PRODUCTION OF SOAP FROM *SESAMUM INDICUM* (SESAME SEED)

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ABSTRACT

Sesame (*Sesamum indicum L.)* seed is the oldest oil seed crop and the highest content of fats and oils among other seeds oil. The fats and oils of sesame contain good proteins and vitamins for body health and food. This research presents the study of soap from undehulled (coated) and dehulled sesame seed oil which derived from a soxhlet extraction method. Different treats of sesame seed were exposed to experiment that determine the value of physiochemical, such as saponification value (SV), acid value (AV), iodine value (IV), free fatty acid value (FFA) and peroxide value (PV). Sesame oils were saponified with two types of alkali which sodium hydroxide (NaOH) and potassium hydroxide (KOH) to give a solid and liquid soap, respectively. The analysis of the soap was carried out to determine the pH value, foam height, colour in solution, and solubility in water and foam structure. Further analysis of the soap was testing with a dirty cloth, hand washing and laboratory glassware.

Results from the analysis were exposed that sesame seed can produce either solid or liquid soap with high of conditioning agent. Dehulled oil has saponification value of 186.5 mg KOH/ g, acid value of 109.4 g I_2/100g, peroxide value of 14.8 meq H_2O_2/g, and free fatty acid value of 49.3 % (oleic) and undehulled seed oil has values 210.4 mg KOH/ g, 1.34 g I_2/100g, 106.3 meq H_2O_2/g, 8 and 39.48 % (Oleic), respectively. In conclusion, the objective of this study is achieved where sesame seed soap can be established, whether from undehulled or dehulled oil. In future, sesame oil is potentially to use as an oil foundation in making soap.

ABSTRAK

Bijian bijan (Sesamum indicum L.) adalah tanaman bijian yang tertua dan mempunyai kandungan lemak dan minyak yang tertinggi antara minyak bijian lain. Lemak dan minyak bijan mengandungi protein dan vitamin yang baik untuk kesihatan pada badan dan makanan. Tujuan kajian ini adalah untuk menghasilkan sabun daripada minyak bijan yang berkulit dan tidak berkulit, di mana minyak bijan di perolehi daripada cara pengekstrak soxhlet. Bijian bijan yang berkulit dan tidak berkulit didedahkan dalam eksperimen yang menentukan nilai fisiokimia, seperti nilai saponifikasi (SV) , nilai asid (AV), nilai iodin (IV) , nilai asid lemak (FFA) dan nilai peroksida (PV). Minyak bijan disaponifikasikan dengan dua jenis alkali iaitu sodium hidroksida (NaOH) dan potassium hidroksida (KOH) untuk menghasilkan sabun pepejal dan cecair. Analisis sabun dijalankan untuk menentukan nilai pH , ketinggian buih, warna larutan, kelarutan dalam air dan struktur buih. Analisis sabun selanjutnya adalah menguji sabun dengan kain kotor, mencuci tangan dan aparatus makmal.

Hasil daripada analisis mendedahkan bahawa bijian bijan boleh menghasilkan sabun sama ada pepejal atau cecair dengan adanya pelembap semula jadi. Minyak bijan tidak berkulit mengandungi nilai saponifikasi iaitu 186,5 mg KOH / g, nilai asid 109.4 g I2/100g , nilai peroksida 14.8 meq H2O2 / g, dan nilai nilai asid lemak sebanyak 49.3 % (oleik), manakala minyak bijan berkulit mengandungi 210,4 mg KOH / g, 1.34 g I2/100g , 106.3 meq H2O2 / g , 8 dan 39,48 % (oleik). Kesimpulannya, objektif kajian ini dicapai di mana sabun bijian bijan boleh dihasilkan daripada minyak bijan yang berkulit atau tidak berkulit. Pada masa akan datang , minyak bijan mempunyai potensi untuk digunakan sebagai minyak asas dalam membuat sabun.

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LIST OF ABBREVIATIONS

cm^3	cubic centimetre
%	percentage
cm	centimetre
mg	milligram
g	gram
ω	omega
^oC	temperature
M	molarity
mL	mile litre
N	normality
meq	measured in equivalent
s	second
i.e.	in example

LIST OF ABBREVIATIONS

AV	Acid Value
CH_3OH	Methanol
FFAV	Free Fatty Acid
HCl	Hydrochloric Acid
IV	Iodine Value
KOH	Potassium Hydroxide
LD	Liquid Dehulled soap
LU	Liquid Undehulled soap
NaOH	Sodium Hydroxide
PV	Peroxide Value
QRec	QRec Chemical Company LTD
R&M	R&M Supplier
$RCOOCH_3$	Functional group
RCOONa	Functional Group
SAFC	Sigma Aldrich Fluka Company
SU	Solid Undehulled soap
SD	Solid Dehulled soap
SV	Saponification Value
UK	United Kingdom

1 INTRODUCTION

1.1 Motivation and statement of problem

Sesame (*Sesamum Indicum L.*) is an important oilseed crop in the world. It is also known as benniseed (Africa), benne (Southern United States), gingelly (India), sim-sim (Hebrew) and tila (Sanskrit). The seed is a member of the *Pedaliaceae family*, which is family of a flowering plant. Sesame seed generally were originated found in Africa and the Middle East, because both species are similar. Then, the seeds were brought to India, Burma, China, Japan and Unites States along the late seventeenth century. Until today, sesame seed was found in every country and it has become an annual plant production (Hwang, 2005). Sesame seed grows in the tropical and subtropical area. It is commonly known as cultivated species, which depending on the variety varies in height and conditions. It has a large taproot and a diverse surface mat of feeder roots, which makes it resistant to drought. Figure 1 shown the plant of sesame, the stems have branches and densely hairy. The leaves also are hairs on the both sides and also highly variable in shape and size. The lower leaves are dull green in color, 3-17.5 cm long and 1-7 cm wide, while the upper leaves is 1-2 cm long. Sesame has large, white and bell-

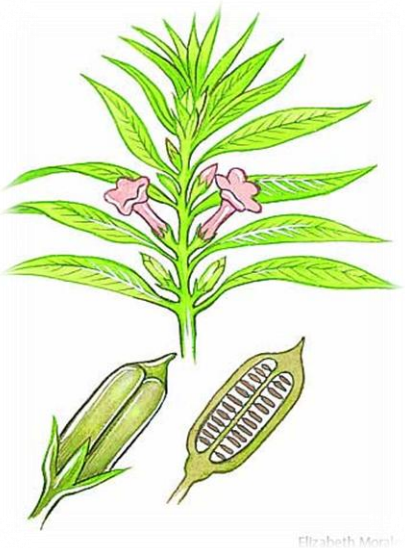

Figure 1.1: The plant of sesame

shaped of flowers. The sesame fruits are a capsule with dimensions; 2-5 cm long and 0.5-2 cm in diameter. The capsule may have 4-8 rows of seeds in each sesame fruits capsule (Hwang, 2005). Generally, sesame crop was first recorded in Babylon and Assyria over 4000 years ago (Hwang, 2005). The sesame seed was used for making wine and cakes, while the sesame oil was used for cooking, medicinal, and cosmetic purposes. Until today, the application is still using in several of usage. Basically, the sesame oil was used for cooking, massage, health treatment for the body (Bamigboye et al., 2010). However, the application of sesame oil is widely spread in the production of cosmetic of moistures and lotions, margarines, pharmaceuticals, paints, lubricants and soaps making.

As the application is widely spread, sesame oil contains one primary vitamin and other beneficial substances. Primary vitamin that included in sesame oil is Vitamin E, which contains 40 mg from 100 mg of oil. Sesame oil has antioxidant properties, thus, free radicals or waste products in the body that can lead to cell damage and disease may be eliminated. Sesame seed provides copper, iron, manganese, magnesium, phosphorus, and zinc, along with vitamin B1, the amino acid tryptophan and fibre. The major content that sesame oil provides is fatty acids (Grooper et al., 2009). However, sesame seed has a different value of fatty acid between undehulled (whole) and dehulled seeds. Undehulled seed, basically higher amount of undesirable oxalic acid, which it could complex with the fatty acid in sesame oil (Abou-Gharbia et al., 1997). Fatty acids are the simplest of the lipids. It can be found in the vegetables or fruits oil and animal fat. Sesame oil provides a variety of varying fatty acids like palmitic, palmitoleic, stereos, linolenic and archidic. Those fatty acids include less than 20% saturated fatty acid in sesame oil, while the rest more than 80% of total fatty acids are oleic and linoleic acid (Grooper et al., 2009). Basically, oleic and linoleic acids were used in the production of soap making. In the soap making field, the fatty acid like oleic acid and linoleic acid is mixed with an acoustic alkali through direct saponification (Aiwize and Achebo, 2012). Direct saponification is a traditional method which fats and oils are saponified with an alkali solution. Alkali solutions that may use in the production of soap are sodium hydroxide solution and potassium hydroxide solution (Burke, 2005).

Oils or fatty acid that used in soap production usually can be extracted by using several of methods, such as hot water flotation, bridge press, ram press and modern laboratory method; solvent extraction and enzymatic extraction (Warra, 2011). In solvent extraction method, soxhlet extraction with n-hexane solvent is the most popular method used in the experiment. The solvent of n-hexane is more suitable for free fatty acid extraction compared to others hydrocarbon solvent, such as ethanol. This is because n-hexane is a non-polar solvent, while ethanol is a polar solvent, which is suitable for bio-active compound.

Recently, sesame (*sesamum indicum L.)* is widely used in Malaysia as cooking oil, cereals, foods and other production. The properties that found in sesame oil are often used in cosmetic industry, such as oleic acid, linoleic acid, stearic acid and palmitic acid (Shahidi, 2005). Therefore, this study is conducted due to the factors of the availability of sesame seed in Malaysia. Indian is the major supplier of sesame seed in Malaysia. In fact, Malaysia imported 23521 tons in 2006 from Indian (APEDA AgriXchange, n.d.) and increased 47% in 2007. Besides that, other factors that give the ability to conducting this study is the higher demand of essential oil as pharmaceutical, aromatherapy aid and cosmetic ingredients give large opportunities for global marketing. Sesame oil has a pleasant odour comes from the extracted oil. Therefore, the opportunity to produce a good smell of soap is higher and it can save the cost for expenses of essential oil. Last but not least, Sesame oil contains of high fatty acid, such as oleic acid and linoleic acid. These fatty acids provide a conditioning agent, which moisturizer that has the ability to nourish skins. Moreover, the cost of oleic acid in the market is high. Thus, the opportunity to produce a great soap with a great moisturizer is higher and it also can save the cost for expenses of fatty acid for conditioning agent. Due to these factors, the initiative to invent a new soap from new edible oils, the sesame oil is conducted.

This research establishes the production of soap from sesame oil. Therefore, undehulled and dehulled sesame seed had been selected with the solvent extraction, which is a soxhlet extraction method.

1.2 Objectives

The objective of this study is:

i) To establish the production of soap by undehulled (whole) and dehulled sesame seed using soxhlet method

1.3 Scope of this research

To achieve the objectives of the study:

i) Extract oil from sesame seed with whole (undehulled) and dehulled seed using soxhlet extraction method
ii) Determine the physiochemical analysis of acid value (AV), saponification value (SV), iodine value (IV), fatty acid value (FV) and peroxide value (PV)
iii) Conduct the saponification with two alkaline solution; (NaOH and KOH)
iv) Determine the characteristic analysis of foam height, pH value and to observe the colour of the solution, the odour, the solubility of soap and foam structure
v) Test the application of the soap produced by tested it to dirty cloth, hand washing and laboratory glassware.

1.4 Main contribution of this work

As the previous journals that found were only about the physiochemical analysis of the sesame oil, however this study is to establishes the production of soap from sesame oil and to come out with real soaps by using different kind of oil, such undehulled seed and dehulled seed.

1.5 Organisation of this thesis

The structure of the reminder of the thesis is outlined as follow:

Chapter 2 provides the information of the past research related to the sesame seed. Besides that, this chapter will be includes the description of fatty acid, soxhlet extraction, saponification and the alkalis.

Chapter 3 tells how the research will be made. This is included the flow diagram process from the beginning till the end of the process. Besides that, this chapter tells the detail operation of this research, which includes the soxhlet extraction method, physiochemical analysis, direct saponification for soap making and soap testing.

Chapter 4 is discussed about the result that collected from the journal of the American oil chemist's society, journal of pure and applied sciences, and journal of agriculture science and technology. The experiment was able to performed completely for dehulled and the invention of the experiment, undehulled seeds. This chapter also discussed about the soap analysis that produced using the formula that generated from the research experiment during Industrial Training. In order to establish soap from undehulled and dehulled oil, soap will be tested for its availability.

Chapter 5 are presented the conclusion and recommendation for this study. All methodologies, scopes and objectives that successfully tested and produced a yield of standard were presented in this chapter.

2 LITERATURE REVIEW

2.1 Overview

This chapter is provides the information of the past research related to the sesame seed, where sesame seed was early found in African and Asia. Besides that, this chapter will be includes the description of fatty acid that found in sesame oil, such as oleic acid, linoleic acid, stearic acid and palmitic acid. In addition, the information of soxhlet extraction also provides in this chapter, where there are many methods that used in gaining the sesame oil. Next, the saponification of the previous study was also been discussed in this study, followed by the alkalis.

2.2 Introduction

This study presents the information about sesame seed from the previous research.

2.3 Previous work on Sesame seed

Sesame (*Sesamum Indicum L.*) is an important oilseed crop in the world and it was ranked ninth from the top thirteen among the edible seed crop (Hwang, 2005). Sesame seed grows in the tropical and subtropical area. It is commonly known as cultivated species, which depending on the variety varies in height and conditions. Based on Anon (2008), the world production of sesame crop was estimated about 3.66 million tonnes, where Asia and Africa produced 2.55 and 0.95 million tonnes, respectively.

2.3.1 Nutrient and Composition of Sesame seed

Sesame seed has a variety of colour and composition content of fat and proteins. It also has different content of lipid between undehulled (whole) and dehulled seed. Table 2-1 shows the proximate composition of sesame seed from Nigerian with different colour and undehulled and dehulled sesame. White sesame has the highest crude fat than the black and brown sesame, however, has the lowest of moisture content. According to Bamigboye et al. (2010) research, it was found that sesame seed with low moisture

content is expected of its long shelf life and keeping quality. These might be the disadvantage for microorganisms inside food to grow well.

Table 2-1: Proximate Composition of Sesame seed (%)

Sesame	Crude Fat	Crude protein	Carbohydrates	Crude Fiber	Ash	Moisture
Black sesame	48.4-56.7	22.8-30.3	3.4-10.8	2.8-7.2	4.4-5.5	4.6-6.4
White sesame	50.1-51.7	22.6-24.1	7.9-13.2	5.3-7.5	4.2-4.5	4.4-4.7
Brown sesame	46.3-53.1	21.8-27.6	4.7-13.6	3.7-7.3	3.9-5.4	5.0-8.2
Nigerian undehulled sesame	51.5	20.0	12.5	6.0	5.0	5.0
Nigerian dehulled sesame	55.0	24.3	10.4	2.0	3.0	5.3

(*Retrieved from Hwang, 2005)*

The percentage of dehulled sesame seed on the table also shows higher than the undehulled sesame seed. This is because the hull of sesame seed has the largest amount of undesirable oxalic acid and crude fiber, which gives bitter and dark color to the food (Abou-Gharbia et al. (1997).

Sesame seed also contains of antioxidants that inhibit the development of rancidity in the oil (Tunde-Akintunde et al., 2012). According to Xu et al (2005), as cited in Budowski (1964) found that the stability of sesame seed was highly oxidation than other plant seeds

2.3.2 Fatty acids

Fatty acids are the main constituent of oils and fats. The configuration of fatty acids is almost entirely straight chain aliphatic carboxylic acids. According to Arild & Christian (2005), it found that 30-35% was represented as total energy intake in industrial and most dietary source of fatty acids, such as vegetable oils, dairy products, meat products, grain and fatty fish. Many type of common saturated fatty acid in plants and place, such as oleic, linoleic, stearic, palmitic and so on.

In sesame seed, 80% of the fatty acids are included in extracting oil (Hwang, 2005). The fatty acids that may found in the extracted oil of sesame seeds are palmitic, stearic, oleic and linoleic acid. The value of the composition is based on the following table.

Table 2-2: Oil contents and Characteristic of Sesame seed

Characteristics	**White sesame seed (wt %)**	
	Unroasted	Roasted
Palmitic	8.7	8.7
Stearic	4.9	5.1
Oleic	46.8	47.2
Linoleic	39.6	39.0

(*Retrieved from Mohamed and Awatif, 1998)*

Based on Table 2-2, the data show the oil contents in sesame seed with different characteristics, which unroasted and roasted sesame seed. Roasted and unroasted seeds give higher of oleic acid content, followed by linoleic acid at 47.2 and 39.0 wt%, and 46.8 and 39.6 wt%, respectively. Therefore, sesame oil can be classified as oleic-linoliec acid group (Warra, 2011).

2.3.2.1 Oleic acid, Linoleic acid, Stearic acid and Palmitic acid

According to Arild & Christian (2005), fatty acid may found in animals, plants and microorganisms. In example, oleic acid (18:1 ω-9) may be found in plant and animals, but somehow also found in microorganism, followed by palmitic acid, stearic acid and linoleic acid.

In soap production, the fatty acid was used due to the characteristic that shown in Figure 2.1. Based on the figure, stearic and palmitic acid were used as to harden the soap, while oleic and linoleic acid were used as conditioning agent. Myristic acid was used as cleansing, harden and can produce bubbly lather soap.

	Hardness	Cleansing	Bubbly Lather	Creamy Lather	Conditioning
Lauric	Yes	Yes	Yes		
Myristic	Yes	Yes	Yes		
Palmitic	Yes			Yes	
Stearic	Yes			Yes	
Ricinoleic			Yes	Yes	Yes
Oleic					Yes
Linoleic					Yes
Linolenic					Yes

Figure 2.1: Characteristic of fatty acids (Retrieved from http://www.soapcalc.net/info/SoapQualities.asp)

2.3.3 Types of Extraction

There are many methods to gain the oil from the seed, such as hot water flotation, bridge press, ram press and modern laboratory method; solvent extraction and enzymatic extraction (Warra, 2011). However, in this work, solvent extraction by using soxhlet method is chosen by a few considerations.

2.3.3.1 Soxhlet Extraction Method

In solvent extraction, soxhlet extractor is a piece of laboratory apparatus invented in 1879 by Franz von Soxhlet. It was originally designed for the extraction of a lipid from a solid material. However, soxhlet extractor is not limited to the extraction of lipids.

The soxhlet extraction with n-hexane solvent is the most popular method used in the experiment (Uzun et al, 2006; Saydut et al, 2008; Latif and Anwar, 2011; Mohamed and Awatif, 1997). The solvent of n-hexane is more suitable for free fatty acid extraction compared to the ethanol because n-hexane is a non-polar solvent, while ethanol is a polar solvent, which is suitable for bio-active compound. Based on Liauw et al (2008) research, it was determined that n-hexane solvent produced 44.29% of oil yields from neem seeds compared to ethanol solvent produced 41.11% of oil yields.

In soxhlet extractor, solid material containing some of the desired compound is placed inside a thimble, which made from thick filter paper, and loaded into main chamber of the soxhlet extractor. Then, soxhlet extractor will placed onto a flask, which containing the solvent. The process will continue to chamber that containing the solid material slowly fills with warm solvent. After the extraction, solvent is removed and typically by rotary evaporator. The excess of solvent in the extracted sample is being evaporated (anonymous, n.d.).

2.3.4 Direct Saponification

The extracted oil from sesame seed is usually used for massage, health treatment for the body and cooking (Bamigboye et al, 2010). It is also used in producing of cosmetic, margarine, pharmaceuticals, paints, lubricants and soap making. In the soap making field, the fatty acid like oleic acid and linoleic acid is mixed with an acoustic alkali through direct saponification (Aiwize and Achebo, 2012). Direct saponification is a traditional method which fats and oils are saponified with an alkali solution (Ogoshi and Miyawaki, 1985).

$$RCOOCH_3 + NaOH \longrightarrow RCOONa + CH_3OH \qquad (1)$$

The expression above (1) is the chemical reaction that takes by the extracted oil with an alkali.

2.3.4.1 Alkali solution

There are two types of alkali solution that used in the producing of soap; potassium hydroxide (KOH) and sodium hydroxide (NaOH). Typically, potassium hydroxide is used to produce a liquid soap and sodium hydroxide is used to produce solid soap.

2.4 Summary

This paper previously, there are only studies about saponification for chemical analysis in order to prove that sesame oil can be used in producing a product (Warra, 2011; Bamigboye et al, 2010; Abou-Gharbia et al, 1997; Latif and Anwar, 2010; Uzun et al, 2005; and Saydut et al, 2007). Hence, this work aims to prepare oil from whole (undehulled) and dehulled sesame seed in order to compare the chemical analysis by using soxhlet apparatus and to utilize the extracted seed oil for soap production through solid and liquid composition. At present, soaps that produce from sesame oil may be used more extensively than the soap that produced from other vegetable oils. In fact, the production of sesame plant may affect the local economy due to the increasing of uses sesame oil as the main fatty acid in the soap. However, this work is not yet known whether it will benefit positively or negatively to the users. Thus, this will be one of the objectives of this study.

3 MATERIALS AND METHODS

3.1 Overview

This study presents how the research will be made. This is included the flow diagram process from the beginning till the end of the process. Besides that, this chapter tells the detail operation of this research, which includes the soxhlet extraction method, physiochemical analysis, direct saponification for soap making and soap testing.

3.2 Introduction

This study presents the soxhlet extraction method by using n-hexane as the solvent of this solvent extraction technique.

3.3 Chemicals

This paper presents the chemicals that used in this study, such as sodium hydroxide pellets (99%), potassium hydroxide pellets (85%), and iodine monochloride for synthesis, sodium thiosulphate anhydrous (97%) and hydrochloric acid fuming (37%) were obtained from Merck, Germany. The sorbitol 70 wt% in solution water was obtained from Sigma Aldrich, Malaysia. While sodium carbonate hydrates (99%), starch indicator, 1% in water indicator was obtained from Sigma Aldrich, Germany and Fluka, Malaysia respectively. Furthermore, potassium iodide (99.9%), propylene glycol and sodium polyphosphate extra pure (65% reagent) were obtained from R&M Marketing (U.K), SAFC (Singapore) and QRec, respectively. Hexane of analytical reagent grade (99.06%) was obtained from Fisher Scientific, U.K for the soxhlet extraction solvent.

3.4 Raw material

The main raw material that used in this research was white sesame seed (*Sesamum Indicum*). It was purchased from the local market (i.e Giant, Kuantan), where the

sample was selected based on the ability easy to get in local market and frequently used in society.

3.5 Equipments

There are many types of equipment that used in this experiment. However, the major equipments were listed on the following:-

a) Soxhlet extraction unit

b) Distillation Assembly

c) Oven

d) Stainless steel blender

e) Hot plate

f) Analytical balance

g) pH meter

h) Thermometer

i) Glassware

3.6 Experimental work

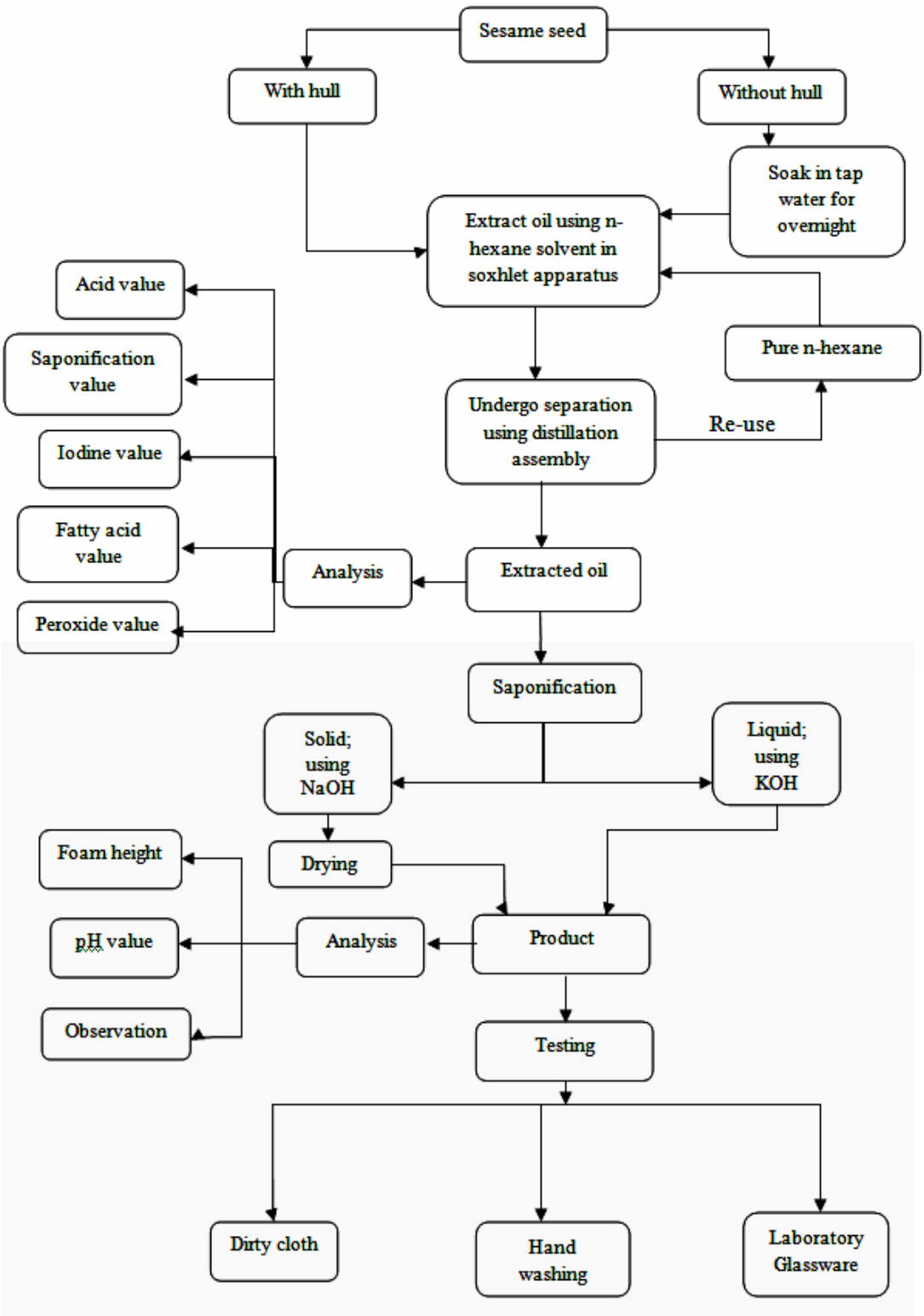

Figure 3.3: Flow chart of Experimental Work

3.7 Sample Preparation

As the sesame seed were obtained from local market in Kuantan, the seed was divided into 50:50 and labelled as undehulled and dehulled seeds. Both labelled seeds were washed to make sure the seed is clean. The labelled dehulled seeds were soaked into tap water for overnight (Bamigboye et al., 2010). Then, the seeds were dried at 60^{o}C for overnight. After dried, the seeds were finely grounded by using stainless steel blender. The grounded seed were dried at 45^{o}C for overnight (Wara et al., 2011); to make sure the seeds are dry completely.

3.8 Extraction of Sesame seed using Soxhlet Extractor

After the sample preparation, 100 grams of grounded seeds are subjected to extract at 60^{o}C using n-hexane (purity 99.6%) solvent. The sample is put inside the soxhlet neck, which at the bottom and the top was covered by wools (Figure 3.2.2). The heating mantle is switched on for vaporizing the solvents to start the extraction. The solvent will eventually fill the neck, and then fill the round bottom flask back with the extract. After finishing the extraction about 8 hours, the heating mantle was switched off, to let the apparatus cools down for a while. The extracted oil and solvents was collected from the bottom flask and transferred into the distillation assembly apparatus to separate the excess solvents from the extracted oil. Then, the yield of the extracts per raw material is calculated.

$$Yield\ (\%) = \frac{Oil\ weig\ ht}{sample\ \ weig\ ht} x100\% \qquad (2)$$

After extraction and evaporation is finish, the extracted seed oil was stored in freezer at 2^{o}C for subsequent physiochemical analysis.