SEPARATION OF OIL AND WATER BY USING COCONUT COIR

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I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Chemical Engineering (Chemical).

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Special Dedication to my supervisor, my family members,
my friends, my fellow colleague and all faculty members
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ABSTRACT

The aims for this study are to examine the emulsion stability and to evaluate the effectiveness of using coconut coir in the separation of oil and water (emulsion). Crude oil and distilled water were used as the raw materials. To complete the objective, there are three main (3) parts of experiment need to be done. The first part was the pre-treatment of the coconut coir. In this part, the required particle sizes of the coir which varied between 630 µm to 2 mm were determined and also had been modified, weighing and packaging. While in the second part the formation and stabilization of the oil-in-water (O/W) emulsion was examined. The O/W ratios were varied from 50-75:50-25 and during the stability part the emulsion was verified by visually observes on separation layer for each of the samples of emulsion. As for the emulsifier, Tween 20 at different concentration 1%, 3% and 5% of total volume was used in formation of O/W emulsion. Last but not least, the last part studied the separation of oil and water by using coconut coir. The optimum condition of O/W emulsion is discovered by the correlation between the emulsifier concentrations, and oil-water ratio. Here, the effectiveness of prepared coconut coir is analyzed in order to adsorbed the raw crude oil, and water by considering the effect of size particles and the modification of the coconut coir. Then, the amount of oil and water being adsorbed and recover from the coconut coir are weighed to see the efficiency of the coconut coir. The most stable of O/W emulsion was found at 5% of emulsifier concentration at 75-25% of oil-water ratio. The adsorption of oil was better at 630µm particle size of unmodified coir. Therefore, the effectiveness of the coconut coir in order to separate the most stable emulsion into oil and water would be required.

Key words: O/W emulsion stabilization, Coconut coir, Tween 20, Separation

ABSTRAK

Kajian ini telah diadakan bagi mengkaji pembentukan kestabilan emulsi dan menilai tahap keberkesanan penggunaan sabut kelapa dalam proses pengasingan minyak dan air (emulsi). Minyak mentah dan air suling digunakan dalam menjayakan kajian ini. Kajian ini terbahagi kepada tiga (3) bahagian yang utama. Bahagian yang pertama adalah mengenai pra-rawatan yang dilakukan terhadap sabut kelapa. Dalam bahagian ini, saiz sabut kelapa iaitu 630 µm dan 2 mm ditentukan, ditimbang, dan diubahsuai. Manakala, dalam bahagian yang kedua (2) pula pembentukan dan kestabilan emulsi minyak didalam air dijalankan. Nisbah minyak-air dilakukan dalam mengikut nisbah yang berbeza-beza daripada 50-75:50-25 dan dalam kajian mengenai kestabilan emulsi, ianya dijalankan dengan memerhatikan lapisan pemisahan air pada setiap sampel. Bagi ejen pengemulsi iaitu *Tween 20* digunakan pada kepekatan yang berbeza iaitu pada 1%, 3% dan 5% dalam proses penyediaan emulsi ini. Pada bahagian terakhir (3), sabut kelapa yang diubah suai dan tidak diubahsuai digunakan bagi menganalisis kebolehan sabut kelapa untuk menjerap minyak mentah. Didapati bahawa keadaan optima bagi pembentukan dan kestabilan emulsi telah ditemui dengan mengaitkan kepekatan bahan pengemulsi dengan nisbah miyak-air. Manakala, keberkesanan abut kelapa dianalisis dalam usaha untuk mejerap minyak mentah dan air dengan mengambil kira aiz, dan pengubahsuaian yang dilakukan kepada abut kelapa. Keputusan bagi emulsi yang paling stabil adalah apabila kepekatan pengemulsi ialah 5 % dan nisbah minyak-air pada 75-25%. Penjerapan minyak yang optima didapati lebih berkesan pada saiz 630µm bagi sabut kelapa yang tidak mengalami penubahsuaian.Maka, keberkesanan sabut kelapa dalam memisahkan emulsi yang paling stabil kepada minyak dan air adalah berkesan.

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LIST OF ABBREVIATIONS

O/W	Oil-in-Water emulsion
W/O	Water-in-Oil emulsion
µm	Micrometer
mm	Millimetre
rpm	Revolution per minutes

CHAPTER 1

INTRODUCTION

1.1 Background of Research Study

Bradley et al. (2006) was investigate that coconut palm tree can also be known as *Cocos Nucifera* trees which grow abundantly along the coast line of countries within 15° of the equator. They are grown well in saline soil, sandy and tropical climate. Besides, a healthy coconut tree will produce about 120 watermelon-sized husks per year, each with a coconut embedded inside. Besides, based on the project that has been done by the School of Education, University of Queensland Australia (2010), it stated that probably coconuts grew originally in South Asia, Malaysia and Polynesia.

Moreover, the coconut provides a nutritious source of juice, meat, oil, and also milk that has fed and nourished populations around the world for generations. On the other hand, many populace of island think coconut is a staple in the diet and provides the majority of the food eaten. Almost one third of the world's population depends on coconut to some degree for their economy and their food. Amongst these cultures the coconut has a long and respected history (Coconut research centre, 2010).

Coconut is highly nutritious and rich in vitamins, minerals, and fibre. It is classified as a "functional food" because it gives many health benefits beyond its nutritional content. Coconut oil is of unusual interest because it possesses healing properties far beyond that of any other dietary oil and is extensively used in traditional medicine among Asian and Pacific populations. Pacific Islanders consider coconut oil to be the cure for all illness. The coconut palm is so well valued by them as both a source of medicine and food that it is called "The Tree of Life." Only recently has current medical science unlocked the secrets of coconut's amazing healing powers (Coconut Research Centre, 2010).

1.2 Problem Statement

In Malaysia, the coconut tree is a plant that has many uses in all aspects of life. All parts such as coconut fruits, stems, and leaves have its own uses. After use or taken its content, the left part is called as waste materials. For examples, coconut husk, coir, shell and others.

The effort to utilize these waste materials in Malaysia is still low. The disposal of these materials is expensive and may lead to environmental problem (Zuim et al., 2011). Besides, many of these wastes has been thrown away and burnt. This phenomenon will give bad effect to the environment.

So, in order to reduce environmental problems, maximum recycling of the wastes should be established. This can directly increase the profitability of the agriculture industry and thus can help to reduce the environmental pollution. In addition, coconut coir is one of the main waste materials that can pollute the environment. Therefore, new alternative need to be discover in order to reduce the environmental problems.

Recently, there is an alternative application for coir fibre to be used in composite materials had been studied (Ezekiel et al., 2010; Tran et al., 2011). Before this the coir fibre from the husk is used for the production of non-food products such as yarns and ropes, mats, padding of mattresses and brushes This had been done in Southern India and Sri Lanka which already have a long standing tradition and is the major production areas of a wide range of diversified coir products (Rodríguez et al., 2011).

1.3 Objectives of Study

At the end of this study, it is necessary

i. To examine the emulsion stability using emulsifier in different concentration at different volume ratio of oil-in-water emulsion

ii. To evaluate the effectiveness usage of coconut coir in separation of oil and water

1.4 Scopes of Study

There are important task to be carried out in order to achieve the objectives of this study. The important scopes that have been identified for this research are;

- Determine the optimum condition for oil in water emulsion stability by;

    i. Test the effectiveness of emulsifier concentration on emulsions stability

        a. 1 %
        b. 3 %
        c. 5%

    ii. Investigate the efficiency of different volume ratio of oil in water emulsion in stabilizing emulsions

        a. 50-50%
        b. 65-35%
        c. 75-25%

- Determine the optimum condition for coconut coir for separation of oil and water by;

i. Analyse the efficiency of modification coconut coir in order to adsorb oil

   a. Modified with sulphuric acid

   b. Unmodified

ii. Determine the volume adsorbed and recover by the different particle size of coconut coir

   a. 2 mm

   b. 630 µm

1.5 Rationale and Significance

i. Coconut coir is the cheapest raw material that can be use.

ii. Coconut coir is easy to get and environmental friendly.

iii. It will lead to maximise the uses of coconut coir widely in Malaysia.

iv. It will boost up the coconut supplier or industries earnings as well as to increase the Malaysia's economy.

v. The important thing is reduce the contaminations or in other word save the earth.

vi. Lastly, able to increase the living standards and income of the local workers in Malaysia.

CHAPTER 2

LITERATURE REVIEW

2.1 Coconut

The scientific name for the coconut tree is *Cocos nucifera L.*(Abdul Khalil et al., 2006; Adeyi & Oladayo, 2010; Asasutjarit et al., 2007; Bradley et al., 2006; Defoirdt et al., 2010; Koschek et al., 2007; Rodríguez et al., 2011; Tran et al., 2011;Young, 1983).

Researchers concede that the coconuts are grown extensively in coastal areas of tropical countries (Bradley et al., 2006; van Dam et al., 2004), for example with consider to the implantation of coconut trees in Sri Lanka (Rodríguez et al., 2011; van Dam et al., 2004), Malaysia (Abdul Khalil et al, 2006), Philippine (van Dam et al., 2006), Vietnam (Tran et al., 2011), Brazil (Koschek et al., 2007; Zuim et al.,2011) and others.

In Malaysia, one of the most important crops is coconut palm (*Cocosnucifera L.*). Besides, statistics showed that total production of coconut palm based on states are increased from 512,699 in 2006 to 555,120 metric ton in 2008 (MOA,2009).

There are varieties types of coconut in Malaysia which includes Malayan Tall, MAWA, MATAG, Malayan short and Wangi. Perak, Johor, Selangor, Sarawak and Sabah are the top producer of coconut (MOA, 2009).

2.1.1 Coconut coir

The coir fibre is obtained from coconut husk and because of its high content of lignin; it is also one of the hardest natural fibres. Similarly, Praveen et al. (2008) acknowledge that coir is hard and though organic fibre has been extracted from the husk of the coconut.

In addition to that, van Dam et al. (2006) also report that fibre were manually extracted from the husk and separated from the pith. Figure 2.1 shows the parts inside of the coconut. It consists of 4 parts which are husk, skin, shell and copra.

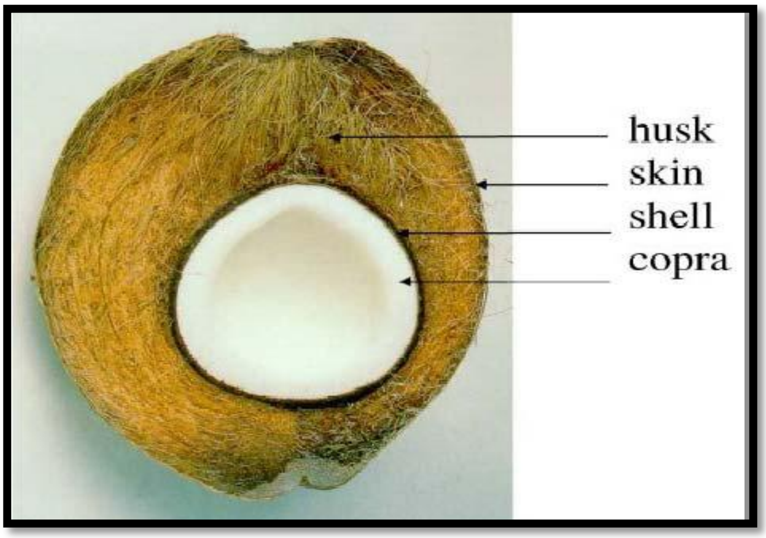


Figure 2.1: Coconut and husk

Source: (Bradley et al., 2006)

2.1.2 Chemical composition in coconut coir

In order to proceed for this study, one needs to know the chemical composition of the fibre. From van Dam et al. (2006) researched its state that chemical compositions of coir fibre of different varieties are similar. Table 2.1 shows that each chemical composition (lignin, hemi-cellulose, alpha cellulose, and extractive) has the similar amount in percent for each varieties of coconut. Besides, many researchers agree that coconut contain high content of lignin (Abdul Khalil et al., 2006; Adeyi & Oladayo, 2010).

Table 2.1: Chemical composition of coir fibre and pith of different coconut varieties

variety	hot water extractive (%)	alpha cellulose (%)	hemi-cellulose (%)	uronic acid (%)	acid insoluble lignin(%)	acid soluble lignin (%)	total lignin (%)	ash (%)
CATD Fiber	2.3 (0.1)	35.1 (0.3)	16.8 (0.3)	4.6 (1)	32.7 (1.1)	1.0(0.3)	33.6 (0.9)	2.6 (0.4)
CATD Pith	9.8	20.8	15.7	6.6	42.4	1	43.5	6.1
AGAT Fiber	2.9 (0.0)	35.1 (0.8)	17.4 (0.1)	3.8 (1)	34.6 (0.1)	1.4 (0.1)	36 (0.1)	3 (0.3)
AGAT Pith	15	21.1	13.4	5.5	44.6	1.5	46.1	7.9
TAGT Fiber	2 (0.1)	34.9 (0.5)	17.6 (0.8)	4.9 (0)	34.5 (0.2)	1.3(0.1)	35.8 (0.1)	1.6 (0.2)
TAGT Pith	11.9	22.7	16.6	5.7	43.7	1.3	44.9	3.5
LAGT Fiber	2.1 (0.2)	35.2 (0.3)	17.6 (0.5)	4.7 (2)	34.6 (0.5)	1.3(0.1)	35.7 (0.8)	2.7 (0.3)
LAGT Pith	12.6	20	15.5	5.5	45.7	1.2	46.9	7.2
RIT Fiber	2 (0.4)	33.3 (0.2)	18 (0.3)	4.8 (2)	35.4 (0.3)	1.3(0.1)	36.6 (0.1)	2.3 (0)
RIT Pith	12.6	20.5	15	5.3	45.9	1.4	47.3	5.5
BAYT Fiber	1.8 (0.5)	35.5	17.3 (0.5)	3.5 (2)	34.9 (0.6)	1.2 (0.1)	35.9 (0.8)	2.5 (0.1)
BAYT Pith	10.9	23.1	12.3	66	44.2	1	45.2	6.6

Source: (van Dam et al., 2006)

2.1.3 Physical properties

2.1.3.1 Colour

There are too many varieties color of coconut which can be from pale yellow to dark brown. Neutrally, its depends on the varieties of coconuts, maturity, time lapsing between husking and retting, quality of water used for retting and the duration of retting (Jayasekara et al., 2010)

2.1.3.2 Impurities

Impurities' degree in mattress and mixed coir is based on the predetermined contract between the buyer and the supplier. Other than coir, including husk and this considered as an impurity in coir (Jayasekara et al., 2010).

2.1.3.3 Texture

It is solid, resilient, spongy and elastic. Resilience showed the amount of energy kept up in a body when one unit volume is stressed or compressed. Furthermore, after releasing the compression force, it is measured as a percentage deviation from the original volume (Jayasekara et al., 2010).

2.2 Emulsification

It is a process where two immiscible liquid that are normally mixed, one liquid which are discontinuous, dispersed or internal phase becoming dispersed in form of small droplets or globule in the other liquid which are continuous, dispersing or external phase (Abdurahman et al., 2010; SitiNurul Huda, 2009)

2.2.1 Type of Emulsion

Referring to Siti Nurul Huda (2009) research, in most emulsions, one of the liquids is in aqueous while the other is hydrocarbon and usually comes in form of oil. There are two types of emulsion that can distinguish by determined which kind of liquid form the continuous phase (external phase). This can be shown in Figure 2.2 below, they are;

· Water-in-oil (W/O) for water droplets dispersed in oil.

· Oil-in-water (O/W) for oil droplets dispersed in water.

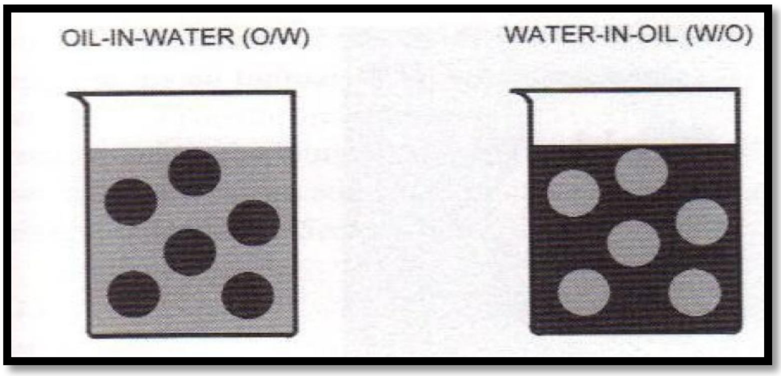


Figure 2.2: Oil-in-water (O/W) and water-in-oil emulsion (W/O)

Source: (SitiNurul Huda, 2009)

Factors of the type of emulsion formed are;

a. Type of emulsifying agent used
b. Relative proportions of the phases
c. Method of preparation of the emulsion

2.2.2 Interfacial Tension

Interfacial tensions occur when energy increased from an imbalance in the cohesive forces of the two liquids to form the smallest possible interfacial area. The interfacial tension for oil and water are high and that is why these two fluids cannot be mix. The higher of the interfacial tension make the emulsion become less stable and therefore decrease the ability to form an emulsion.

2.2.3 Emulsifying agent (emulsifier)

Emulsifier is added to the emulsion solution in order to stabilize the water and the oil. This emulsifier is use widely in our daily life. For example are detergent creams and lotions. The emulsifier used will decreased the interfacial tension between the oil and water in order to form an emulsion. The stability of an emulsion depends on the factors of emulsion formed discuss in section 2.2.1.

Types of emulsifying agents are;

a. Naturally occurring material such as protein and phospholipids.

b. Synthetic materials like ester of glycerol, propylene glycol, sorbitan ester of fatty acids and cellulose ethers.

c. Kindly divided solids such as bentonite and carbon black.

2.3 Demulsification

Demulsification is a reverse from emulsification process. Meaning that, it is a process of oil and water separation form emulsion. But because of the demulsification mechanism of the demulsifier is quite complicated therefore not all of crude oil emulsion can break by the demulsifier. Typical demulsification techniques include electrical, chemical, thermal, mechanical or acoustic method (Abdurahman et al., 2010; SitiNurul Huda, 2009)

2.3.1 Demulsifying agent (demulsifier)

Both emulsifier and demulsifier act as the surface-active agent (surfactant). The difference is the function of itself. Demulsifier is added to the emulsion solution and act as separator which is to separate the oil and water. (Abdurrahman et al., 2010)

The essential needs on demulsifiers are the abilities to have one or more of the following behaviours ("Demulsification in petroleum recovery");

a. The oil or water interface must be strong with the capability to destabilize the protective film around the droplet and/or to adjust the contact angle of the solids which may be part of the interfacial film;
b. Ability to flocculate the droplets;
c. Ability to uphold coalescence by opening path for water's natural attraction to water; and
d. Promotion of film drainage and thinning of the interdroplet lamella by raised compressibility and put changes to the interfacial viscosity.

2.4 Characterization

2.4.1 Temperature

Liebold et al. (1975) said that the temperature at which certain crude oils containing higher hydrocarbon chains are demulsified are from 40°C to 80°C. In common, temperature of between 50°C and 80°C are maintained and these give the optimum results.

2.4.2 Particle size

Referring to Nduka et al. (2008), the particles size is one of the major determining factors. The particle size enhance the mopping ability as follow – carbonized 325µm > uncarbonized 325 µm> carbonized 625µm> uncarbonized 625µm. Therefore the smaller the size particle the more it will adsorb the oil. Thus, for this study the particle size that will be used are gross fibre and fine fibre.

2.4.3 Rheological models

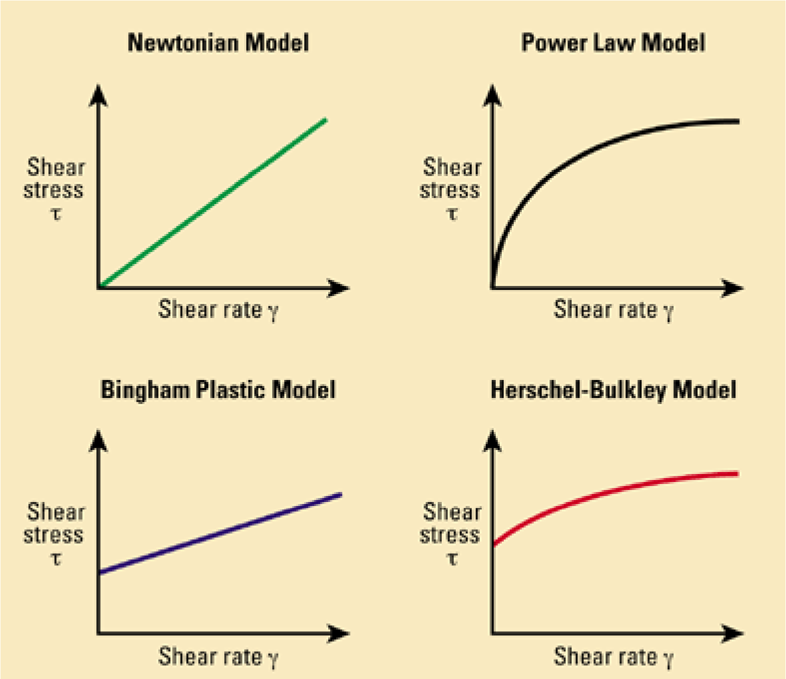


Figure 2.3: X-Y plots of rheological models

Source: Schlumberger, 2011

.

Fluids are described as Newtonian or non-Newtonian depending on their response to shearing. Shear stress of a Newtonian fluid (upper left) is proportional to the shear rate. Most drilling mud are non-Newtonian fluids, with viscosity decreasing as shear rate increases, and correspond more closely to one of the other three models shown (Schlumberger, 2011).

2.5 Oil-water separation

From Siti Nuurul Huda (2009) study, separation process in chemistry and chemical engineering is used to transform a mixture of substances into two or more different products. These separated products could be differing in terms of physical or chemical properties.

Oil can exist in water in several ways;

a. Free oil – created by oil droplets with a diameter go over for about 30 microns. It increases rapidly to the water surface when given a sufficient quiescent settling period (Surthesan, S. S., 1999).

b. Mechanical dispersions – fine oil droplets allocations ranging in size from less than 1 micron to 30 microns and having stability because of electrical charges and other forces, but due to the presence of surface active materials (Surthesan, S. S., 1999).

c. Chemical emulsions – the oil droplets distribution is similar to mechanical dispersion, but which have additional stability due to chemical, the surface active agents present at the oil and water interface may create an interaction (Surthesan, S. S., 1999).

d. Dissolved oil – dissolved in a chemical sense; the removal by normal physical means is impossible (Surthesan, S. S., 1999).

e. Oil that holds to the surface of particulate materials – known as oil wet solids (Surthesan, S. S., 1999).

2.5.1 Types of separation process

There are many ways to separate a solution into two or more different products such as;

a. Adsorption
b. Crystallization
c. Distillation
d. Drying
e. Evaporation
f. Extraction
g. Filtration
h. Centrifugation
i. Sedimentations
j. Precipitations and many more.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter describes the materials, apparatus, equipment and experimental method used in this study. The experiment was divided into three main (3) parts; the first part was the pre-treatment of the coconut coir, while the second part examined the formation and stabilization of the oil-in-water emulsion and the last part studied the separation of oil and water by using coconut coir.

3.2 Materials and Apparatus

1. Brookfield Rotational Digital Viscometer Model LV/DV-III (equipped with a water bath thermostat, and a spindle set)
2. Grinder
3. Vibratory Sieve-shaker

4. Stirrer (three-blade propeller)
5. Apparatus (500ml/250ml/50ml glass beaker, 100ml/10ml measuring cylinder, 1000ml conical flask, 100-1000μm micropipette, aluminum foil, filter paper, cotton fabric)
6. 10L light crude oil, Bintulu crude oil
7. Surfactant: Tween 20
8. Coconut coir
9. Sulphuric acid (H_2SO

3.3 Sample preparation and Experimental procedures

3.3.1 Pre-treatment method

3.3.1.1 Grinding

The purpose of this process is to grind the raw coconut coir into a medium/small particle size of fibre. This equipment is known as grinder. It's only suitable for a fibre material such as coconut coir, sugar cane, empty fruit bunch and etc. For safety purpose, the grinding must be operating with laboratory assistant (J.P) existence. In Figure 3.1 below, its shows the step on how to operate the grinder.

STEP 1
- Tied up the sack at the place where the product will come out

STEP 2
- Turn the power on

STEP 3
- Insert the sample (coconut coir) little by little

STEP 4

- Off the machine and take out the product

Figure 3.1: Flowchart for grinding process of the coconut coir.

3.3.1.2 Sieving

After grinding, the next step is the sieving process. The purpose of this process is to determine the particle size needed (630µm and 2mm) to be used in this study. This process is using vibratory sieve shaker. Figure 3.2 below shows the flowchart for the sieving process.

STEP 1

- Prepare a stack of sieves. A sieve with a larger opening is placed above a sieve with smaller openings.

↓

STEP 2

- Pour the soil (cocout coir) into the stack of sieves from the top

↓

STEP 3

- Place the cover on the top of the sieves

↓

STEP 4
Run the stack of sieves through the sieve shaker for about 5 minutes and set it amplitude to 0.5 – 1.0

STEP 5
Stop the sieve shaker and remove the stack of sieves.

Figure 3.2: Flowchart for sieving process of the coconut coir.

3.3.1.3 Modification

Then, after determine the particle sizes the next process is the modification process of the coconut coir by using the Sulphuric acid (H_2SO_4). The process is shows in the Figure 3.3 below.

STEP 1
The coconut coir is wash with soap and let it sun dried for 1 day. Take half of the sample dried for the modification part.

STEP 2
Prepare 1M of Sulphuric acid (H_2SO_4) solution and immersed the coconut coir in the solution for 1/2 day for each particle size.

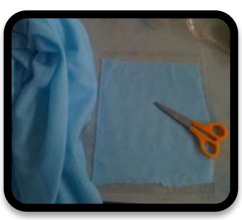
STEP 3
After that, the coconut coir is wash with water to remove all the reacted chemical and let it sun dried

STEP 4
Further dried it using oven at 200°C for 30 minutes

Figure 3.3: Flowchart for modification process of the coconut coir.

3.3.1.4 Weighing and packaging method

Here, the sample for this study was prepared. Cloth, thread, scissors was used. Figure 3.4 below shows the method to weight and packaging the samples.



STEP 1
- Cutting the cloth for the pillowcase/bags (12 cm x 12cm)

STEP 2
- 3 g of coconut coir for each particle size were weight and noted

STEP 3

- 6 samples for each particle size of unmodified and modified coir was prepared and weight noted.

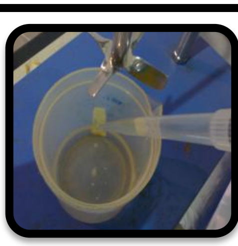
Figure 3.4: Flowchart for weighing and packaging process of the modified and unmodified coir

3.3.2 Emulsification

This study is not only focus on the preparation of the emulsion that can be seen in the figure 3.5 below, but also the identification of the stability of the emulsion based on two methods which are visually observed (Table 3.1) and the second method is the characterization of the emulsion using Brookfield equipment.

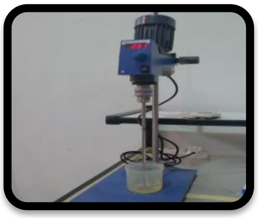
STEP 1

- Sample of distilled water is prepared in the volume of 50ml in the 250ml gradually beakers



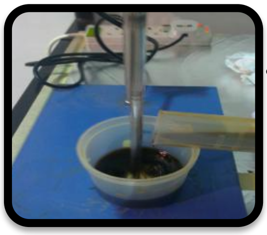
STEP 2

- The surfactant (Tween 20) with 1% cocentration from 100ml oil-water volume is added into this sample of water. This surfactant must be added into the continuous phase which is water.



STEP 3

- This water and surfactant then is agitated vigorouly using a standard blade propeller at speed 900rpm and the temperature range of 25 to 30°C (room temperature) for 2 minutes.



STEP 4

- 50ml of crude oil (Bintulu crude oil) is added into the beaker cotaining of water and surfactant after 2 minutes water and surfactant is agitated. The volume ratio of this emulsion is 50-50% oil-in-water emulsion.

STEP 5

- After 8 minutes oil, water and surfactant are agitated, a droplet of this emulsion is put on the filter paper to check whether it is the oil in water or water in oil emulsion.

STEP 6

- The oil in water emulsion then is put into the 100ml measuring cylinder. After 30minutes, the volume of water is read. The water volume i read in 5hours for every 30 minutes. The water volume reading is recorded in table 3.1.

STEP 7

- Step 1-6 is repeated by prepare the volume ratio of the Bintulu crude oil emulsion for 65-35% and 75-25% oil-in water. For every volume ratio of the emulsion, the surfactant must be added in concentration of 1%, 3%, 5%.

Figure 3.5: Flowchart for emulsion preparation for different volume ratio

Table 3.1 (a): Experimental data for volume of water layer of emulsion at room temperature using different concentration of surfactant for 50-50% oil-in-water emulsion

	50-50% O/W		
TWEEN 20	1% (1mL)	3%(3mL)	5%(5mL)
Time,(min)	$V_{H2O,ml}$	$V_{H2O,ml}$	$V_{H2O,ml}$
0			
30			
60			
90			
120			
150			
180			
210			
240			
270			
300			

Table 3.1 (b): Experimental data for volume of water layer of emulsion at room temperature using different concentration of surfactant 65-35% oil-in-water emulsion

	65-35% O/W		
TWEEN 20	1% (1mL)	3%(3mL)	5%(5mL)
Time,(min)	$V_{H2O,ml}$	$V_{H2O,ml}$	$V_{H2O,ml}$
0			
30			
60			
90			
120			
150			
180			
210			
240			
270			
300			

Table 3.1(c): Experimental data for volume of water layer of emulsion at room temperature using different concentration of surfactant 75-25% oil-in-water emulsion

	75-25% O/W		
TWEEN 20	1% (1mL)	3%(3mL)	5%(5mL)
Time,(min)	$V_{H2O,ml}$	$V_{H2O,ml}$	$V_{H2O,ml}$
0			
30			
60			
90			
120			
150			
180			
210			
240			
270			
300			

3.3.3 Demulsification (Adsorption)

Adsorption is one of the separation processes. The separation of oil and water by using coconut coir process is also known as demulsification method where the coconut coir acts as the demulsifier. Before proceed with the emulsion solution, this study was done first with the adsorption of 75ml crude oil and water only by the sample. This is to determine the effectiveness of the coir whether it's adsorbed oil more or water. Then the study proceeds with the emulsion in which prepared in section 3.3.2. Here the emulsion used is the most stable one that had been identify in the section 3.3.2.

STEP 1

- Weight the initial sample before immersed for 2mm particle size.

STEP 3

- Prepared 75ml of crude oil (Bintulu crude oil)

STEP 4

- Immersed the sample into the crude oil for 10 minutes.

STEP 5

- After 10 minutes hung the sample in air for 5 minutes to drain off unadsorbed surface liquid. The quantity of liquid actually adsorbed by the coir was determined by weighing and recorded in the table 3.2.

STEP 6

- The amount of liquids recovered was obtained by pressing the pillowcase at room temperature and recorded in the table 3.2. Repeat step 1-6 for the water and emulsion solution.

Figure 3.6: Flowchart for the adsorption of coconut coir for different type of fluid

Table 3.2: Experimental data for the adsorption of coconut coir for each particles size for both modified/unmodified coir and recovery of the fluid (oil and water)

			Before immersed + pillow bag (g)			After immersed (10+5min) & After press+pillow bag (g)			Amount of oil recovered after press (ml)		
			1	2	AVERAGE	1	2	AVERAGE	1	2	AVERAGE
crude oil (75 ml)	3g modified coir	630µm			±			±			±
		2mm			±			±			±
	3g unmodified coir	630µm			±			±			±
		2mm			±			±			±
emulsion 75-25% (o/w)	3g modified coir	630µm			±			±			±
		2mm			±			±			±
	3g unmodified coir	630µm			±			±			±
		2mm			±			±			±
water (75 ml)	3g modified coir	630µm			±			±			±
		2mm			±			±			±
	3g unmodified coir	630µm			±			±			±
		2mm			±			±			±

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

Findings from this study are discussed in two parts; the first part studied the formation and stabilization of oil-in-water (O/W) emulsion, while the second part discusses the separation of oil-in-water (demulsification) by using coconut coir.

4.2 The formation and stabilization of Emulsion

For first part of this study, Tween 20 (emulsifier) which acts as a stabilizing agent (emulsifier) was used. The purpose for using Tween 20 is to produce an O/W emulsion and to make the interface between water or oil droplets in suspension stabilized. Dispersion of these two immiscible liquids is known as emulsion. In order to form an emulsion, stabilizing agent is necessarily to be added to the immiscible liquids and the effect on the stability of emulsion form will be observed.

4.2.1 Effect of different oil-water ratio on Emulsion Stability

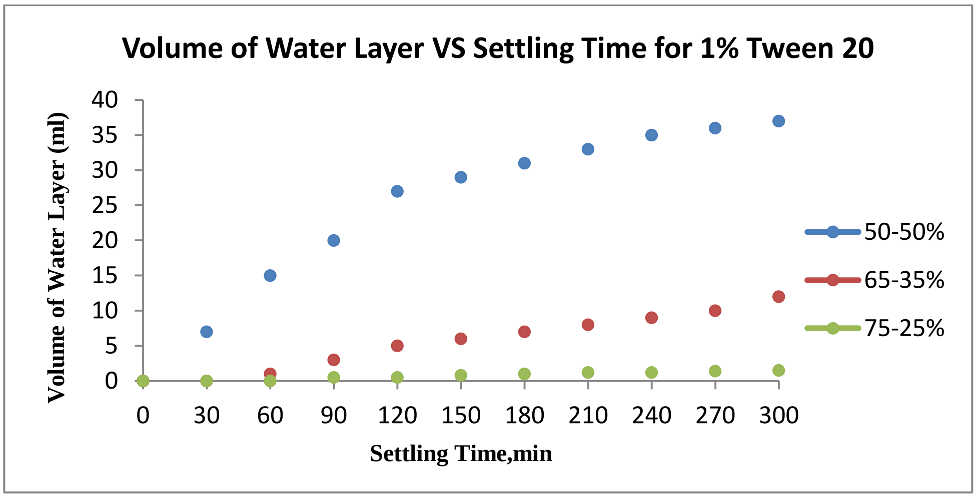


Figure 4.1(a): Volume of water layer versus settling time for separation of water layer for emulsion at 1% concentration of Tween 20

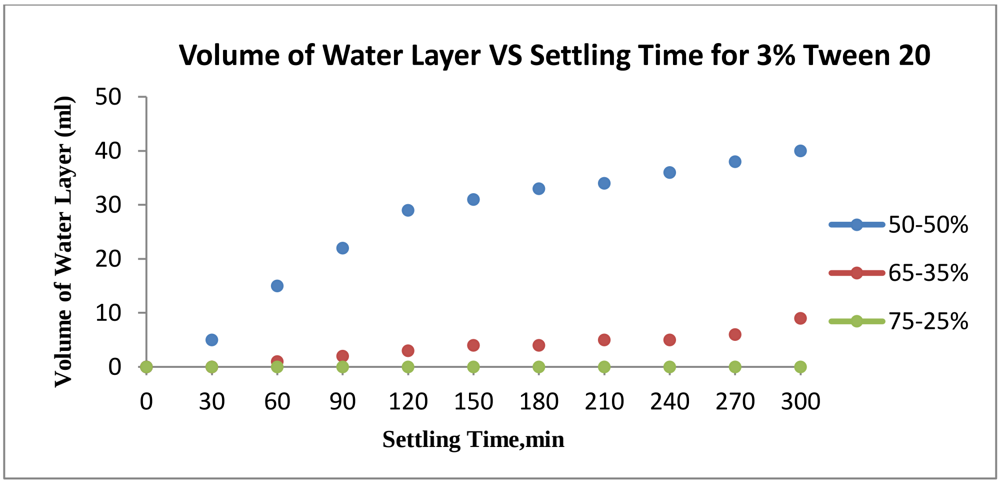


Figure 4.1(b): Volume of water layer versus settling time for separation of water layer for emulsion at 3% concentration of Tween 20

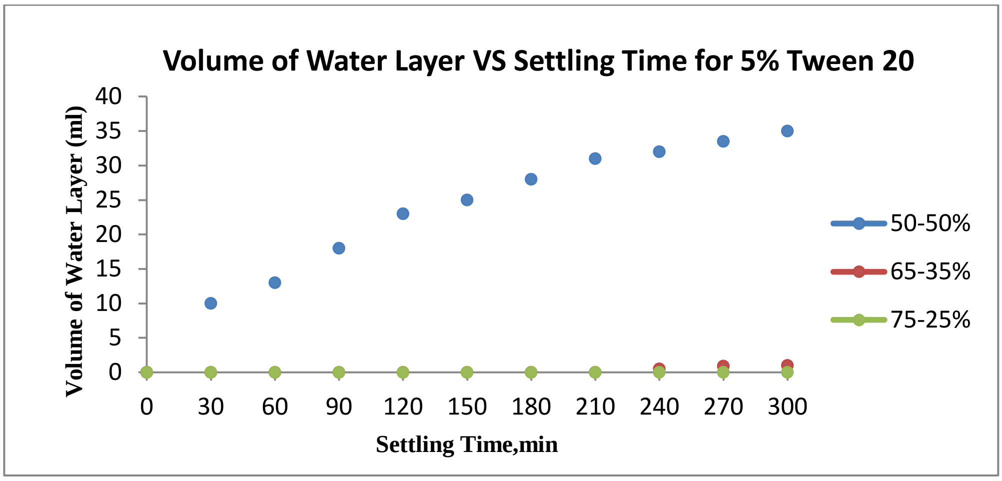


Figure 4.1(c): Volume of water layer versus settling time for separation of water layer for emulsion at 5% concentration of Tween 20

This experiment was done for 5 hours for each emulsion samples. The stability of the emulsions was observing visually and determined by measuring the water level from the emulsion at 27°C as a function of time. In terms of emulsion characterization, this study used different in emulsifier's concentrations to get a most stable for O/W emulsion. (See appendix A: water-oil separation layer) The volume of water settled at the bottom of volumetric cylinder was measured for every 30 min for 5 hours.

Figure 4.1 a, b and c above shows the same trends for the effect of volume ratio of water on emulsion stability. From the observation, for each of the emulsifier concentrations (1%, 3% & 5%), as the volume ratio of water is less, the emulsions become more stable. When the water quantity in the emulsion sample is small, the viscosity of that emulsion is high. So that the molecule's velocity in the emulsion is small and make it hard to move and separate water from emulsion. Surface tension between water and oil in that emulsion become high. Therefore, smallest amount of water separate from the emulsion will give a result as stable emulsion. From the result for emulsion sample for 75-25% O/W give the lower volume of water level for each graph.

4.2.2 Effect of emulsifier concentration on emulsion stability

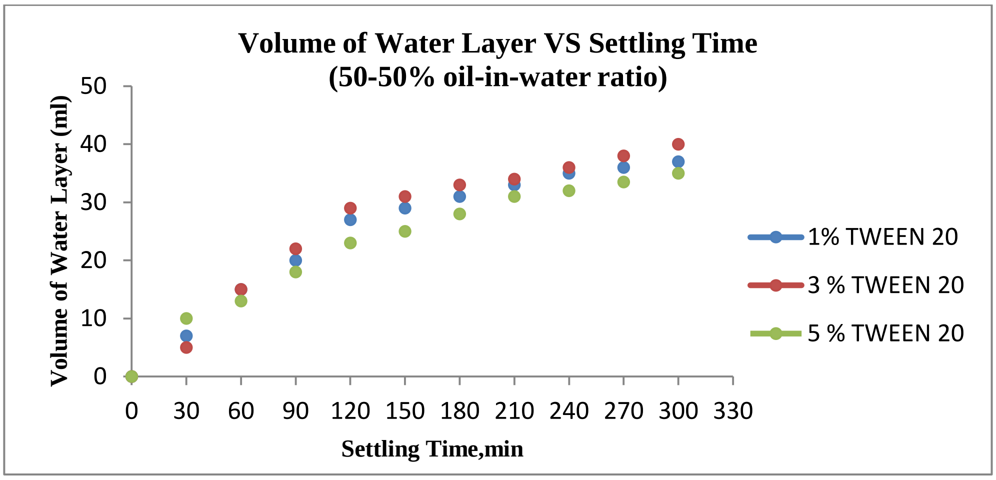


Figure 4.2(a): Experimental data for volume of water layer of emulsion at room temperature using different concentration of surfactant for 50-50% oil-in-water emulsion

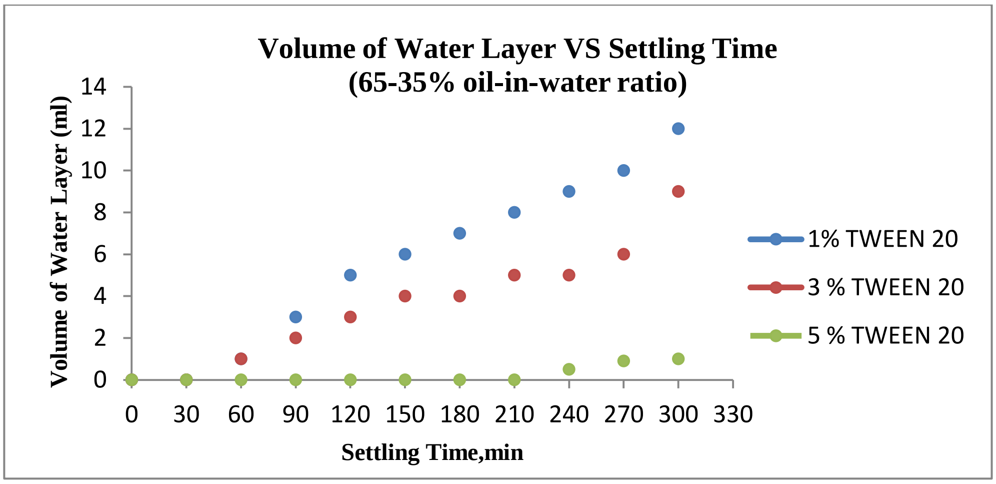


Figure 4.2(a): Experimental data for volume of water layer of emulsion at room temperature using different concentration of surfactant for 65-35% oil-in-water emulsion

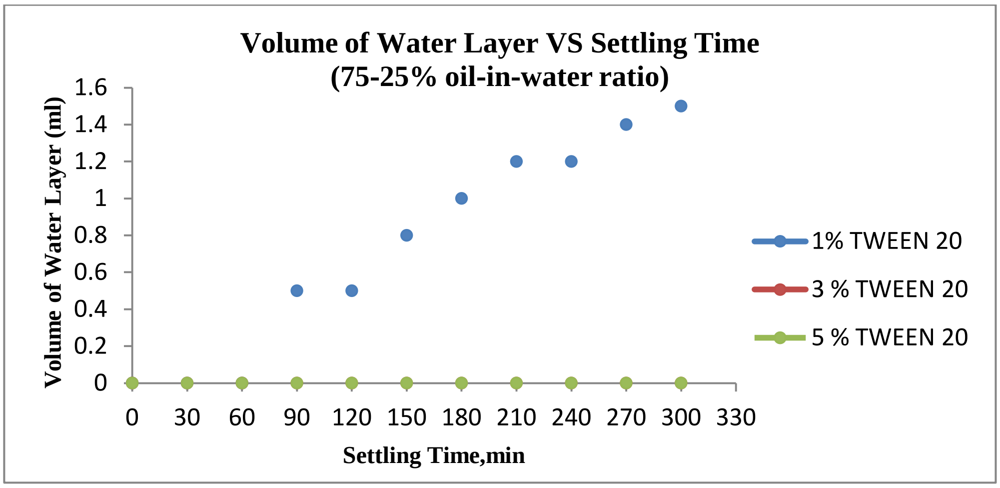


Figure 4.2(c): Experimental data for volume of water layer of emulsion at room temperature using different concentration of surfactant for 75-25% oil-in-water emulsion

From the observation, less the volume of the water layer separate from the emulsion will make the emulsion more stable. In other words, the emulsion is high in concentration. The result for 50-50%, 65-35% and 75-25% volume ratio of O/W separation added with three different concentrations of Tween 20 is shown in Figure 4.2 a, b and c.

As shown in Figures 4.2 a, b and c below, as the concentration of the emulsifier is increase from 1% to 5% the emulsion stability is increasing. From the result for emulsion sample for 5% concentration of emulsifier (Tween 20) at 75-25% O/W emulsion ratio give the lower volume of water level and appeared the most significant on emulsion stability. As the concentration for the Tween 20 increased, the viscosity of oil-in-water emulsion also increased significantly. Stability was evaluated via the ratio of total water separated from the emulsion.

4.2.3 Correlation between viscosity and emulsion stability

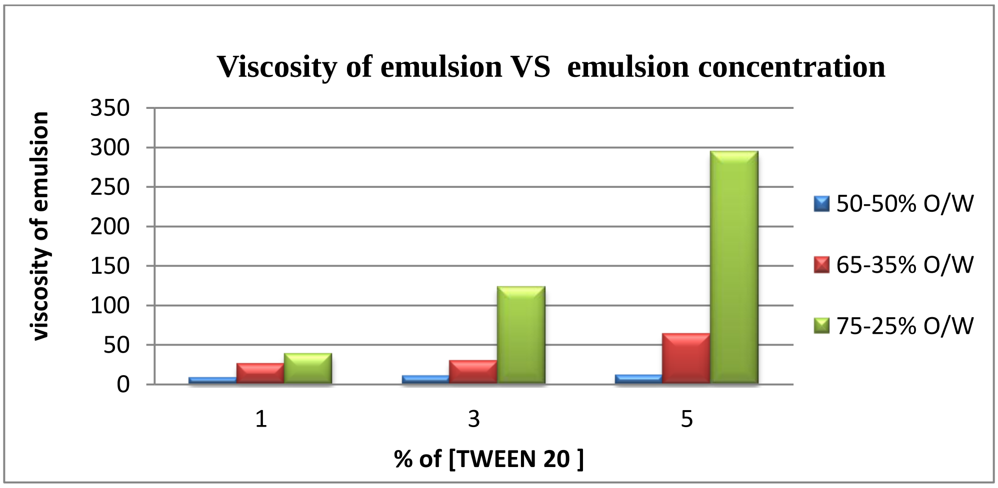


Figure 4.3: Experimental data for emulsion viscosity for each concentration of emulsion at different values of oil-in-water volume ratio

To guarantee the stability for each emulsion so that it will not just by referring to the visual observation, a significant of emulsion stability studies is done where the viscosity of each sample of emulsions should be analyzed. This study is done by using Brookfield (viscometer) in FKKSA laboratory. In theory, the higher the viscosity characterizes the higher of the emulsion stability. For that reason, the viscosity analysis is a necessarily to ensure that the viscosity of the emulsions is proportional to its stability as shown Figure 4.3 above. Both had shows the viscosity as the concentration of emulsifier is varied.

From Figure 4.3 above, it can be conclude that as the concentration of the emulsifier is increase, the viscosity will be increase too. Thus the more stable emulsion can be seen. That means the surface tension between both liquid is high and the interfacial tension is low. As expected in previous study in section 4.2.1, as the water volume in emulsion decreased the emulsion becomes more stable. The emulsion is most stable with 75-25% oil-in-water ratio and therefore given the higher number in viscosity reading.

Therefore, from both section 4.2.1 and 4.2.2 the most stable emulsion can be identify. The most stable emulsion is emulsion at 75-25% o/w volume ratio with 5% concentration of Tween 20(emulsifier). This emulsion only separated into oil and water after 5 days.

4.3 Separation of oil and water (emulsion) by using coconut coir

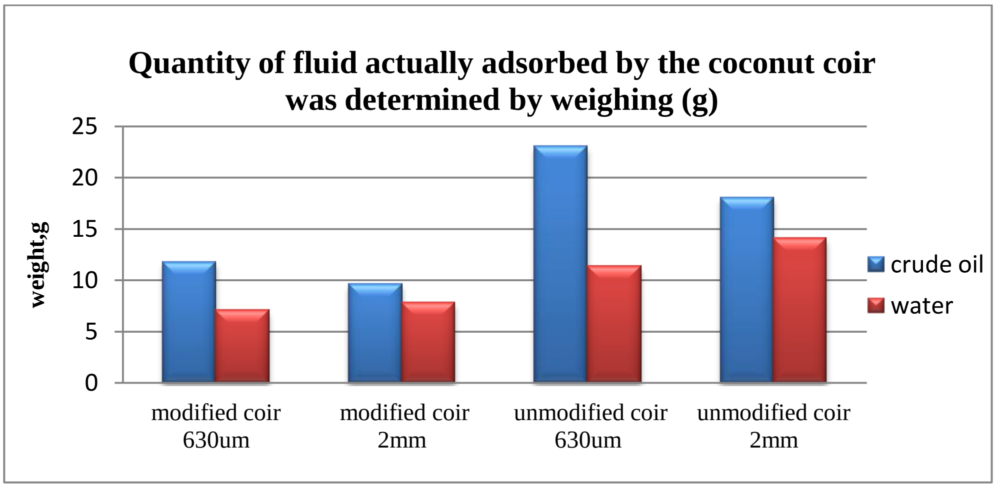


Figure 4.4(a): Experimental data quantity of fluid actually adsorbed by the coconut coir was determined by weighing (g)

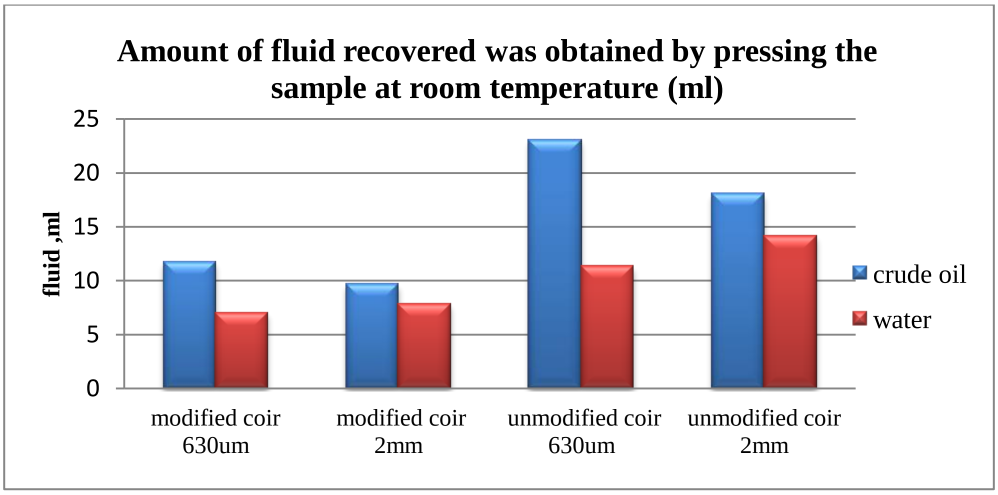


Figure 4.4(b): Experimental data for amount of fluid recovered (ml) was obtained by pressing the sample at room temperature

In this study, one of the separation processes which are adsorption was used. Adsorption is a process that happen when a liquid or gas accumulates on the surface of a solid or a liquid (adsorbent), forming a molecular or atomic films (the adsorbate). Totally different from absorption, in which a substance diffuses into a liquid or solid to form a solution (Scott, 2011). This method includes the usage of the natural waste known as coconut coir. The coir of aromatic dwarf coconut will be used ad it was collected from Kg. Melayu Subang in Shah Alam. Besides, the coir fibre was obtained from the coconut and it was manually extracted from the husk. The different in particle size (630 µm and 2mm); modification coir was used in order to identify the effectiveness of the coir to separate oil and water.

In terms of particle size and type of fluid being adsorbed, the results of the study reveal that the amount of the adsorption of the coir for the crude oil at 630µm particle size were far greater than the adsorption at 2mm particle size as shown in figure 4.4(a). But the result was different for the adsorption of the water by coir where at 2mm particle size adsorbed more water rather than the adsorption of water at 630µm particle size. Theoretically, smaller the particle size the bigger the surface area. This might be one of the reasons why the 630µm adsorbed more oil rather than 2mm particle size. On the other hand, the reason for the water not to be adsorbed more by the smaller particle because of the physical characteristic of the coconut coir itself. Coconut coir is a fibre that contains high percentage of fatty acid (avoid water-hydrophobic). So, the ability for the coconut coir adsorbed the water will be decrease when the particle size is decrease.

While for the modification part, as explain earlier (methodology) there are two type of coir which is modified coir (immersed in sulphuric acid solution) and the other one is the unmodified coir. The purpose for the modification study is to modify the structure of the coir itself or to improve the specific adsorption for the coir. As shown in Figure 4.4(a) above, the unmodified coir for both particle sizes is more efficient to adsorb oil or water rather than the modified coir. The result is totally different from Nduka et. Al (2008) in which it has been found that modified coir is more effective to adsorb oil. This is because of the nature of the coir (sorbent) features likes availability of pores/voids, convolution, lumen, lacunae, inter-fibril/fibre spaces, specific adsorption sites and also the viscosity and molecular sizes of the fluid itself.

Based on the result, since the coir is more efficient to adsorb oil rather than water therefore another study is conducted. This study involves the emulsion which is the most stable one from section 4.2. This study wants to justify the efficiency of the coir to adsorb oil.

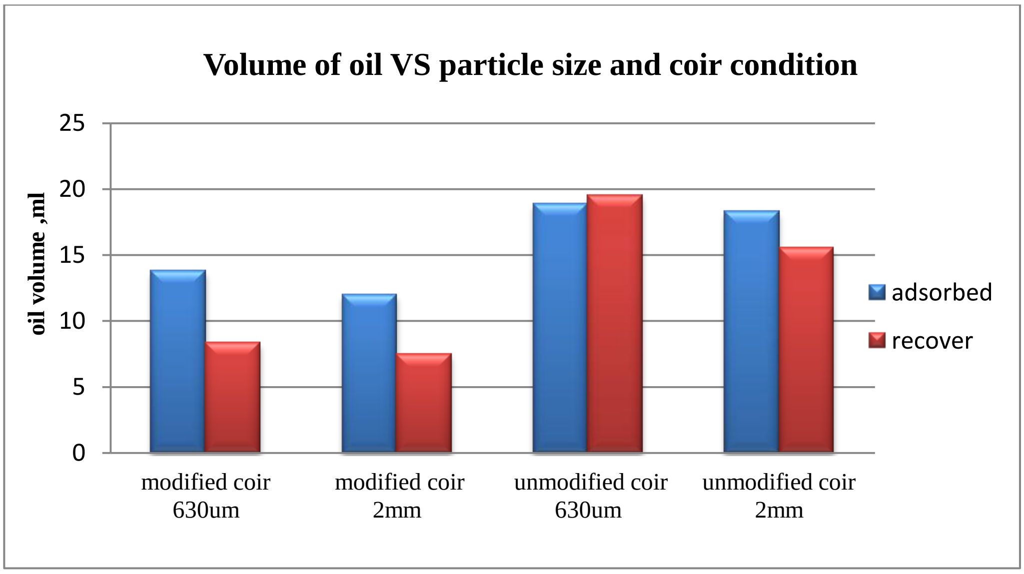


Figure 4.5: Experimental data for amount of oil being adsorbed and recover from 75-25% of O/W emulsion

Figure 4.5 shows the volume being adsorbed by the coir after immersed the coir into the 75-25% o/w emulsion. Unmodified coir, 630μm give the highest volume of oil being adsorbed and recover rather than the others. After immersed and pressed the pillowcase that contain coir, the volume of the adsorbed and recover only can be noted after 3 days. This is to ensure that the coir is only adsorbing the oil not water

After 3 days, noticed that for the adsorb oil, the readings was almost 100% oil. Only 0.5-1% of water can be seen on the bottom of the measuring cylinder. But for the recover, the fluid seen too be 100% oil.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusions

In conclusion, the optimum condition for the emulsion is when the ratio of the oil-water is at 75-25% of the total volume. Then, the emulsifier (Tween 20) concentration for 5 % of the total volume also contributes to the stable emulsion. Besides that, the most effective coir is for unmodified coir fat 630µm. Then with these two optimum conditions, it is applied for the separation study. The study demonstrated the coir fibre exhibit good performance to adsorb oil from emulsion solution. Due to the low cost and the performance, coconut coir is the new alternative that can use for separation of oil and water (Ibrahim et al, 2009).

5.2 Recommendations

For the study of emulsion formation and stabilization, there are several parts that can be improved to obtain a better understanding of physical and chemical characteristics of the emulsions. More parameter such as temperature, speed of the agitation and varieties of emulsifier should be used in order to determine their potential to give a stable emulsion.

Besides, more details should be given for the study of the separation of oil and water by using coconut coir. Should be, a specific equipment likes organic elementary analysis be used for a better understanding of the chemical composition of the coconut coir.

Smaller particle size of the coconut coir should be use in the future and try to dilute it with any chemical so it can become a liquid solution. This is for the better procedure in order to separate the emulsion into oil and water phase.

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APPENDIX A

Volume of water layer versus settling time for separation of water layer for emulsion at 1% concentration of Tween 20

	50-50% O/W	65-35%O/W	75-25% O/W
Time,(min)	$V_{H2O,ml}$	$V_{H2O,ml}$	$V_{H2O,ml}$
30	7	0	0
60	15	1	0
90	20	3	0.5
120	27	5	0.5
150	29	6	0.8
180	31	7	1
210	33	8	1.2
240	35	9	1.2
270	36	10	1.4
300	37	12	1.5

Volume of water layer versus settling time for separation of water layer for emulsion at 3% concentration of Tween 20

	50-50% O/W	65-35% O/W	75-25% O/W
Time,(min)	$V_{H2O,ml}$	$V_{H2O,ml}$	$V_{H2O,ml}$
0	0	0	0
30	5	0	0
60	15	1	0
90	22	2	0
120	29	3	0
150	31	4	0
180	33	4	0
210	34	5	0
240	36	5	0
270	38	6	0
300	40	9	0

Volume of water layer versus settling time for separation of water layer for emulsion at 5% concentration of Tween 20

	50-50% O/W	65-35% O/W	75-25% O/W
Time,(min)	$V_{H2O,ml}$	$V_{H2O,ml}$	$V_{H2O,ml}$
0	0	0	0
30	10	0	0
60	13	0	0
90	18	0	0
120	23	0	0
150	25	0	0
180	28	0	0
210	31	0	0
240	32	0.5	0
270	33.5	0.9	0
300	35	1	0

Experimental data for volume of water layer of emulsion at room temperature using different concentration of surfactant for 50-50% oil-in-water emulsion

	50-50% O/W		
TWEEN 20	1% (1mL)	3%(3mL)	5%(5mL)
Time,(min)	$V_{H2O,ml}$	$V_{H2O,ml}$	$V_{H2O,ml}$
0	0	0	0
30	7	5	10
60	15	15	13
90	20	22	18
120	27	29	23
150	29	31	25
180	31	33	28
210	33	34	31
240	35	36	32
270	36	38	33.5
300	37	40	35

Experimental data for volume of water layer of emulsion at room temperature using different concentration of surfactant for 65-35% oil-in-water emulsion

	65-35% O/W		
TWEEN 20	1% (1mL)	3%(3mL)	5%(5mL)
Time,(min)	$V_{H2O,ml}$	$V_{H2O,ml}$	$V_{H2O,ml}$
0	0	0	0
30	0	0	0
60	1	1	0
90	3	2	0
120	5	3	0
150	6	4	0
180	7	4	0
210	8	5	0
240	9	5	0.5
270	10	6	0.9
300	12	9	1

Experimental data for volume of water layer of emulsion at room temperature using different concentration of surfactant for 75-25% oil-in-water emulsion

	75-25% O/W		
TWEEN 20	1% (1mL)	3%(3mL)	5%(5mL)
Time,(min)	$V_{H2O,ml}$	$V_{H2O,ml}$	$V_{H2O,ml}$
0	0	0	0
30	0	0	0
60	0	0	0
90	0.5	0	0
120	0.5	0	0
150	0.8	0	0
180	1	0	0
210	1.2	0	0
240	1.2	0	0
270	1.4	0	0
300	1.5	0	0

APPENDIX B

Experimental data for emulsion viscosity for each concentration of emulsion at different values of oil-in-water volume ratio

% TWEEN20	viscosity (AVG)		
	50-50%	65-35%	75-25%
1	**8.10 ± 0.30**	**25.80 ± 0.00**	**38.70 ± 0.30**
3	**9.75 ± 0.15**	**29.70 ± 0.30**	**123.30 ± 0.30**
5	**12.00 ± 0.00**	**63.90 ± 0.30**	**293.90 ± 0.00**

Experimental data for amount of fluid recover obtained by pressing the ample at room temperature

			Before immersed + pillow bag (g)			Amount of sorbate recovered was obtained by pressing the pillow at room temperature (ml)		
			AVERAGE			AVERAGE		
crude oil (75 ml)	3g modified coir	630µm	**5.40**	±	**0.10**	**11.00**	±	**0.50**
		2mm	**4.95**	±	**0.35**	**7.00**	±	**0.00**
	3g unmodified coir	630µm	**5.95**	±	**0.25**	**28.50**	±	**0.50**
		2mm	**5.15**	±	**0.05**	**14.50**	±	**0.50**
emulsion 75-25% (o/w)	3g modified coir	630µm	**5.80**	±	**0.00**	**8.35**	±	**0.45**
		2mm	**5.55**	±	**0.35**	**7.50**	±	**0.50**
	3g unmodified coir	630µm	**5.45**	±	**0.25**	**19.50**	±	**4.50**
		2mm	**5.60**	±	**0.20**	**15.50**	±	**4.50**
water (75 ml)	3g modified coir	630µm	**5.05**	±	**0.05**	**0.00**	±	**0.00**
		2mm	**5.20**	±	**0.20**	**0.60**	±	**0.60**
	3g unmodified coir	630µm	**4.90**	±	**0.00**	**8.60**	±	**0.80**
		2mm	**4.85**	±	**0.05**	**15.00**	±	**1.00**