phase from the aqueous phase. Contact time, concentration of surfactant, weight of samples and types of surfactant on oil yield were tested to identify optimum extraction conditions. After optimum conditions were achieved, solvent based extraction using hexane was done by soxhlet method for comparison. The oil collected from both extractions was compared based on their extraction efficiencies, physical appearance of oil and Fourier Transform Infrared Spectroscopy (FTIR) characterization. The appearance of oil extracted using surfactant showed lighter yellow colour and did not produced any odour compared to hexane extraction. Optimum extraction of 87.15% was achieved at weight of sample of 1g, 30 minutes extraction at 150 shakes/min, and 0.015M surfactant concentration while the extraction efficiency for mixed surfactant of GE-60 and Brij-30 was 92.93% in the ratio of Brij-30 to GE-460 of 3:1. Percentage of extraction using solvent is 91.37% which is higher compared to microemulsion-based extraction. The FTIR characterization on both methods shows not much difference due to the same source of origin. The formation of oil was proven by ester formation that was detected at 1743cm⁻¹.

Keywords: Soxhlet, microemulsion, Brij-30 surfactant, peanut

ABSTRAK
Minyak sayuran merupakan satu alternatif lain bagi menggantikan biodiesel pada masa kini. Pengekstrakan minyak sayuran menggunakan surfaktan merupakan satu kaedah yang lebih baik berbanding pelarut heksana yang berbahaya kepada manusia. Dalam kajian ini, kadar peratusan dan keadaan fizikal minyak yang telah diekstrak menggunakan Brij-30, GE-460 and surfaktan campuran dinilai pada keadaan optimum. Pengekstrakan berasaskan mikroemulsi dilakukan dengan mencampurkan surfaktan dengan sampel kacang tanah yang telah dikisar (0.2-0.5mm) dan digoncangkan pada kelajuan 150 rpm. Ini membolehkan minyak terbebas daripada sampel sebagai satu fasa berasingan dari fasa akues. Masa sentuhan, kepekatan surfaktan, berat sampel dan jenis-jenis surfaktan untuk mengekstrak minyak telah dikaji untuk mendapatkan keadaan pengekstrakan yang optimum. Selepas kadar optimum telah dicapai, pengekstrakan menggunakan heksana telah dibuat melalui keadaan soxhlet sebagai perbandingan. Minyak yang terkumpul daripada kedua-dua kaedah pengekstrakan telah dibandingkan berdasarkan peratusan minyak terhasil, sifat fizikal minyak dan keadaan spektroskopi inframerah (FTIR). Keadaan fizikal minyak yang diekstrak menggunakan surfaktan menunjukkan warna kuning yang lebih cerah dan tidak menghasilkan sebarang bau berbanding pengekstrakan menggunakan heksana. Pengekstrakan optimum 87.15% telah dicapai apabila berat sampel 1g, 30 minit masa pengekstrakan pada 150 guncangan / min dan kepekatan surfaktan 0.015M manakala peratusan pengekstrakan untuk surfaktan campuran oleh GE-60 and Brij 30 ialah 92.93% pada kadar nisbah 3:1 Brij-30 dan GE-460. Peratusan pengekstrakan menggunakan pelarut lebih tinggi iaitu 91.37% berbanding dengan pengekstrakan berasaskan mikroemulsi. Percirian FTIR menggantikan kedua-dua kaedah tidak menunjukkan perbezaan yang ketara kerana datang daripada sumber yang sama. Pembentukan minyak yang terhasil boleh dibuktikan melalui kumpulan sebatian asid dan alkohol yang dikesan pada 1743cm⁻¹.

Kata kunci: Soxhlet, mikroemulsi, Brij 30 bahan permukaan, kacang tanah
1.0 Introduction

The occurrence of death, birth and migration happened everyday in our country. According to *Malaysia Demographics Profile* (2011), the total number of people in our country is 28,728,607. Since the human population increasing from year to year, the demand for energy also increase gradually. However, these demands affect the world’s renewable sources such as fossil fuels in producing energy supply to human beings. The lack of energy supply and environmental restrictions leads to search for alternate methods for biofuel production. Vegetable oils have been considered as renewable fuels which is a great substitute for biodiesel fuel (Nguyen et al., 2010). Vegetable oil is extracted from plants and can be produced from oil-bearing crops such as soybean (*Glycine max*), corn (*Zea mays*), peanut (*Arachis hypogaea*) and palm kernel (Naksuk et al., 2009).

Vegetable oil can be extracted through several methods and processes. Examples of method that are used for vegetable oil extraction are solvent-based extraction, microemulsion-based extraction, aqueous-based extraction, mechanical extraction and supercritical fluids. In this project, we focused on microemulsion-based extraction. This method is considered as a clean technology since the surfactants used are non-toxic and biodegradable. The interfacial tension between aqueous extracting system and oil vegetable seeds can be lowered by surfactants through micelle formation (Naksuk et al., 2009). Surfactants are surface-active substances. Holmberg and Jönsson (2003) reported that surfactant have a tendency to absorb at any surfaces and interfaces. There are four types of surfactant which are anionic, cationic, nonionic and zwitterionic surfactant. Surfactants have the most versatile functions such as emulsification, detergency, wetting and spreading, solubilizing, foaming and defoaming, lubricity, biocidal, anti-static and corrosion inhibition (Hargreaves and Hargreaves, 2003).
Due to its unique structure, surfactants provide smoother transition between hydrophilic and hydrophobic regions of the interface. Thus, this will provide suitable environment for solubilizing hydrophilic and hydrophobic molecules (Kadioglu et al., 2010).

In this project, the effectiveness in extracting oil from plants using single surfactant and mixed surfactant will be investigated. The characteristics of each surfactant will be studied. A few batch studies will be carried out such as the concentration of surfactants, types of surfactant, weight of samples and contact time in order to get the highest percentage of oil extraction. A nonionic surfactant which is Brij-30, anionic surfactant GE-460 and their mixture are going to be used for extraction process.

Although vegetable oil can be extracted by using solvent-based extraction method, it is not a major and applicable method nowadays since it is hazardous to human being as well as the environment. The volatile compound n-hexane is used in solvent-based extraction to compare its extraction yield and appearance of oil with microemulsion-based extraction under optimum conditions.

The significance of this study was to use a more environmental friendly approach in extracting vegetable oil where biodegradable surfactants were used. In fact, nonionic surfactant Brij-30 was chosen to identify its optimization and its efficiency for vegetable oil extraction since there was no research done using this surfactant.
Thus, the objectives of this study were:

1. To determine the optimum conditions to extract oil from peanut.
2. To identify effect of type of surfactants on yield of oil extracted.
3. To compare the yield of extraction using different surfactants.
4. To compare the quality of oil extracted using microemulsion-based and solvent-based methods.
2.0 Literature Review

2.1 Vegetable Oil

Vegetable oil processes involving extraction and processing of oils and fats from vegetable sources. This oil was extracted from various types of fruits, seeds and nuts. Black (2007) reported that vegetable oils are used widely in food industry. Vegetable oil can be manufactured from the seeds of vegetables which have yellow flowers, much resembling when in blossom. Apart from that, it is also been used in non-food applications such as biodiesel, composites, lubricants, foams, coatings, surfactants and oleo chemicals. In this project, oil was extracted from peanut. Fatty acids composition in peanut is about 80-85% unsaturated and 17-20% saturated fatty acids which are responsible for stabilization in cooking or deep frying (Erickson, 1990). Thus, peanut oil can be saponified and treated chemically for the manufacture of soaps and detergents. It also can be used in cosmetic products.

2.2 Microemulsion

Microemulsion is a thermodynamically stable homogeneous mixture of oil and water that were stabilized by surfactants and co-surfactants. According to Torchilin (2006), microemulsion was denoted as a transparent dispersion of oil and water which is thermodynamically stable, stabilized by an interfacial film of amphiphilic molecules. Microemulsion is an isotropic solution of water and oil which stabilized by an appropriate surfactant or linker molecules. Furthermore, microemulsion can be exploited in triglyceride application since low interfacial tension (IFT) can be achieved. This is because it is an essential characteristic in oil and grease detergency as well as water-based extraction of triglycerides from oil seeds or algae (Phan et al., 2011). There are three types of microemulsion which are, oil-in-water, water-in-oil and middle phase microemulsion.
Microemulsion also exhibit unique properties such as producing ultralow interfacial tension and ultrahigh solubilization (Do et al., 2008). Apart from that, microemulsion also related with the critical micelle concentration of surfactants. Micelle formation can be formed if there is an equilibrium involving clusters of surfactants which contain 50-150 molecules above CMC. Thus, an appropriate concentration of surfactant should be applied to stabilize the microemulsion solution.

2.3 Surfactants

Surfactant is a substance which is used relatively in cleaning solution which are able to remove any dirt from substances, textiles and other solids when dissolves in water (Perkins, 1998). The molecular structure of surfactant consists of hydrophobic group which has very little attraction for the solvent (solvent repellent) and hydrophilic group which is strongly attracted to solvent (solvent loving) in aqueous solution (Yildirimtabil, 2006). The hydrophobic tail will repels water and attracted to oil and grease in dirt while the hydrophilic head is more attracted to water molecule.

Surfactant is a wetting agent material that can greatly reduce the surface tension of water which enables the cleaning solution to wet a surface more quickly. The low surface tension of water make the dirt and grease from dirty clothes, dishes and other surface can be lifted easily and preventing them from being suspended in dirty water (Perkins, 1998). The hydrophobic head of surfactant can form hydrogen bonds with water and pulls the stain away from fabric that removes the stain particles surrounding it. The hydrophilic tail of surfactant will emulsify the oily soils by keeping them dispersed. Surfactants are widely used in aqueous cleaners to provide detergency, emulsification, antistatic agent, wetting agent and many more (Salager, 2002).
The four major classifications of surfactant are described based on the type of dirt and fabric which they work the best and how they can cope with water hardness.

2.3.1 Anionic Surfactant

Anionic surfactant is a substance that is water soluble and negatively charge in aqueous solution (Oliver, 2004). Important types of anionic surfactants are carboxylates, sulfonates, sulfates and phosphates where the sulfonates types make them useful in dry cleaning because they are soluble in organic solvents (Perkins, 1998). The anionic surfactant hydrophobic chain should be in linear alkyl group with a chain length of 12-16 carbon atoms in achieving optimum detergency which make it more effective and degradable than the branched ones (Tharwat et al., 2005). Anionic surfactants account for about 50 % of the world production and the most commonly used anionic surfactants are alkylbenzene sulfonates, lauryl sulfates, lignosulfonates and soaps (Salager, 2002).

2.3.1.1 Glycolic acid ethoxylate lauryl ethers (GE-460)

Glycolic acid ethoxylate lauryl ethers bearing the commercial name GE-460 is an anionic ethoxylated alcohol. The average molecular weight of GE-460 is 460. The molecular formula is CH₃(CH₂)₁₁₋₁₃O(CH₂CH₂O)₄₋₅CH₂COOH. The hydrophile-lipohile balance (HLB) is 10.4. The HLB differentiate the contribution of their molecular structures to the solubilization process. The CMC value is 130mg/L and the micellar size is 7.6nm (Tau et al., 2005).

2.3.2 Cationic Surfactant

Cationic surfactants have a positively charged ion and hydrophilic in nature which have the ability to adhere to and modify solid surfaces (Perkins, 1998). Cationic surfactant can be
divided into alkyl amine, ethoxylated amine, alkyl imidazolines and quaternaries (Fainerman et al., 2001). They also reported that cationic surfactant does not provide effective cleaning and mainly used in the application concern with germicidal, hydrophobisation and antistatic effect. This type of surfactant are widely used in textile, as fabric softeners, fixatives for anionic dyes and dyeing rate retarders for cationic dyes (Perkins, 1998). Examples of cationic surfactant are esterquat and mono alkyl quaternary system.

2.3.3 Nonionic Surfactant

Nonionic means that are no ionic constituents are present and ionically inert. Nonionic surfactants are widely used in textiles and the most common type of surfactant is polyoxyethylenated linear and polyoxyethylenated alkylphenols (Perkins, 1998). Besides that, these surfactants have a polyhydroxyl unit as the polar group and hydrophilic group which is non-dissociable type which make the surfactant cannot ionize in aqueous solution (Salager, 2002). Nonionic surfactants are described as synthetic surfactant and the example of this surfactant is polyglycerol esters.

2.3.3.1 Polyoxyethylene (4) lauryl ether (Brij-30)

Brij-30 is a nonionic surfactant which also known as polyoxyethylene (4) lauryl ether. The molecular formula for Brij-30 is $C_{12}H_{25}(OCH_2CH_2)_4OH$. The HLB for Brij-30 is 9.7. The critical micelle concentration (CMC) for Brij-30 is in the range of 7 – 15.00 mgL$^{-1}$ (Yeh et al., 1998).

2.3.4 Zwitterionic Surfactant

Amphoteric or zwitterionic surfactants develop a negative or positive charge depending on whether the solution is alkaline or acidic. Amphoteric surfactant can exhibit anionic,
cationic and non-ionic dissociation in solution depending on the pH and acidity of water (Salager, 2002). This type of surfactant is suitable for the usage in personal care and household cleaning products. In acid pH solutions, the molecule behaves like a cationic surfactant, whereas in alkaline pH solutions they behave like anionic surfactant (Tharwat et al., 2005). They are chemically stable both in acids and alkalis. Furthermore, amphoteric surfactants are more compatible with all other classes of surfactants because it is highly soluble and effective although in the presence of high concentrations of electrolytes, acids and alkalis. Although this type of surfactant can form mixed micelles in aqueous solutions and soluble in water, the solubility character shows minimum isoelectric point (Tharwat et al., 2005). Imidazolines and betaines are the major amphoteric surfactant.

2.3.5 Solubilisation of oil with surfactants

Surfactants are capable in increasing aqueous solubility of a hydrophobic compound. The hydrophobic cores of micelles offer good affinity for hydrophobic solutes to stay in micelles as well as increase the aqueous solubility by using surfactant (Tau et al., 2005). In a study by Tau et al. (2005), five ethoxylated surfactants with small difference in molecular structure and HLB were chosen to prove the solubilisation process of free fatty acids. The results shown that mixed surfactant of Tergitol 15-S-5 and Tergitol 15-S-7 which has the lowest HLB values was the most hydrophobic surfactant because they can solubilize 50-100% palmitic acid at. Schulz and Moya (1998) reported that molecular structure and HLB number of a surfactant is essential in determining the solubilisation of aqueous solution. Surfactants with low HLB values can provide a more hydrophobic environment characteristics and bigger micellar core volumes compared to surfactant with high HLB. Thus, it is important to choose the appropriate surfactant in extracting vegetable oil.
2.4 Oil Extractions

According to Volkov (2001), microemulsion-based extractions of biomolecules consist of biomolecule containing aqueous solution in contact with a surfactant containing lipophilic phase. Several studies have been conducted to extract vegetable oil with different techniques. Do and Sabatini (2010) reported that there were different processing parameters on the vegetable oil extraction efficiency such as pH, surfactant concentration, extraction time, shaking speed, solid-to-liquid ratio and salinity level. From the evaluation of their study, aqueous extended-surfactant based method produced better crude oil quality in terms of free fatty acid content. The peanut and canola seeds produced 93-95% extraction efficiency of oil at optimum conditions where seed to liquid ratio at 1-5, as well as the extraction process occurred for 30 minutes at 150 shakes/minutes. The oil compound also contains free fatty acid and the alkyl-ethoxylate-sulfate is the best surfactant in extracting the oil.

The microemulsion-based extraction that was conducted by Naksuk et al. (2009) from palm kernel oil shows that different parameters could affect the amount of oil extracted. Firstly, the effect of grain size in which the smaller size of seed produced higher extraction efficiency compared to large size of vegetable seeds. There are three standard sizes of the vegetable oil seed which are coarse size (larger than 0.425mm), fine size (between 0.212 and 0.425mm) and very fine size (less than 0.212mm). They claimed that the largest grain size showed the lowest efficiency while the most appropriate grain size is between 0.212 to 0.425mm. The smaller size of seeds provides larger surface area that enhanced the surfactant monomer interaction with the surface and thus releasing oil from the seeds. Secondly, the contact time also affect the oil extraction. The extraction that occurred for 30 minutes produced the maximum efficiency in extracting oil while extraction after 30 minutes decreased the extraction efficiency. Contact time at 30 minutes considered as the
optimum time in extracting oil and decreases the interfacial tension. Thirdly is the effect of palm kernel load. They also found that the optimum weight for extraction efficiency is 1.0g seeds loading. When the mass of palm kernel load increases the extraction efficiency decreases due to less penetration of surfactant monomer into the kernel. Furthermore, Naksuk et al. (2009) also proved that mixed-surfactant solution produced extraction efficiency at range 93.99 – 94.13% by mixing 3 wt% Comperlan KD and 0.1 wt% Alfoilral 145-5PO at optimum grind kernel size.

Do and Sabatini (2010) investigated the efficiencies of canola and peanut oil extraction with surfactant. They found that the extraction efficiency increases as the concentration surfactant increases below the critical microemulsion concentration. The fraction of oil extracted did not change with increasing surfactant concentration above the critical microemulsion concentration. In addition, Do and Sabatini (2010) found that the best concentration for anionic surfactant is in range of 0.15 wt% - 0.35 wt% below the CMC values since the surfactant monomers aggregate to form micelles at above CMC values. At 0.15 wt% surfactant concentration, extended-surfactant solutions are able to produce ultralow IFT between aqueous extraction and vegetable oil phases. Based on their study, when water was used as the extraction medium, it produced the lowest extraction efficiency of 40%. Anionic surfactant; alkyl-propoxylate-surfactant (APS) produced higher extraction efficiency of 65% while mixture of water and APS surfactant produced stable-emulsion like phases which are not desirable in the extraction process. Both anionic surfactants; alkyl-propoxylate-ethoxylate-sulfactant (APES) and APS yielded very high peanut oil extraction efficiencies of 92 – 95%.
Next, vegetable oil can be extracted using pressing. However, it was not an optional method since it did not produce a higher yield as the solvent-based extraction. In addition to increase the oil yield, usually extraction using pressing will be followed by solvent method (Alesovski et al., 1998). Vegetable oil also can be extracted by solvent-based extraction method using hexane. This method has several advantages since it produces higher yield and less turbid oil than mechanical extraction as well as low operating cost compared to supercritical fluid extraction (Maria et al., 2008).
3.0 Materials and Methods

3.1 Materials

The nonionic surfactant Brij-30 and anionic surfactant GE-460 are from Sigma and used as received. The peanut was purchased at the local market. All the solutions were prepared with deionized water.

3.2 Methods

3.2.1 Sample Preparation

The peanut was dried at 100°C in an oven for a duration ranging from 30 minutes to 24 hours to remove moisture. After drying, the peanut samples were weighed and recorded. The samples were dried again in oven and the weights were recorded. The process of drying and weighing the peanuts samples were repeated a few times until constant weight was obtained. After the constant weight was obtained, the peanut samples were ground using mortar and pestle and sieved with 0.2-0.5mm mesh size metal sieve. The ground samples were kept in desiccators to prevent the humidity absorption from surroundings and to maintain the low humidity of atmosphere. The samples were divided into different weight ranging from 0.03g to 5g.

3.3 Oil Extraction

3.3.1 Batch Studies

The optimum conditions in extracting oil were identified based on the batch studies conducted. The optimum conditions for oil extraction were determined by a few parameters which are contact time, concentration of surfactant, weight of samples and types of surfactant.
3.3.1.1 Contact time

The contact time indirectly indicates the rate of reactions on detachment of oil from seeds. The contact time was varied from 15 minutes to 180 minutes. The mixture of surfactant solution and peanut was agitated on a rotary shaker at 150 rpm. The oil collected was transferred into petri dish and dried with water bath to eliminate water. The oil extracted was placed in desiccator to prevent humidity absorption from surroundings. The extraction efficiency at each time was calculated using equation 3.1.

3.3.1.2 Concentration of surfactants

Surfactant concentration was varied from below till above the CMC to study the effect of surfactant concentration on oil seed extraction. The concentrations of surfactant used were 0.005-0.030 M. Percentage of extraction efficiency was calculated using equation 3.1.

3.3.1.3 Weight of samples

The ground peanut samples were prepared with particle size of 0.2-0.5mm. The size was fixed but the weight was varied in the range of 0.03 – 5.0g. The extraction efficiency of the oil collected was calculated based on equation 3.1.

3.3.1.4 Types of surfactant

Effect of nonionic surfactant Brij-30, anionic surfactant GE-460 and their mixture on oil extraction were also studied. The volume for single surfactant system was fixed at 20 mL. As for the mixed surfactant, the volume used was in the ratio of 3:1 (Brij-30 : GE-460). The extraction efficiencies of the oil collected were calculated based on equation 3.1.
3.3.2 Extraction Using Hexane

Hexane extraction is conducted by using Soxhlet extraction method. The ground samples were weighed according to optimum weight from microemulsion-based extraction and were inserted into thimble as shown in Figure 3.1 (The Extraction Procedure, n.d.). Then, the round bottom flask was filled with hexane and heated to evaporate the hexane. The temperature was controlled in order to get an evenly drops of condensed hexane and the samples were left for eight hours. The rotary evaporator was used to obtain solvent free oil. The extraction experiments were conducted in triplicates.

Figure 3.1: Soxhlet Apparatus (The Extraction Procedure, n.d.)